Supporting Information

Systematic Synthesis of Novel Phosphoglycolipid Analogues as Potential Agonists of GPR55

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1. Biological experiments – Materials and methods

Animals

All animal experiment protocols were reviewed and approved by the Wako Animal Experiments Committee at RIKEN. Fertilized Boris Brown chicken eggs were purchased from a local supplier (Inoue Poultry Farm, Sagamihara, Japan) and incubated in a rocking egg incubator at 38 °C until the embryos had developed to Hamburger and Hamilton Stage 36 (*Hamburger and Hamilton*, 1951).

Cell culture

Neuronal cell culture was carried out as previously described (*Guy et al*., 2019). Dorsal root ganglia (DRG) were dissected from Stage 36 embryos and DRG sensory neurons were extracted and dissociated into single cells by trypsinization. Dissociated neurons were suspended in Leibowitz's L15 medium (Life Technologies, CA) with N2 Supplement (Life Technologies), 20 ng/ml nerve growth factor (NGF; Promega, WI) and 750 μg/ml bovine serum albumin (Life Technologies), and seeded onto 3 cm glass-based dishes that had been coated with 9 μg/cm^2 mouse laminin (Life Technologies). Seeded dishes were placed in a 37 °C incubator with 5% CO_2 for 1 – 4 hours before use in the turning assay.

In vitro *axon turning assay*

Axon turning assays (*Lohof et al*., 1992) were performed as described previously (*Guy et al.*, 2019) with some modifications. Test compounds were dissolved in a vehicle of 0.001% (v/v) DMSO/PBS and used at an in-pipette concentration of 20 μM. On an Olympus IX51 inverted microscope (Olympus Corporation, Tokyo, Japan), a growth cone, the tip of single extending axons, was introduced to a microscopic concentration gradient of test compounds and imaged for 45 minutes using a QICAM Fast 1394 (QImaging, Burnaby, Canada) CCD digital camera controlled by Metavue software (version 7.8.2.0, Molecular Devices, CA). This microscopic concentration gradient was produced by repeated pulsatile ejection of test compound solution through a glass micropipette (borosilicate glass with filament, catalog # BF100-50-10, Sutter Instrument, CA) the tip of which was placed 100 μm away from the growth cone at an angle of 45°. Micropipettes were made using a P-97 Flaming/Brown micropipette puller (Sutter Instrument), designed to have a steep shoulder and an approximate tip diameter of approximately 1 μm. Nitrogen gas pressure of 1 – 4 psi was applied to the test solution within the pipette by connection to a PV820 Picopump (World Precision Instruments, FL) electrically gated pressure control system. The frequency (2 Hz) and duration (20 ms) of positive gas pressure was controlled by a pulse generator (model AWG-50, ELMOS, Osaka, Japan). This pulsatile ejection of test solution from the pipette tip induces and maintains a microscopic concentration gradient of test compound in the culture medium for the

duration of the assay (*Lohof et al*, 1992, *Zheng et al*, 1994). At a distance of 100 μm from the pipette tip, test compound concentration is approximately 1000 times lower than the in-pipette concentration ((*Lohof et al*, 1992, *Ming et al*, 1997). At the end of the assay, the turning angle (the angle between the growth cone's starting position and its final position, measured relative to its axon) and axon extension (in microns) were calculated using Metavue software. Growth cones that bifurcated, collapsed, retracted or failed to extend more than 10 μm during the 45 minutes of the assay were discounted from the study.

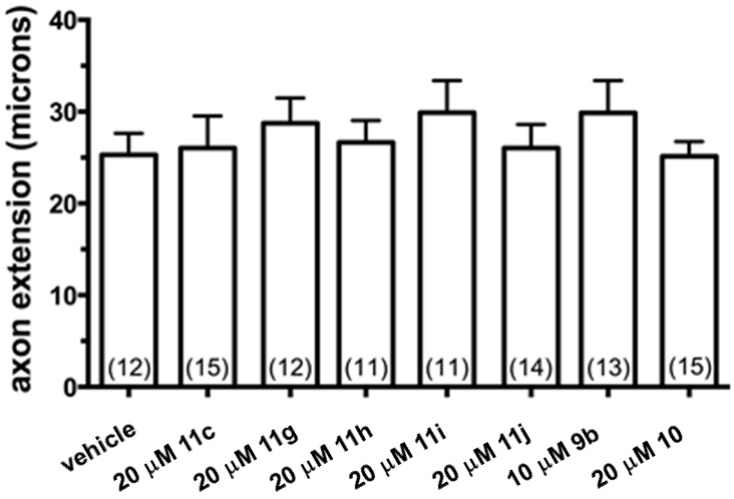

Figure S. Axon growth is unaffected by treatment with LPGlc analogues. Bars show mean axon extension in microns ± SEM, numbers in parentheses indicate the number of axons tested. No significant difference in axon extension was observed.

Methods References

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2. Chemical experiments and compound characterization

2-1. General procedures

Air and/or moisture sensitive reactions were carried out under argon atmosphere with anhydrous solvents. Reactions were monitored by thin-layer chromatography (TLC) (Kieselgel 60 F254, EM Science). Compounds were detected by UV light and/or Hanessian's stain and purified by column chromatography (silica gel 60, 40-100 mesh, EM Science). Solvents were removed lower than 40 °C under reduced pressure. Synthesized compounds were analyzed by ^{1}H NMR and ^{13}C NMR (JEOL ECX 400 spectrometer). ^{1}H NMR analyses were conducted with $CDCl_3$, CD_3OD or pyridine-d_5 referenced to $CHCl_3$ at 7.26 ppm, to CD_3OD at 3.31 ppm or to pyridine at 7.22 ppm, respectively. ^{13}C NMR analyses were referenced to $CDCl_3$ at 77.0 ppm, CD_3OD at 49.00 or pyridine-d_5 at 123.87 ppm. Synthesized compounds were assigned by standard 2D experiments. MALDI-TOF mass spectra were recorded on a SHIMADZU Compact MALDI AXIMA-CFR spectrometer with 2,5-dihydroxybenzoic acid (DHBA) as the matrix. HRMS determinations were performed with a JEOL Accustom JMS-T700LCK mass spectrometer with CF_3CO_2Na as the internal standard. All other reagents were purchased from Kanto Chemicals Co. Inc., Tokyo Chemical Industries Co., Ltd., FUJIFILM Wako Pure Chemical Co., Aldrich Chemical Co. and Avanti Polar Lipids, Inc.

2-2. Synthesis and compound characterization

10-(Benzyloxy)decan-1-ol (13a)

To a suspension of 1,10-decanediol (**12a**) (857 mg, 4.92 mmol) and sodium hydride (197 mg, 4.92 mmol) in dry DMF (16 mL) was added benzyl bromide (195 µL, 1.64 mmol). The mixture was stirred at 60 °C for 21 h. The reaction was quenched with water and the mixture was diluted with EtOAc followed by washing with brine three times and then dried over Na_2SO_4. After filtration and evaporation under reduced pressure, the crude product was purified using column chromatography (silica gel, hexane / EtOAc = 8 / 1) to afford **13a** (colorless oil, 173 mg, 40% yield). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37-7.26 (m, 5H), 4.50 (s, 2H), 3.63 (t, *J* = 6.7 Hz, 2H), 3.46 (t, *J* = 6.7 Hz, 2H), 1.64-1.52 (m, 4H), 1.38-1.26 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.6, 128.3, 127.6, 127.4, 72.8, 70.4, 63.0, 32.7, 29.7, 29.4, 29.4, 29.4, 29.3, 26.1, 25.6; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{17}H_{28}O_2Na$ 287.1982, found 287.1989

(((10-Bromodecyl)oxy)methyl)benzene (14a)

To a solution of **13a** (269 mg, 1.02 mmol) and tetrabromomethane (677 mg, 2.04 mmol) in dry THF (5 mL) was added triphenylphosphine (535 mg, 2.04 mmol). The mixture was stirred at ambient temperature for 2 h. After evaporation under reduced pressure, the crude product was purified using column chromatography (silica gel, hexane to hexane / EtOAc = 8 /1) to afford **14a** (yellow oil, 332 mg, 99%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.35 (m, 4H), 7.31-7.27 (m, 1H), 4.51 (s, 2H), 3.47 (t, *J* = 6.7, 2H), 3.41 (t, *J* = 7.2, 2H), 1.89-1.82 (m, 2H), 1.66-1.59 (m, 2H), 1.44-1.30 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.5, 128.2, 127.4, 127.3, 72.7, 70.3, 33.8, 32.7, 29.6, 29.3, 29.3, 29.2, 28.6, 28.0, 26.0; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{17}H_{27}BrONa$ 349.1143, 351.1123 found 349.1146, 351.1122

(S)-2-(3-((10-(Benzyloxy)decyl)thio)-2-((*tert*-butyldimethylsilyl)oxy)propyl)isoindoline-1,3-dion e (19a)

To a solution of **18**[1] (135 mg, 0.384 mmol) and sodium hydride (18.0 mg, 0.461 mmol) in dry DMF (0.9 mL) at 0 ºC under argon atmosphere was added a solution of **14a** (151 mg, 0.461 mmol) in dry DMF (1.0 mL). The mixture was stirred at ambient temperature for 8 h. The reaction was quenched with water at 0 ºC and the mixture was diluted with EtOAc, followed by washing with brine three times and then dried over Na_2SO_4. After filtration and evaporation under reduced pressure, the crude product was purified using column chromatography (silica gel, hexane / EtOAc = 8 /1) to afford **19a** (yellow oil, 174 mg, 78% yield). $[\alpha]_D^{26}$ = -26.6 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.83 (m, 2H), 7.74-7.70 (m, 2H), 7.36-7.26 (m, 4H), 4.50 (s, 2H), 4.17 (m, 1H), 3.93-3.78 (m, 2H), 3.46 (t, *J* = 6.7 Hz, 2H), 2.68-2.52 (m, 4H), 1.64-1.52 (m, 4H),

1.39-1.25 (m, 12H), 0.81 (s, 9H), 0.050 (s, 3H), -0.12 (s, 3H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.2, 138.6, 133.9, 132.0, 128.3, 127.6, 127.4, 123.1, 72.8, 70.5, 69.1, 43.1, 37.4, 33.0, 29.7, 29.6, 29.5, 29.4, 29.2, 28.8, 17.7, -4.6, -4.9; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{34}H_{51}NO_4SSiNa$ 620.3206, found 620.3199

(S)-2-(3-((10-(Benzyloxy)decyl)thio)-2-hydroxypropyl)isoindoline-1,3-dione (20a)

20a (yellow oil, 154 mg, 83% yield) was synthesized from **19a** (227 mg, 0.366 mmol) in the reported method.[1] $[\alpha]_D^{26}$ = 5.20 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.88-7.84 (m, 2H), 7.75-7.72 (m, 2H), 7.35-7.26 (m, 5H), 4.50 (s, 2H), 4.03 (m, 1H), 3.86 (m, 2H), 3.46 (t, *J* = 6.7 Hz, 3H), 2.82-2.51 (m, 4H), 1.64-1.53 (m, 4H), 1.38-1.24 (m, 12H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 138.6, 134.1, 131.9, 128.3, 127.6, 127.4, 123.4, 72.8, 70.4, 68.2, 42.9, 37.5, 32.6, 29.7, 29.6, 29.4, 29.4, 29.1, 28.7, 26.1; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{28}H_{37}O_4SNa$ 506.2340, found 506.2341

(S)-1-Amino-3-((10-(benzyloxy)decyl)thio)propan-2-ol (21a)

To a solution of **20a** (114 mg, 0.236 mmol) in MeOH (2.4 mL) was added hydrazine monohydrate (57.0 μL, 1.18 mmol). The mixture was stirred under reflux for 2 h. After evaporation under reduced pressure, 2 N aq. K_2CO_3 (236 μL) and H_2O (2 mL) were added to the mixture and then the crude product was extracted with $CHCl_3$ three times and dried over Na_2SO_4. After filtration and evaporation under reduced pressure, **21a** was obtained (white solid, 69.0 mg, 69% yield). **21a** was used for next step without further purification. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.23 (m, 5H), 4.49 (s, 2H), 3.64 (m, 1H), 3.45 (t, *J* = 6.7 Hz, 2H), 2.87 (m, 1H), 2.70-2.61 (m, 2H), 2.61-2.50 (m, 3H), 1.64-1.53 (m, 4H), 1.40-1.24 (m, 12H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.5, 128.2, 127.5, 127.3, 72.7, 70.4, 46.3, 37.0, 32.4, 29.6, 29.6, 29.4, 29.3, 29.0, 28.7, 26.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{20}H_{36}NO_2S$ 354.2464, found 354.2467

3-(((S)-3-((10-(Benzyloxy)decyl)thio)-2-hydroxypropyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11a)

To a solution of **28a**[1] (19.0 mg, 0.0636 mmol) and **21a** (22.5 mg, 0.0636 mmol) in EtOH (636 μL) was added DIPEA (11.0 μL, 0.0636 mmol). The mixture was stirred at ambient temperature overnight. After evaporation under reduced pressure, the crude product was purified using column chromatography (silica gel, chloroform / methanol = 10 / 1 → 5 / 1) to afford **11a** (white solid, 25.0 mg, 64% yield). $[\alpha]_D^{26}$ = -37.4 (c 1.0 in pyridine); ^{1}H NMR (pyridine-d_5, 400 MHz): δ 7.49 (d, *J* = 7.2 Hz, 2H), 7.40 (m, 2H), 7.32 (t, *J* = 7.2 Hz, 2H), 4.55-4.50 (m, 3H), 4.43-3.97 (m, 7H), 3.48 (t, *J* = 6.7 Hz, 2H), 2.94 (d, *J* = 6.3 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 1.68-1.51 (m, 4H), 1.41-1.14 (m, 14H); ^{13}C NMR (pyridine- d_5, 100 MHz): δ 186.4, 184.7, 170.7, 169.4, 140.0, 129.0, 128.3, 128.1,

86.3, 80.7, 79.1, 75.7, 73.2, 71.4, 71.2, 71.0, 62.0, 50.0, 37.8, 33.3, 30.5, 30.3, 30.1, 30.0, 30.0, 29.8, 29.4, 26.8; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{30}H_{46}N_2O_9SNa$ 633.2822, found 633.2825

1,13-Tridecanediol (12b)

To a solution of lithium aluminum hydride (LAH) (2.8 g, 0.0738 mol) in dry THF (246 mL) on ice bath was slowly added tridecanedioic acid (3.0 g, 0.0123 mol). The mixture was stirred under reflux for 5.5 h, followed by quenching with water and 1 N aq. NaOH on ice bath. The crude product was extracted with EtOAc three times and dried over Na_2SO_4. After filtration and evaporation under reduced pressure, **12b** was obtained (white solid, 2.56 g, 97% yield). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.63 (t, *J* = 6.7 Hz, 4H), 1.58 (m, 4H), 1.50-1.20 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 63.0, 32.7, 29.5, 29.5, 29.3, 25.7; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{13}H_{28}O_2Na$ 239.1987, found 239.1991

13-(Benzyloxy)tridecan-1-ol (13b)

13b (colorless oil, 254 mg, 60% yield) was synthesized from **12b** (308 mg, 1.29 mmol) in the same manner as **13a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.34-7.33 (m, 4H), 7.29-7.27 (m, 1H), 4.49 (s, 2H), 3.56 (t, *J* = 6.7, 2H), 3.46 (t, *J* = 7.2, 2H), 1.66-1.59 (m, 2H), 1.57-1.50 (m, 2H), 1.38-1.28 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.3, 128.0, 127.4, 127.2, 72.6, 70.2, 62.4, 32.5, 29.5, 29.4, 29.4, 29.2, 25.9, 25.6; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{20}H_{34}O_2Na$ 329.2456, found 329.2455

(((13-Bromotridecyl)oxy)methyl)benzene (14b)

14b (colorless oil, 516 mg, 85% yield) was synthesized from **13b** (511 mg, 1.55 mmol) in the same manner as **14a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.34 (m, 4H), 7.31-7.27 (m, 1H), 4.51 (s, 2H), 3.47 (t, *J* = 6.7 Hz, 2H), 3.41 (t, *J* = 7.2 Hz, 2H), 1.89-1.82 (m, 2H), 1.65-1.58 (m, 2H), 1.44-1.27 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.6, 128.3, 127.6, 127.4, 72.8, 70.5, 34.0, 32.8, 29.7, 29.5, 29.5, 29.4, 29.4, 28.7, 28.1, 26.1; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{20}H_{33}BrONa$ 391.1612, 393.1592, found 391.1609, 393.1587

(S)-2-(3-((13-(Benzyloxy)tridecyl)thio)-2-((*tert*-butyldimethylsilyl)oxy)propyl)isoindoline-1,3-dione (19b)

19b (colorless oil, 611 mg, 75% yield) was synthesized from **14b** (578 mg, 1.48 mmol) and in the same manner as **19a**. $[\alpha]_D^{26}$ = -35.8 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.80 (m, 2H), 7.73-7.68 (m, 2H), 7.37-7.23 (m, 5H), 4.49 (s, 2H), 4.17 (m, 1H), 3.93-3.78 (m, 2H), 3.46 (t, *J* = 6.7 Hz, 2H), 2.68-2.52 (m, 4H), 1.64-1.52 (m, 4H), 1.39-1.25 (m, 18H), 0.80 (s, 9H), 0.048 (s, 3H), -0.12 (s, 3H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.2, 138.6, 133.8, 132.0, 128.2, 127.5, 127.4, 123.1, 72.7, 70.4, 69.1, 43.1, 37.4, 32.9, 29.7, 29.6, 29.5, 29.4, 29.1, 28.8, 26.1, 25.5, 17.7, -4.7, -5.0;

HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{37}H_{57}NO_4SSiNa$ 662.3675, found 662.3671

(S)-2-(3-((13-(Benzyloxy)tridecyl)thio)-2-hydroxypropyl)isoindoline-1,3-dione (20b)

20b (colorless oil, 386 mg, 57% yield) was synthesized from **19b** (818 mg, 1.24 mmol) in the same manner as **20a**. $[\alpha]_D^{26}$ = 3.40 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.82 (m, 2H), 7.72-7.69 (m, 2H), 7.34-7.32 (m, 4H), 7.29-7.27 (m, 1H), 4.50 (s, 2H), 4.05 (m, 1H), 3.86-3.83 (m, 2H), 3.46 (t, *J* = 6,7 Hz, 2H), 2.77-2.54 (m, 4H), 1.65-1.54 (m, 4H), 1.41-1.24 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.4, 138.5, 133.9, 131.7, 128.1, 127.4, 127.2, 123.2, 72.6, 70.3, 68.1, 42.8, 37.2, 32.4, 29.6, 29.5, 29.4, 29.3, 29.3, 29.0, 28.6, 26.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{31}H_{44}NO_4S$ 526.2991, found 526.2989

(S)-1-Amino-3-((13-(benzyloxy)tridecyl)thio)propan-2-ol (21b)

21b (white solid, 58.0 mg, quant.) was synthesized from **20b** (76.0 mg, 0.139 mmol) in the same manner as **21a**. ^1H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.32 (m, 4H), 7.29-7.25 (m, 1H), 4.49 (s, 2H), 3.63 (m, 2H), 3.45 (t, *J* = 6.7 Hz, 2H), 2.87 (dd, *J* = 3.6, 13 Hz, 1H), 2.71-2.50 (m, 5H),1.60 (m, 4H), 1.40-1.23 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.6, 128.2, 127.5, 127.4, 72.7, 70.4, 46.4, 37.0, 32.4, 29.7, 29.6, 29.5, 29.4, 29.4, 29.1, 28.8, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{23}H_{42}NO_2S$ 396.2936, found 396.2932

3-(((S)-3-((13-(Benzyloxy)tridecyl)thio)-2-hydroxypropyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11b)

11b (white solid, 37.0 mg, 88% yield) was synthesized from **21b** (25.9 mg, 0.0619 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -56.8 (c 1.0 in pyridine); ^1H NMR (pyrdine-d_5, 400 MHz): δ 7.49 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 1H), 4.55-4.50 (m, 3H), 4.41-3.95 (m, 7H), 3.49 (t, *J* = 6.3 Hz, 2H), 2.93 (d, *J* = 6.3 Hz, 2H), 2.64 (t, *J* = 7.6 Hz, 2H), 1.70-1.54 (m, 4H), 1.45-1.17 (m, 20H); ^{13}C NMR (pyrdine-d_5, 100 MHz): δ 186.3, 184.7, 170.7, 169.4, 140.0, 129.0, 128.2, 128.0, 86.3, 80.7, 79.1, 75.7, 73.2, 71.5, 71.3, 71.0, 62.0, 50.0, 37.8, 33.3, 30.5, 30.3, 30.2, 30.1, 30.1, 29.8, 29.5, 26.9; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{33}H_{53}N_2O_9S$ 653.3472, found 653.3474

1,15-Pentadecanediol (12c)

12c (white solid, 4.17 g, quant.) was synthesized from 15-pentadecanolactone (3.75 g, 0.0156 mol) in the same manner as **12a**. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.63 (t, *J* = 6.7 Hz, 4H), 1.57 (m, 4H), 1.40-1.10 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 63.0, 32.7, 29.5, 29.5, 29.5, 29.4, 25.7; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{15}H_{32}O_2Na$ 267.2300, found 267.2300

15-(Benzyloxy)pentadecan-1-ol (13c)

13c (colorless oil, 274 mg, 24% yield) was synthesized from **12c** (205 mg, 0.767 mmol) in the same manner as **13a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.34-7.33 (m, 4H), 7.30-7.27 (m, 1H), 4.50 (s, 2H), 3.63 (t, *J* = 6.3 Hz, 2H), 3.46 (t, *J* = 6.7 Hz, 2H), 1.65-1.53 (m, 4H), 1.40-1.26 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.7, 128.3, 127.6, 127.4, 72.8, 70.5, 63.0, 32.7, 29.7, 29.6, 29.5, 29.4, 29.4, 26.1, 25.7; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{22}H_{38}O_2Na$ 357.2770, found 357.2762

(((15-Bromopentadecyl)oxy)methyl)benzene (14c)

14c (colorless oil, 197 mg, 68% yield) was synthesized from **13c** (247 mg, 0.691 mmol) in the same manner as **14a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.35 (m, 4H), 7.31-7.27 (m, 1H), 4.51 (s, 2H), 3.48 (t, *J* = 6.7 Hz, 2H), 3.41 (t, *J* = 6.7 Hz, 2H), 1.90-1.83 (m, 2H), 1.66-1.59 (m, 2H), 1.45-1.28 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.7, 128.2, 127.4, 72.8, 70.5, 33.9, 32.8, 29.7, 29.6, 29.5, 29.5, 29.4, 29.4, 28.7, 28.1, 26.1; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{22}H_{37}BrONa$ 419.1925, 421.1905 found 419.1928, 421.1908

(S)-2-(3-((15-(Benzyloxy)pentadecyl)thio)-2-((*tert*-butyldimethylsilyl)oxy)propyl)isoindoline-1,3-dione (19c)

19c (colorless oil, 300 mg, 67% yield) was synthesized from **14c** (182 mg, 0.435 mmol) in the same manner as **19a**. $[\alpha]_D^{26}$ = -37.2 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.85-7.83 (m, 2H), 7.72-7.70 (m, 2H), 7.34-7.33 (m, 4H), 7.30-7.27 (m, 1H), 4.50 (s, 2H), 4.19-4.16 (m, 1H), 3.93-3.78 (m, 2H), 3.46 (t, *J* = 6.3 Hz, 2H), 2.56 (t, *J* = 7.2 Hz, 2H), 1.64-1.53 (m, 4H), 1.37-1.25 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.6, 133.8, 132.0, 128.2, 127.5, 127.3, 123.0, 72.7, 70.4, 69.1, 29.7, 29.6, 29.5, 29.4, 29.4, 29.1, 28.7, 26.1, 25.5; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{39}H_{61}NO_4SSiNa$ 690.3988, found 690.3981

(S)-2-(3-((15-(Benzyloxy)pentadecyl)thio)-2-hydroxypropyl)isoindoline-1,3-dione (20c)

20c (colorless oil, 113 mg, 49% yield) was synthesized from **19c** (276 mg, 0.400 mmol) in the same manner as **20a**. $[\alpha]_D^{26}$ = 2.80 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.87-7.84 (m, 2H), 7.75-7.71 (m, 2H), 7.34-7.33 (m, 4H), 7.30-7.27 (m, 1H), 4.50 (s, 2H), 4.03 (m, 1H), 3.86-3.84 (m, 2H), 3.46 (t, *J* = 6.7 Hz, 2H), 2.90 (m, 1H), 2.78-2.73 (m, 1H), 2.63-2.53 (m, 3H), 1.64-1.54 (m, 4H), 1.35-1.25 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.6, 134.0, 131.8, 128.2, 127.5, 127.3, 123.3, 72.7, 70.4, 68.1, 42.9, 37.4, 32.5, 29.6, 29.6, 29.5, 29.5, 29.4, 29.1, 28.7, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{33}H_{48}NO_4S$ 554.3304, found 554.3303

(S)-1-Amino-3-((15-(benzyloxy)pentadecyl)thio)propan-2-ol (21c)

21c (white solid, 55.0 mg, 94% yield) was synthesized from **20c** (75.5 mg, 0.131 mmol) in the

same manner as **21a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.33-7.32 (m, 4H), 7.29-7.23 (m, 1H), 4.49 (s, 2H), 3.63 (m, 1H), 3.45 (t, *J* = , 2H), 2.89-2.85 (, 1H), 2.69-2.62 (m, 2H), 2.56-2.50 (m, 3H), 1.63-1.53 (m, 4H), 1.35-1.24 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.6, 128.2, 127.3, 72.7, 70.4, 46.4, 37.0, 32.4, 29.7, 29.6, 29.5, 29.5, 29.4, 29.4, 29.1, 28.8, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{25}H_{46}NO_2S$ 424.3249, found 424.3249

3-(((S)-3-((15-(Benzyloxy)pentadecyl)thio)-2-hydroxypropyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11c)

11c (white solid, 41.0 mg, 84% yield) was synthesized from **21c** (30.4 mg, 0.0717 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -24.0 (c 1.0 in pyridine); ^{1}H NMR (pyridine-d_5, 400 MHz): δ 7.49 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 2H), 4.55-4.48 (m, 3H), 4.43-3.97 (m, 7H), 3.49 (t, = 6.3 Hz, 2H), 2.93 (d, *J* = 6.3 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 1.70-1.54 (m, 4H), 1.43-1.18 (m, 24H); ^{13}C NMR (pyridine- d_5, 100 MHz): δ 186.3, 184.6, 170.6, 169.4, 140.0, 129.0, 128.2, 128.0, 86.3, 80.6, 79.1, 75.7, 73.2, 71.4, 71.3, 70.9, 62.0, 49.9, 37.8, 33.3, 30.5, 30.3, 30.2, 30.2, 30.2, 30.1, 29.8, 29.5, 26.8; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{35}H_{56}N_2O_9SNa$ 703.3604, found 703.3599

10-(Naphthalen-2-ylmethoxy)decan-1-ol (13d)

13d (white solid, 342 mg, 66% yield) was synthesized from **12a** (857 mg, 4.92 mmol) in the same manner as **13a**.^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.85 (m, 2H), 7.79 (s, 1H), 7.48 (m, 3H), 4.68 (s, 2H), 3.62 (t, *J* = 6.7 Hz, 2H), 3.52 (t, *J* = 6.7 Hz, 2H), 1.70-1.61 (m, 2H), 1.59-1.51 (m, 2H), 1.41-1.27 (m, 12H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.1, 133.2, 132.8, 128.0, 127.7, 127.6, 126.2, 125.9, 125.6, 72.8, 70.4, 62.9, 32.7, 29.7, 29.4, 29.4, 29.3, 26.1, 25.6; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{21}H_{30}O_2Na$ 337.2143, found 337.2138

2-(((10-Bromodecyl)oxy)methyl)naphthalene (14d)

14d (white solid, 373 mg, 95% yield) was synthesized from **13d** (332 mg, 0.984 mmol) in the same manner as **14a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.81 (m, 3H), 7.80 (s, 1H),7.52-7.45 (m, 3H), 4.68 (s, 2H), 3.52 (t, J = 6.7 Hz), 3.41 (t, J = 6.7 Hz, 2H), 1.85 (m, 2H), 1.65 (m, 2H), 1.48-1.27 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.1, 133.2, 132.8, 128.0, 127.7, 127.6, 126.1, 125.9, 125.6, 72.8, 70.4, 33.9, 32.7, 29.7, 29.3, 29.3, 29.2, 28.6, 28.0, 26.1; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{21}H_{29}BrONa$ 399.1299, 401.1279, found 399.1295, 401.1275

(S)-2-(2-((*tert*-Butyldimethylsidlyl)oxy)-3-((10-(naphthalen-2-ylmethoxy)decyl)thio)propyl)isoindoline-1,3-dione (19d)

19d (yellow oil, 172 mg, 69% yield) was synthesized from **14d** (148 mg, 0.371 mmol) in the same

manner as **19a**. $[\alpha]_D^{26}$ = -24.6 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.85-7.79 (m, 5H), 7.77 (s, 1H), 7.70-7.66 (m, 2H), 7.49-7.43 (m, 3H), 4.66 (s, 2H), 4.19 (m, 1H), 3.94-3.78 (m, 2H), 3.51 (t, *J* = 6.7 Hz, 2H), 2.65 (t, *J* = 7.2 Hz, 2H), 2.56 (t, *J* = 7.2 Hz, 2H), 1.68-1.53 (m, 4H), 1.42-1.23 (m, 12H), 0.817 (s, 9H), 0.0617 (s, 3H), -0.109 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.1, 136.1, 133.8, 133.1, 132.8, 131.9, 127.9, 127.7, 127.5, 126.1, 125.9, 125.6, 123.0, 72.8, 70.4, 69.1, 43.0, 37.3, 32.9, 29.6, 29.5, 29.4, 29.3, 29.1, 28.7, 26.1, 25.5, 17.7, -4.7, -5.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{38}H_{54}NO_4SSi$ 648.3543, found 648.3539

(S)-2-(2-Hydroxy-3-((10-(naphthalen-2-ylmethoxy)decyl)thio)propyl)isoindoline-1,3-dione (20d)

20d (yellow oil, 127 mg, 90% yield) was synthesized from **19d** (170 mg, 0.254 mmol) in the same manner as **20a**. $[\alpha]_D^{26}$ = 2.60 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.80 (m, 5H), 7.77 (s, 1H), 7.72-7.67 (m, 2H), 7.48-7.44 (m, 3H), 4.66 (s, 2H), 4.03 (m, 1H), 3.83 (d, *J* = 4.9 Hz, 2H), 3.50 (t, *J* = 6.7 Hz, 2H), 2.98 (br, 1H), 2.77-2.57 (m, 2H), 2.54 (t, *J* = 7.2 Hz, 2H), 1.67-1.53 (m, 4H), 1.41-1.23 (m, 12H) ; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 136.1, 134.0, 133.1, 132.8, 131.8, 128.0, 127.7, 127.6, 126.1, 125.9, 125.6, 125.6, 123.3, 72.8, 70.4, 68.1, 42.8, 37.4, 32.5, 29.6, 29.6, 29.4, 29.3, 29.0, 28.7, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{32}H_{40}NO_4S$ 534.2678, found 534.2674

(S)-1-Amino-3-((10-(naphthalen-2-ylmethoxy)decyl)thio)propan-2-ol (21d)

21d (yellow solid, 94.0 mg, 72% yield) was synthesized from **20d** (170 mg, 0.306 mmol) in the same manner as **21a**. ^1H NMR ($CDCl_3$, 400 MHz): δ 7.85-7.80 (m, 3H), 7.78 (s, 1H), 7.49-7.44 (m, 3H), 4.66 (s, 2H), 3.66 (m, 1H), 3.50 (t, *J* = 6.7 Hz, 2H), 2.90 (dd, *J* = 3.2, 13 Hz, 2H), 2.72-2.55 (m, 2H), 2.52 (t, *J* = 7.2 Hz, 2H), 1.67-1.53 (m, 4H), 1.41-1.23 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.1, 133.2, 132.8, 128.0, 127.7, 127.6, 126.1, 125.9, 125.6, 72.8, 70.4, 70.2, 46.2, 37.0, 32.4, 29.7, 29.6, 29.4, 29.3, 29.1, 28.7, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{24}H_{38}NO_2S$ 404.2623, found 404.2620

3-(((S)-2-Hydroxy-3-((10-(naphthalen-2-ylmethoxy)decyl)thio)propyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11d)

11d (white solid, 15.0 mg, 59% yield) was synthesized from **21d** (15.9 mg, 0.0372 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -41.8 (c 1.0 in pyridine); ^1H NMR ($CDCl_3$, 400 MHz): δ 8.00-7.90 (m, 4H), 7.65 (d, *J* = 8.5 Hz, 1H), 7.55-7.47 (m, 2H), 4.72 (s, 2H), 4.54 (d, *J* = 9.9 Hz, 1H), 4.45-3.97 (m, 7H), 3.55 (t, *J* = 6.7 Hz, 2H), 2.94 (d, *J* = 6.3 Hz, 2H), 2.64 (t, J = 7.2 Hz, 2H), 1.68 (m, 2H), 1.57 (m, 2H), 1.44-1.13 (m, 14H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 186.4, 184.7, 170.7, 169.5, 137.6, 134.2, 133.7, 128.7, 128.6, 128.5, 126.9, 126.8, 126.6, 126.5, 86.4, 80.7, 79.2, 75.8, 73.2, 71.5, 71.3,

71.0, 62.0, 50.0, 37.8, 33.3, 30.5, 30.3, 30.1, 30.1, 30.0, 29.8, 29.4, 26.9; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{34}H_{49}N_2O_9S$ 661.3159, found 661.3153

13-(Naphthalen-2-ylmethoxy)tridecan-1-ol (13e)

13e (white solid, 492 mg, 46% yield) was synthesized from **12b** (675 mg, 2.82 mmol) in the same manner as **13a**. ^1H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.84 (m, 3H), 7.81 (s, 1H), 7.52-7.47 (m, 3H), 4.69 (s, 2H), 3.61 (t, *J* = 6.7 Hz, 2H), 3.53 (t, *J* = 6.7 Hz, 2H), 1.72-1.65 (m, 2H), 1.60-1.53 (m, 2H), 1.44-1.29 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.0, 133.1, 132.8, 127.9, 127.6, 127.5, 126.1, 125.8, 125.5, 72.7, 70.3, 32.6, 29.6, 29.4, 29.3, 29.3, 26.0, 25.6; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{24}H_{36}O_2Na$ 379.2613, found 379.2609

2-(((13-Bromotridecyl)oxy)methyl)naphthalene (14e)

14e (white solid, 453 mg, 82% yield) was synthesized from **13e** (475 mg, 1.25 mmol) in the same manner as **14a**. ^1H NMR ($CDCl_3$, 400 MHz): δ 7.84-7.82 (m, 3H), 7.78 (s, 1H), 7.48-7.46 (m, 3H), 4.67 (s, 2H), 3.51 (t, *J* = 6.7 Hz, 2H), 3.41 (t, *J* = 6.7 Hz, 2H), 1.89-1.82 (m, 2H), 1.68-1.61 (m, 2H), 1.44-1.25 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.2, 133.2, 132.9, 128.0, 127.8, 127.6, 126.2, 126.0, 125.7, 125.7, 72.9, 70.5, 34.0, 32.8, 29.7, 29.5, 29.5, 29.4, 29.4, 28.7, 28.1, 26.2; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{24}H_{35}BrONa$ 441.1769, 443.1749 found 441.1772, 443.1755

(S)-2-(2-((*tert*-Butyldimethylsilyl)oxy)-3-((13-(naphthalen-2-ylmethoxy)tridecyl)thio)propyl)isoindoline-1,3-dione (19e)

19e (colorless oil, 434 mg, 74% yield) was synthesized from **14e** (363 mg, 0.823 mmol) in the same manner as **19a**. $[\alpha]_D^{26}$ = -24.4 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.87-7.81 (m, 5H), 7.78 (s, 1H), 7.73-7.69 (m, 2H), 7.49-7.44 (m, 3H), 4.67 (s, 2H), 4.18 (m, 1H), 3.94-3.78 (m, 2H), 3.51 (t, *J* = 6,7 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 2.56 (t, *J* = 7.2 Hz, 2H), 1.68-1.52 (m, 4H), 1.40-1.23 (m, 18H), 0.807 (s, 9H), 0.0514 (s, 3H), -0.119 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.2, 136.2, 133.9, 133.2, 132.9, 132.0, 128.0, 127.8, 127.6, 126.2, 125.9, 125.7, 125.7, 123.1, 72.9, 70.5, 69.1, 43.1, 37.4, 33.0, 29.7, 29.6, 29.5, 29.4, 29.2, 28.8, 26.1, 25.6, 17.7, -4.6, -4.9; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{41}H_{60}NO_4SSi$ 690.4012, found 690.4009

(S)-2-(2-Hydroxy-3-((13-(naphthalen-2-ylmethoxy)tridecyl)thio)propyl)isoindoline-1,3-dione (20e)

20e (colorless oil, 348 mg, 61% yield) was synthesized from **19e** (679 mg, 0.954 mmol) in the same manner as **20a**. $[\alpha]_D^{26}$ = 3.60 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.87-7.81 (m, 5H), 7.77 (s, 1H), 7.74-7.70 (m, 2H), 7.49-7.44 (m, 3H), 4.66 (s, 2H), 4.03 (m, 1H), 3.85-3.84 (m, 2H), 3.50 (t, *J* = 6.7 Hz, 2H), 2.78-2.56 (m, 2H), 2.54 (t, *J* = 7.2 Hz, 2H), 1.67-1.53 (m, 4H),

1.40-1.24 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.0, 133.9, 133.1, 132.7, 131.7, 127.9, 127.7, 127.5, 126.1, 125.8, 125.6, 123.2, 72.7, 70.4, 68.1, 42.8, 37.3, 32.5, 29.6, 29.5, 29.4, 29.3, 29.0, 28.6, 26.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{35}H_{46}NO_4S$ 576.3148, found 576.3145

(S)-1-Amino-3-((13-(naphthalen-2-ylmethoxy)tridecyl)thio)propan-2-ol (21e)

21e (white solid, 48.0 mg, 94% yield) was synthesized from **20e** (65.2 mg, 0.109 mmol) in the same manner as **21a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.83-7.81 (m, 3H), 7.78 (s, 1H), 7.48-7.46 (m, 3H), 4.66 (s, 2H), 3.64 (m, 2H), 3.50 (t, *J* = 6.7 Hz, 2H), 2.89-2.85 (dd, *J* = 3.2, 13.0 Hz, 1H), 2.69-2.56 (m, 3H), 2.51 (t, *J* = 7.2 Hz, 2H), 1.67-1.53 (m, 4H), 1.41-1.24 (m, 18H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.1, 133.2, 132.8, 128.0, 127.8, 127.6, 126.2, 125.9, 125.7, 125.6, 72.8, 70.5, 70.3, 46.3, 37.0, 32.4, 29.7, 29.6, 29.5, 29.4, 29.4, 29.1, 28.8, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{27}H_{44}NO_2S$ 446.3093, found 446.3084

3-(((S)-2-Hydroxy-3-((13-(naphthalen-2-ylmethoxy)tridecyl)thio)propyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11e)

11e (white solid, 35.0 mg, 89% yield) was synthesized from **21e** (25.4 mg, 0.0542 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -48.6 (c 1.0 in pyridine); ^{1}H NMR (pyridine-d_5, 400 MHz): δ 8.10-7.90 (m, 4H), 7.64 (d, *J* = 8.5 Hz, 1H), 7.52-7.48 (m, 2H), 4.72 (s, 2H), 4.52 (d, *J* = 11.2 Hz, 1H), 4.43-3.97 (m, 7H), 3.55 (t, *J* = 6.3 Hz, 2H), 2.94 (d, *J* = 4.9 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 1.70 (m, 2H), 1.58 (m, 2H), 1.48-1.17 (m, 20H); ^{13}C NMR (pyridine-d_5, 100 MHz): δ 186.3, 184.6, 170.6, 169.4, 137.6, 134.2, 133.7, 128.7, 128.5, 128.5, 126.9, 126.8, 126.6, 126.5, 86.3, 80.6, 79.1, 75.7, 73.2, 71.4, 71.2, 71.0, 62.0, 50.0, 37.8, 33.3, 32.4, 30.5, 30.3, 30.2, 30.1, 30.1, 29.8, 29.5, 26.9; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{37}H_{55}N_2O_9S$ 703.3628, found 703.3628

15-(Naphthalen-2-ylmethoxy)pentadecan-1-ol (13f)

13f (yellow oil, 350 mg, 27% yield) was synthesized from **12c** (851 mg, 3.18 mmol) in the same manner as **13a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.84-7.82 (m, 2H), 7.78 (s, 1H), 7.49-7.45 (m, 3H), 4.67 (s, 2H), 3.63 (t, *J* = 6.7 Hz, 2H), 3.51 (t, *J* = Hz, 2H), 1.68-1.53 (m, 4H), 1.28 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.2, 133.2, 132.9, 128.0, 127.8, 127.6, 126.2, 125.9, 125.7, 125.7, 72.9, 70.5, 63.0, 32.7, 29.7, 29.6, 29.5, 29.5, 29.4, 29.4, 29.4, 26.1, 25.7; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{26}H_{40}O_2Na$ 407.2926, found 407.2923

2-(((15-Bromopentadecyl)oxy)methyl)naphthalene (14f)

14f (white solid, 268 mg, 47% yield) was synthesized from **13f** (495 mg, 1.22 mmol) in the same manner as **14a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.83 (m, 3H), 7.80 (s, 1H), 7.51-7.47 (m, 3H), 4.69 (s, 2H), 3.53 (t, *J* = 6.7 Hz, 2H), 3.41 (t, *J* = 7.2 Hz, 2H), 1.90-1.83 (m, 2H), 1.71-1.63 (m, 2H),

1.45-1.29 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.0, 133.1, 132.7, 127.8, 127.6, 127.4, 125.9, 125.7, 125.5, 72.7, 70.3, 33.6, 32.6, 29.6, 29.5, 29.4, 29.4, 29.3, 29.3, 28.6, 28.0, 26.0; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{26}H_{39}BrONa$ 469.2082, 471.2062, found 469.2081, 471.2062

(S)-2-(2-((*tert*-Butyldimethylsilyl)oxy)-3-((15-(naphthalen-2-ylmethoxy)pentadecyl)thio)propyl) isoindoline-1,3-dione (19f)

19f (yellow oil, 300 mg, 60% yield) was synthesized from **14f** (317 mg, 0.675 mmol) in the same manner as **19a**. $[\alpha]_D^{26}$ = -25.6 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.81 (m, 3H), 7.78 (s, 1H), 7.48-7.45 (m, 3H), 4.66 (s, 2H), 4.18 (m, 1H), 3.94-3.78 (m, 2H), 3.50 (t, *J* = 6.7 Hz, 2H), 2.64 (t, *J* = 7.6 Hz, 2H), 2.56 (t, *J* = 7.2 Hz, 2H), 1.68-1.53 (m, 4H), 1.37-1.26 (m, 22H), 0.812 (s, 9H), 0.0560 (s, 3H), -0.114 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.0, 133.6, 133.0, 132.7, 131.8, 127.8, 127.6, 127.4, 125.9, 125.7, 125.5, 122.8, 72.7, 70.3, 69.0, 42.9, 37.3, 32.8, 29.6, 29.4, 29.4, 29.3, 29.3, 29.0, 28.6, 26.0, 25.4; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{43}H_{64}NO_4SSi$ 718.4325, found 718.4318

(S)-2-(2-Hydroxy-3-((15-(naphthalen-2-ylmethoxy)pentadecyl)thio)propyl)isoindoline-1,3-dion e (20f)

20f (white solid, 120 mg, 79% yield) was synthesized from **19f** (180 mg, 0.242 mmol) in the same manner as **20a**. $[\alpha]_D^{26}$ = 2.80 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.87-7.82 (m, 5H), 7.78 (s, 1H), 7.75-7.71 (m, 2H), 7.52-7.46 (m, 3H), 4.67 (s, 2H), 4.04 (m, 1H), 3.85 (m, 2H), 3.51 (t, *J* = 6.7 Hz, 2H), 2.78-2.53 (m, 4H), 1.66-1.54 (m, 4H), 1.42-1.24 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.1, 133.9, 133.2, 132.8, 131.8, 127.9, 127.7, 127.5, 126.1, 125.9, 125.6, 123.3, 72.8, 70.4, 68.2, 42.9, 37.4, 32.5, 42.9, 37.4, 32.5, 29.7, 29.6, 29.5, 29.5, 29.4, 29.1, 28.7, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{37}H_{50}NO_4S$ 604.3461, found 604.3453

(S)-1-Amino-3-((15-(naphthalen-2-ylmethoxy)pentadecyl)thio)propan-2-ol (21f)

21f (white solid, 69.0 mg, quant.) was synthesized from **20f** (87.0 mg, 0.139 mmol) in the same manner as **21a**. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.83-7.81 (m, 3H), 7.77 (s, 1H), 7.48-7.45 (m, 3H), 4.66 (s, 2H), 3.63 (m, 1H), 3.50 (t, *J* = 6.7 Hz, 2H), 2.86 (dd, *J* = 3.1, 12.5 Hz, 1H), 2.69-2.51 (m, 5H), 1.67-1.54 (m, 4H), 1.41-1.24 (m, 22H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.1, 133.2, 132.8, 128.0, 127.7, 127.6, 126.1, 125.9, 125.7, 125.6, 72.8, 70.5, 70.3, 46.3, 37.0, 32.4, 29.7, 29.6, 29.5, 29.5, 29.4, 29.4, 29.1, 28.8, 26.1; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{29}H_{48}NO_2S$ 474.3406, found 474.3404

3-(((S)-2-Hydroxy-3-((15-(naphthalen-2-ylmethoxy)pentadecyl)thio)propyl)amino)-4-(glucosyla mino)cyclobut-3-ene-1,2-dione (11f)

11f (white solid, 44.0 mg, 95% yield) was synthesized from **21f** (30.5 mg, 0.0614 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -47.4 (c 1.0 in pyridine); ^{1}H NMR (pyridine-d_5, 400 MHz): δ 8.00-7.91 (m, 4H), 7.64 (d, J = 8.1 Hz, 1H), 7.54-7.47 (m, 2H), 4.72 (s, 2H), 4.53 (d, J = 9.4 Hz, 1H), 4.44-3.97 (m, 7H), 3.56 (t, J = 6.7 Hz, 2H), 2.94 (d, J = 6.3 Hz, 2H), 2.65 (t, J = 7.2 Hz, 2H), 1.70 (m, 2H), 1.59 (m, 2H), 1.48-1.17 (m, 24H); ^{13}C NMR (pyridine- d_5, 100 MHz): δ 186.4, 184.7, 170.6, 169.4, 137.6, 134.2, 133.7, 128.7, 128.6, 128.5, 126.9, 126.8, 126.6, 126.5, 86.4, 80.7, 79.2, 75.8, 73.2, 71.5, 71.2, 71.0, 62.0, 50.0, 37.8, 33.4, 30.5, 30.3, 30.3, 30.2, 30.2, 30.1, 29.9, 26.9; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{39}H_{59}N_2O_9S$ 731.3941, found 731.3936

2-((S)-2-((*tert*-Butyldimethylsilyl)oxy)-3-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propyl)isoindoline-1,3-dione (19g)

19g (colorless oil, 160 mg, 61% yield) was synthesized from **17**[2] (152 mg, 0.406 mmol) in the same manner as **19a**. $[\alpha]_D^{26}$ = -11.4 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.84-7.67 (m, 4H), 5.35 (m, 8H), 4.16 (m, 1H), 3.91-3.76 (m, 2H), 2.81 (m, 6H), 2.62 (t, *J* = 6.7 Hz, 2H), 2.56 (t, *J* = 7.2 Hz, 2H), 2.04 (m, 4H), 1.58 (m, 2H), 1.43 (m, 2H), 1.37-1.22 (m, 6H), 0.868 (t, *J* = 6.7 Hz, 3H), 0.789 (s, 9H), 0.0342 (s, 3H), -0.135 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.1, 133.8, 132.0, 130.3, 129.6, 128.4, 128.2, 127.9, 127.8, 127.4, 123.0, 69.1, 43.0, 37.4, 32.8, 31.4, 29.2, 29.1, 28.6, 27.1, 26.7, 25.5, 22.4, 17.6, 14.0, -4.7, -5.0; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{37}H_{57}NO_3SSiNa$ 646.3726, found 646.3720

2-((S)-2-Hydroxy-3-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propyl)isoindoline-1,3-dione (20g)

20g (yellow oil, 41.0 mg, 88% yield) was synthesized from **19g** (56.6 mg, 0.0875 mmol) in the same manner as **20a**. $[\alpha]_D^{26}$ = 0.160 (c 1.0 in $CHCl_3$) ; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.89-7.69 (m, 4H), 5.34 (m, 8H), 4.03 (m, 1H), 3.85 (m, 2H), 2.88-2.58 (m, 8H), 2.56 (t, *J* = 7.2 Hz, 2H), 2.05 (m, 4H), 1.60 (m, 2H), 1.45 (m, 2H), 1.38-1.23 (m, 6H), 0.877 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 134.0, 131.9, 130.4, 129.6, 128.5, 128.2, 128.1, 128.0, 127.8, 127.5, 123.4, 68.2, 42.9, 37.4, 32.5, 31.4, 29.2, 29.2, 28.6, 21.1, 26.7, 25.6, 22.5, 14.0; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{31}H_{43}NO_3SNa$ 532.2861, found 532.2879

(S)-1-Amino-3-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propan-2-ol (21g)

To a solution of **20g** (19.0 mg, 0.0373 mmol) in MeOH (373 μL) was added methyl hydrazine[3] (12.0 μL, 0.373 mmol). The mixture was refluxed for 2.5h. After evaporation under vacuum, the crude product was purified using column chromatography (silica gel, $CHCl_3$ / MeOH = 20 / 1 containing 1% (v/v) Et_3N) to afford **21g** (colorless oil, 15.0 mg, quant.). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.39 (m, 8H), 4.01 (br, 1H), 3.22-2.88 (m, 2H), 2.82 (m, 6H), 2.64 (m, 2H), 2.53 (t, *J* = 7.2

Hz, 2H), 2.06 (m, 4H), 1.63-1.22 (m, 10H), 0.884 (t, *J* = 6.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 130.4, 129.6, 128.5, 128.2, 128.1, 128.1, 127.8, 127.5, 67.7, 44.6, 36.6, 31.5, 29.3, 29.2, 28.7, 27.2, 26.7, 25.6, 22.5, 14.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{23}H_{42}NOS$ 380.2987, found 380.2983

3-(((S)-2-Hydroxy-3-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11g)

11g (yellow oil, 10.0 mg, 67% yield) was synthesized from **21g** (9.10 mg, 0.0226 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -57.0 (c 1.0 in pyridine); ^{1}H NMR (pyridine-d_5, 400 MHz): δ 5.56-5.40 (m, 8H), 4.52 (d, *J* = 9.9 Hz, 1H), 4.44-3.98 (m, 8H), 3.10-2.90 (m, 8H), 2.65 (t, *J* = 7.2 Hz, 2H), 2.11 (m, 4H), 1.62 (m, 2H), 1.47-1.21 (m, 8H), 0.855 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (pyridine-d_5, 100 MHz): δ 186.3, 184.7, 170.7, 169.5, 131.0, 130.5, 129.2, 129.1, 128.7, 128.7, 128.6, 128.4, 86.3, 80.7, 79.2, 75.7, 71.5, 71.3, 62.1, 50.0, 37.8, 33.2, 32.0, 29.9, 29.9, 29.4, 26.3, 23.2, 23.1, 14.5; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{33}H_{53}N_2O_8S$ 637.3523, found 637.3520

(S)-2-(Thiiran-2-ylmethyl)isoindoline-1,3-dione (23)

To a solution of **22**[1] (2.90 g, 0.0143 mol) in MeOH (48 mL) was added thiourea[4] (2.20 g, 0.0285 mol). The mixture was stirred at ambient temperature for 22 h. After evaporation under vacuum, the crude product was purified using column chromatography (silica gel, hexane / EtOAc = 6 / 1) to afford **23** (white solid, 2.33 g, 74% yield). $[\alpha]_D^{26}$ = 1.56 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.85-7.81 (m, 2H), 7.74-7.70 (m, 2H), 4.12 (dd, *J* = 4.5, 14 Hz, 2H), 3.59 (dd, *J* = 7.2, 14 Hz, 2H), 3.13 (m, 1H), 2.45 (d, *J* = 5.8 Hz, 1H), 2.41 (d, *J* = 4.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.8, 134.0, 131.7, 123.3, 42.8, 30.9, 24.6; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{11}H_{10}NO_2S$ 220.0432, found 220.0425

(S)-2-(Acetylthio)-3-(1,3-dioxoisoindolin-2-yl)propyl acetate (24)

To a solution of **23** (1.90 g, 0.00867 mol) and sodium acetate (142 mg, 0.00173 mol) in acetic anhydride (36 mL) was added acetic acid (3.6 mL). The mixture was refluxed for 38 h. After evaporation under vacuum, the crude product was purified using column chromatography (silica gel, hexane / EtOAc = 8 / 1 to 4 / 1) to afford **24** (brown oil, 1.89 g, 68% yield). $[\alpha]_D^{26}$ = -26.4 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.82-7.80 (m, 2H), 7.70-7.68 (m, 2H), 4.26-4.12 (m, 3H), 3.97-3.87 (m, 2H), 2.24 (s, 3H), 2.01 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 193.6, 170.3, 167.7, 134.0, 131.6, 64.1, 41.6, 38.3, 30.4, 20.5; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{15}H_{15}NO_5SNa$ 344.0569, found 344.0564

(S)-2-(3-Hydroxy-2-mercaptopropyl)isoindoline-1,3-dione (25)

To a solution of **24** (512 mg, 1.59 mmol) in MeOH (6 mL) was added 4 N HCl/dioxane (2 mL). The mixture was stirred at ambient temperature for 2 h. After evaporation under vacuum, the crude product was purified using column chromatography (silica gel, hexane / EtOAc = 4 / 1 to 2 / 1) to afford **25** (white solid, 313 mg, 83% yield). $[\alpha]_D^{26}$ = -42.4 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.85-7.82 (m, 2H), 7.75-7.72 (m, 2H), 3.90 (m, 2H), 3.71-3.59 (m, 2H), 3.19 (m, 1H), 3.07 (m, 1H), 1.72 (d, *J* = 9.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 134.2, 131.5, 123.5, 64.4, 40.9, 40.7; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{11}H_{11}NO_3SNa$ 260.0357, found 260.0359

(S)-2-(3-Hydroxy-2-(icosylthio)propyl)isoindoline-1,3-dione (26a)

26a (white solid, 79.0 mg, 73%yield) was synthesized from **25** (52.0 mg, 0.200 mmol) in the same manner as **19a**. $[\alpha]_D^{26}$ = -1.78 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.87-7.84 (m, 2H), 7.74-7.72 (m, 2H), 3.99-3.88 (m, 2H), 3.64 (m, 2H), 3.14 (m, 1H), 2.60 (t, *J* = 2.6 Hz, 2H), 1.56 (t, *J* = 7.6 Hz, 2H), 1.25 (m, 34H), 0.869 (t, *J* = 6.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.6, 134.1, 131.8, 123.4, 62.2, 47.1, 38.4, 31.9, 30.9, 29.6, 29.4, 29.3, 29.1, 28.8, 22.6, 14.1; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{31}H_{51}NO_3SNa$ 540.3487, found 540.3483

(S)-3-Amino-2-(icosylthio)propan-1-ol (27a)

27a (white solid, 72.0 mg, 97% yield) was synthesized from **26a** (97.7 mg, 0.181 mmol) in the same manner as **21a**. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.80-3.71 (m, 2H), 3.03-2.91 (m, 2H), 2.79 (m, 1H), 2.52 (t, *J* = 7.2 Hz, 2H), 1.61-1.53 (m, 2H), 1.37-1.24 (m, 34H), 0.865 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 65.1, 49.9, 44.8, 31.9, 30.8, 30.0, 29.6, 29.5, 29.4, 29.3, 29.1, 28.8, 22.6, 14.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{23}H_{50}NOS$ 388.3613, found 388.3610

3-(((S)-3-Hydroxy-2-(icosylthio)propyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11h)

11h (white solid, 23.0 mg, 72% yield) was synthesized from **27a** (19.6 mg, 0.0479 mg) in the same manner as **11a**. $[\alpha]_D^{26}$ = -6.40 (c 1.0 in pyridine); ^1H NMR (pyridine-d_5, 400 MHz): δ 5.11 (br, 1H), 4.75 (m, 2H), 4.65 (m, 1H), 4.35 (m, 1H), 3.59-3.34 (m, 4H), 1.46 (t, *J* = 7.2 Hz, 3H) ; ^{13}C NMR (pyridine-d_5, 100 MHz): δ 189.4, 189.3, 179.4, 175.2, 85.0, 83.9, 82.2, 78.5, 78.2, 78.0, 74.2, 74.1, 71.0, 70.0, 69.9, 16.0; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{33}H_{60}N_2O_8SNa$ 667.3968, found 667.3972

2-((S)-3-Hydroxy-2-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propyl)isoindoline-1,3-dione (26b)

26b (colorless oil, 90.3 mg, 75% yield) was synthesized from **17** (84.9 mg, 0.226 mmol) in the same manner as **19a**. $[\alpha]_D^{26}$ = -8.32 (c 1.0 in $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz): δ 7.86-7.82 (m,

2H), 7.78-7.68 (m, 2H), 5.35 (m, 8H), 3.93 (m, 2H), 3.63 (m, 2H), 3.13 (m, 1H), 2.79 (m, 6H), 2.62 (t, *J* = 7.2 Hz, 2H), 2.04 (m, 4H), 1.59 (m, 2H), 1.51-1.20 (m, 8H), 0.869 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 168.5, 134.1, 131.7, 130.4, 129.5, 128.4, 128.2, 128.1, 128.0, 127.8, 127.4, 123.4, 62.2, 47.1, 38.3, 31.4, 30.7, 29.2, 29.2, 28.6, 27.1, 26.6, 25.5, 22.5, 14.0; HRMS (ESI) m/z $[M+K]^+$ calculated for $C_{31}H_{43}NO_3SK$ 548.2601, found 548.2615

(S)-3-Amino-2-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propan-1-ol (27b)

27b (colorless oil, 6.73 mg, 92% yield) was synthesized from **26b** (10.0 mg, 0.0193 mmol) in the same manner as **21a**. $[\alpha]_D^{26}$ = -2.60 (c 1.0 in $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.36 (m, 8H), 3.82-3.70 (m, 2H), 3.04-2.77 (m, 9H), 2.53 (t, *J* = 7.2 Hz, 2H), 2.05 (m, 4H), 1.59 (m, 2H), 1.45 (m, 2H), 1.37-1.24 (m,4H), 0.875 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 130.4, 129.5, 128.5, 128.1, 128.0, 127.8, 127.4, 64.9, 49.5, 44.4, 31.4, 30.7, 29.5, 29.2, 28.7, 27.1, 26.7, 25.5, 22.5, 14.0; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{23}H_{42}NOS$ 380.2987, found 380.2986

3-(((S)-3-Hydroxy-2-(((5*Z*,8*Z*,11*Z*,14*Z*)-icosa-5,8,11,14-tetraen-1-yl)thio)propyl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11i)

11i (colorless oil, 9.00 mg, 60% yield) was synthesized from **27b** (9.15 mg, 0.0228 mmol) in the same manner as **11a**. $[\alpha]_D^{26}$ = -41.0 (c 1.0 in pyridine); ^{1}H NMR (pyridine-d_5, 400 MHz): δ 5.58-5.40 (m,8H), 4.56-4.04 (m, 10H), 3.36 (br, 1H), 2.95 (m, 6H), 2.74 (m, 2H), 2.09 (m, 4H), 1.64 (m, 2H), 1.50-1.20 (m, 8H), 0.852 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (pyridine-d_5, 100 MHz): δ 186.3, 184.6, 170.3, 169.4, 130.9, 130.6, 129.1, 129.1, 128.7, 128.7, 128.6, 128.3, 86.5, 80.6, 79.1, 75.8, 70.9, 63.5, 61.8, 50.5, 46.0, 31.9, 31.6, 30.3, 30.3, 29.8, 29.4, 27.7, 26.3, 23.1, 14.5; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{33}H_{52}N_2O_8SNa$ 659.3342, found 659.3342

3-Ethoxy-4-(6-(fluoromethyl)glucosylamino)cyclobut-3-ene-1,2-dione (28b)

28b (brown oil, 40.0 mg, 83%yield) was synthesized from 6-deoxy-6-fluoro-β-D-glucopyranose (26.8 mg, 0.147 mmol) in the reported method.[1] $[\alpha]_D^{26}$ = -11.2 (c 1.0 in MeOH); ^{1}H NMR (CD_3OD, 400 MHz): δ 4.75 (q, *J* = 7.2 Hz, 2H), 4.66 (m, 1H), 4.53 (m, 1H), 3.56-3.35 (m, 5H), 1.46 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (CD_3OD, 100 MHz): δ 189.4, 189.3, 179.4, 175.2, 83.9, 82.2, 78.5, 78.2, 78.0, 74.2, 74.1, 71.0, 70.0, 69.9, 16.0; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{12}H_{16}FNO_7Na$ 328.0807, found 328.0808

3-(6-(Fluoromethyl)glucosylamino)-4-(((S)-2-hydroxy-3-(octadecylthio)propyl)amino)cyclobut-3-ene-1,2-dione (11j)

11j (yellow oil, 5.40 mg, 42%yield) was synthesized from **28b** (6.58 mg, 0.0200 mmol) in the same manner as **11a.** $[\alpha]_D^{26}$ = -10.0 (c 1.0 in pyridine); ^{1}H NMR (pyridine- d_5, 400 MHz): δ 4.32-3.85 (m,

8H), 2.89 (m, 2H), 2.63 (t, *J* = 7.2 Hz, 2H), 1.63-1.54 (m, 2H), 1.41-1.18 (m, 32H), 0.876 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (pyridine- d_5, 100 MHz): δ 186.0, 184.4, 169.6, 168.1, 86.1, 84.5, 82.7, 79.1, 78.2, 78.0, 75.3, 71.1, 70.9, 70.2, 70.1, 66.1, 49.8, 37.5, 33.3, 32.4, 30.3, 30.2, 29.9, 29.9, 29.5, 23.2, 14.6; HRMS (ESI) m/z $[M+H]^+$ calculated for $C_{31}H_{56}N_2O_7FS$ 619.3792, found 619.3795

3-((1-Hydroxy-2-(hydroxymethyl)icosan-2-yl)amino)-4-(glucosylamino)cyclobut-3-ene-1,2-dione (11k)

To a solution of **28a**[1] (26.0 mg, 0.0873 mmol) and 2-amino-2-octadecylpropane-1,3-diol[5] (30.0 mg, 0.0873 mmol) in EtOH (873 μL) was added DIPEA (15.0 μL, 0.0873 mmol). The mixture was stirred at 70 ºC for 14.5 h. After evaporation under vacuum, the crude product was purified using column chromatography (silica gel, chloroform / methanol = 10 / 1 → 5 / 1) to afford **11k** (white solid, 19.0 mg, 36% yield). $[\alpha]_D^{26}$ = 0.400 (c 1.0 in pyridine); ^{1}H NMR (pyridine- d_5, 400 MHz): δ 4.50-4.09 (m, 11H), 2.30 (m, 2H), 1.48 (m, 2H), 1.32-1.10 (m, 30H), 0.828 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (pyridine- d_5, 100 MHz): δ 184.3, 184.0, 170.7, 86.1, 80.8, 79.5, 75.6, 71.5, 65.1, 65.1, 63.4, 62.7, 34.3, 32.4, 31.0, 30.3, 30.2, 29.9, 24.1, 23.2, 14.6; HRMS (ESI) m/z $[M+Na]^+$ calculated for $C_{31}H_{56}N_2O_9Na$ 623.3884, found 623.3880

References

[1] F. Ding, A. T. Guy, P. Greimel, Y. Hirabayashi, H. Kamiguchi, Y. Ito, *Chem. Commun*. **2018**, *54*, 8470-8473.

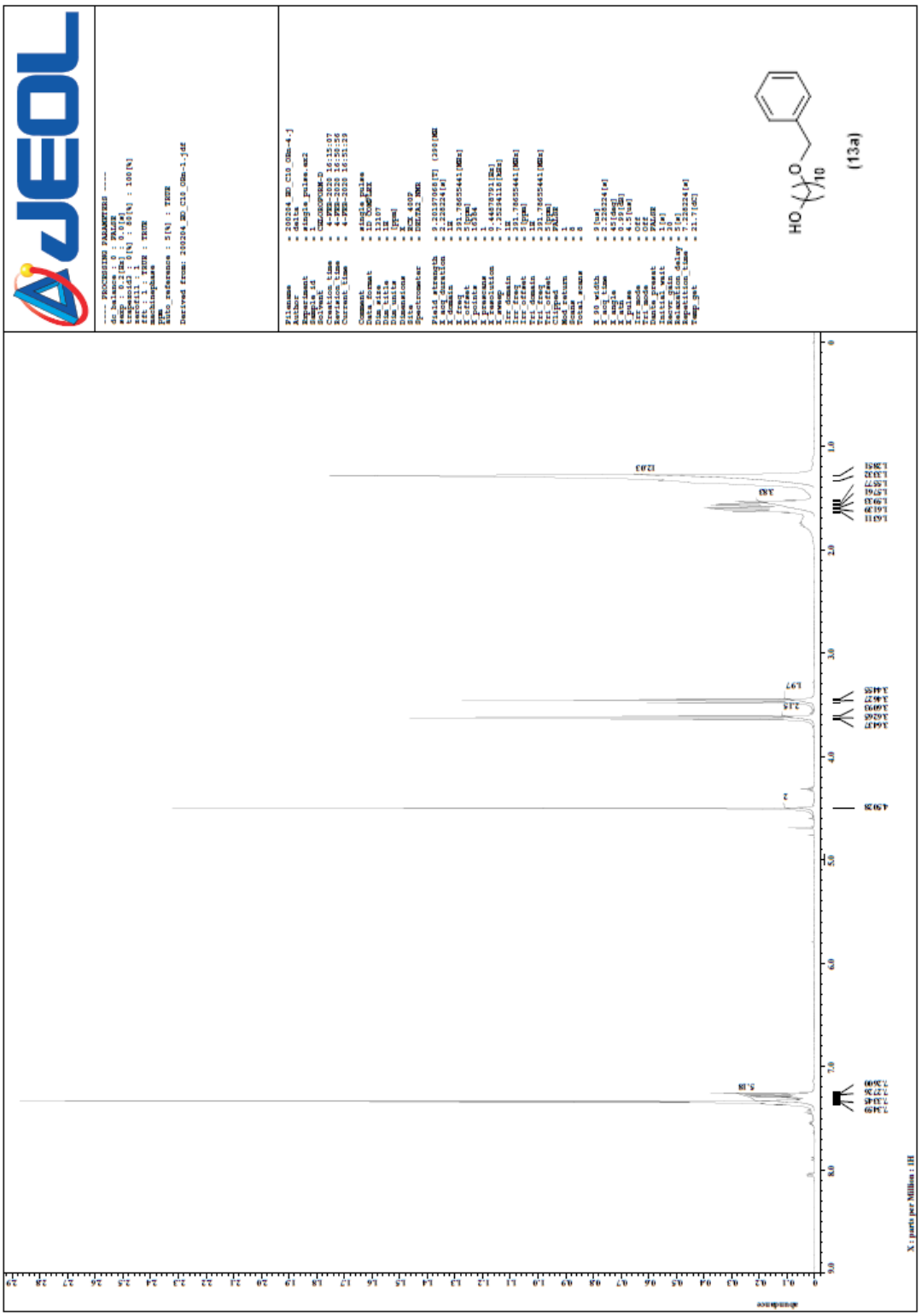
[2] (a) C. J. Easton, L. Xia, M. J. Pitt, A. Ferrante, A. Poulos, D. A. Rathjen, *Synthesis*, **2001**, *3*, 451-457. (b) G. M. Maharvi, A. O. Edwards and A. H. Fauq, *Tetrahedron Lett*. **2010**, *51*, 6426–6428.

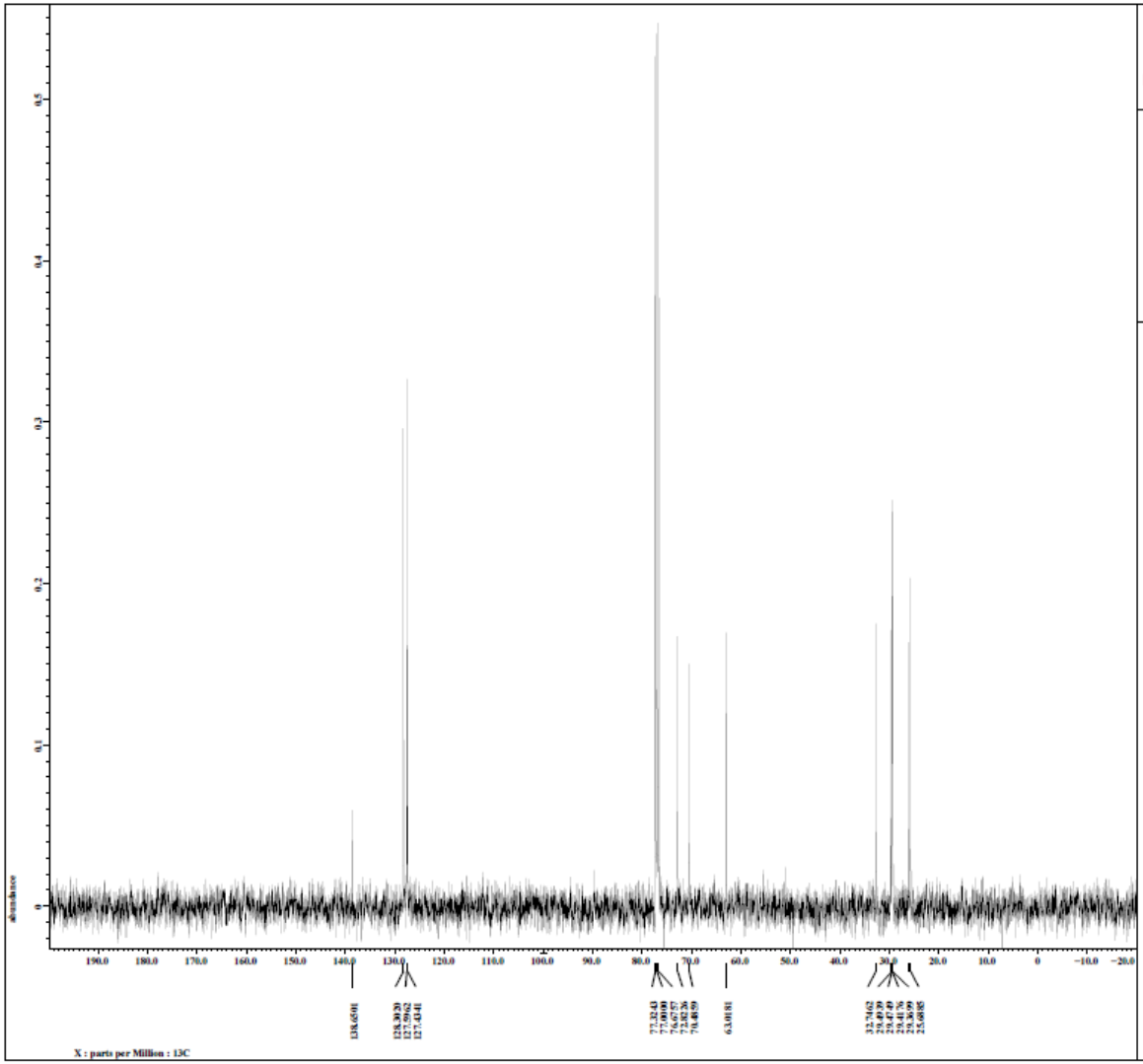
[3] A. L. Smith, C.-K. Hwang, E. Pitsinos, G. R. Scarlato and K. C. Nicolaou, *J. Am. Chem. Soc.* **1992**, *114*, 3136-3138.

[4] B. Tber, Nour-Eddine Fahmi, G. Ronco, P. Villa, D. F. Ewing, G. Mackenzie, *Carbohydr. Res*. **1995**, *267*, 203-215.

[5] T. Fujita, R. Hirose, M. Yoneta, S. Sasaki, K. Inoue, M. Kiuchi, S. Hirase, K. Chiba, H. Sakamoto, M. Arita, *J. Med. Chem.***1996**, *39*, 4451-4459.

2-3. ^{1}H and ^{13}C NMR chart of compounds





JEOL

```
---- PROCESSING PARAMETERS ----
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expp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

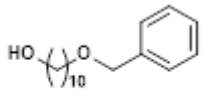
Derived from: 200204_13C_HO_C10_OBn-1.jd
```

```
Filename          = 200204_13C_HO_C10_OBn
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  4-FEB-2020 16:16:04
Revision_time     =  4-FEB-2020 16:54:26
Current_time      =  4-FEB-2020 16:54:49

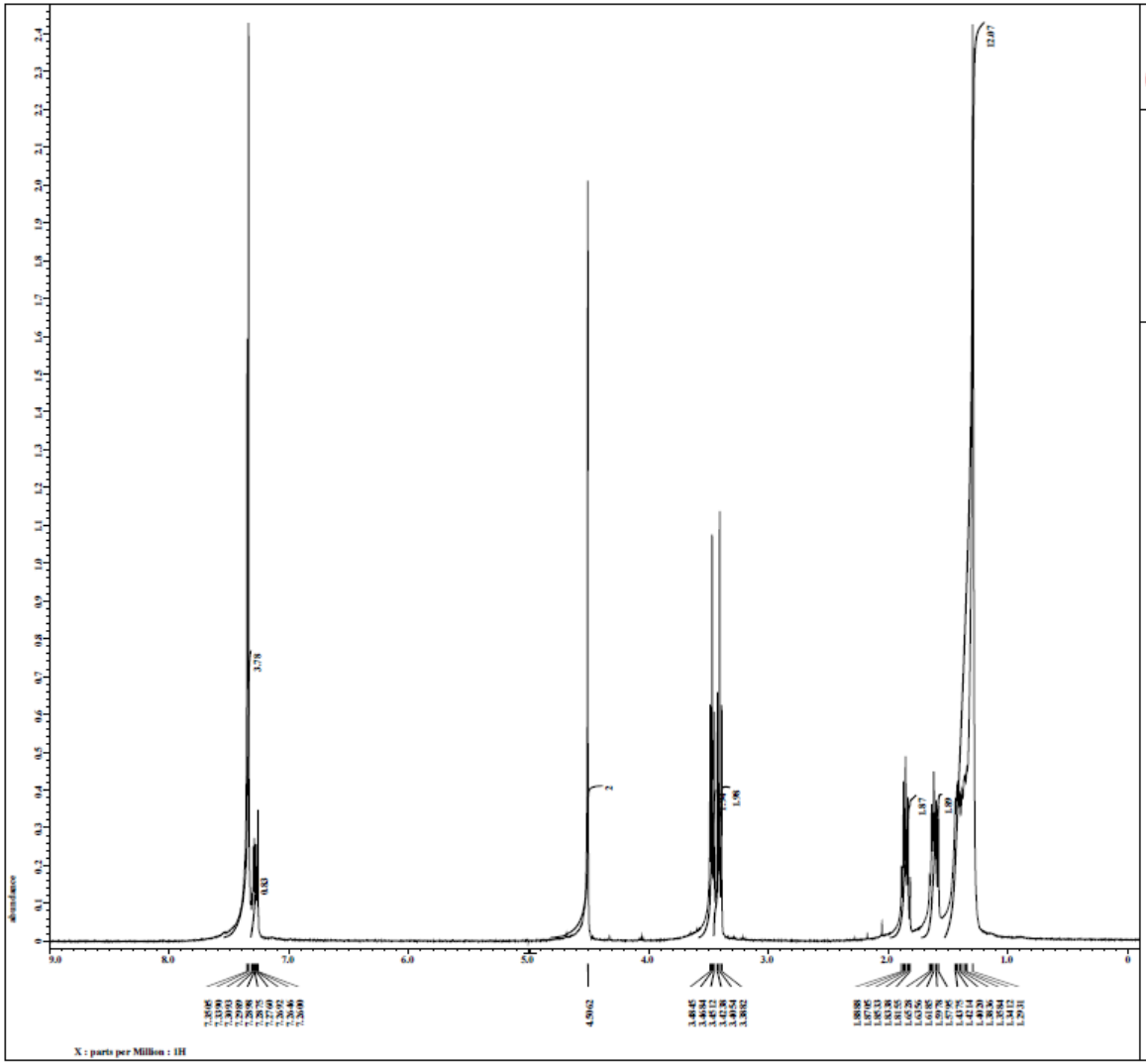
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = NCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T]  (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 42
Total_scans       = 42

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 22[dC]
```



(13a)




```
----- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
manualphase
ppm
auto_reference : 5[%] : TRUE

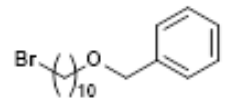
Derived from: 180830_Br_C10_Pb-1.jdf
```

```
Filename          = 180830_Br_C10_Pb-4.jd
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 30-AUG-2018 09:47:16
Revision_time     = 30-AUG-2018 10:28:18
Current_time      = 30-AUG-2018 10:28:45

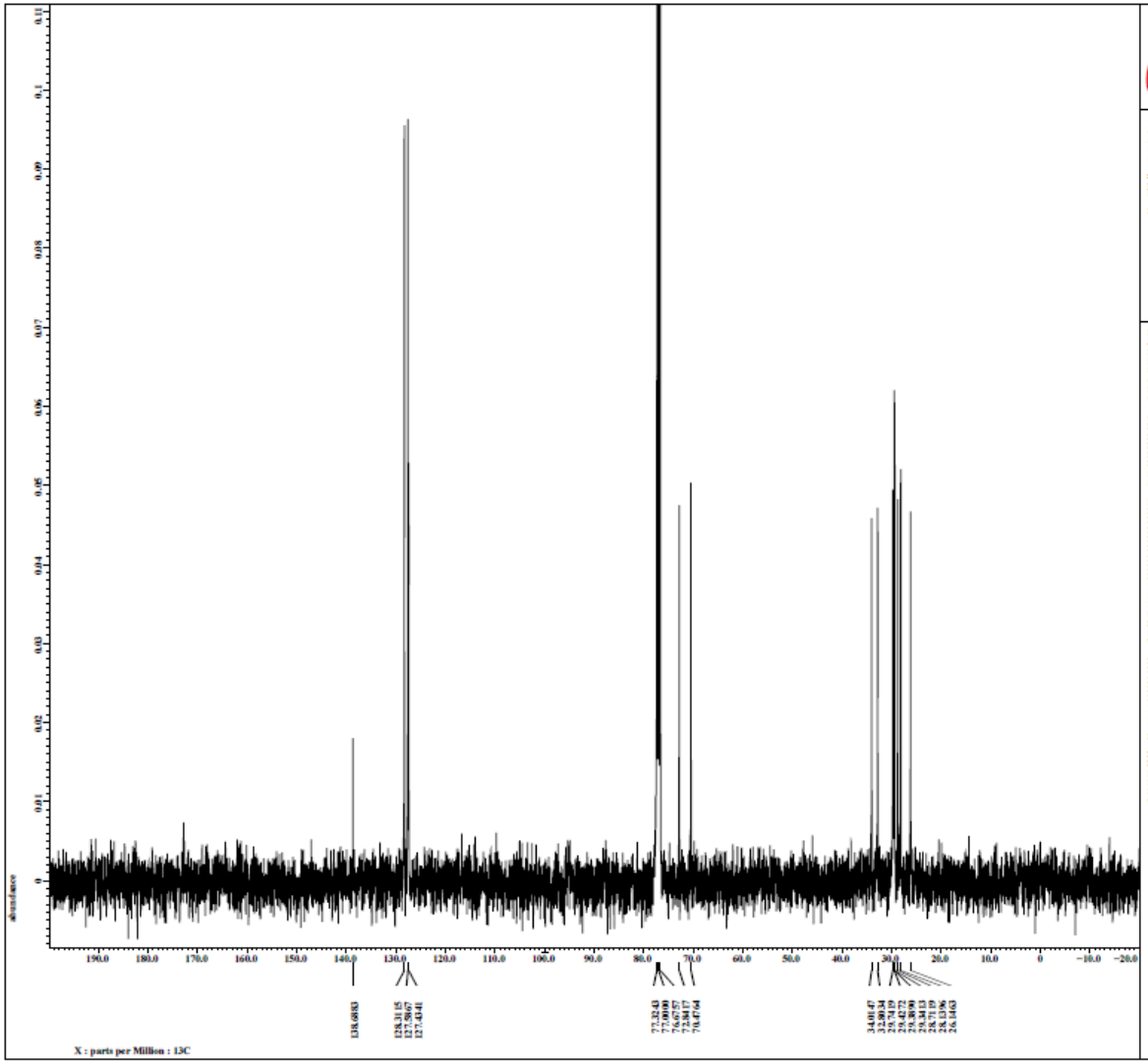
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [PPM]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 24.4[dC]
```



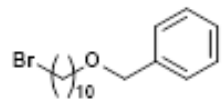
(14a)



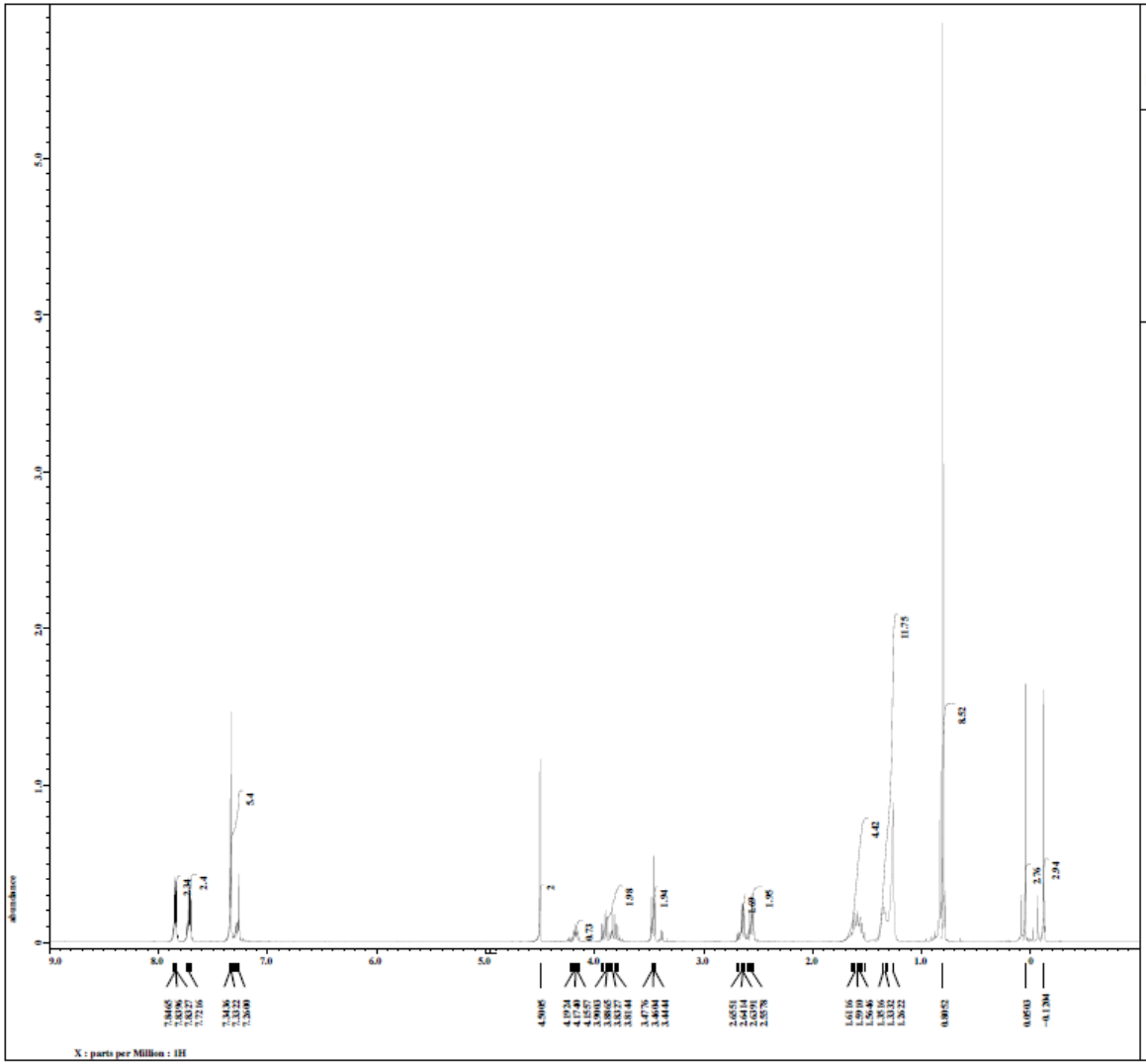
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
manchinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180830_13C_Br_C10_Ph-1.jdf

Filename	= 180830_13C_Br_C10_Ph-
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 30-AUG-2018 09:54:50
Revision_time	= 30-AUG-2018 10:24:11
Current_time	= 30-AUG-2018 10:24:46
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.92358061[Hz]
X_sweep	= 30.78817724[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 128
Total_scans	= 128
X_90_width	= 9.25[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 3.08333333[us]
Irr_atn_dec	= 17.0[dB]
Irr_atn_noe	= 17.0[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 3[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 24.7[dC]



(14a)




```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
expp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
pdm
auto_reference : 5[%] : TRUE

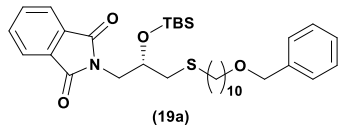
Derived from: 200206_phthN_OTBS_SC10_Fh-
```

```
Filename          = 200206_phthN_OTBS_SC1
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  5-FEB-2020 12:45:03
Revision_time     =  5-FEB-2020 15:05:29
Current_time      =  5-FEB-2020 15:05:53

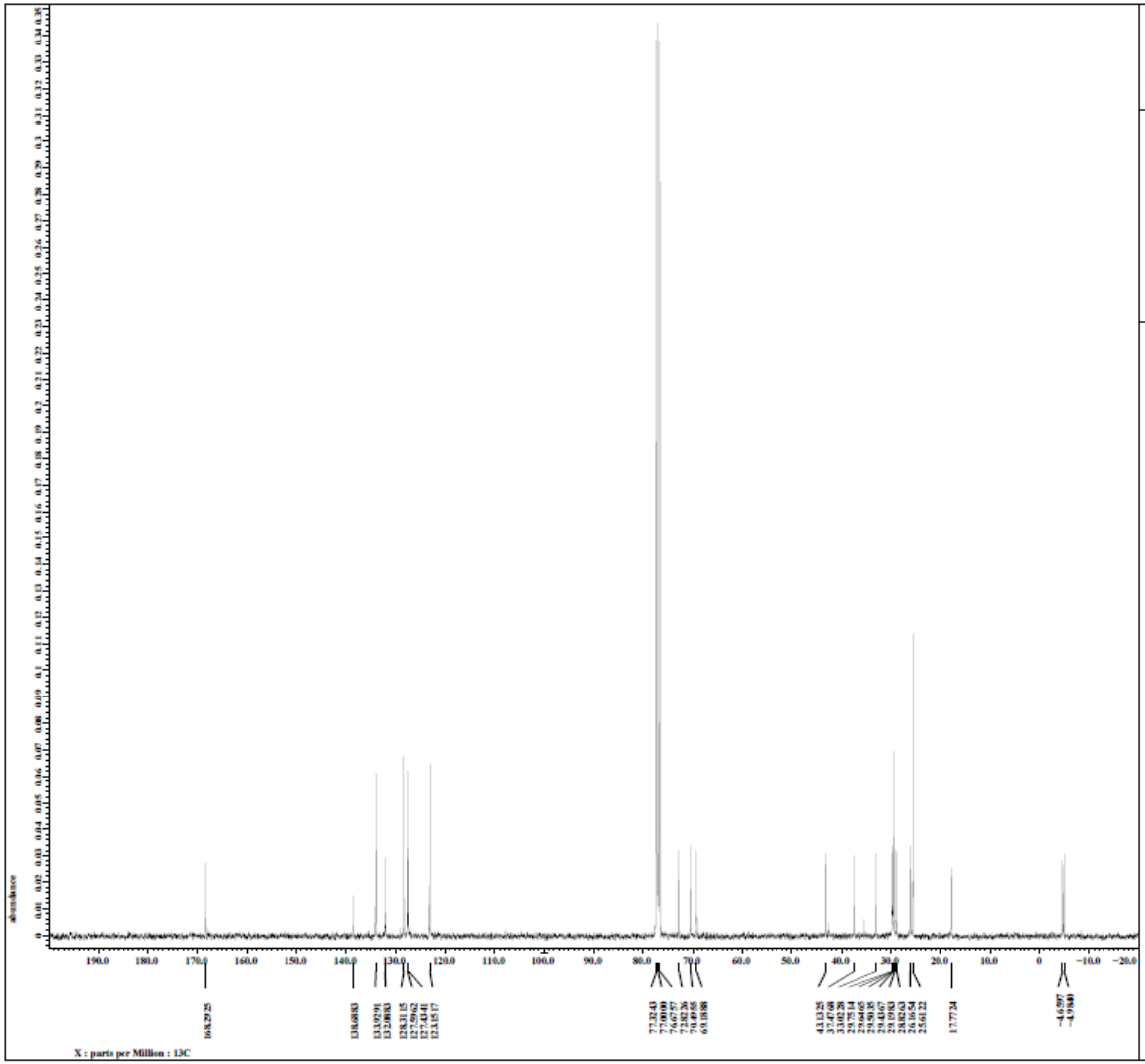
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK_400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 24
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 21.5[dC]
```



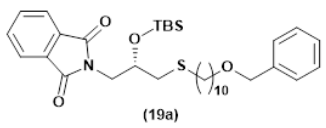
(19a)



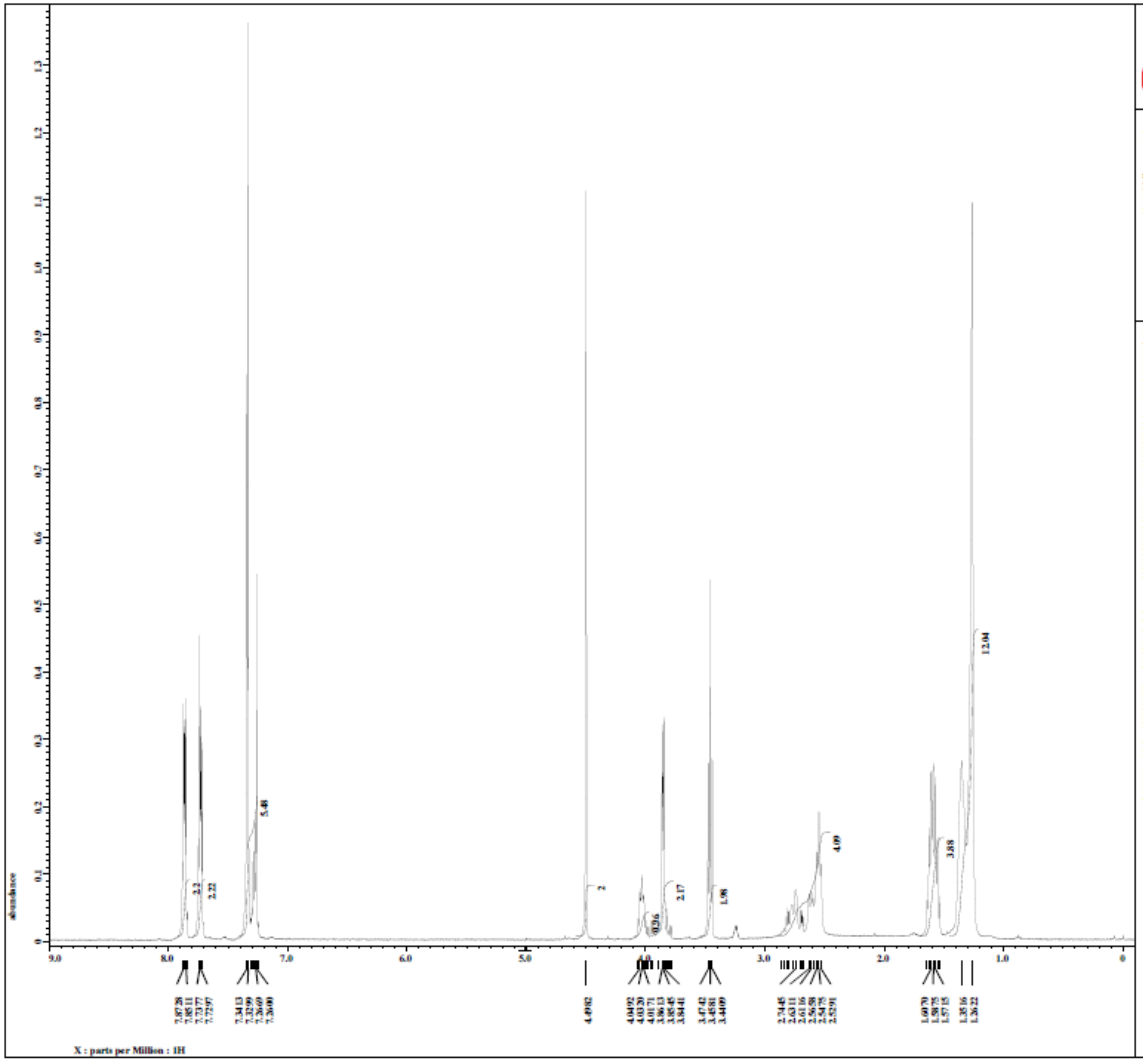
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 200206_13C_phthN_OTBS_SC10

Filename	= 200206_13C_phthN_OTBS
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2020 14:17:22
Revision_time	= 5-FEB-2020 14:52:01
Current_time	= 5-FEB-2020 14:53:52
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78517734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1792
Total_scans	= 1792
X_90_width	= 9.25[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 2.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 52
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 22.2[dC]



(19a)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
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trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ZTM
auto_reference : 5[%] : TRUE

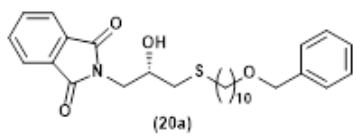
Derived from: 200206_2_phthN_OH_SC10_Fb-
```

```
Filename          = 200206_2_phthN_OH_SC1
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  5-FEB-2020 18:47:08
Revision_time     =  5-FEB-2020 19:24:49
Current_time      =  5-FEB-2020 19:25:23

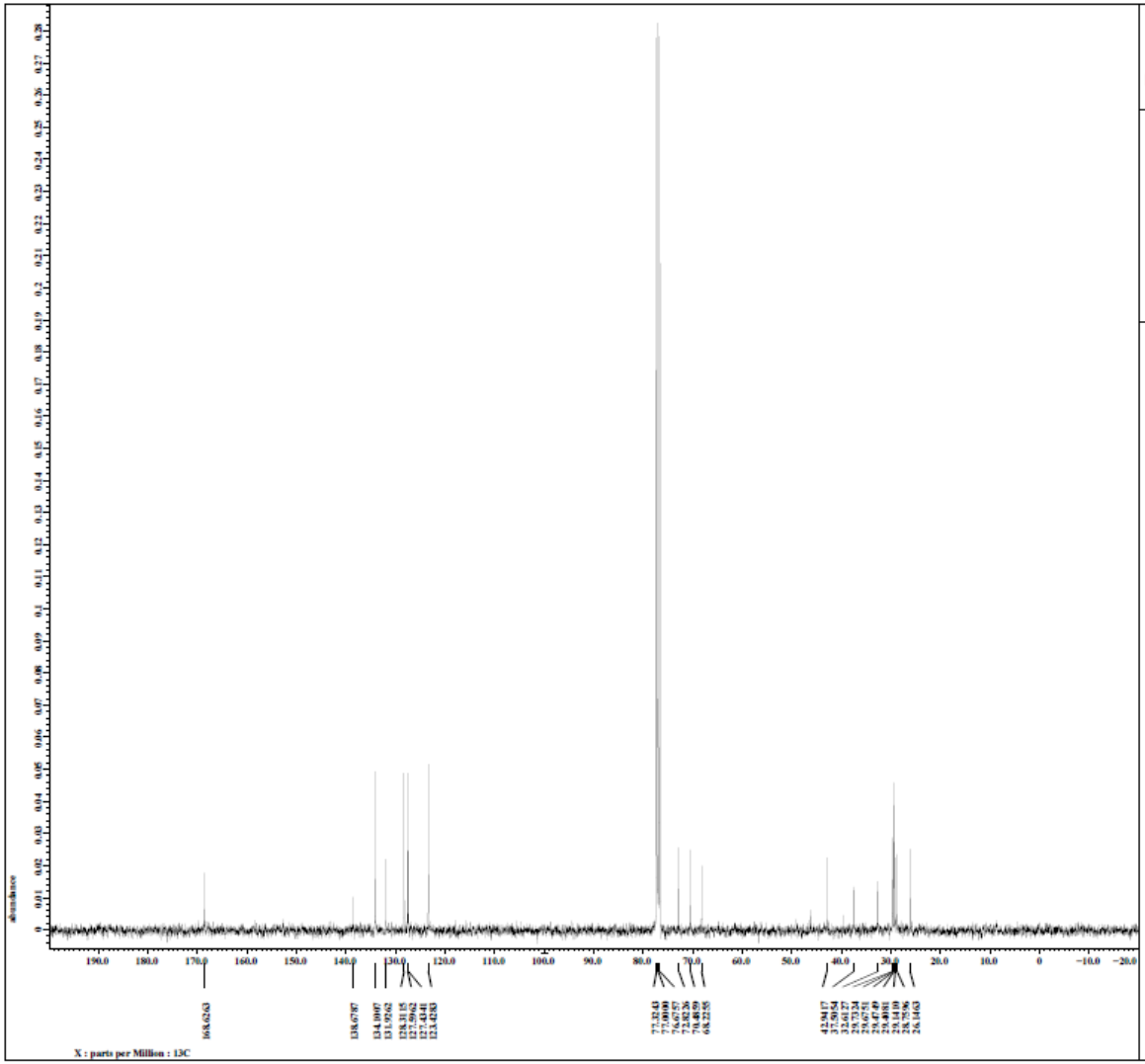
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK_400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[Hz]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22[dC]
```



(20a)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 200206_13C_2_phthN_OH_SC10
```

```
Filename          = 200206_13C_2_phthN_OH
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  5-FEB-2020 19:14:51
Revision_time     =  5-FEB-2020 19:49:57
Current_time      =  5-FEB-2020 19:50:28

Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78517734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 519
Total_scans       = 519

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.00333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 21.9[dC]
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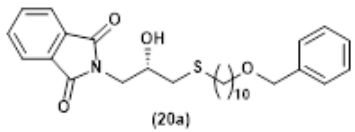
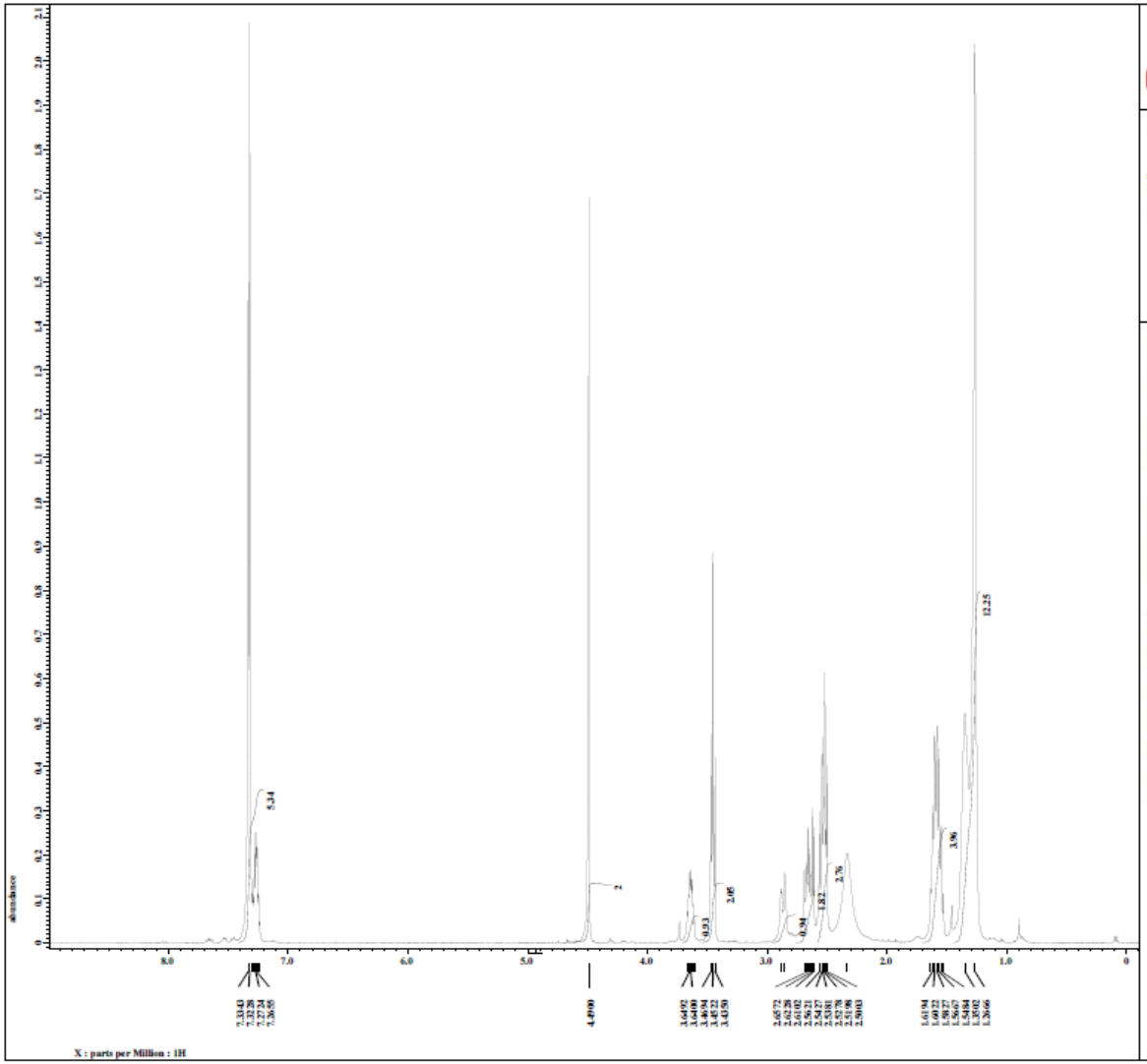


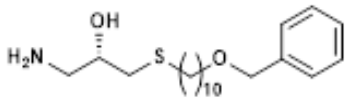
(20a)



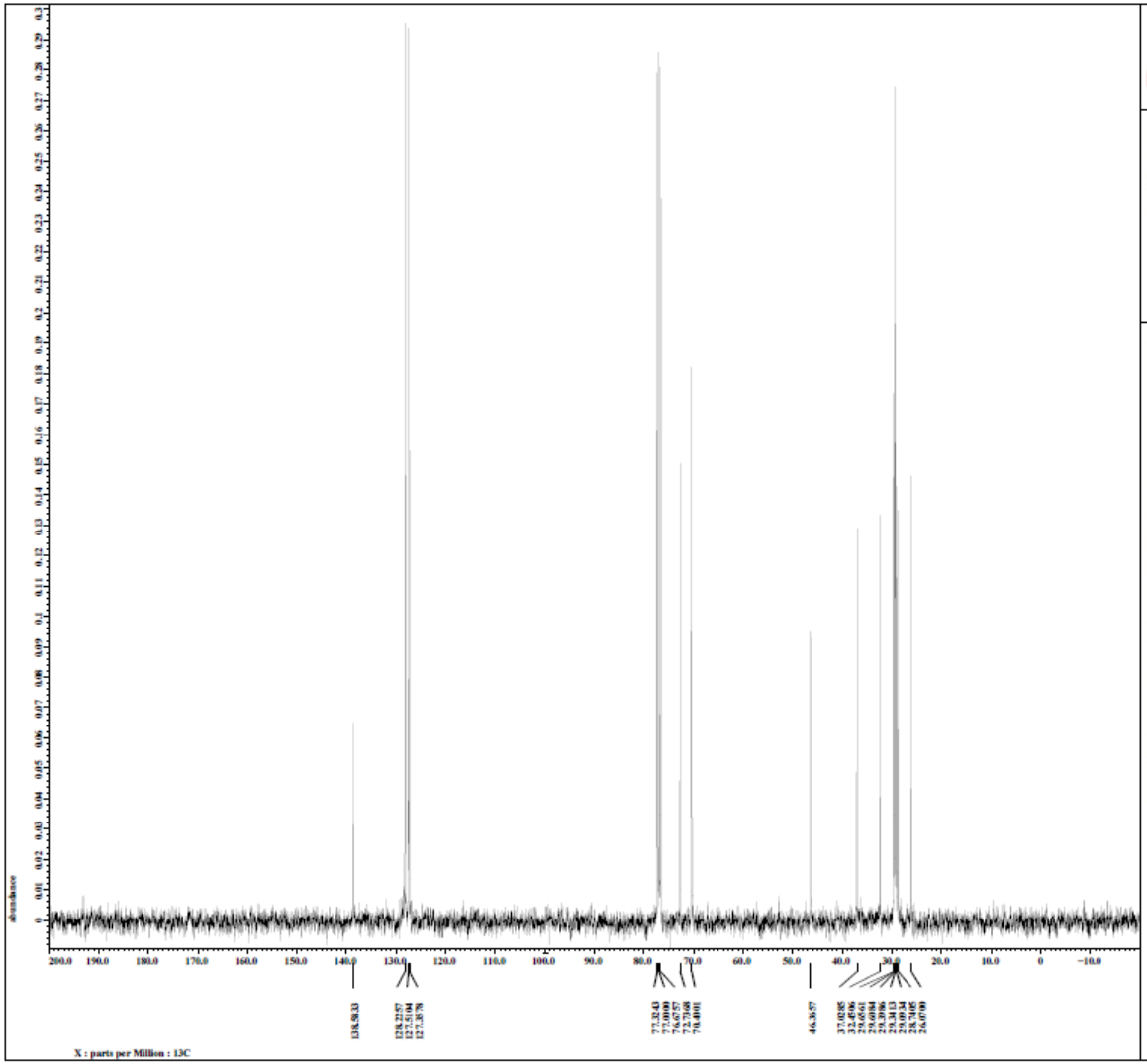
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 200206_HZN_OH_SC10_Ph-1.jd

Filename	= 200206_HZN_OH_SC10_Ph
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2020 18:24:18
Revision_time	= 5-FEB-2020 19:13:09
Current_time	= 5-FEB-2020 19:13:24
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= NCH 4000
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35234110[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 26
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 21.9[dC]



(21a)




```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

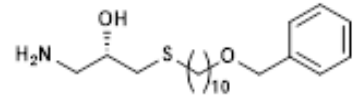
Derived from: 200206_13C_HZN_OH_SC10_Fb-
```

```
Filename          = 200206_13C_HZN_OH_SC1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  5-FEB-2020 18:40:24
Revision_time     =  5-FEB-2020 19:14:41
Current_time      =  5-FEB-2020 19:16:36

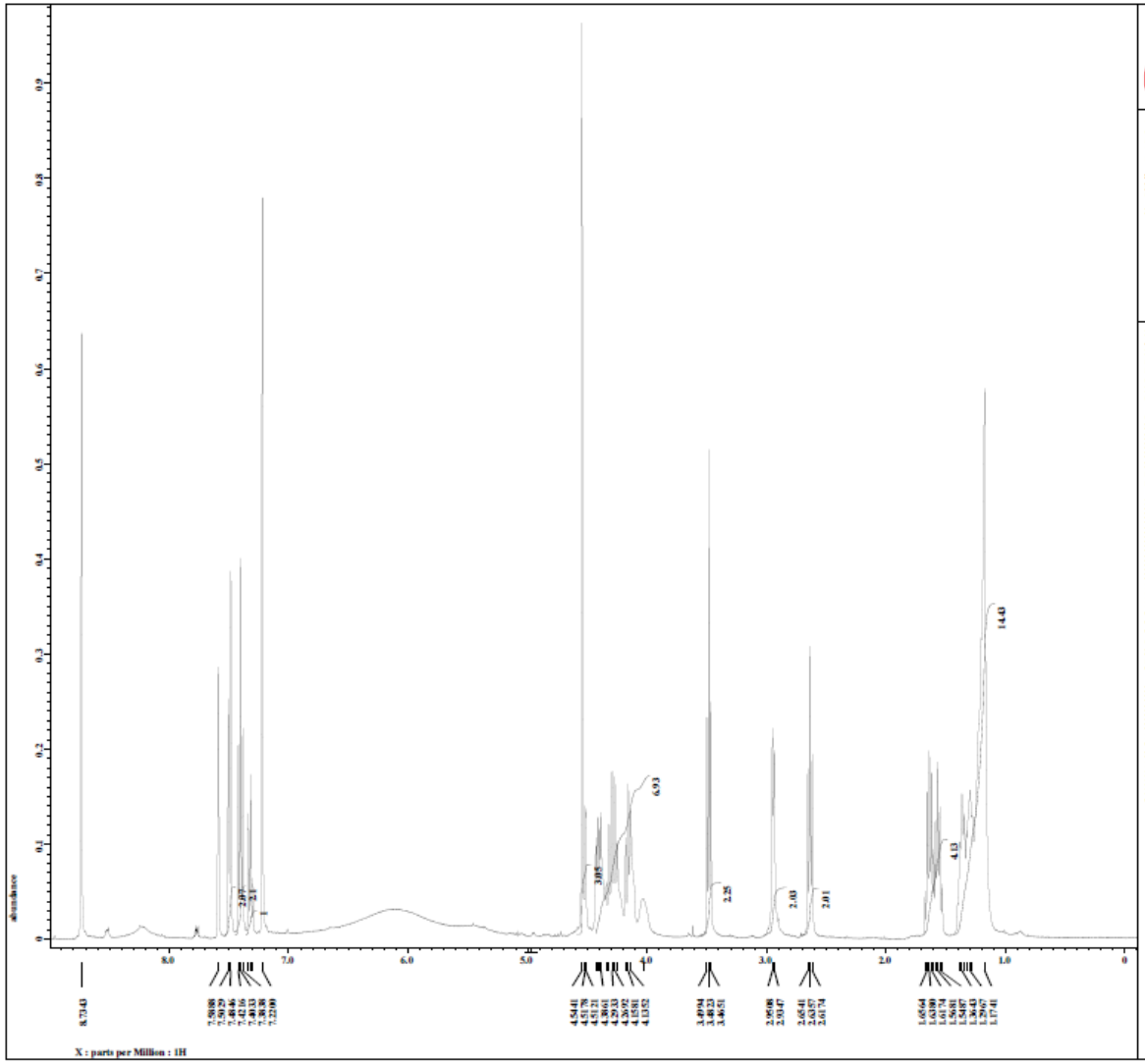
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.75817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 109
Total_scans       = 109

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.2[dC]
```



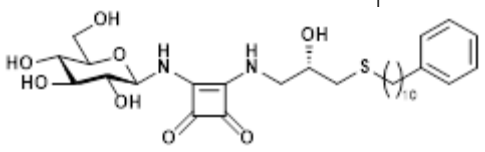
(21a)



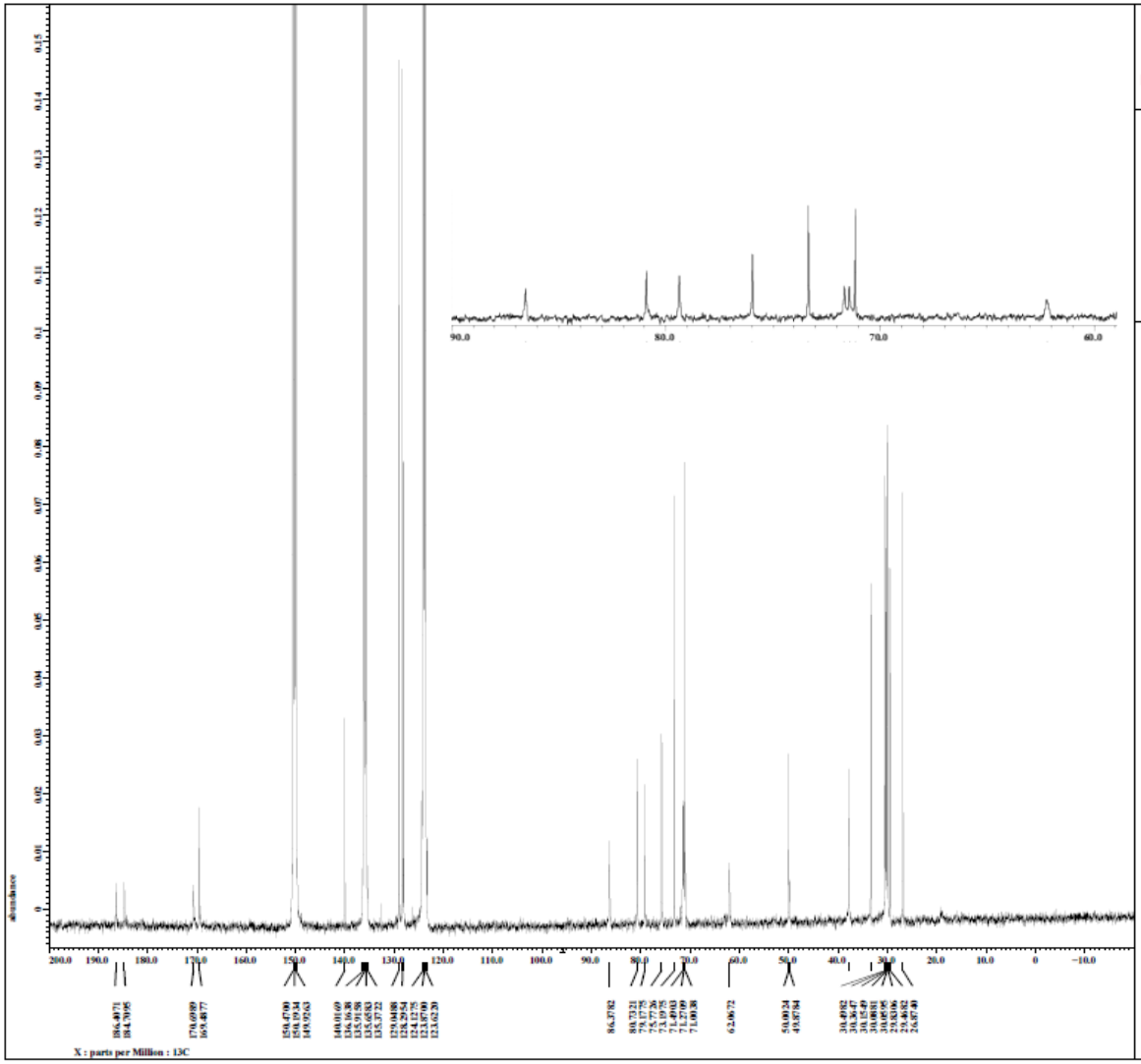
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180517_GlcDNS_Ph_1_10-1.jd

Filename	= 180517_GlcDNS_Ph_1_10
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= PYRIDINE-D5
Creation_time	= 15-MAY-2018 21:32:19
Revision_time	= 4-JUN-2019 16:23:28
Current_time	= 4-JUN-2019 16:23:57
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 20
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 21.6[dC]



(11a)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 2.0[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

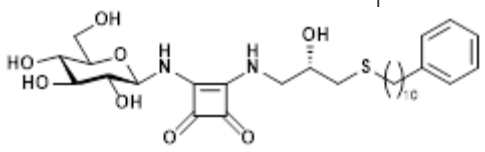
Derived from: 180517_13C_GlcDRS_Ph_1_10-
```

```
Filename          = 180517_13C_GlcDRS_Ph_
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 16-MAY-2018 02:40:52
Revision_time     =  4-JUN-2019 16:25:04
Current_time      =  4-JUN-2019 16:26:05

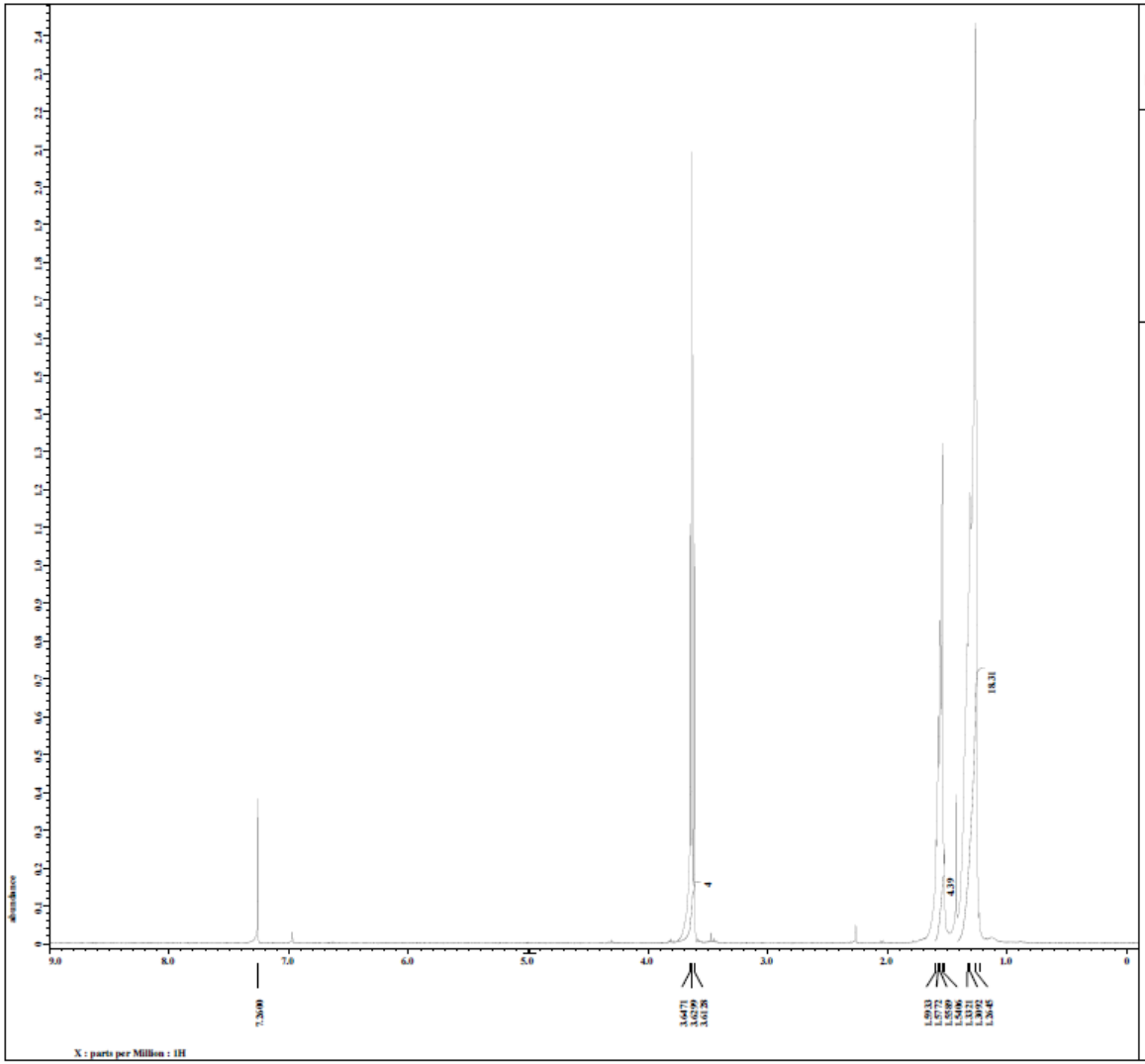
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = 1
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 6016
Total_scans       = 6016

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 21.9[dC]
```



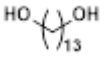
(11a)



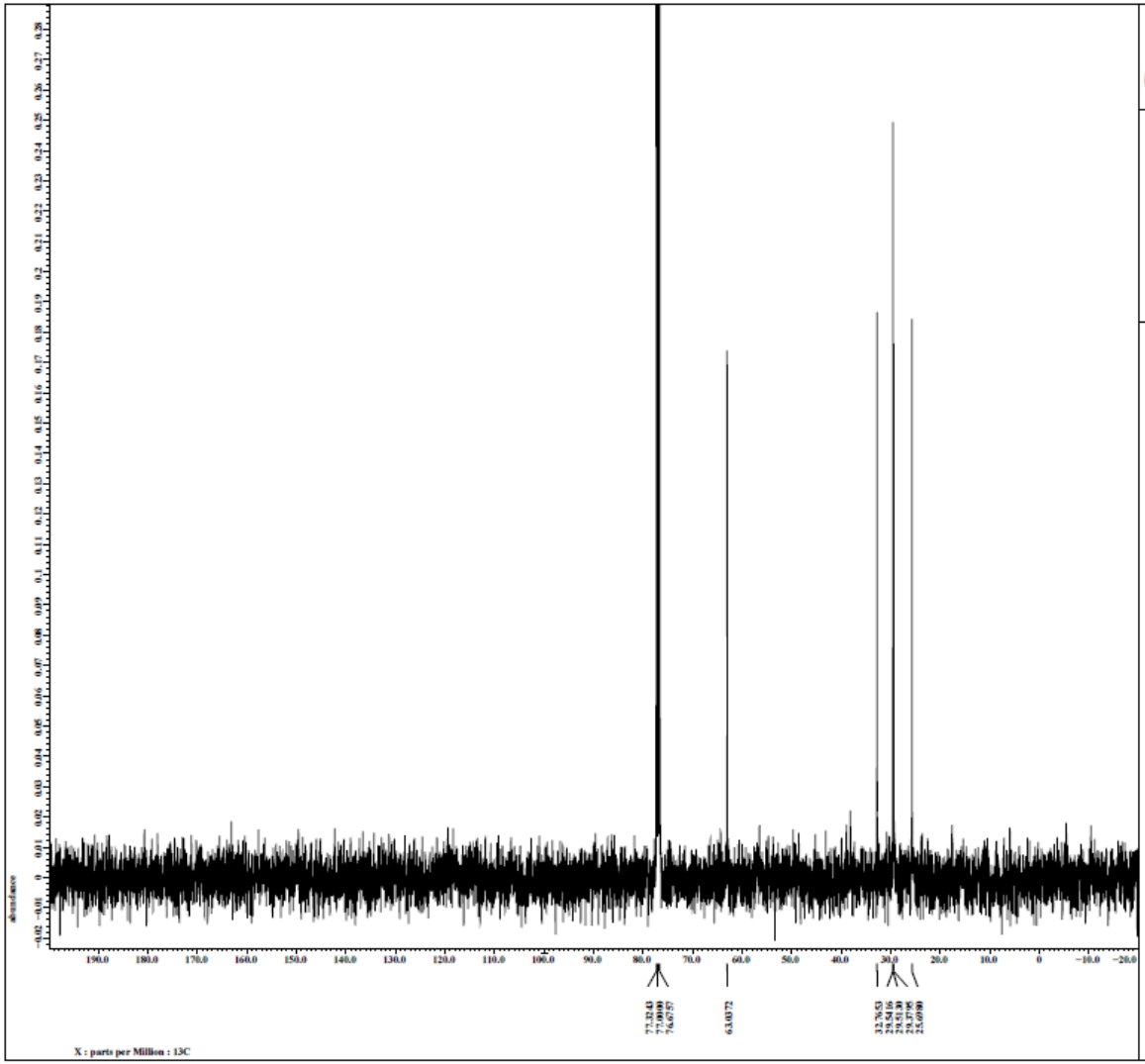
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
copy_ref : X :
auto_reference : 5[%] : TRUE
auto_reference : 5[%] : TRUE

Derived from: 180601_HO_C13_OH-1.jdf

Filename	= 180601_HO_C13_OH-5.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 1-JUN-2018 13:45:16
Revision_time	= 27-JAN-2020 11:29:06
Current_time	= 27-JAN-2020 11:29:47
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 5
Total_scans	= 5
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 26
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 25.2[dC]



(12b)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

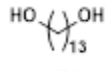
Derived from: 180828_13C_HO_C13_OH-1.jdf
```

```
Filename          = 180828_13C_HO_C13_OH-
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 28-AUG-2018 16:48:55
Revision_time     = 28-AUG-2018 17:35:06
Current_time      = 28-AUG-2018 17:35:39

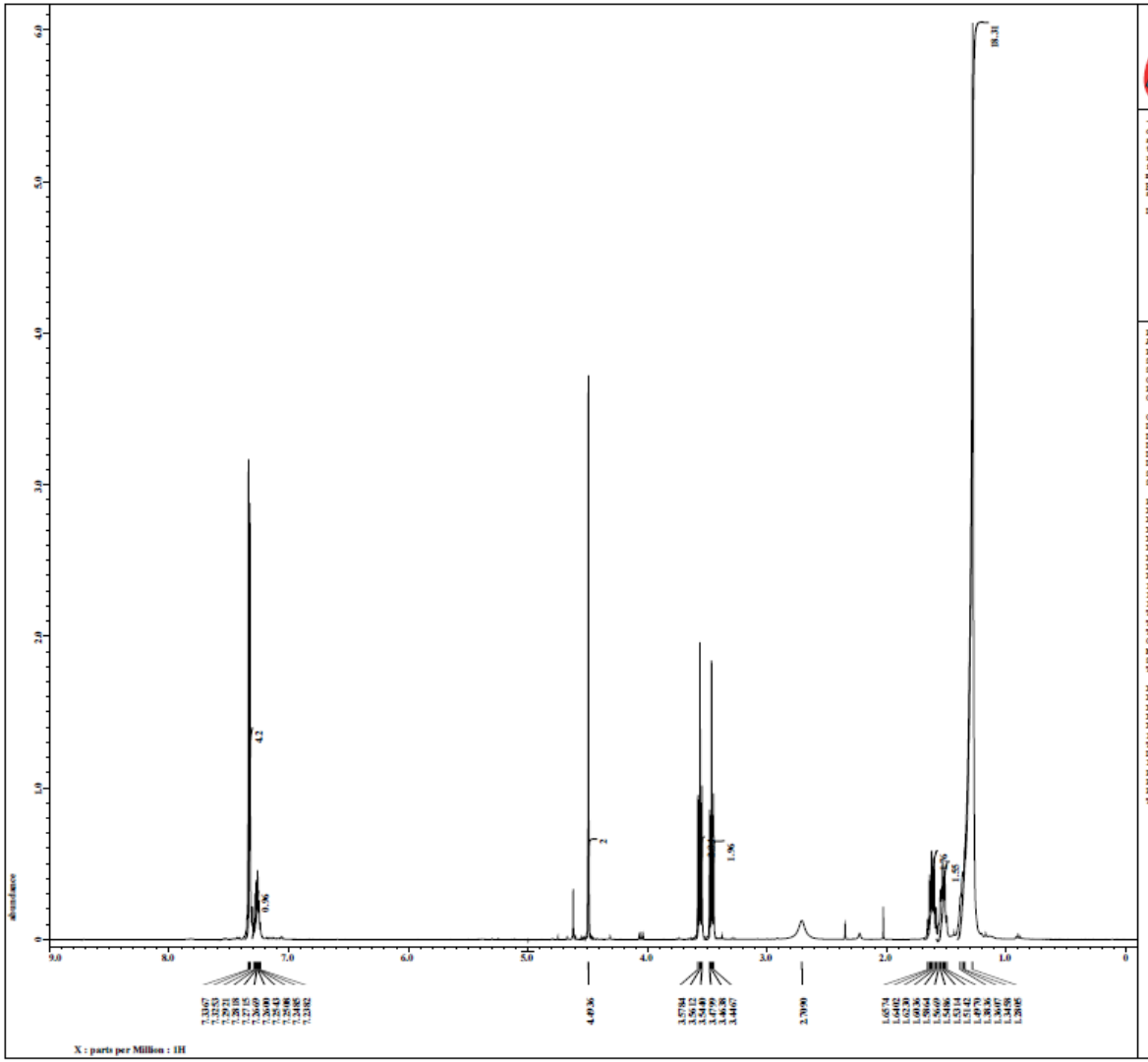
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [PPM]
Dimensions        = X
Site              = RCK 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.76817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 37
Total_scans       = 37

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.0[dB]
Irr_atn_noe       = 17.0[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   = 3.06420464[s]
Temp_get          = 24.8[dC]
```



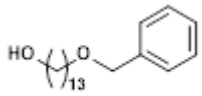
(12b)



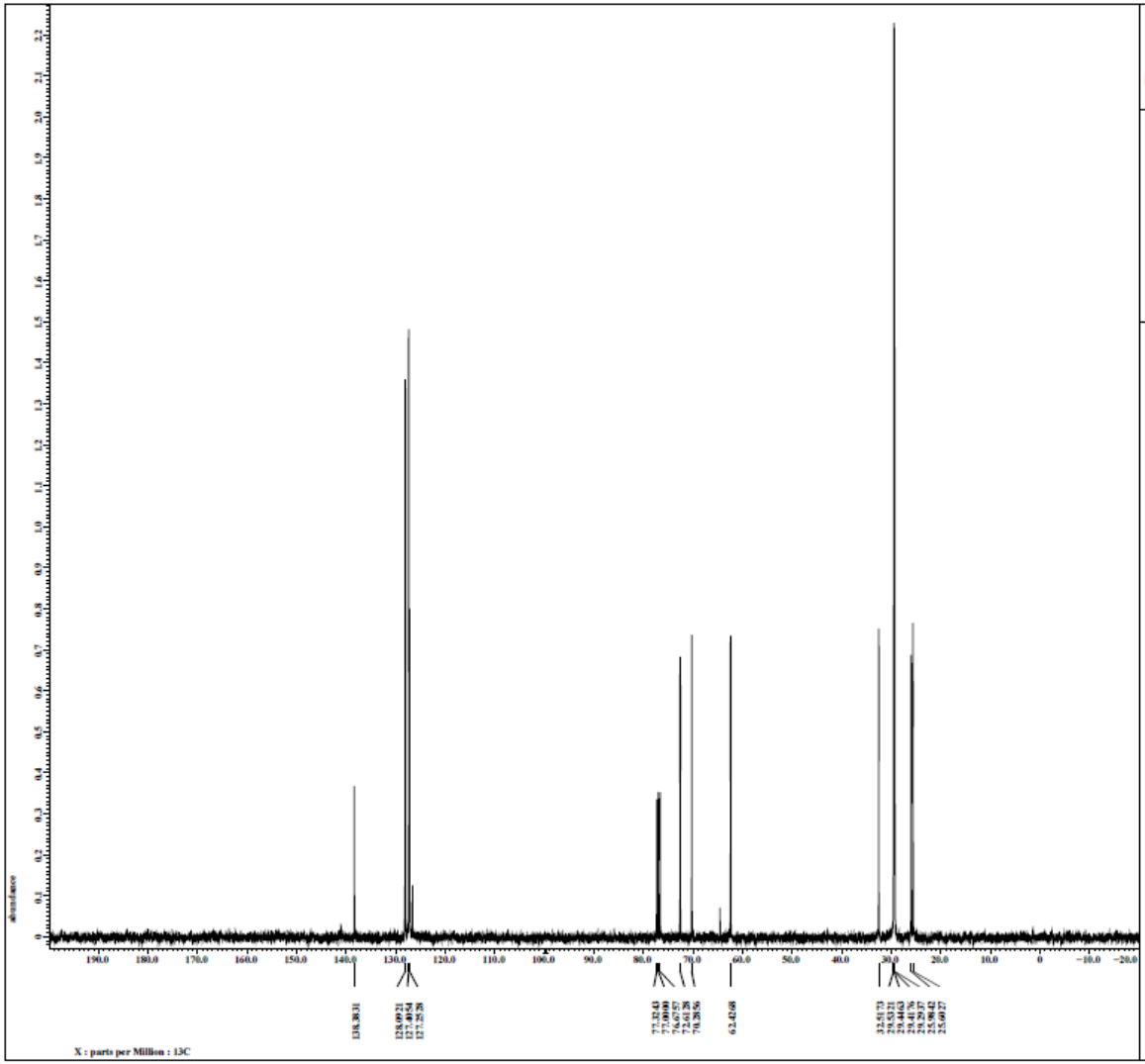
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180611_C13_diol_monoPh-1.j
```

Filename	= 180611_C13_diol_monoP
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 11-JUN-2018 10:14:25
Revision_time	= 28-AUG-2018 14:38:24
Current_time	= 28-AUG-2018 14:39:20
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 7[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 5
Total_scans	= 5
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 18
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 24.7[dC]



(13b)

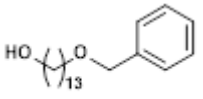


JEOL

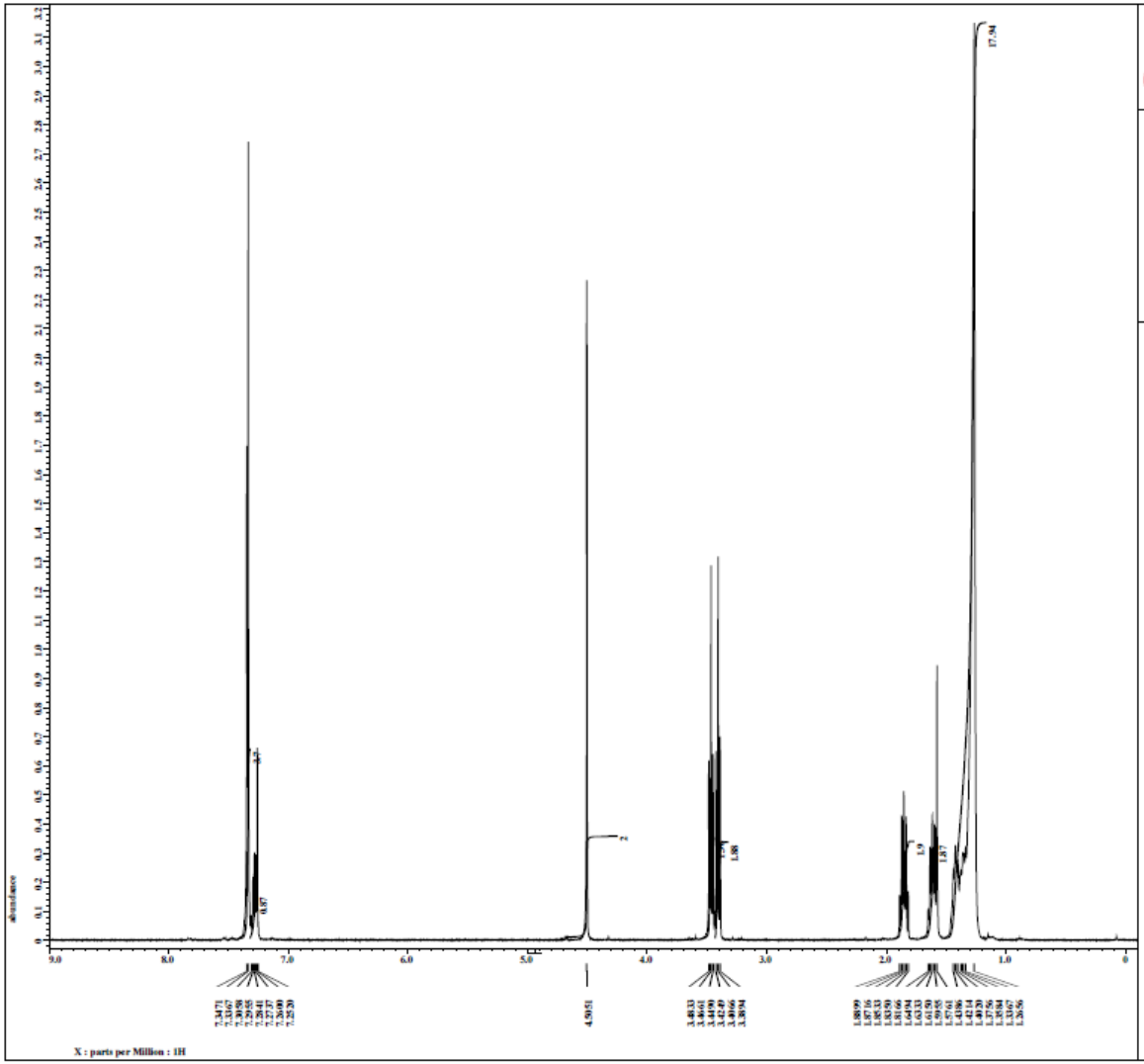
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180611_13C_NO_C13_Ph-3.jdf
```

Filename	= 180611_13C_NO_C13_Ph-
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 11-JUN-2018 10:15:59
Revision_time	= 28-AUG-2018 14:41:15
Current_time	= 28-AUG-2018 14:41:53
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06420464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93858061[Hz]
X_sweep	= 30.75817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 17
Total_scans	= 17
X_90_width	= 9.25[us]
X_acq_time	= 1.06420464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 3.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06420464[s]
Temp_get	= 24.9[dC]



(13b)



JEOL

```
----- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

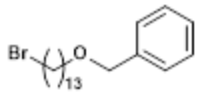
Derived from: 180829_Br_C13_Fb-1.jdf
```

```
Filename          = 180829_Br_C13_Fb-4.jd
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 29-AUG-2018 16:05:13
Revision_time     = 29-AUG-2018 16:25:09
Current_time      = 29-AUG-2018 16:25:25

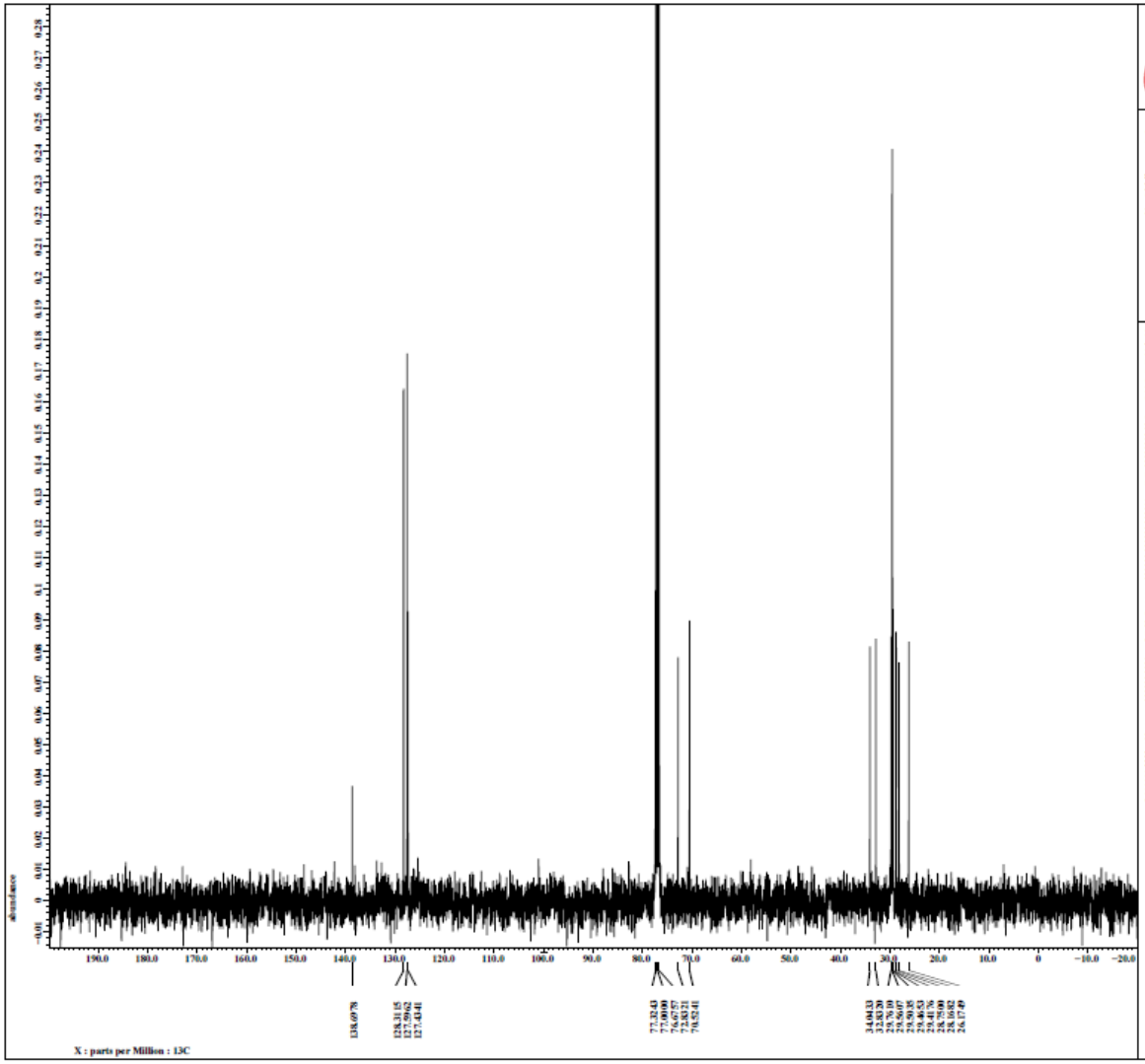
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 131072
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 7
Total_scans       = 7

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 24.5[dC]
```



(14b)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

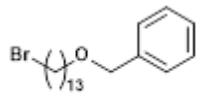
Derived from: 180829_13C_Br_C13_Ph-1.jdf
```

```
Filename          = 180829_13C_Br_C13_Ph-
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 29-AUG-2018 18:14:32
Revision_time     = 29-AUG-2018 18:40:37
Current_time      = 29-AUG-2018 18:42:29

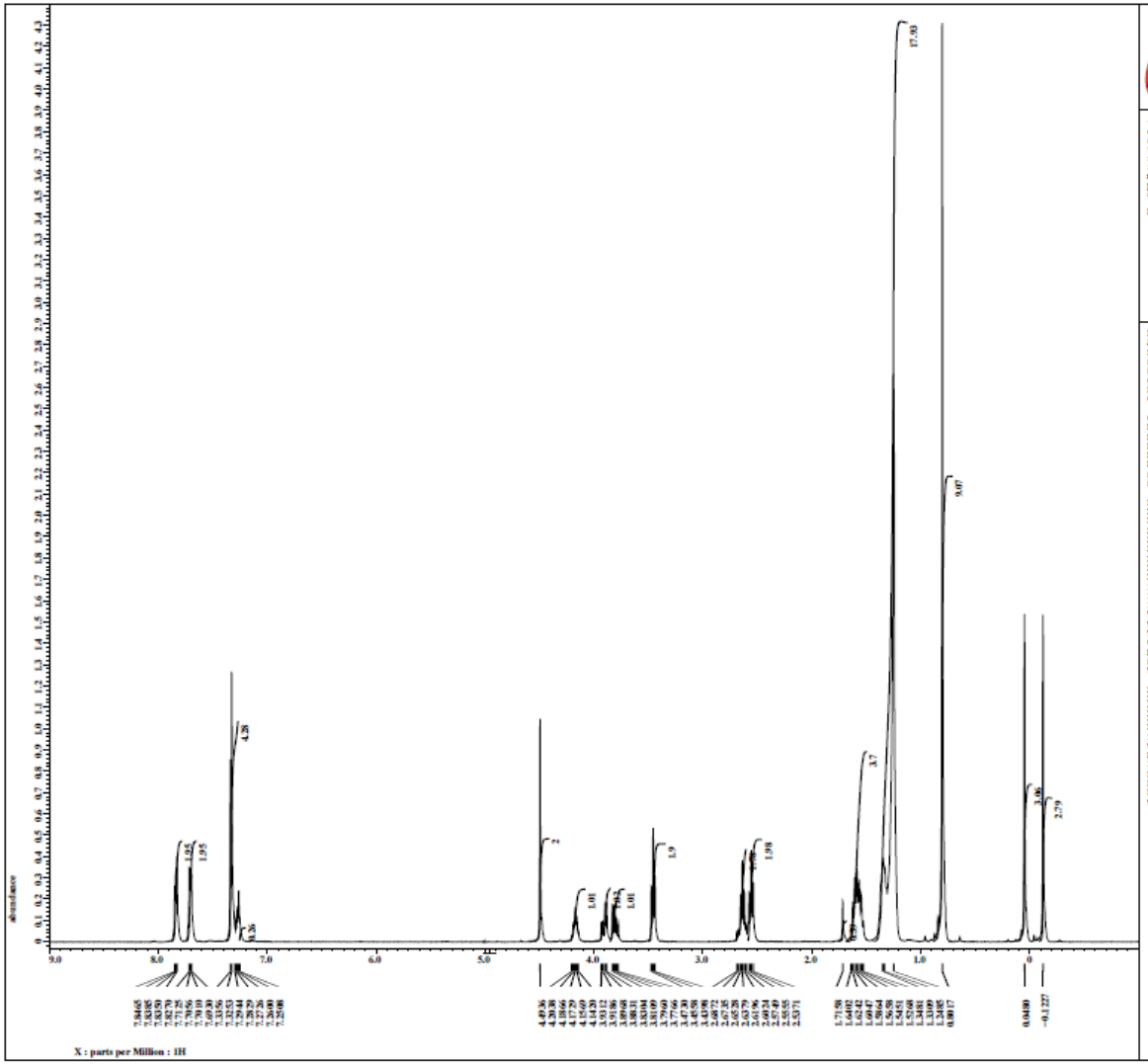
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 109
Total_scans       = 109

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06420464[s]
Temp_get          = 24.7[dC]
```



(14b)



```
---- 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
auto_reference : 5[%] : TRUE

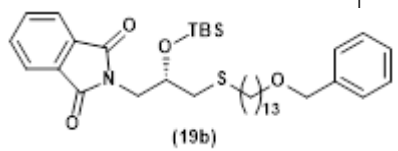
Derived from: 180726_phth_TBS_C13_Ph-1.j
```

```
Filename          = 180726_phth_TBS_C13_P
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 26-JUL-2018 16:53:21
Revision_time     = 26-JUL-2018 19:26:29
Current_time      = 26-JUL-2018 19:27:15

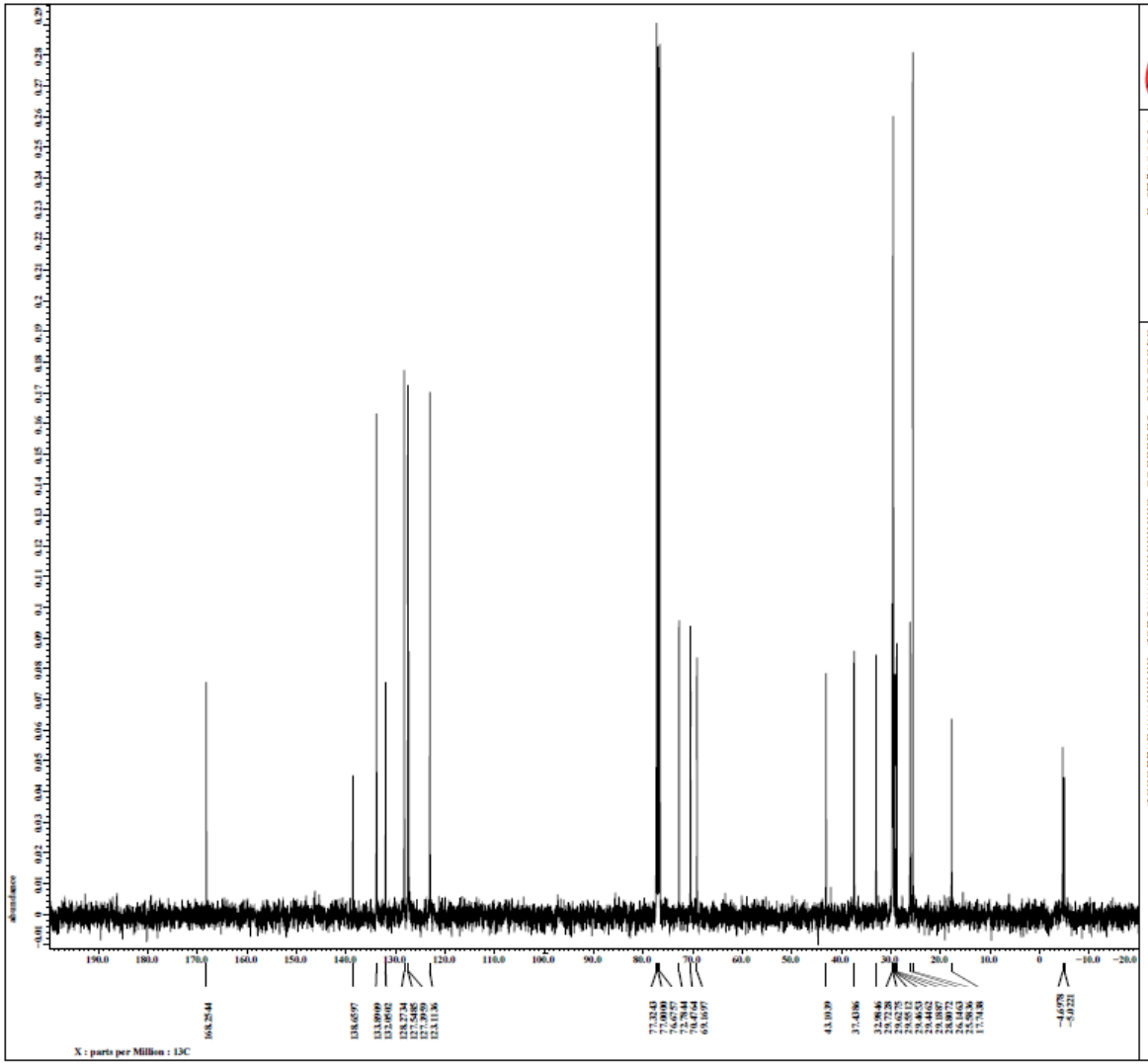
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.3[dC]
```



(19b)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

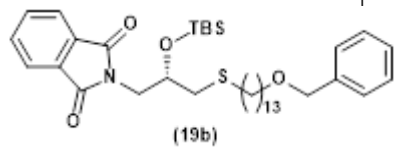
Derived from: 180726_13C_phth_TBS_C13_Fh
```

```
Filename          = 180726_13C_phth_TBS_C
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 26-JUL-2018 16:59:21
Revision_time     = 26-JUL-2018 19:27:51
Current_time      = 26-JUL-2018 19:28:36

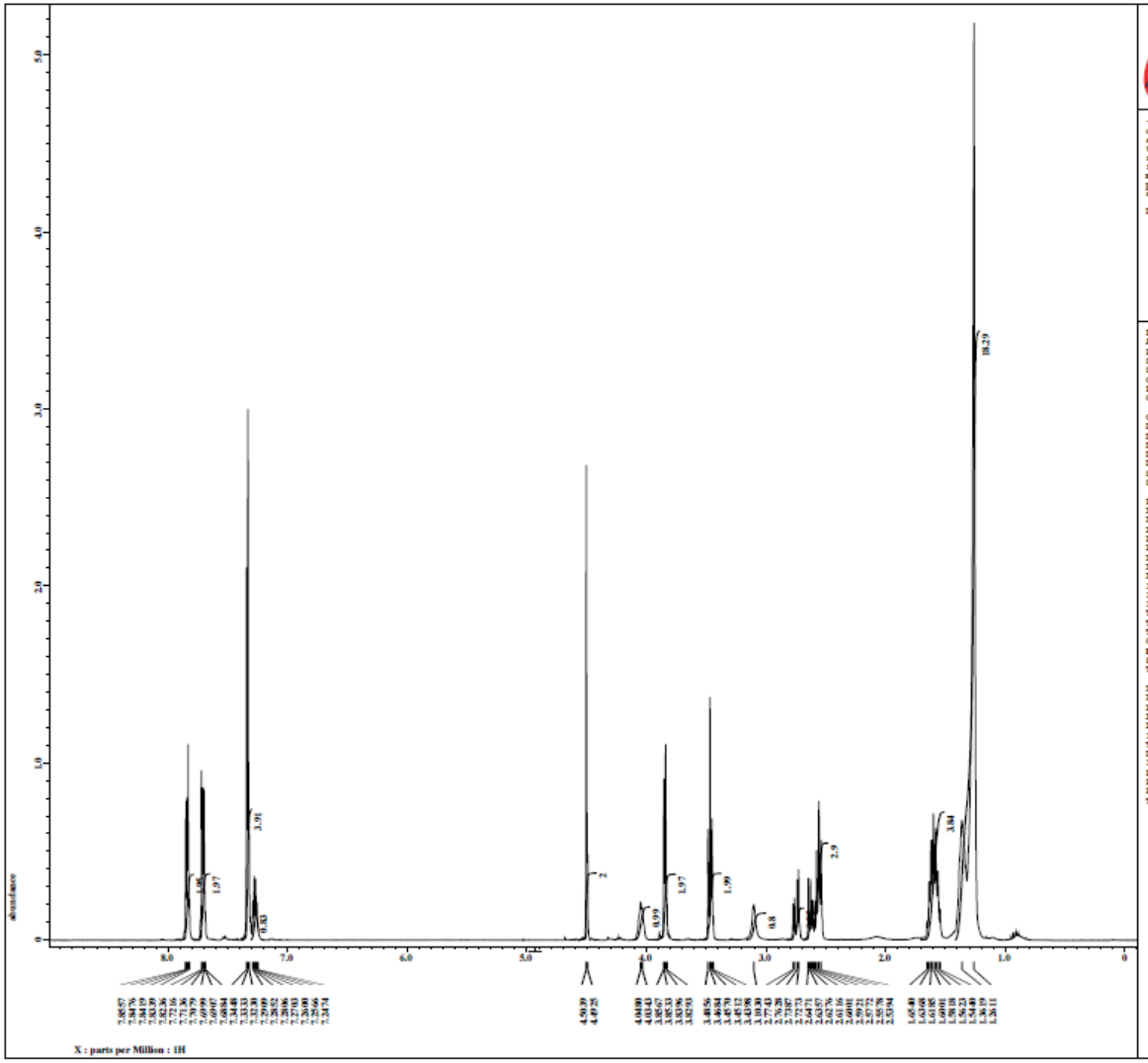
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 97
Total_scans       = 97

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.7[dC]
```



(19b)



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
auto_reference : 5[%] : TRUE

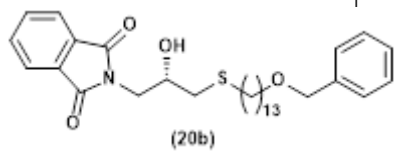
Derived from: 180731_phth_OH_C13_Ph-1.jd
```

```
Filename          = 180731_phth_OH_C13_Ph
Author            = delta
Experiment        = single_pulse.exp2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 31-JUL-2018 14:22:47
Revision_time     =  4-SEP-2018 10:37:54
Current_time      =  4-SEP-2018 10:38:36

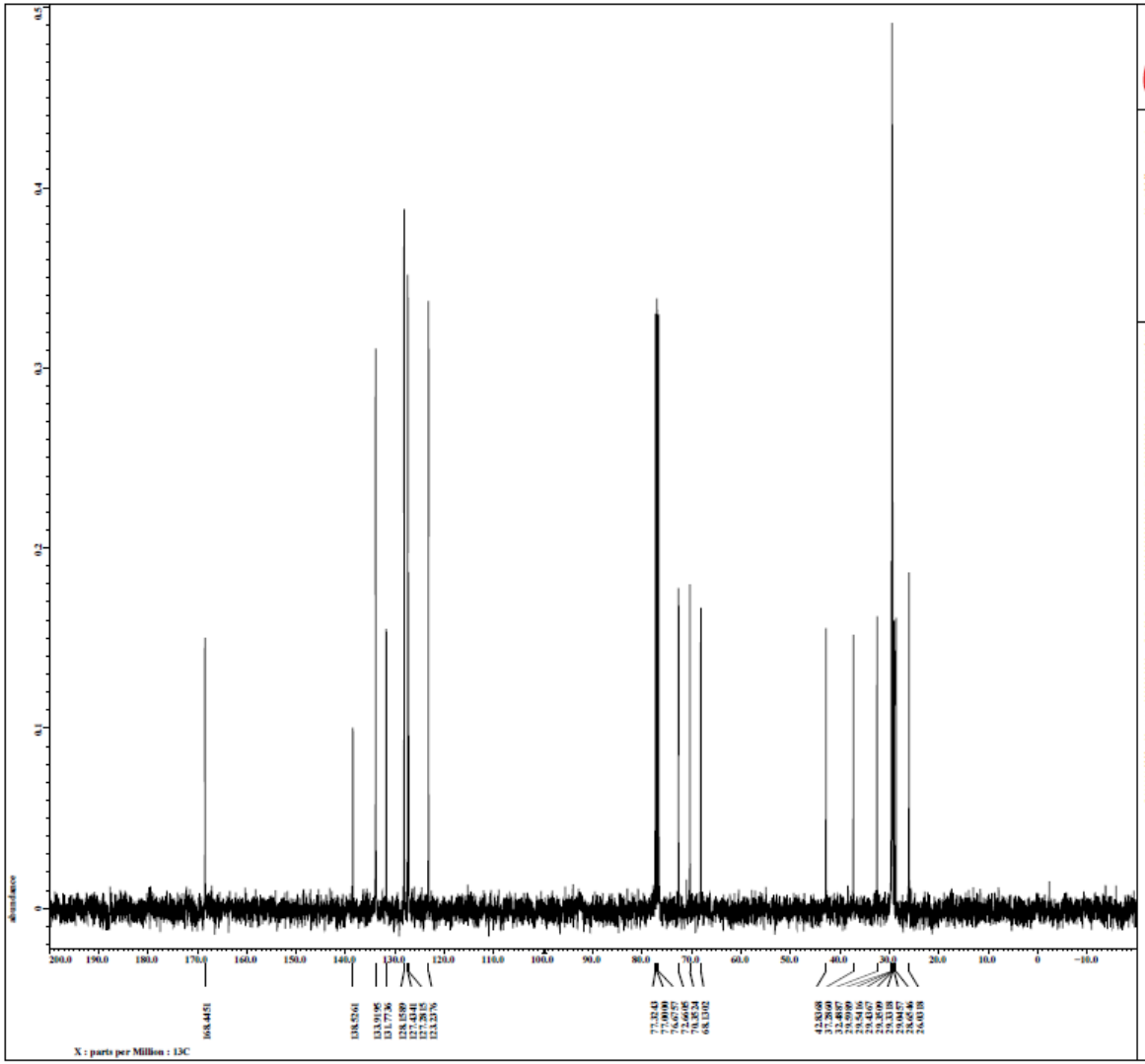
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 28
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 21.9[dC]
```



(20b)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

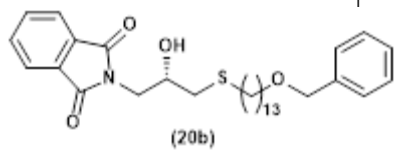
Derived from: 180721_13C_phth_OH_C13_Pb-
```

```
Filename          = 180721_13C_phth_OH_C1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 21-JUL-2018 14:25:26
Revision_time     = 21-JUL-2018 14:56:21
Current_time      = 21-JUL-2018 14:57:46

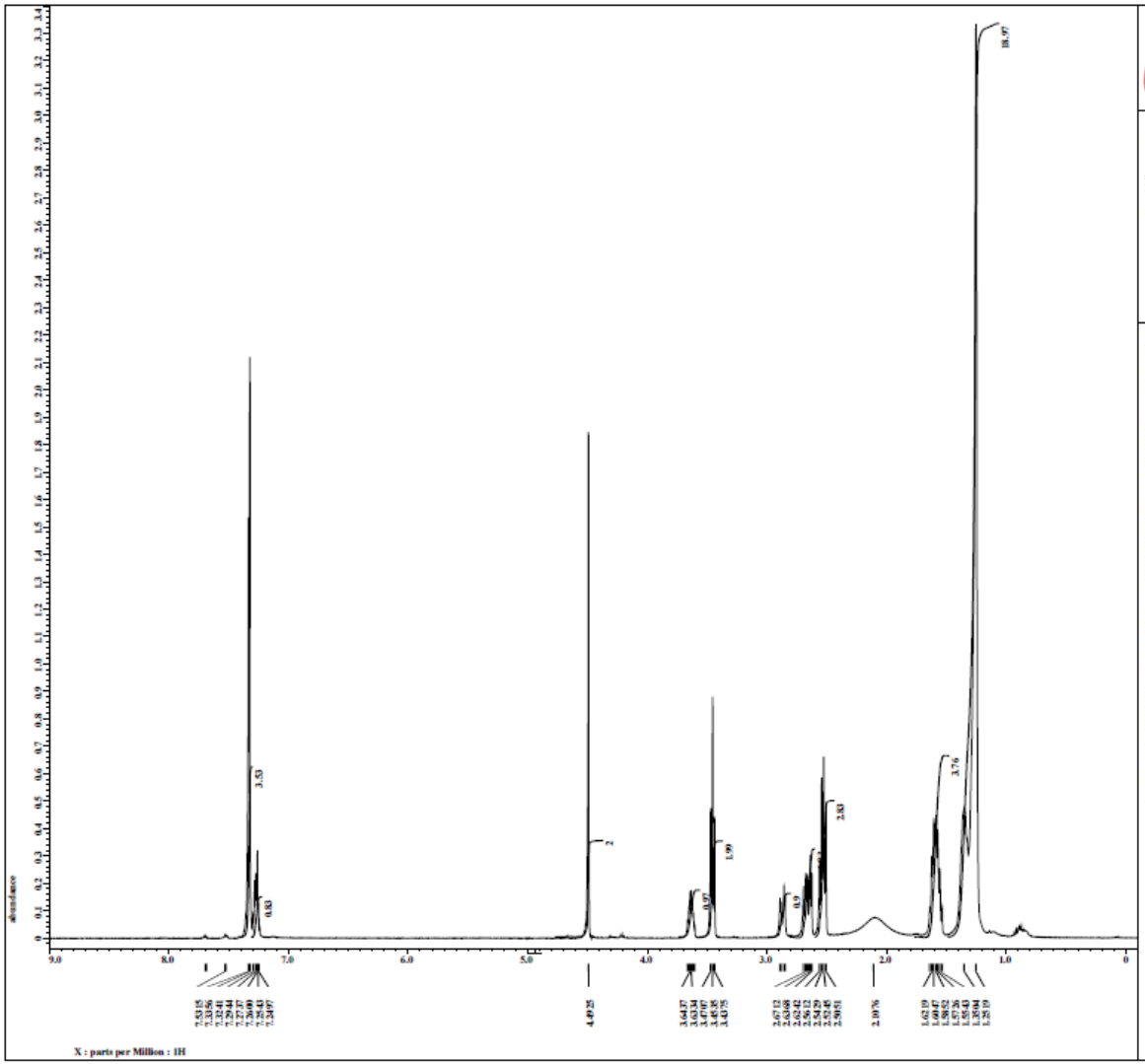
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 25
Total_scans       = 25

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.05333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.2[dC]
```



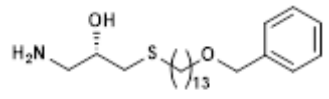
(20b)



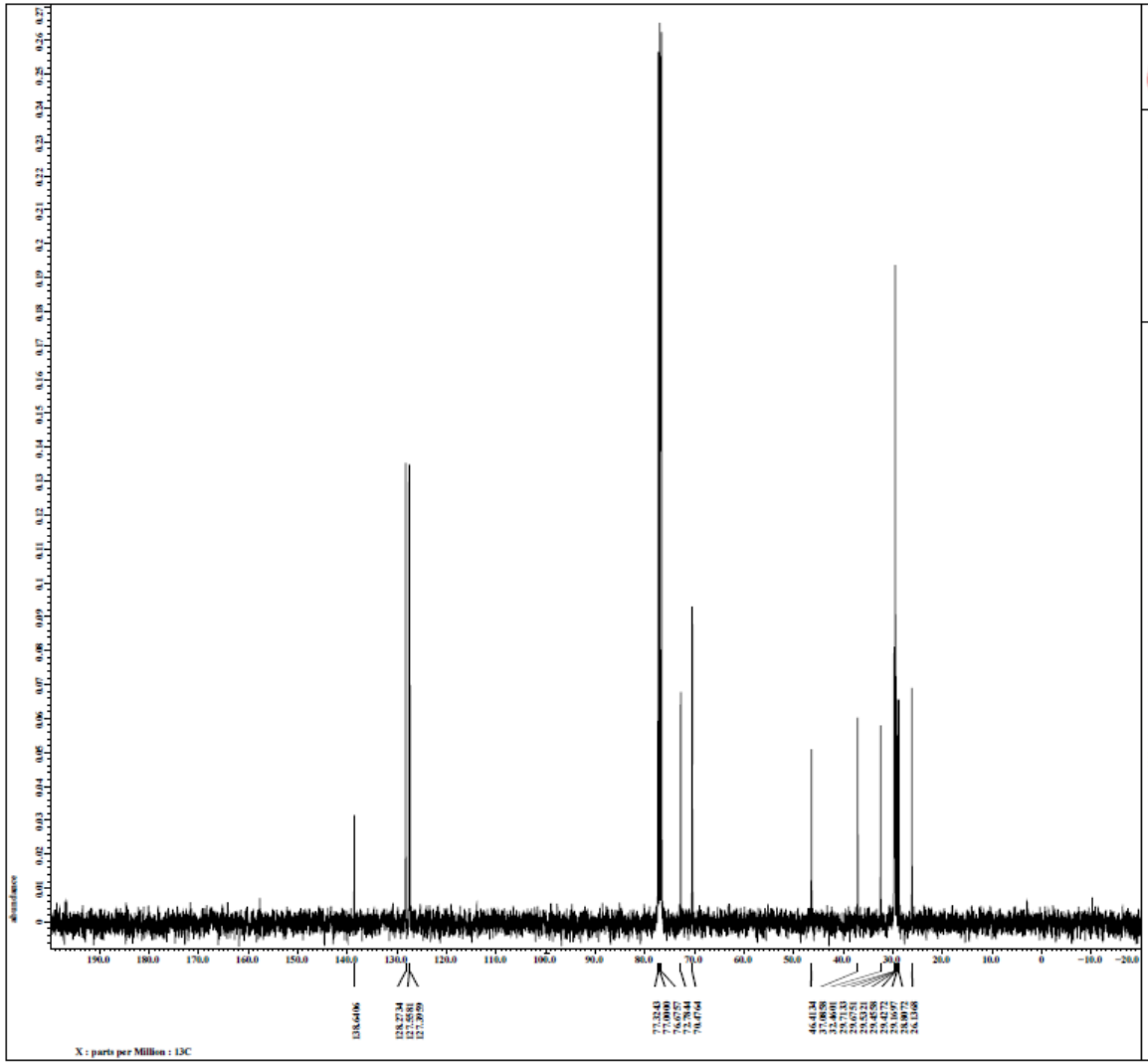
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
expp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 2
fft : 1 : TRUE : TRUE
manualphase
FFT
auto_reference : 5[%] : TRUE

Derived from: 180801_phth_OH_C13_Ph-1.jd
```

Filename	= 180801_phth_OH_C13_Ph
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 1-AUG-2018 09:34:26
Revision_time	= 1-AUG-2018 09:59:14
Current_time	= 1-AUG-2018 09:59:50
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 12107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK_400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 5
Total_scans	= 5
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 24
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 22.4[dC]



(21b)

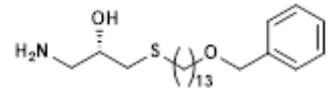


JEOL

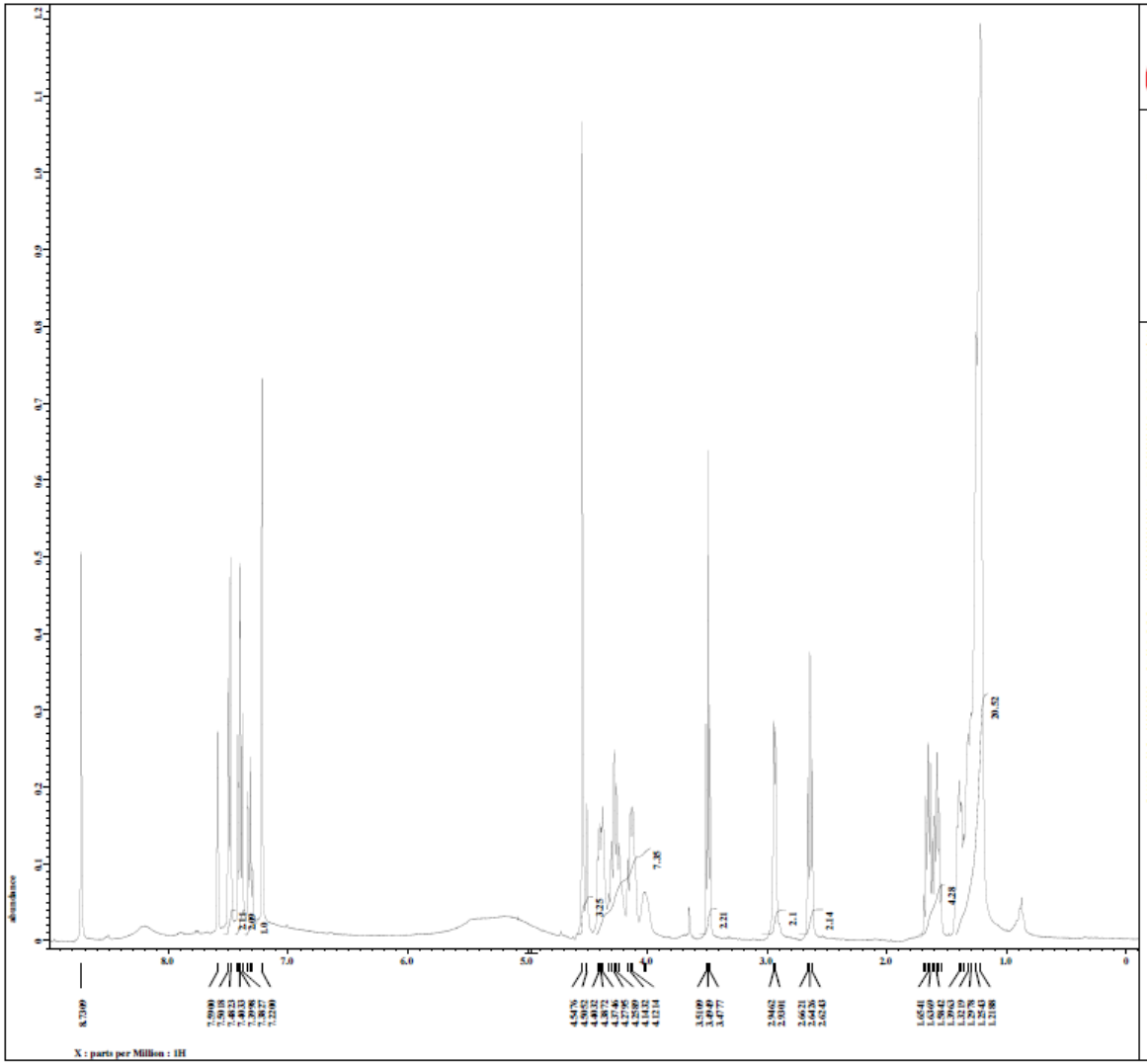
```
---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
expp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

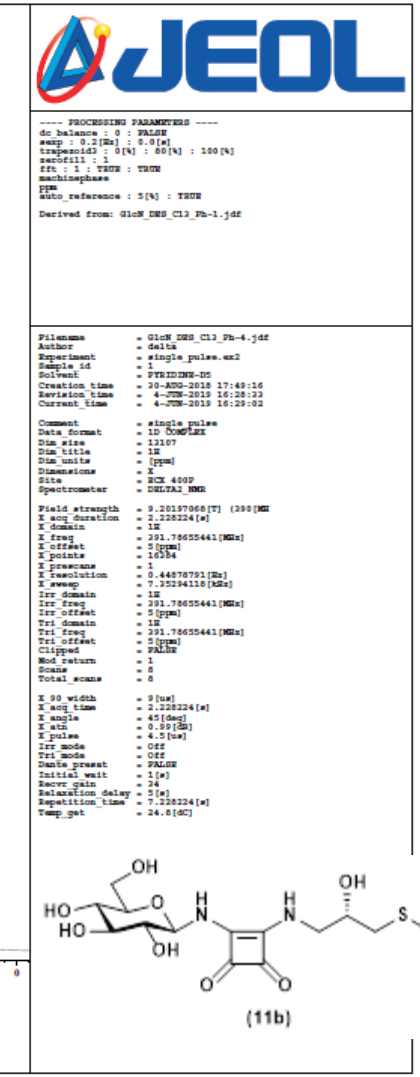
Derived from: 180801_13C_phth_OH_C13_Fh-
```

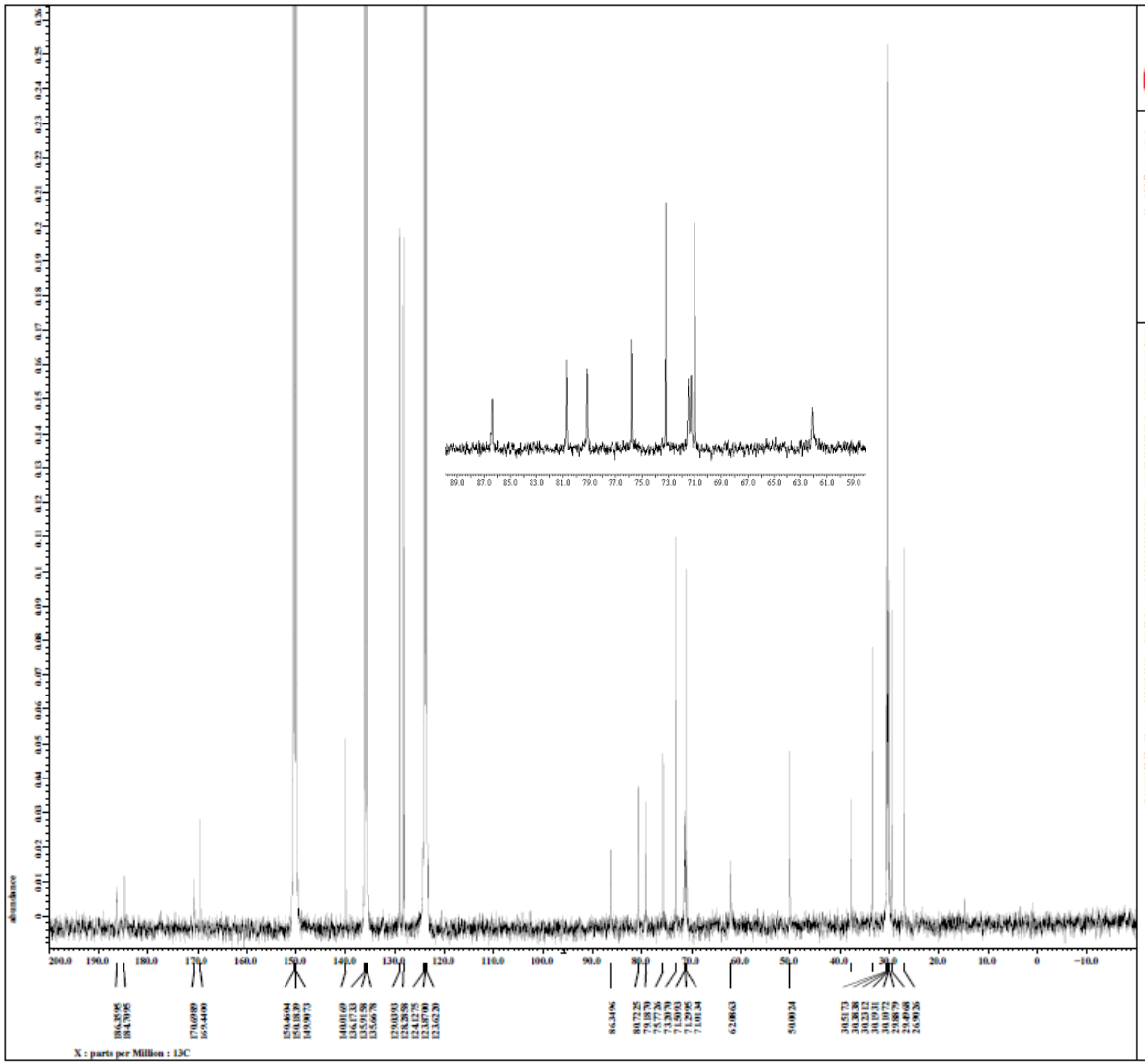
Filename	180801_13C_phth_OH_C1
Author	delta
Experiment	single_pulse_dec
Sample_id	1
Solvent	CHLOROFORM-D
Creation_time	1-AUG-2018 09:41:16
Revision_time	1-AUG-2018 10:02:06
Current_time	1-AUG-2018 10:03:23
Comment	single_pulse decouple
Data_format	1D COMPLEX
Dim_size	26214
Dim_title	13C
Dim_units	[ppm]
Dimensions	X
Site	RCX 400P
Spectrometer	DELTA2_NMR
Field_strength	9.20197068[T] (390[MH
X_acq_duration	1.06420464[s]
X_domain	13C
X_freq	98.51479726[MHz]
X_offset	100[ppm]
X_points	32768
X_prescans	4
X_resolution	0.93958061[Hz]
X_sweep	30.78817734[kHz]
Irr_domain	1H
Irr_freq	391.78655441[MHz]
Irr_offset	5[ppm]
Clipped	FALSE
Mod_return	1
Scans	107
Total_scans	107
X_90_width	9.25[us]
X_acq_time	1.06420464[s]
X_angle	30[deg]
X_atn	4[dB]
X_pulse	3.08333333[us]
Irr_atn_dec	17.8[dB]
Irr_atn_noe	17.8[dB]
Irr_noise	WALTZ
Decoupling	TRUE
Initial_wait	1[s]
Noe	TRUE
Noe_time	2[s]
Recvr_gain	50
Relaxation_delay	2[s]
Repetition_time	3.06420464[s]
Temp_get	22.8[dC]



(21b)


abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.38
3.25
2.21
2.1
2.14
4.28
20.52
2.15
2.69
1.0
8.399
7.599
7.581
7.453
7.403
7.398
7.387
7.229
4.547
4.502
4.482
4.387
4.374
4.279
4.268
4.143
4.124
3.519
3.494
3.477
2.962
2.936
2.662
2.643
2.624
1.654
1.636
1.619
1.594
1.329
1.278
1.261
1.210
X : parts per Million : 1H


---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 0.2[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
Derived from: GlcN_DHS_C13_Ph-1.jdf
Filename = GlcN_DHS_C13_Ph-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = PYRIDINE-D5
Creation_time = 20-AUG-2018 17:49:16
Revision_time = 4-JUN-2019 16:28:33
Current_time = 4-JUN-2019 16:29:02
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 12107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = 1
Site = RCX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 24
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 24.8[dC]
OH
HO
HO
O
OH
H
N
H
N
OH
S
13
O
O
(11b)



X : parts per Million : 13C

JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
PSM
auto_reference : 5[%] : TRUE

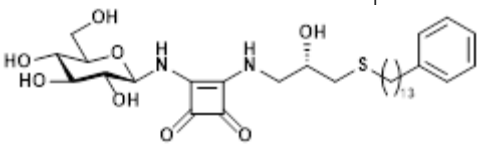
Derived from: 13C_GlcN_DRS_C13_Fh-1.jdf
```

```
Filename          = 13C_GlcN_DRS_C13_Fh-4
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 20-AUG-2015 18:21:45
Revision_time     =  4-JUN-2019 16:30:42
Current_time      =  4-JUN-2019 16:31:11

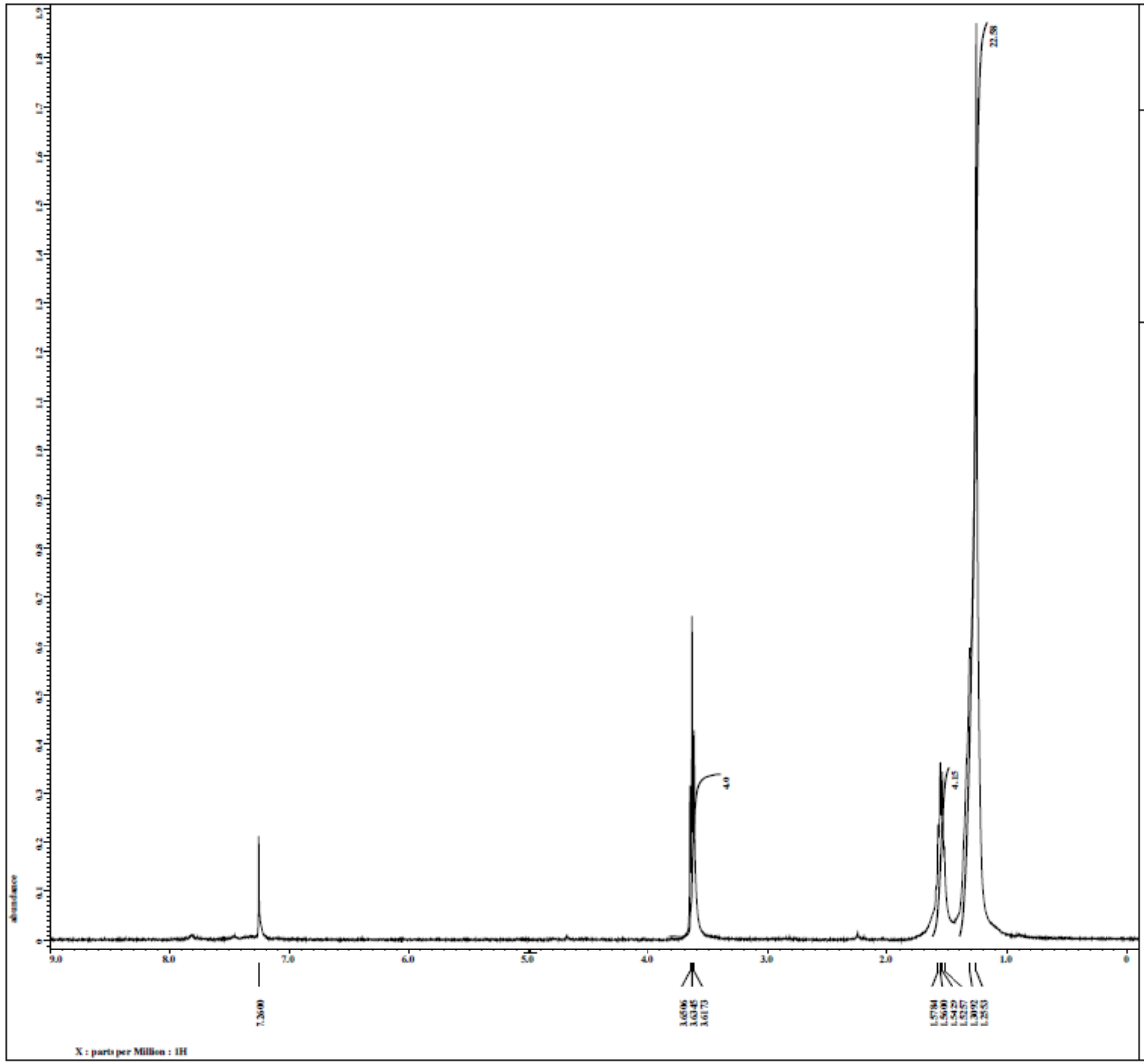
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T]  (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92958061[Hz]
X_sweep           = 30.78817734[KHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 621
Total_scans       = 621

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.05333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06420464[s]
Temp_get          = 25[dC]
```



(11b)



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

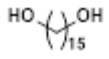
Derived from: 180828_HD_C15_OH-1.jdf

Filename = 180828_HD_C15_OH-4.jd
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 28-AUG-2018 16:59:20
Revision_time = 28-AUG-2018 17:27:56
Current_time = 28-AUG-2018 17:28:22

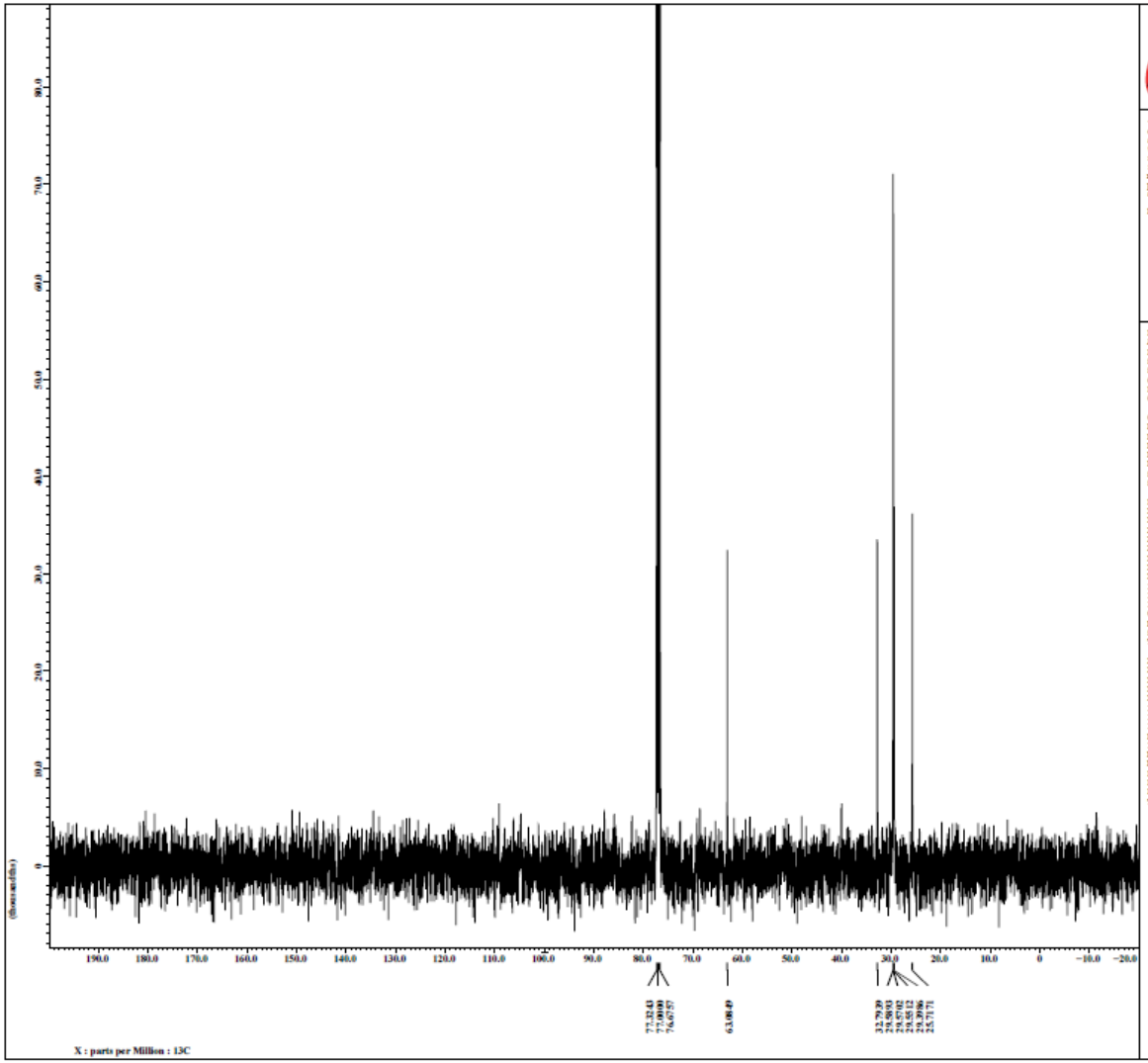
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 40
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 24.6[dC]



(12c)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid1 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

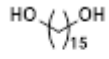
Derived from: 160828_13C_HD_C15_OH-1.jdf
```

```
Filename            = 160828_13C_HD_C15_OH-
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = 1
Solvent             = CHLOROFORM-D
Creation_time       = 28-AUG-2016 17:06:50
Revision_time       = 28-AUG-2016 17:23:01
Current_time        = 28-AUG-2016 17:23:23

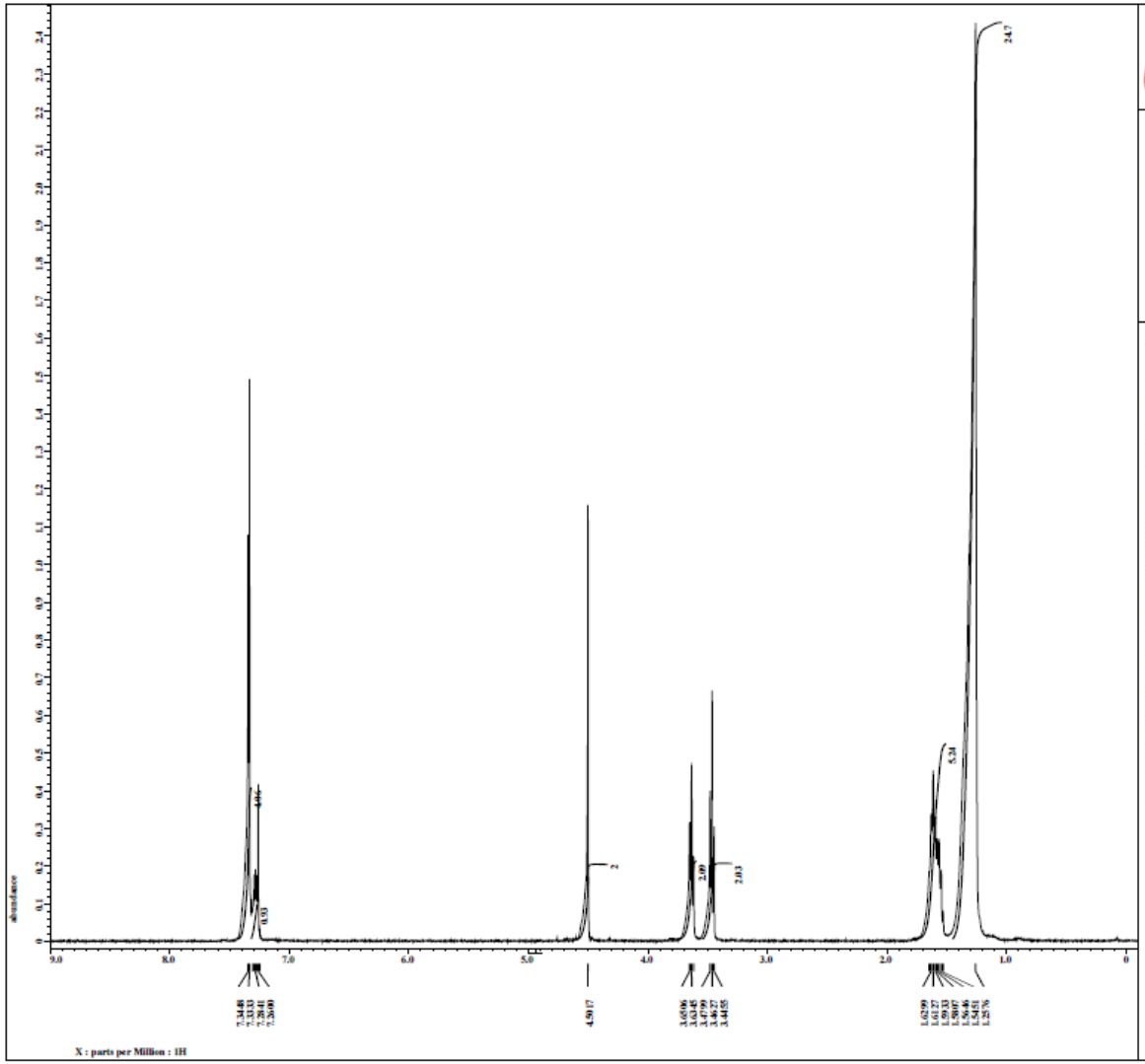
Comment             = single pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCK 400P
Spectrometer        = DELTA2_NMR

Field_strength      = 9.20197068[T] (390[MH
X_acq_duration      = 1.06430464[s]
X_domain            = 13C
X_freq              = 98.51479726[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.92358061[Hz]
X_sweep             = 30.75817734[kHz]
Irr_domain          = 1H
Irr_freq            = 391.78655441[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 128
Total_scans         = 128

X_90_width          = 9.25[us]
X_acq_time          = 1.06430464[s]
X_angle             = 30[deg]
X_atn               = 4[dB]
X_pulse             = 3.08333333[us]
Irr_atn_dec         = 17.8[dB]
Irr_atn_noe         = 17.8[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 50
Relaxation_delay    = 2[s]
Repetition_time     = 3.06430464[s]
Temp_get            = 24.9[dC]
```



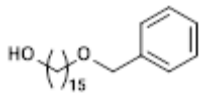
(12c)



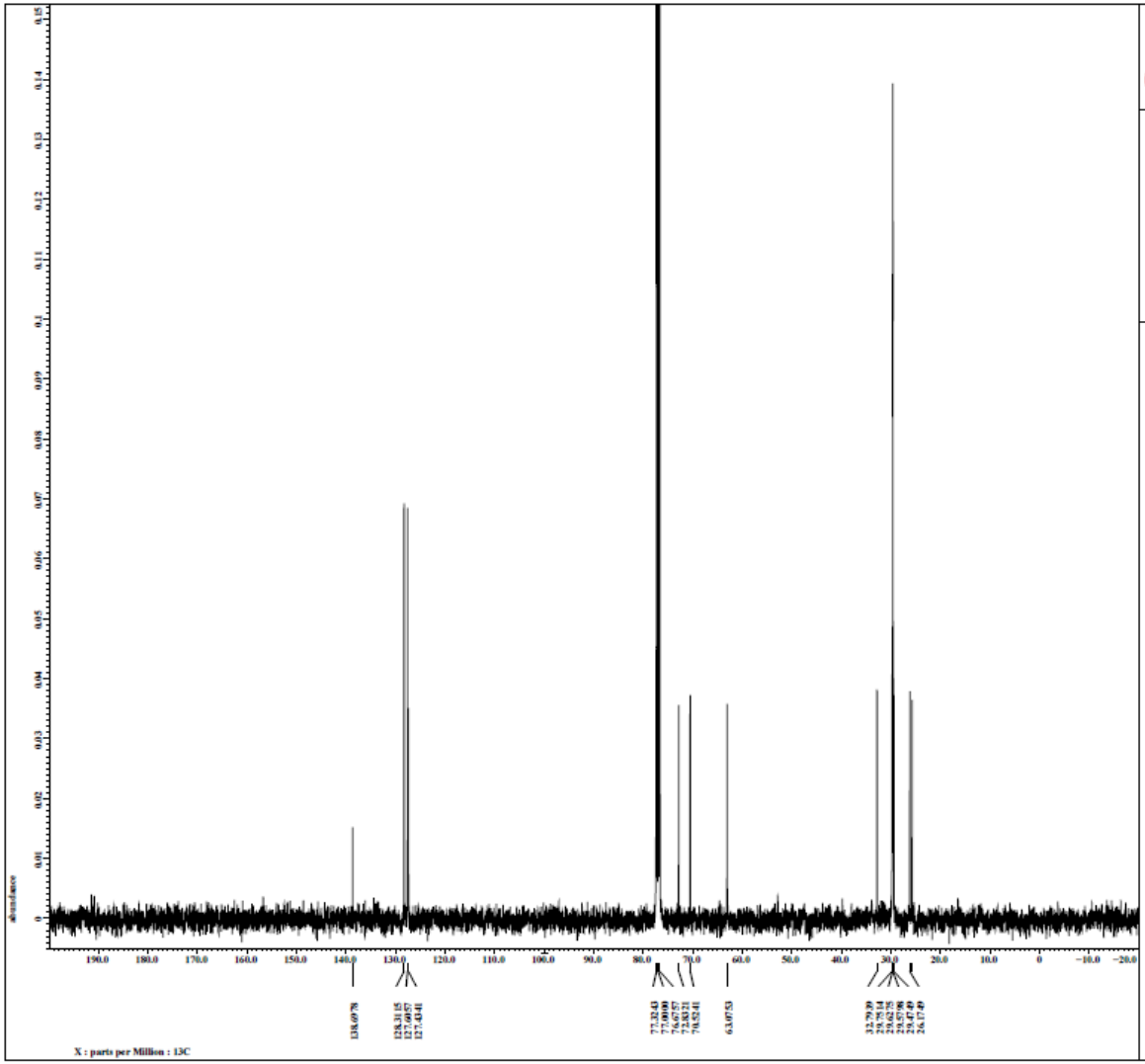
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180823_HD_C15_Ph-1.jdf

Filename	= 180823_HD_C15_Ph-4.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 22-AUG-2018 15:33:08
Revision_time	= 22-AUG-2018 16:00:11
Current_time	= 22-AUG-2018 16:00:38
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [PPM]
Dimensions	= X
Site	= RCK 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 7
Total_scans	= 7
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 24.6[dC]



(13c)



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

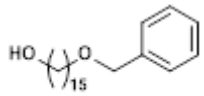
Derived from: 180824_13C_HO_C15_Ph-1.jdf

Filename = 180824_13C_HO_C15_Ph-
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 24-AUG-2018 09:49:44
Revision_time = 24-AUG-2018 10:16:03
Current_time = 24-AUG-2018 10:16:44

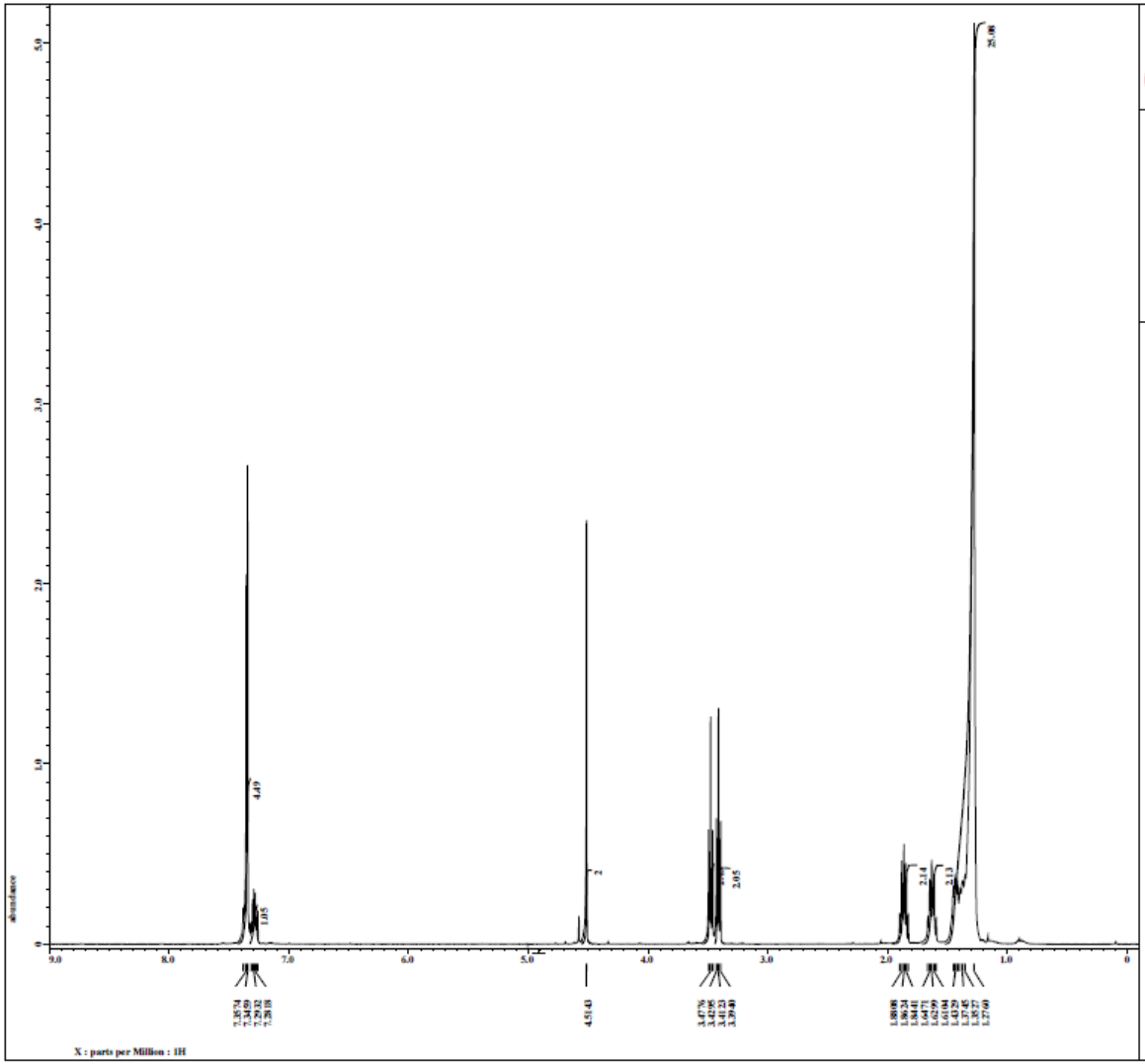
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCX 400F
Spectrometer = DELTA2_NMR

Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.93958061[Hz]
X_sweep = 30.78517734[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 374
Total_scans = 374

X_90_width = 9.25[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4[dB]
X_pulse = 3.00333333[us]
Irr_atn_dec = 17.8[dB]
Irr_atn_noe = 17.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 24.8[dC]



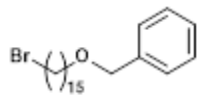
(13c)



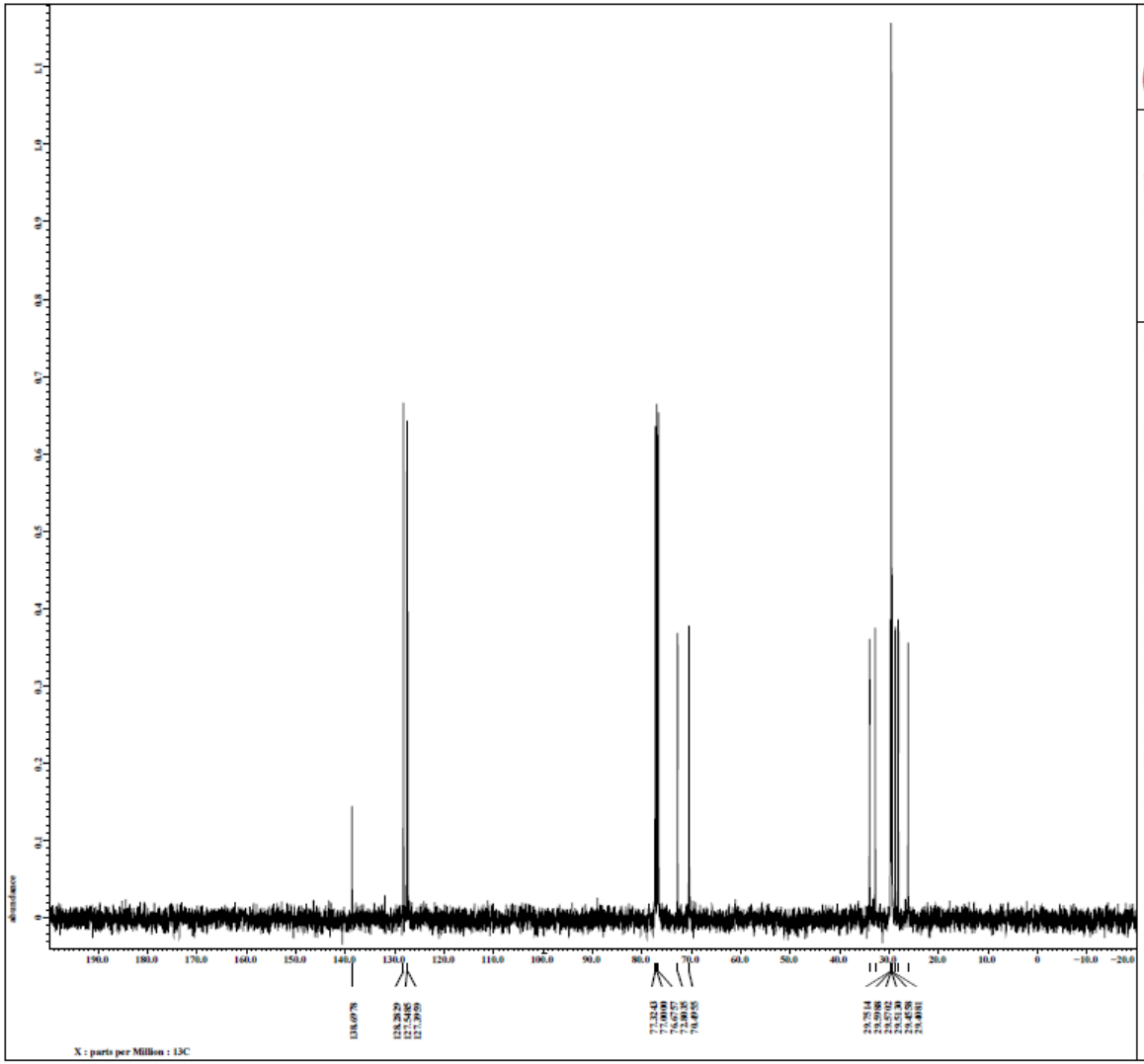
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180823_Br_C15_Pb-1.jdf
```

Filename	= 180823_Br_C15_Pb-4.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-AUG-2018 19:00:41
Revision_time	= 24-AUG-2018 19:43:02
Current_time	= 24-AUG-2018 19:43:41
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [PPM]
Dimensions	= X
Site	= RCK 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44876791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 5
Total_scans	= 5
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 26
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 24.6[dC]



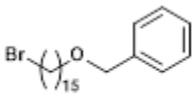
(14c)



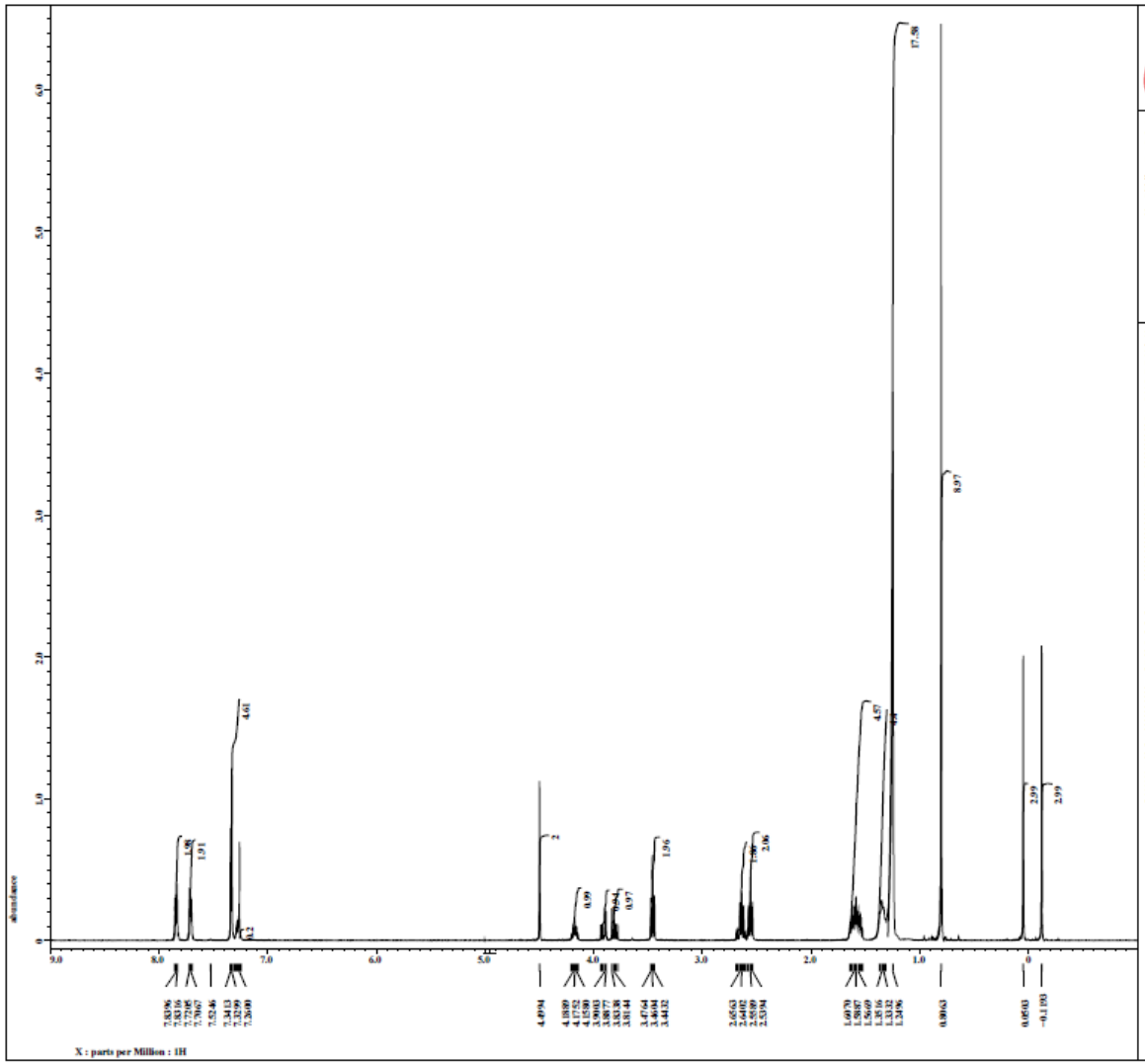
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
manphasephase
ppm
auto_reference : 0[%] : TRUE

Derived from: 180823_13C_Br_C15_Ph-1.jdf

Filename	= 180823_13C_Br_C15_Ph-
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-AUG-2018 19:04:03
Revision_time	= 24-AUG-2018 19:29:50
Current_time	= 24-AUG-2018 19:30:29
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [PPM]
Dimensions	= X
Site	= RCX 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 1.06420464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.75817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 43
Total_scans	= 43
X_90_width	= 9.25[us]
X_acq_time	= 1.06420464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 2.05223232[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 58
Relaxation_delay	= 2[s]
Repetition_time	= 3.06420464[s]
Temp_get	= 24.9[dC]



(14c)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid0 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

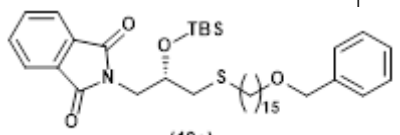
Derived from: 180720_phth_TBS_C15_Ph-1.j
```

```
Filename          = 180720_phth_TBS_C15_P
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 20-JUL-2018 16:52:16
Revision_time     = 20-JUL-2018 18:09:15
Current_time      = 20-JUL-2018 18:09:45

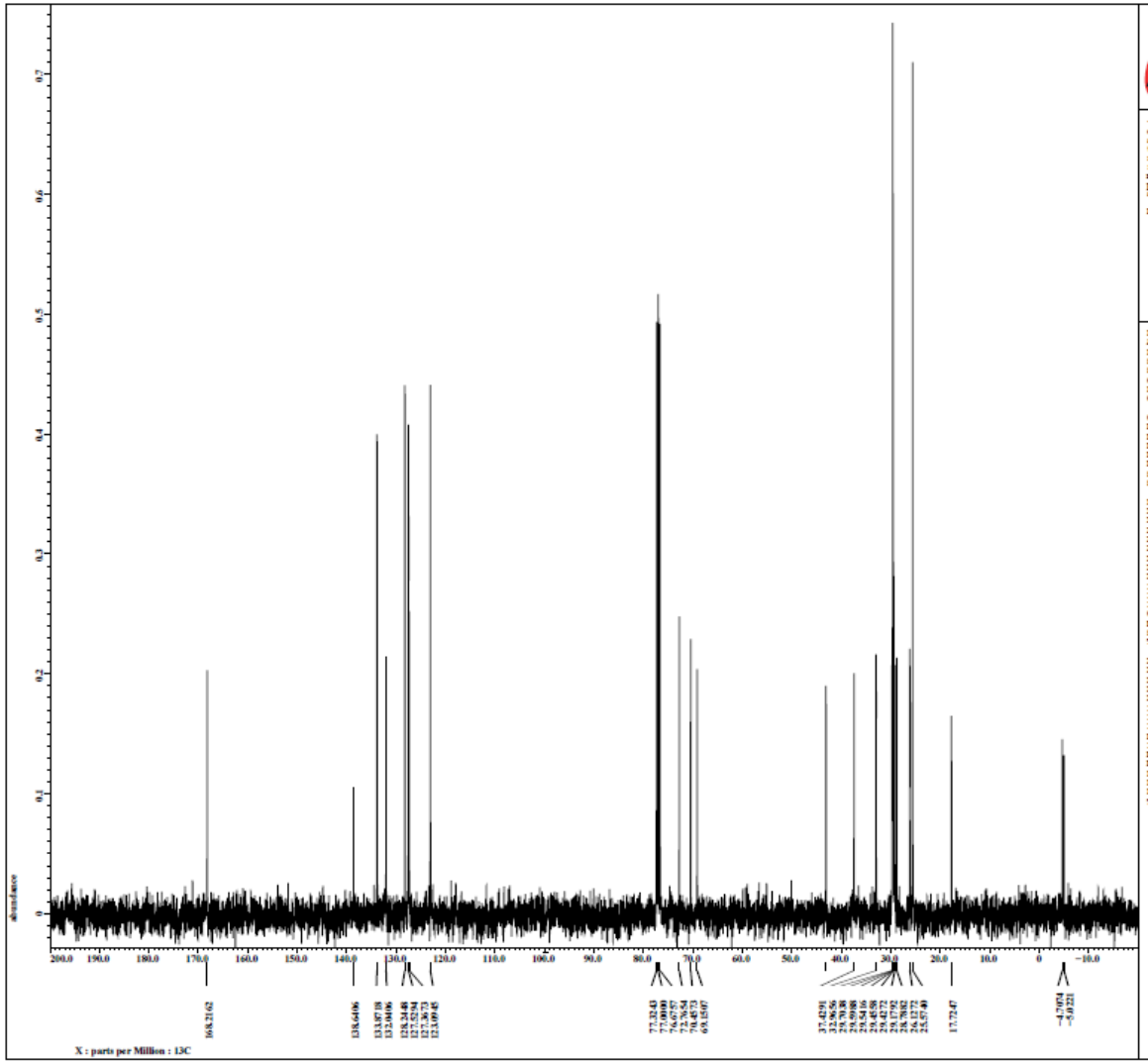
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = 1
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44870791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Decpls_preset     = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.6[dC]
```



(19c)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
PPM
auto_reference : 5[%] : TRUE

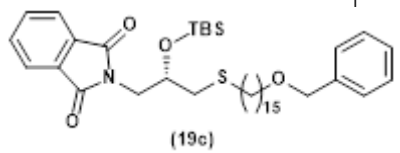
Derived from: 180720_13C_phth_TBS_C15_Ph
```

```
Filename          = 180720_13C_phth_TBS_C
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 20-JUL-2018 16:55:22
Revision_time     = 20-JUL-2018 16:04:56
Current_time      = 20-JUL-2018 16:05:29

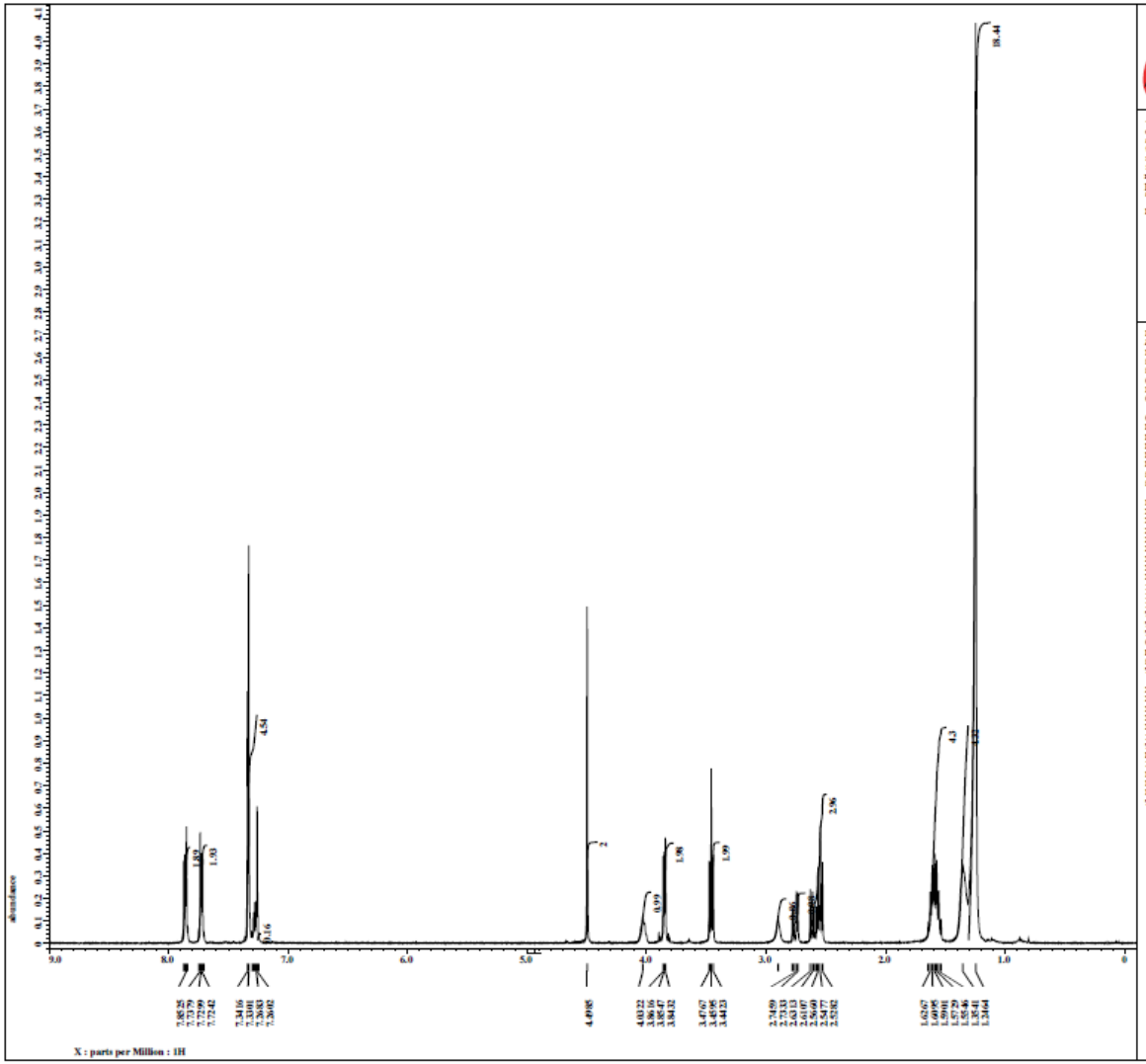
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

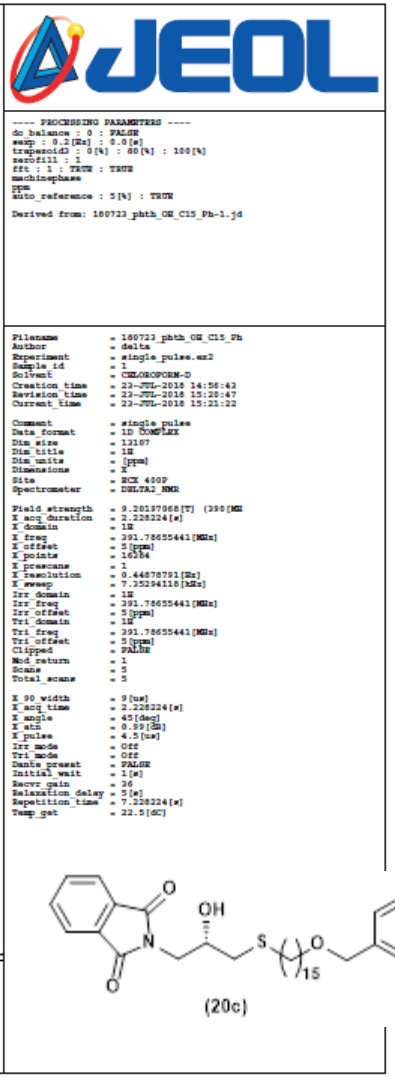
Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 19
Total_scans       = 19

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.6[dB]
Irr_atn_noe       = 17.6[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   = 2.06430464[s]
Temp_get          = 23[dC]
```



(19c)






```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180723_phth_OH_C15_Ph-1.jd
```

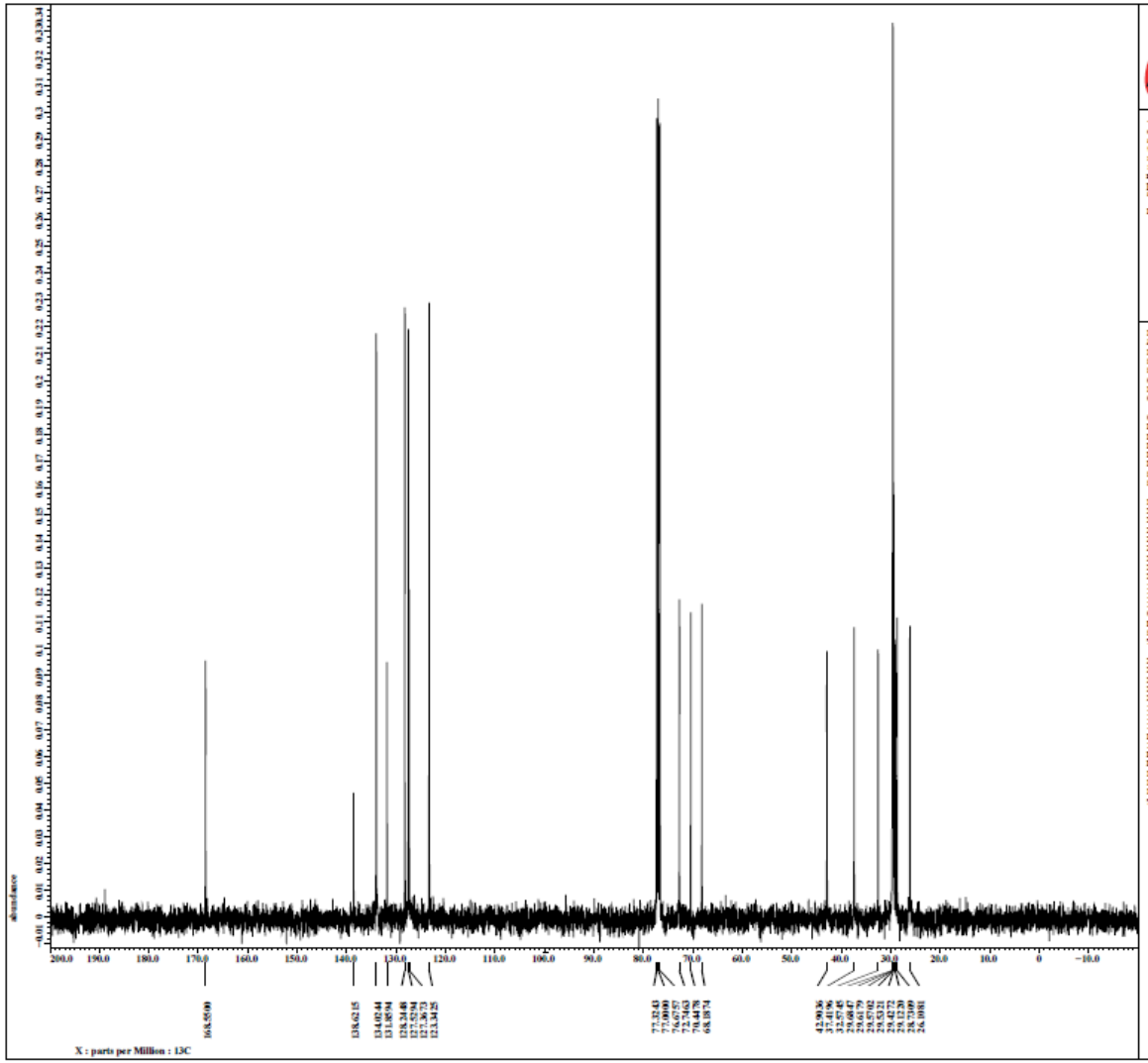
```
Filename          = 180723_phth_OH_C15_Ph
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 23-JUL-2018 14:56:43
Revision_time     = 23-JUL-2018 15:20:47
Current_time      = 23-JUL-2018 15:21:22

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35234118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.5[dC]
```


(20c)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 2.0[Hz] : 0.0[s]
trapezoid1 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
PPM
auto_reference : 5[%] : TRUE

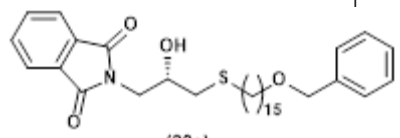
Derived from: 180723_C13_phth_OH_C15_Ph-
```

```
Filename          = 180723_C13_phth_OH_C1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 23-JUL-2018 15:02:36
Revision_time     = 23-JUL-2018 15:24:40
Current_time      = 23-JUL-2018 15:25:37

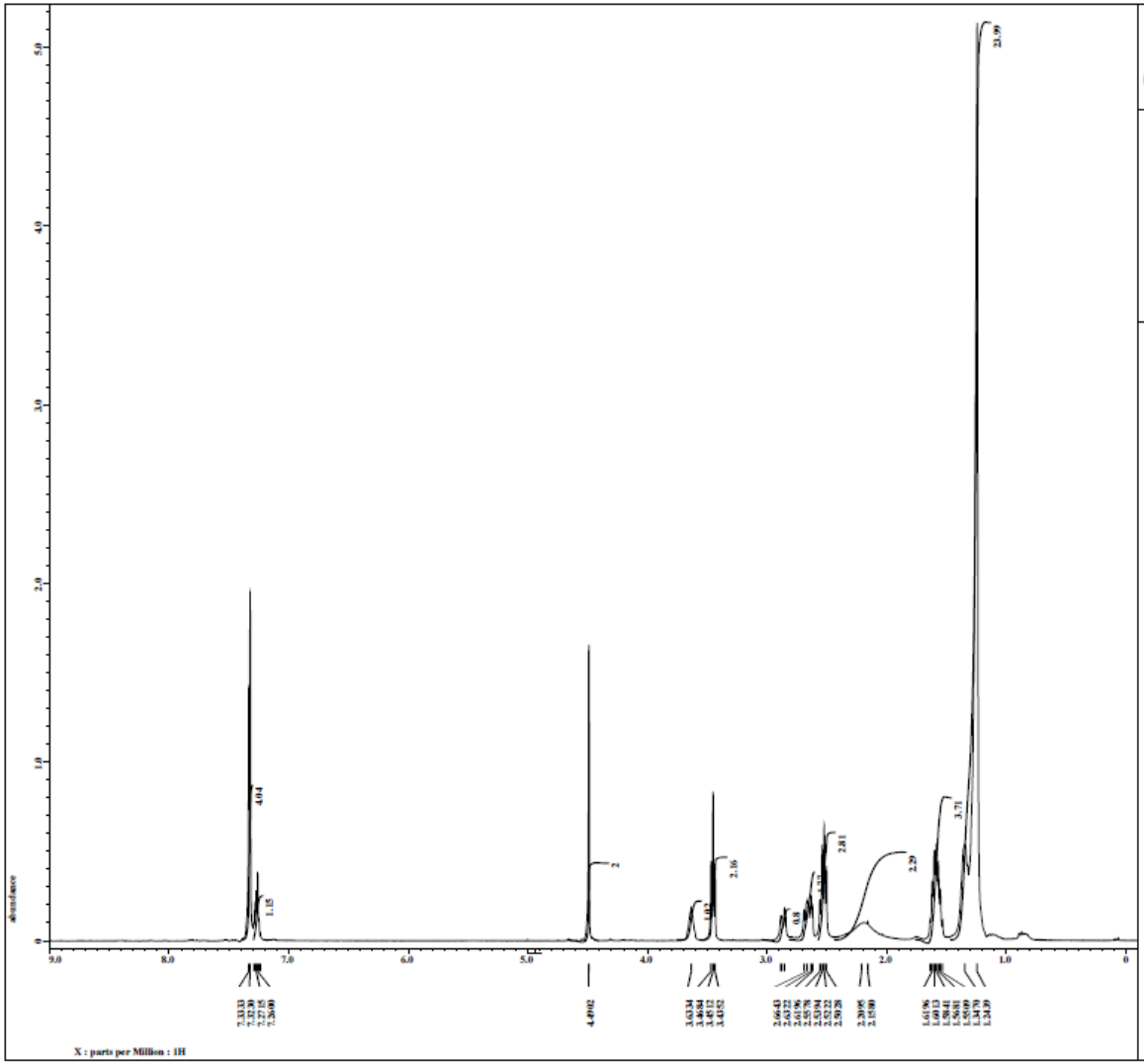
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 77
Total_scans       = 77

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.6[dB]
Irr_atn_noe       = 17.6[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.6[dC]
```



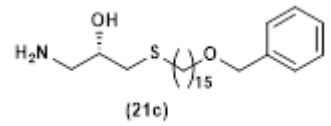
(20c)



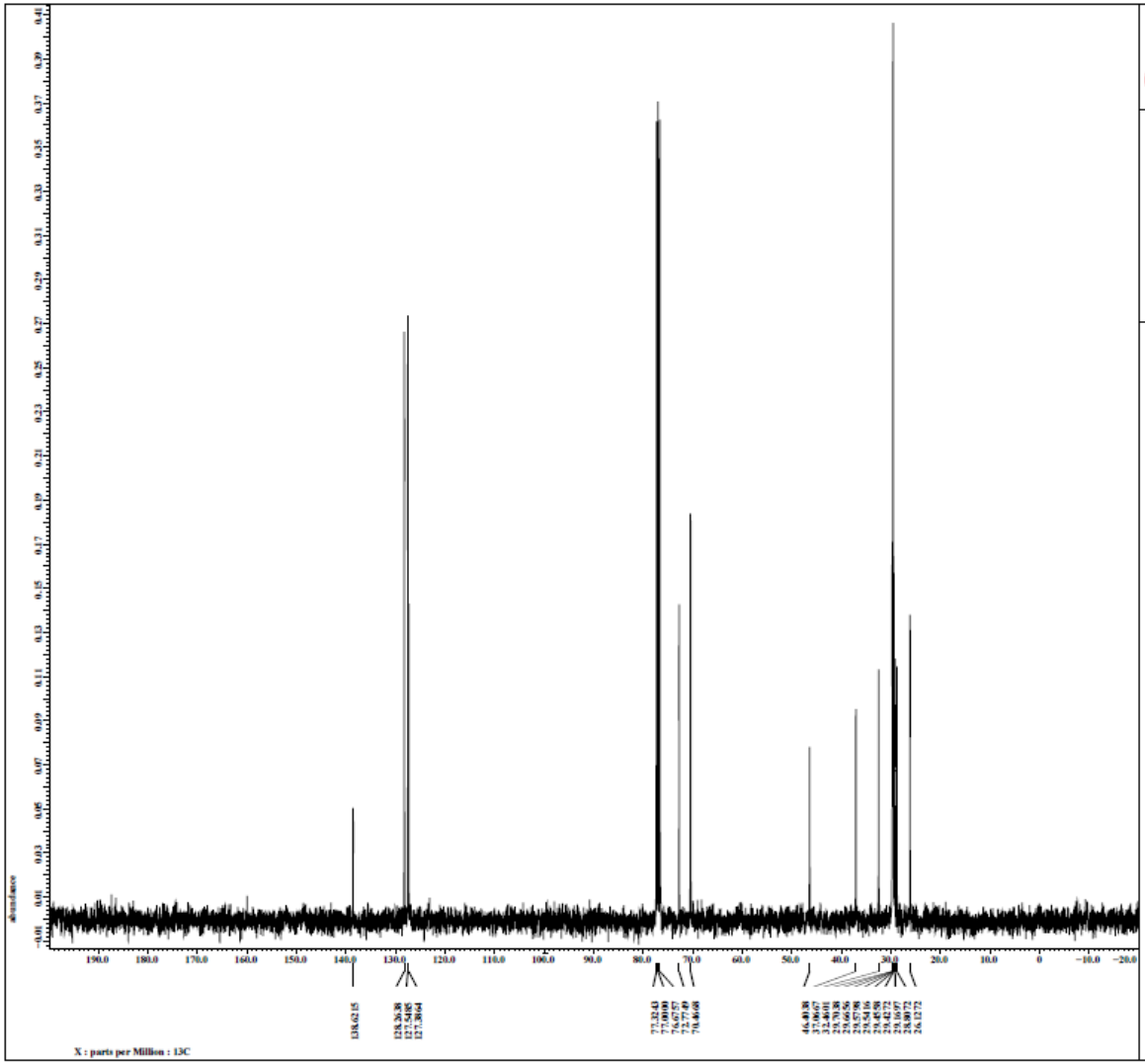
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

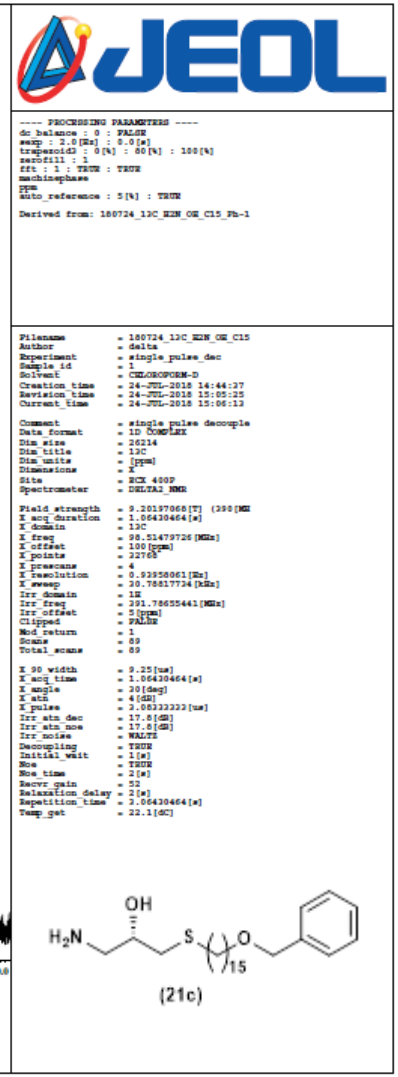
Derived from: 180724_HZN_OK_C15_Ph-1.jdf

Filename	= 180724_HZN_OK_C15_Ph-
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-JUL-2018 14:29:04
Revision_time	= 24-JUL-2018 15:03:20
Current_time	= 24-JUL-2018 15:03:52
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [PPM]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 5
Total_scans	= 5
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 24
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 21.6[dC]



(21c)





```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180724_13C_HZN_OH_C15_Pb-1
```

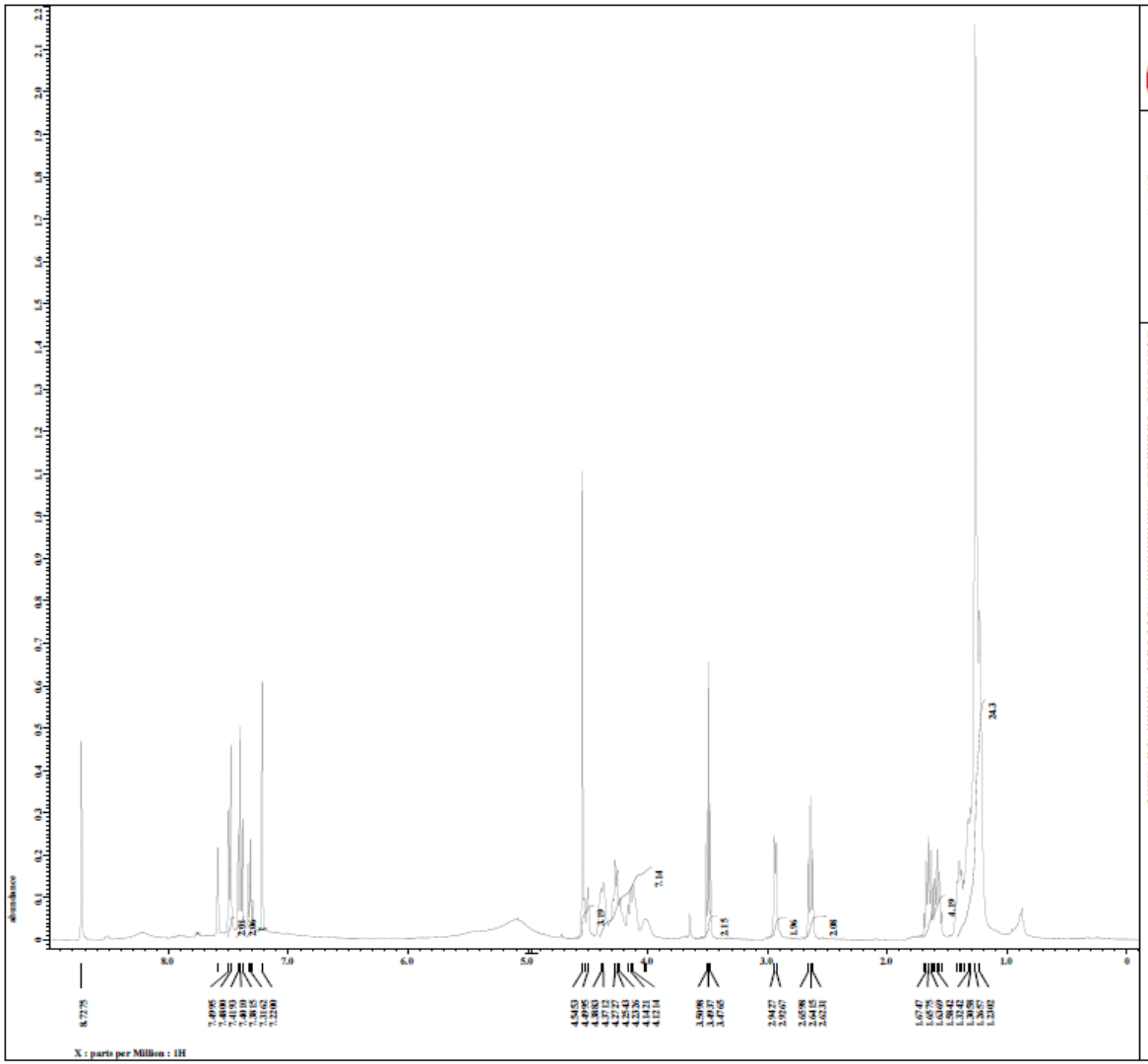
```
Filename          = 180724_13C_HZN_OH_C15
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 24-JUL-2018 14:44:27
Revision_time     = 24-JUL-2018 15:05:25
Current_time      = 24-JUL-2018 15:06:12

Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK_400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.76817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 69
Total_scans       = 69

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.5[dB]
Irr_atn_noe       = 17.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 52
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.1[dC]
```


(21c)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

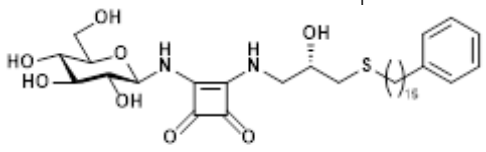
Derived from: GlcN_DNS_C15_Ph-1.jdf
```

```
Filename          = GlcN_DNS_C15_Ph-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 20-AUG-2018 16:25:08
Revision_time     =  4-JUN-2019 16:25:15
Current_time      =  4-JUN-2019 16:25:45

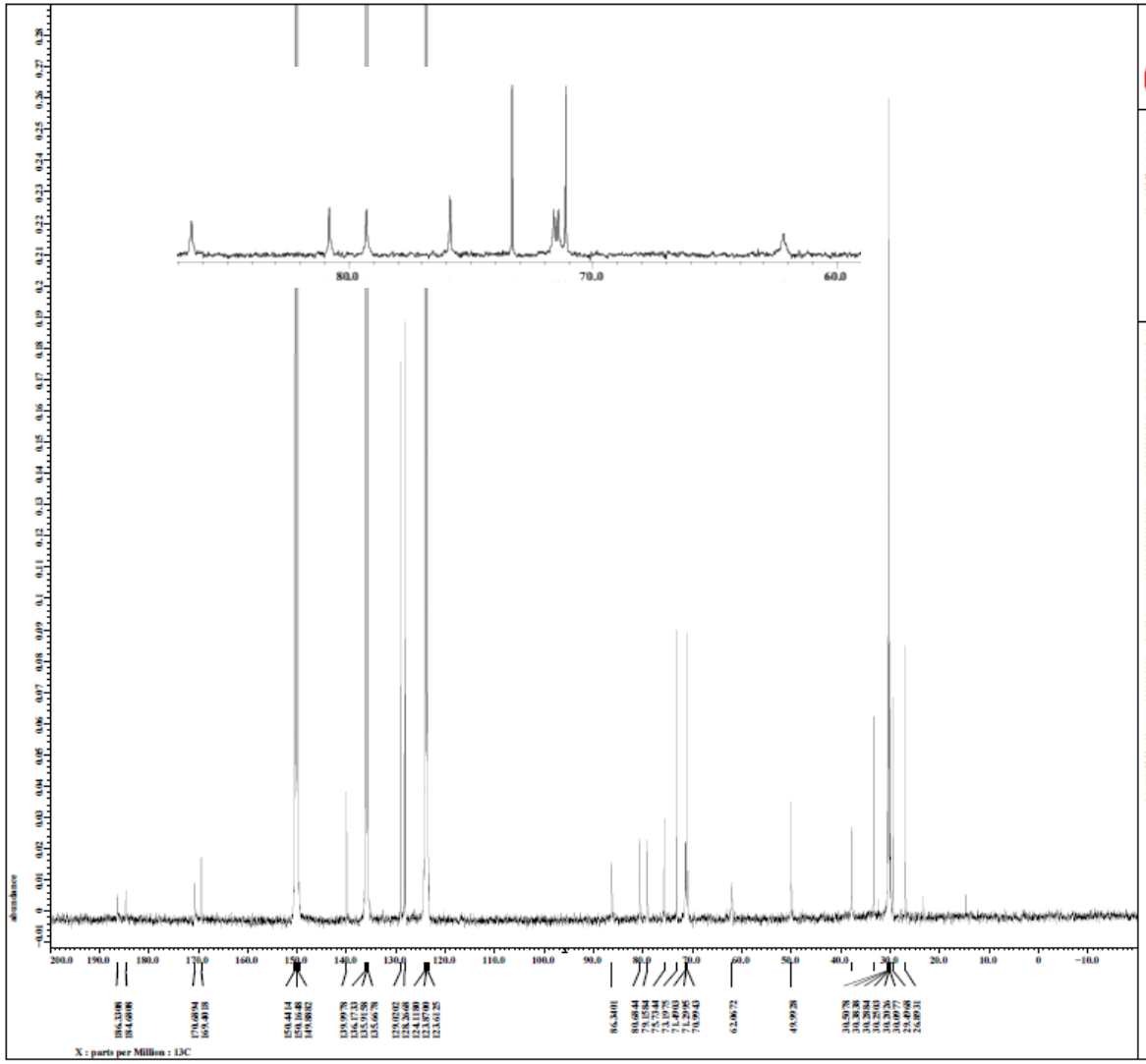
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 24.8[dC]
```



(11c)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

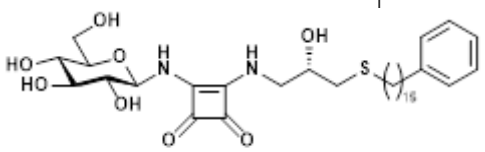
Derived from: 13C_GlcN_DHS_C15_Ph-1.jdf
```

```
Filename          = 13C_GlcN_DHS_C15_Ph-4
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 20-AUG-2018 19:40:00
Revision_time     =  4-JUN-2019 16:22:15
Current_time      =  4-JUN-2019 16:23:08

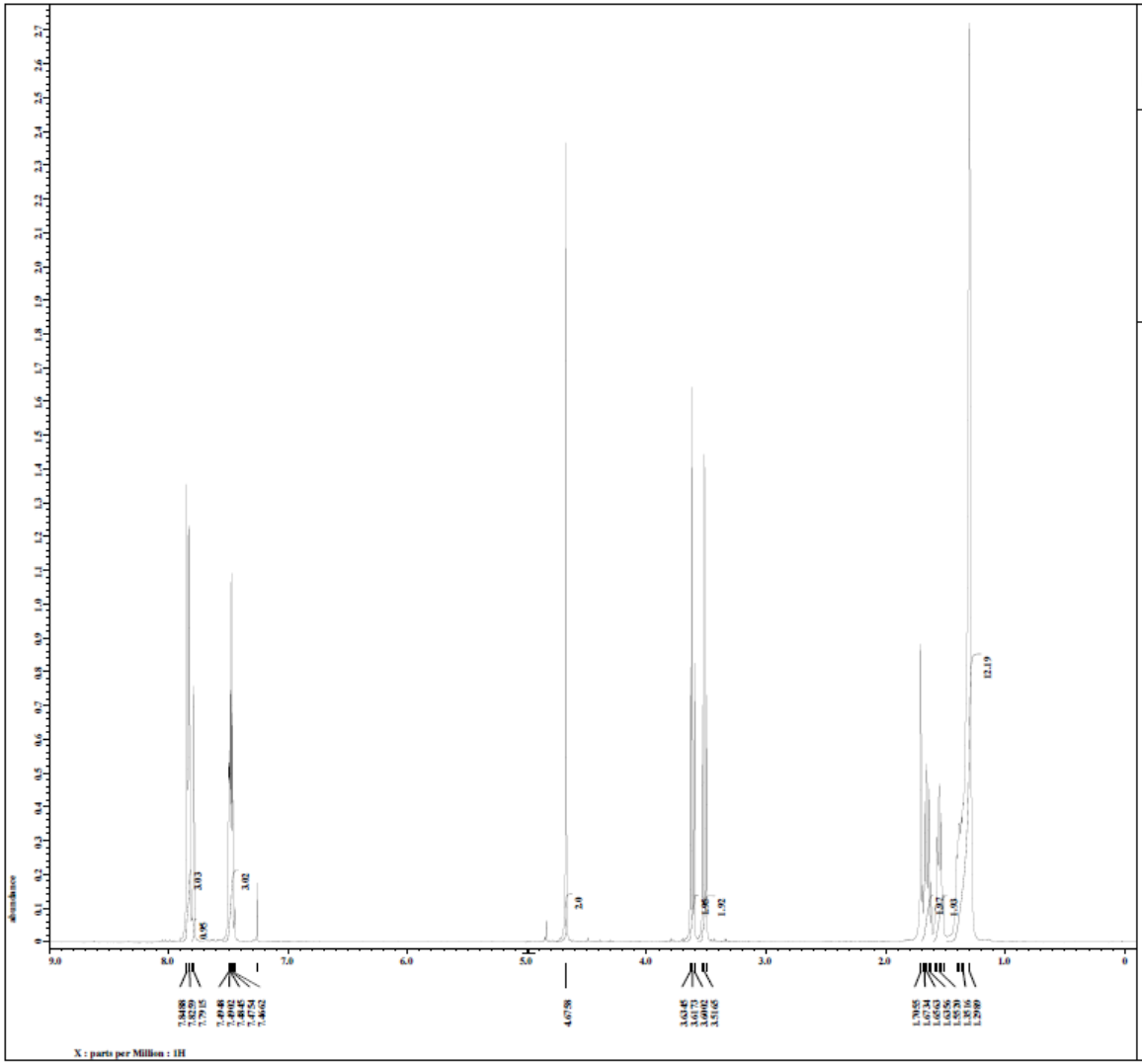
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = NCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1452
Total_scans       = 1452

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 24.9[dC]
```



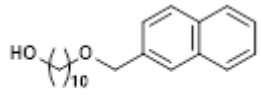
(11c)



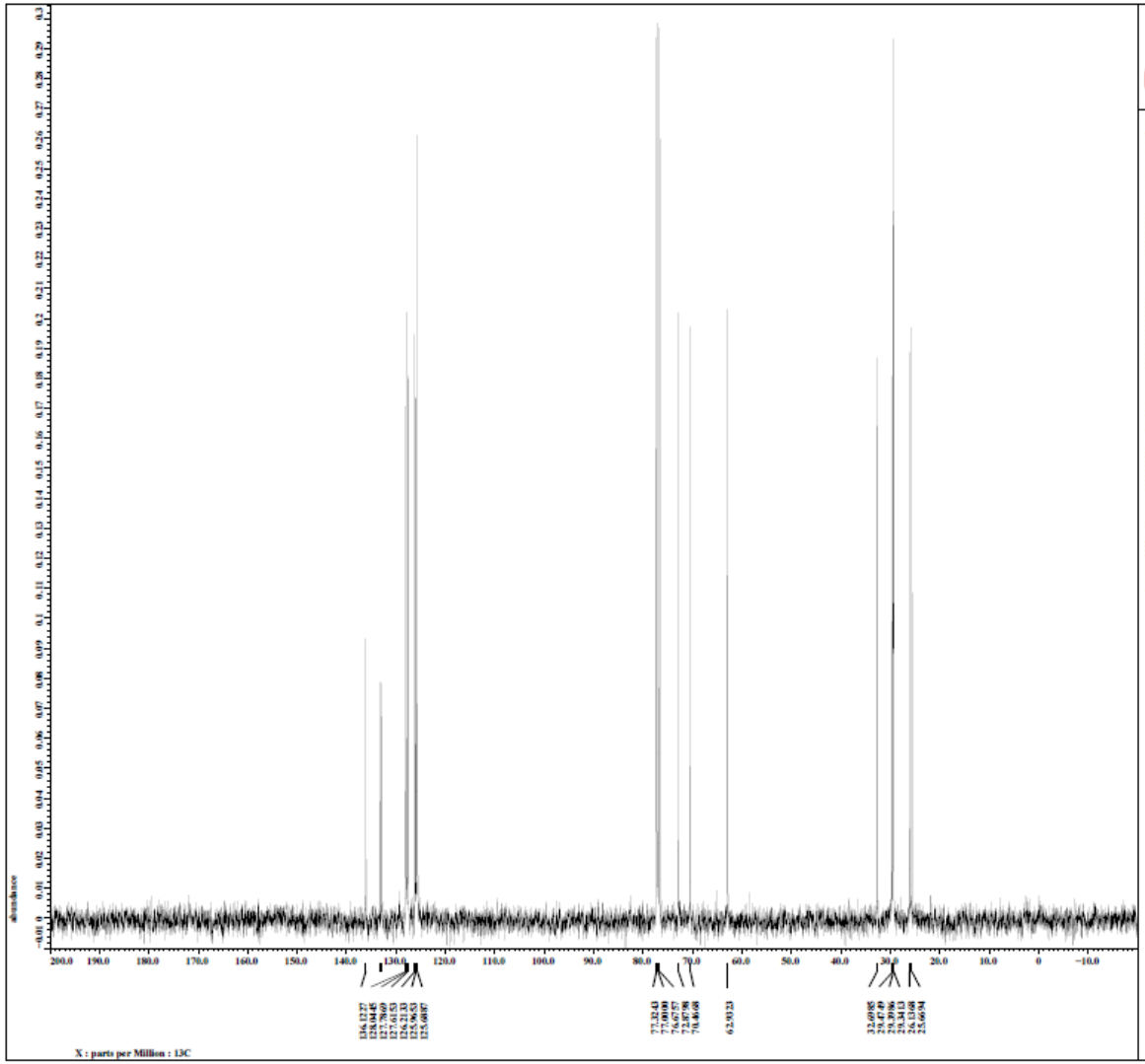
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
auto_reference : 5[%] : TRUE

Derived from: 200204_HO_C10_ONAP-1.jdf

Filename	= 200204_HO_C10_ONAP-6.
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 4-FEB-2020 16:21:50
Revision_time	= 4-FEB-2020 16:59:42
Current_time	= 4-FEB-2020 17:00:24
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35234118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 6
Total_scans	= 6
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 26
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 21.9[dC]



(13d)

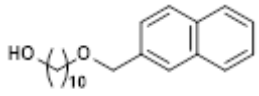


Filename = 200204_13C_ED_C10_ONA
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 4-FEB-2020 16:27:07
Revision_time = 4-FEB-2020 17:29:22
Current_time = 4-FEB-2020 17:40:22

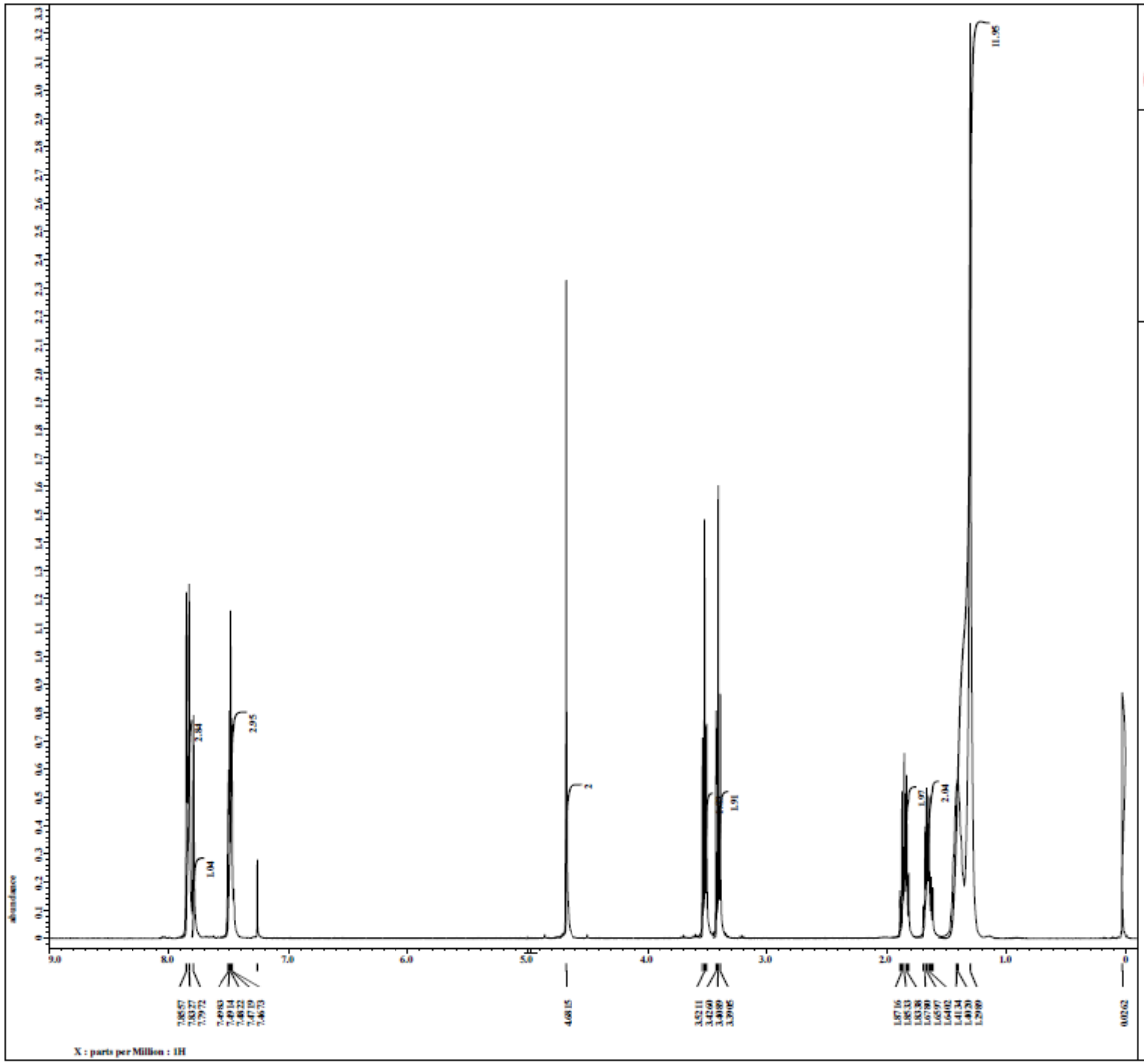
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCK 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.92958061[Hz]
X_sweep = 30.76817734[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 80
Total_scans = 80

X_90_width = 9.25[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4[dB]
X_pulse = 2.85333333[us]
Irr_atn_dec = 17.8[dB]
Irr_atn_noe = 17.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 22.2[dC]



(13d)



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

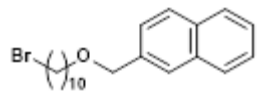
Derived from: 180416_Br_C10_Naph-1.jdf

Filename = 180416_Br_C10_Naph-4.
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 16-APR-2018 20:48:25
Revision_time = 24-AUG-2018 13:35:08
Current_time = 24-AUG-2018 13:35:42

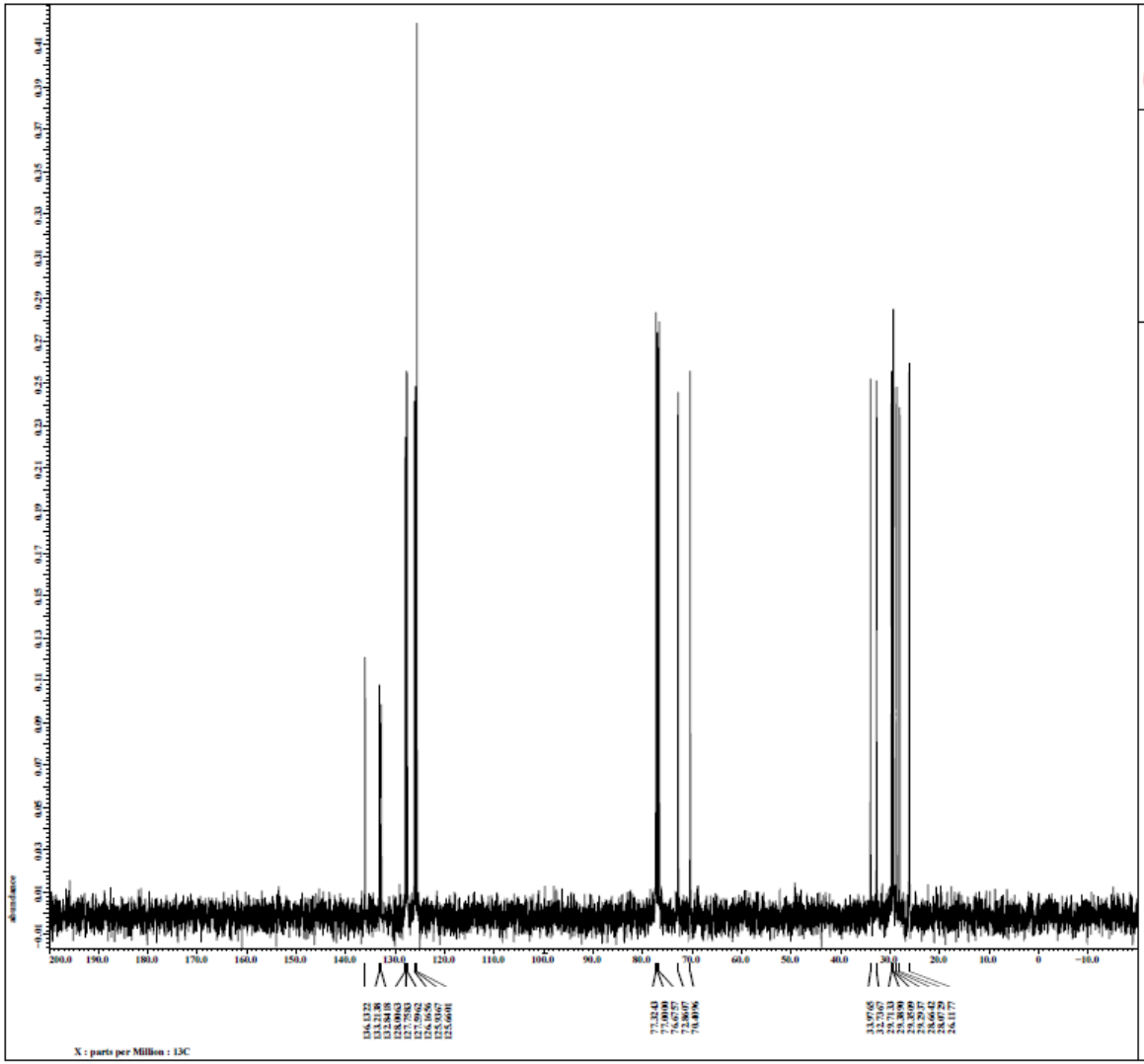
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [PPM]
Dimensions = X
Site = RCX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.20197066[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 28
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 402.1[dC]



(14d)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
expp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
auto_reference : 5[%] : TRUE

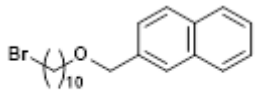
Derived from: 160416_13C_Br_C10_Naph-1.j
```

```
Filename          = 160416_13C_Br_C10_Nap
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 16-APR-2016 20:52:04
Revision_time     = 28-AUG-2018 15:56:25
Current_time      = 28-AUG-2018 15:57:11

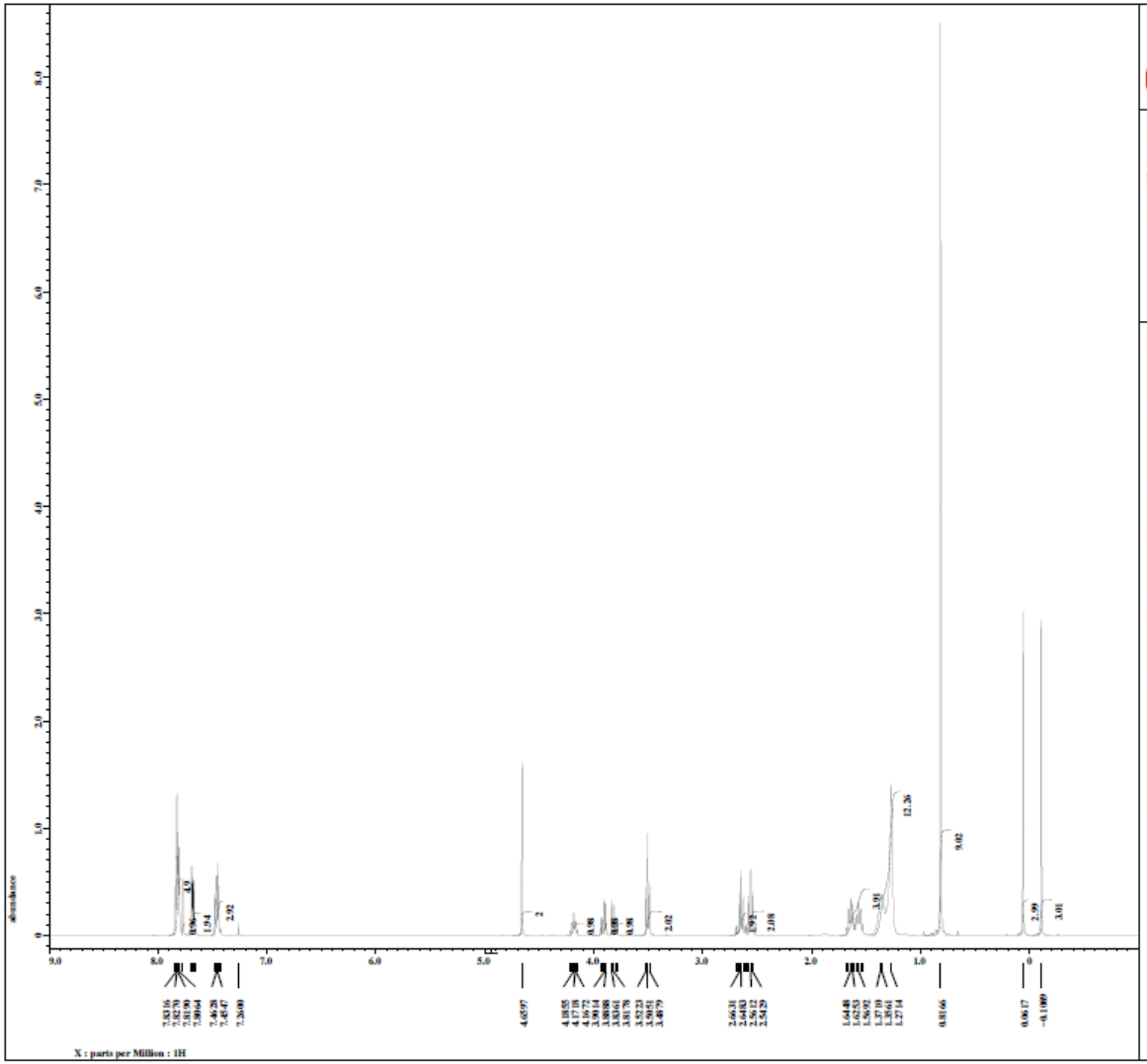
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 22
Total_scans       = 23

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 402.1[dC]
```



(14d)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

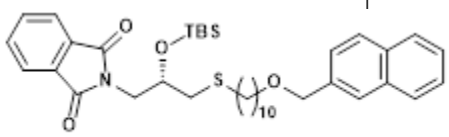
Derived from: 190607_phthN_OTBS_C10_NAP-
```

```
Filename          = 190607_phthN_OTBS_C10
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  7-JUN-2019 15:54:28
Revision_time     =  7-JUN-2019 16:29:28
Current_time      =  7-JUN-2019 16:30:17

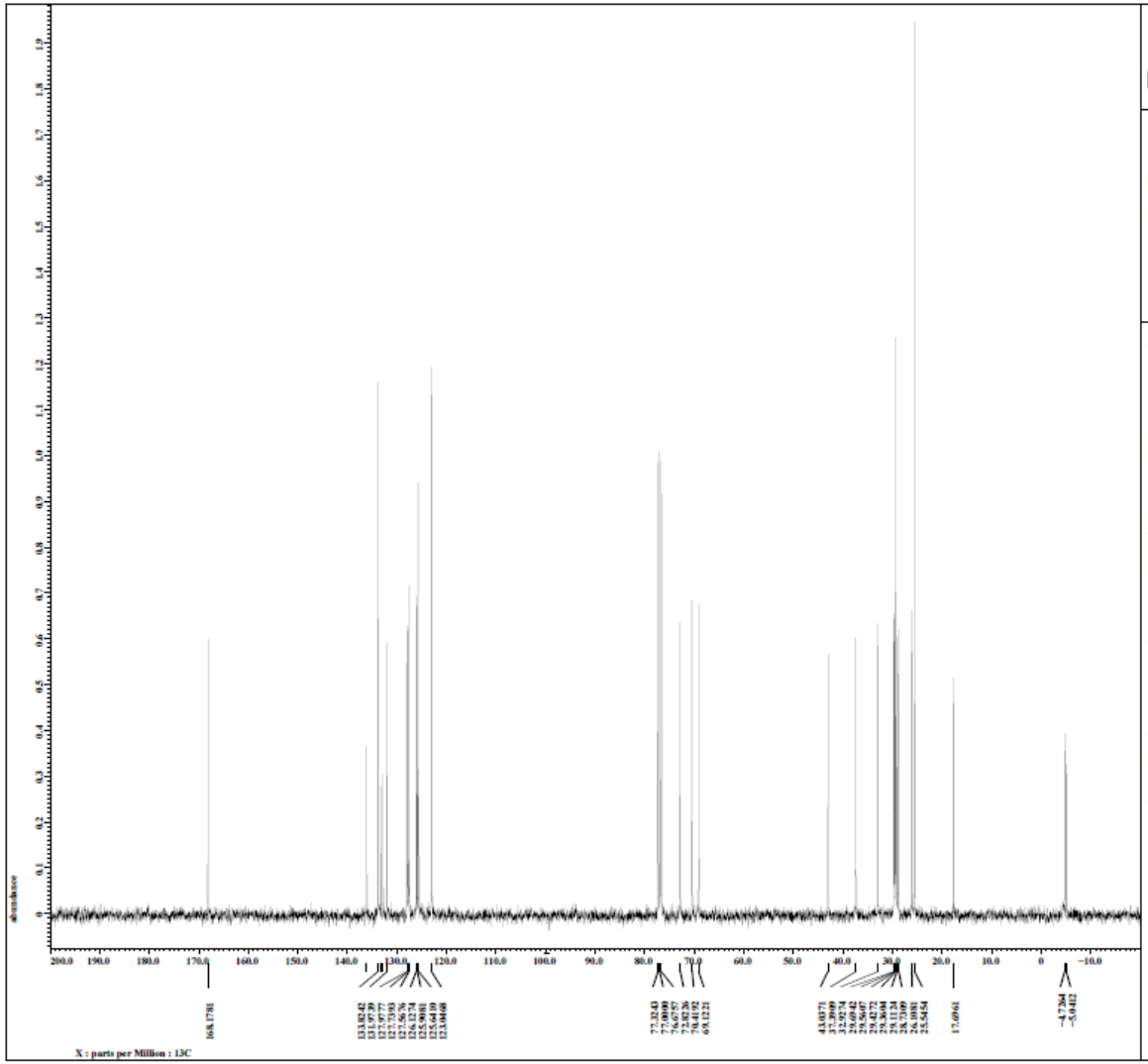
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_swsup           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 460.0[dC]
```



(19d)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

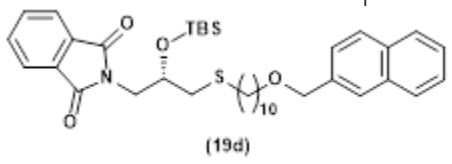
Derived from: 190607_13C_phthN_OTBS_C10_
```

```
Filename            = 190607_13C_phthN_OTBS
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = 1
Solvent             = CHLOROFORM-D
Creation_time       =  7-JUN-2019 15:59:27
Revision_time       =  7-JUN-2019 16:21:34
Current_time        =  7-JUN-2019 16:22:26

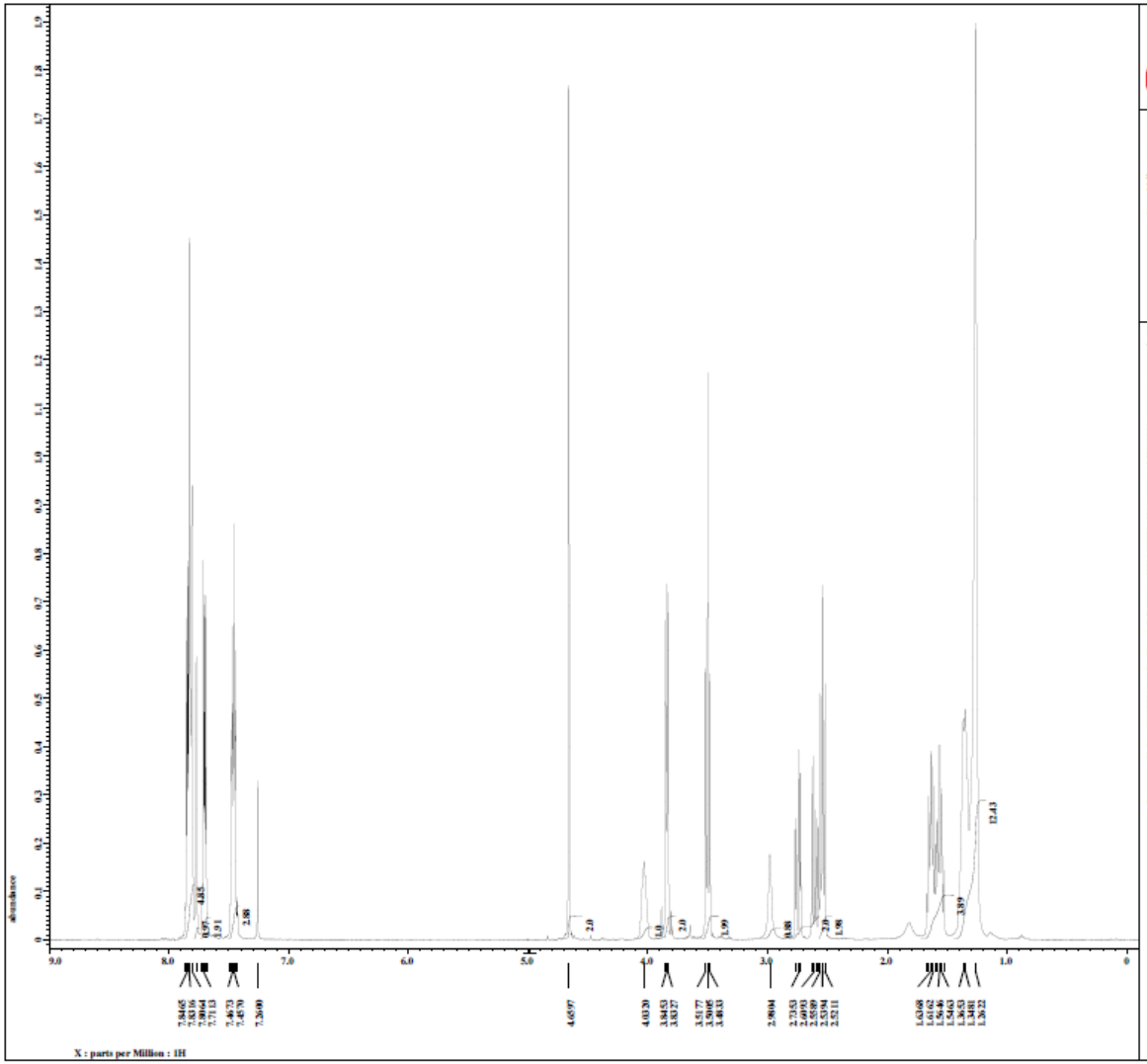
Comment             = single_pulse_decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCK_400P
Spectrometer        = DELTA2_NMR

Field_strength      = 9.20197068[T]  (390[MH
X_acq_duration      = 1.06430464[s]
X_domain            = 13C
X_freq              = 98.51479726[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.92958061[Hz]
X_sweep             = 30.78817734[kHz]
Irr_domain          = 1H
Irr_freq            = 391.78655441[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 85
Total_scans         = 85

X_90_width          = 9.25[us]
X_acq_time          = 1.06430464[s]
X_angle             = 30[deg]
X_atn               = 4[dB]
X_pulse             = 2.65333333[us]
Irr_atn_dec         = 17.8[dB]
Irr_atn_noe         = 17.8[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 60
Relaxation_delay    = 2[s]
Repetition_time     = 3.06430464[s]
Temp_get            = 460.0[dC]
```



(19d)



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

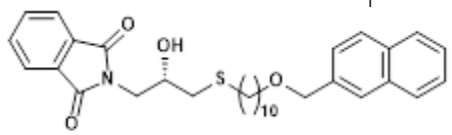
Derived from: 190610_phthN_OH_SC10_NAP-1

Filename = 190610_phthN_OH_SC10_
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 10-JUN-2019 18:49:06
Revision_time = 10-JUN-2019 19:23:29
Current_time = 10-JUN-2019 19:24:04

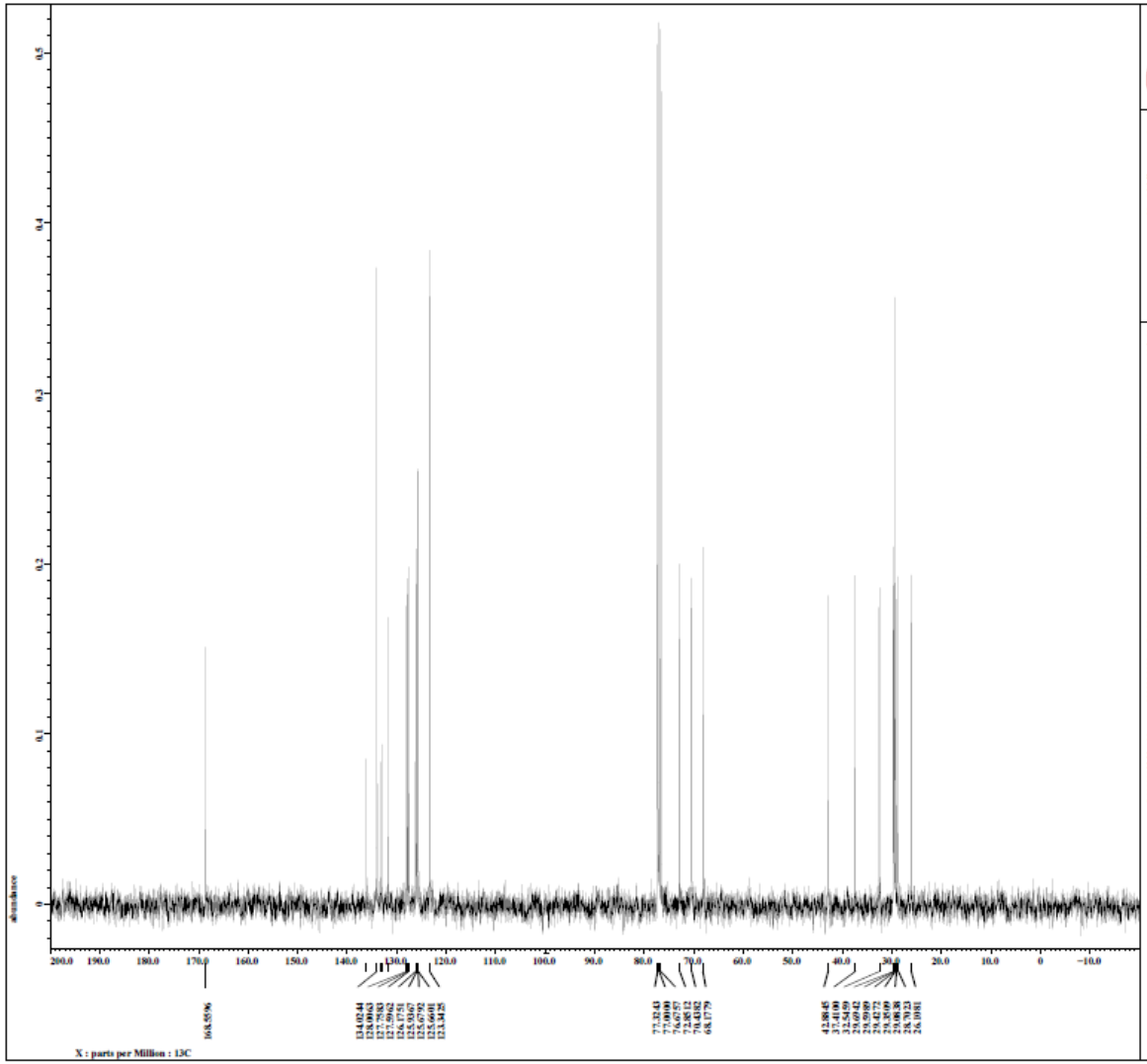
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 12107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 9[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 5
Total_scans = 5

X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 26
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 20.9[dC]



(20d)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 60[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

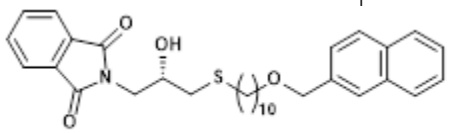
Derived from: 190610_13C_phthN_OE_SC10_N
```

```
Filename          = 190610_13C_phthN_OE_S
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 10-JUN-2019 18:53:00
Revision_time     = 10-JUN-2019 19:26:02
Current_time      = 10-JUN-2019 19:26:26

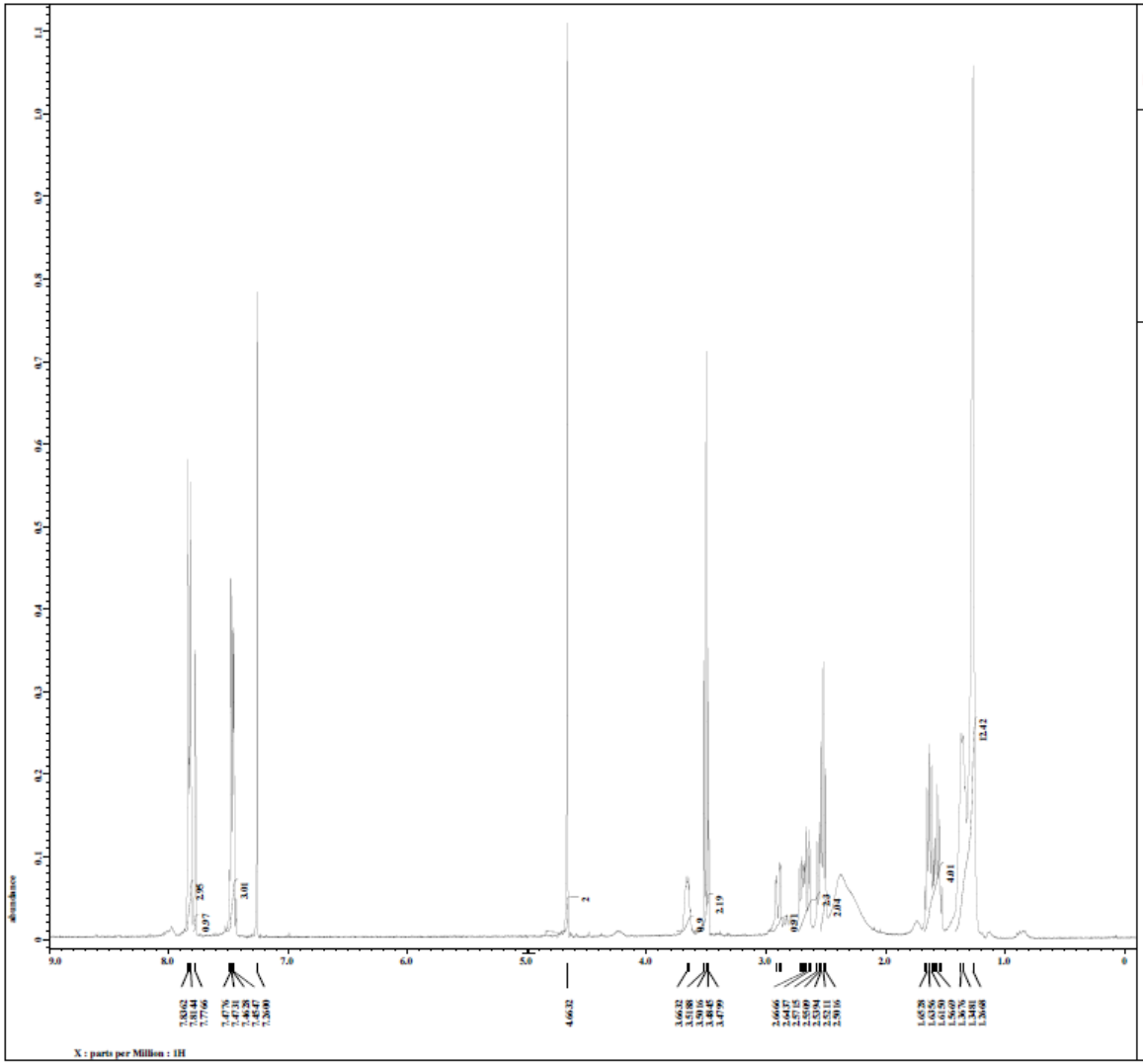
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 55
Total_scans       = 55

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.0[dB]
Irr_atn_noe       = 17.0[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   = 3.06430464[s]
Temp_get          = 21.3[dC]
```



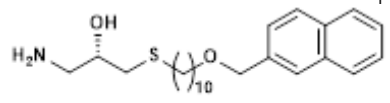
(20d)



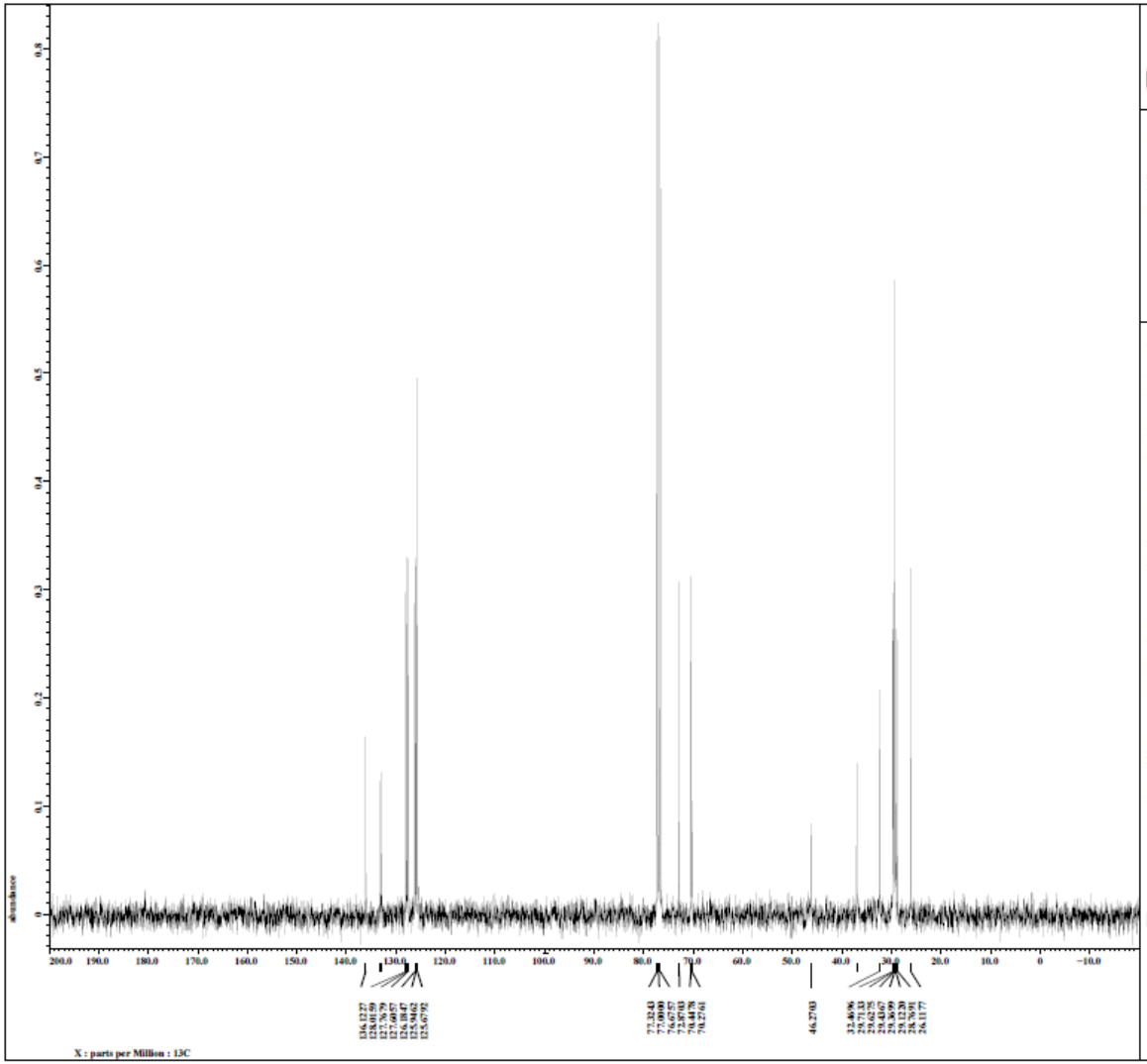
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190611_H2N_OH_SC10_NAP-2.j

Filename	190611_H2N_OH_SC10_NA
Author	delta
Experiment	single_pulse.ex2
Sample_id	1
Solvent	CHLOROFORM-D
Creation_time	11-JUN-2019 12:47:59
Revision_time	11-JUN-2019 13:22:03
Current_time	11-JUN-2019 13:22:31
Comment	single_pulse
Data_format	1D COMPLEX
Dim_size	13107
Dim_title	1H
Dim_units	[ppm]
Dimensions	X
Site	RCX 400P
Spectrometer	DELTA2_NMR
Field_strength	9.20197068[T] (390[MH
X_acq_duration	2.228224[s]
X_domain	1H
X_freq	391.78655441[MHz]
X_offset	5[ppm]
X_points	16384
X_prescans	1
X_resolution	0.44878791[Hz]
X_sweep	7.35234116[kHz]
Irr_domain	1H
Irr_freq	391.78655441[MHz]
Irr_offset	5[ppm]
Tri_domain	1H
Tri_freq	391.78655441[MHz]
Tri_offset	5[ppm]
Clipped	FALSE
Mod_return	1
Scans	7
Total_scans	7
X_90_width	9[us]
X_acq_time	2.228224[s]
X_angle	45[deg]
X_atn	0.99[dB]
X_pulse	4.5[us]
Irr_mode	Off
Tri_mode	Off
Dante_preset	FALSE
Initial_wait	1[s]
Recvr_gain	26
Relaxation_delay	5[s]
Repetition_time	7.228224[s]
Temp_get	22.7[dC]



(21d)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

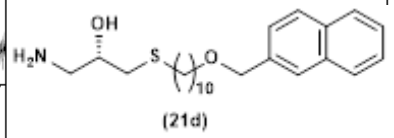
Derived from: 190611_13C_HZN_OH_SC10_NAP
```

```
Filename          = 190611_13C_HZN_OH_SC1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUN-2019 10:28:11
Revision_time     = 11-JUN-2019 11:00:11
Current_time      = 11-JUN-2019 11:01:38

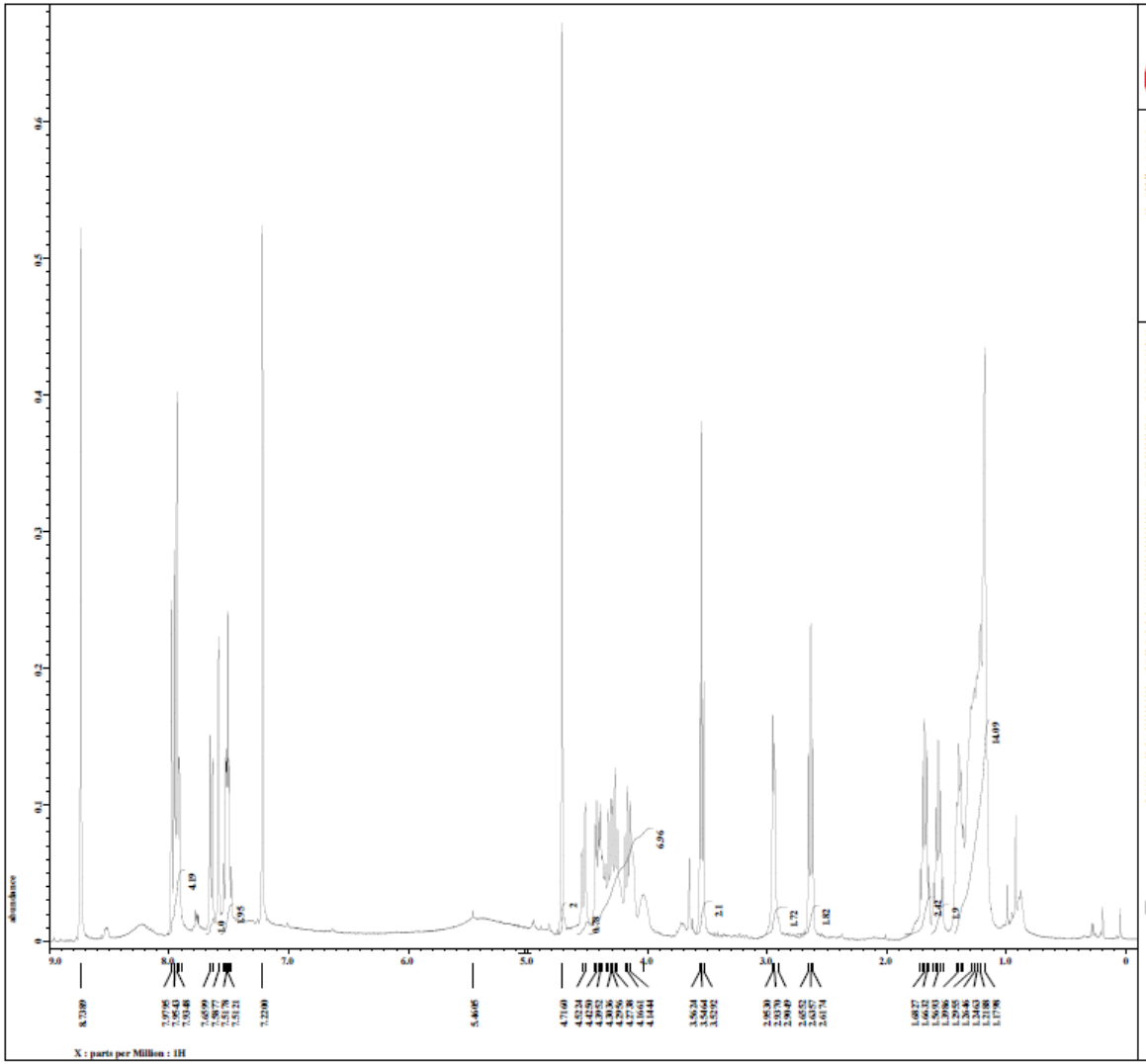
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92358061[Hz]
X_sweep           = 30.75817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 69
Total_scans       = 69

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08222222[us]
Irr_atn_dec       = 17.6[dB]
Irr_atn_noe       = 17.6[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 58
Relaxation_delay  = 2[s]
Repetition_time   = 3.06420464[s]
Temp_get          = 22.2[dC]
```



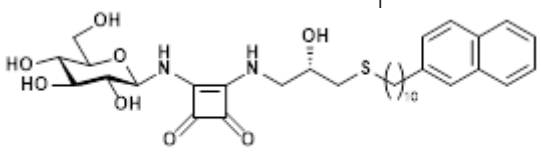
(21d)



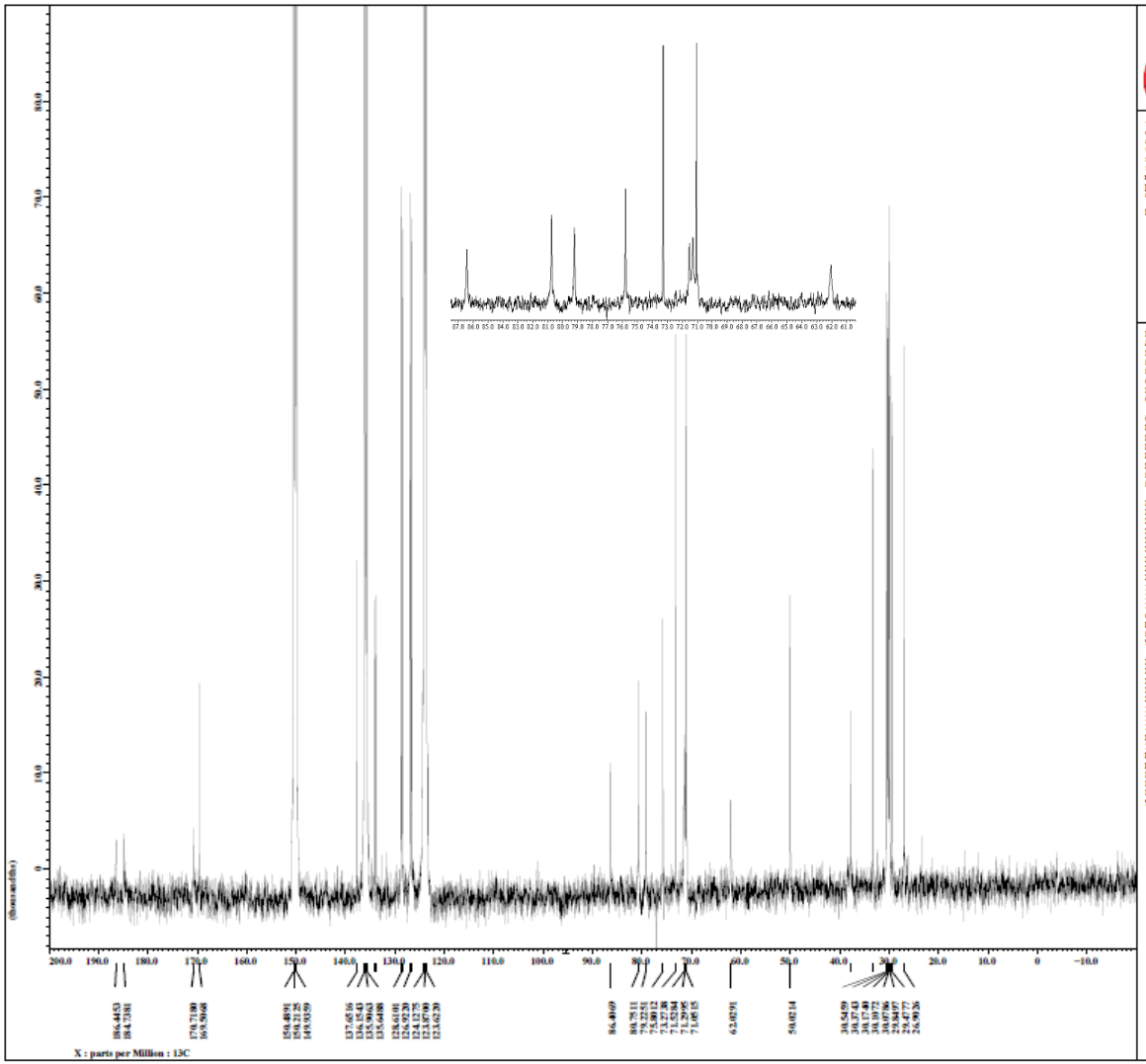
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190612_GlcNDH3_C10_NAP-1.j

Filename	= 190612_GlcNDH3_C10_NA
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= PYRIDINE-D5
Creation_time	= 12-JUN-2019 12:52:26
Revision_time	= 12-JUN-2019 15:00:19
Current_time	= 12-JUN-2019 15:00:57
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.25294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 7
Total_scans	= 7
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 21.9[dC]



(11d)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

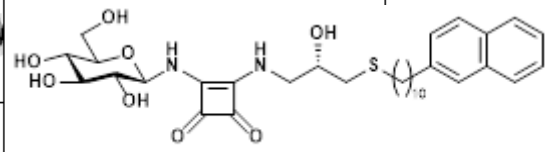
Derived from: 190612_13C_GlcNDRS_C10_NAP
```

```
Filename          = 190612_13C_GlcNDRS_C1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 12-JUN-2019 14:20:54
Revision_time     = 12-JUN-2019 14:55:08
Current_time      = 12-JUN-2019 14:55:48

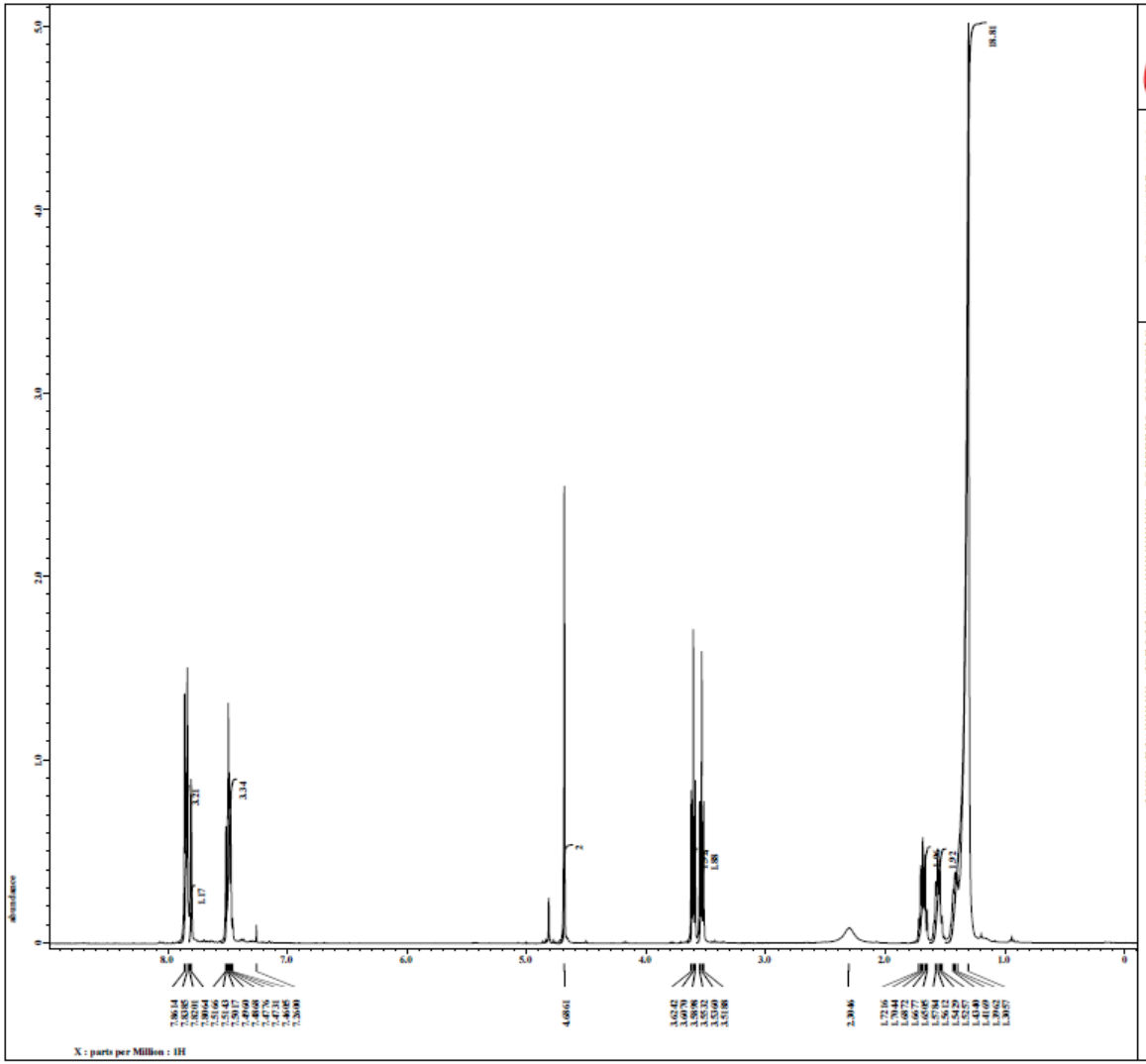
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = NCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92958061[Hz]
X_sweep           = 30.76817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1323
Total_scans       = 1323

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 21.9[dC]
```



(11d)



```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
expp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
1Dpa
auto_reference : 5[%] : TRUE
reference : 7.24[ppm] : 77[ppm]
reference : 77[ppm] : 77[ppm]
reference : 77[ppm] : 77[ppm]
reference : 77[ppm] : 77[ppm]
auto_reference : 5[%] : TRUE

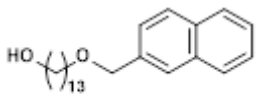
Derived from: 180611_HO_C13_Naph-1.jdf
```

```
Filename          = 180611_HO_C13_Naph-6.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUN-2018 11:43:52
Revision_time     = 28-AUG-2018 15:21:31
Current_time      = 28-AUG-2018 15:22:02

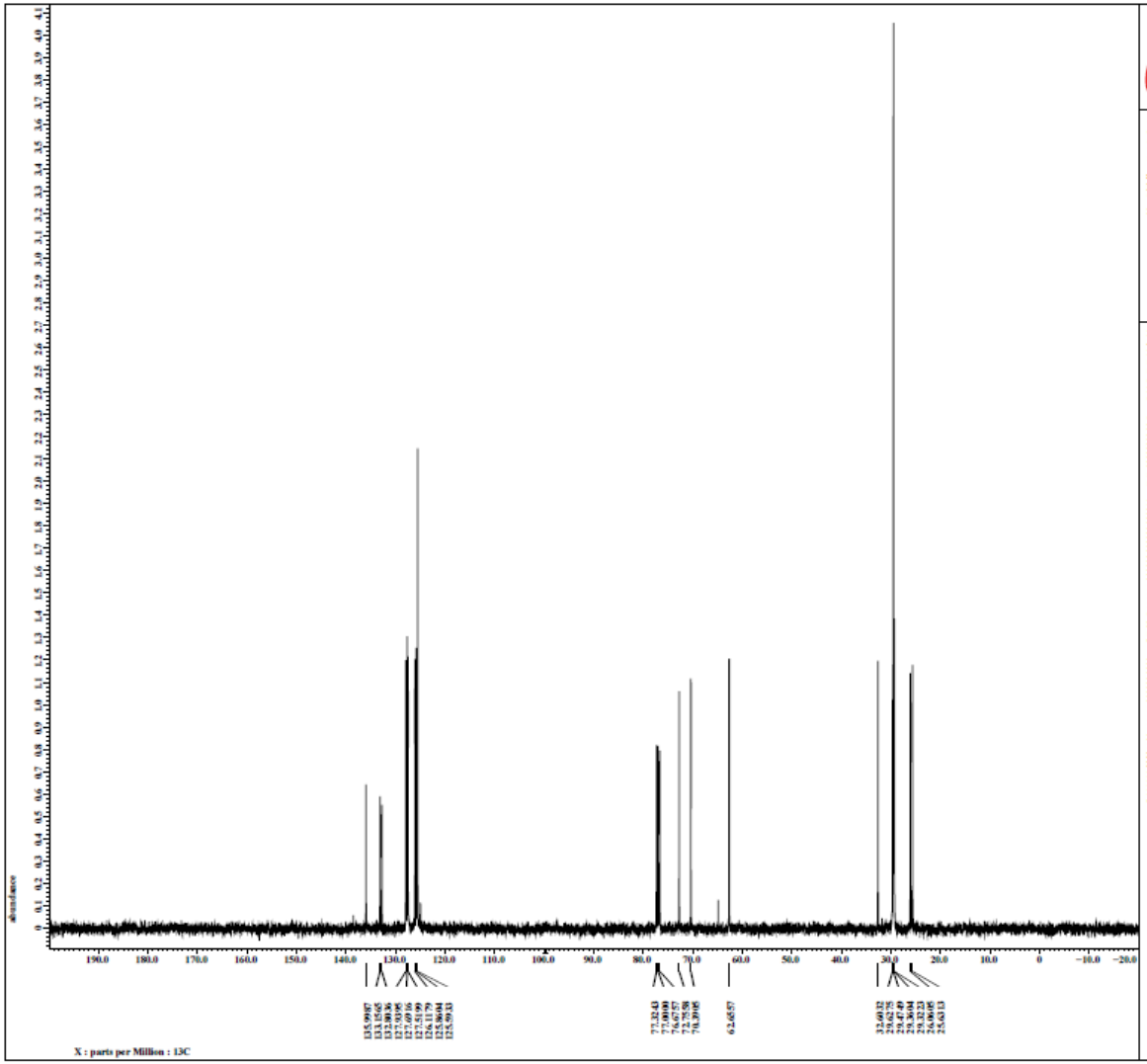
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 25[dC]
```



(13e)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

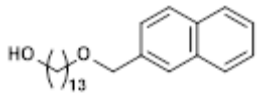
Derived from: 180611_C13_HO_C13_Naph-1.j
```

```
Filename          = 180611_C13_HO_C13_Nap
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUN-2018 11:45:50
Revision_time     = 28-AUG-2018 15:24:31
Current_time      = 28-AUG-2018 15:25:10

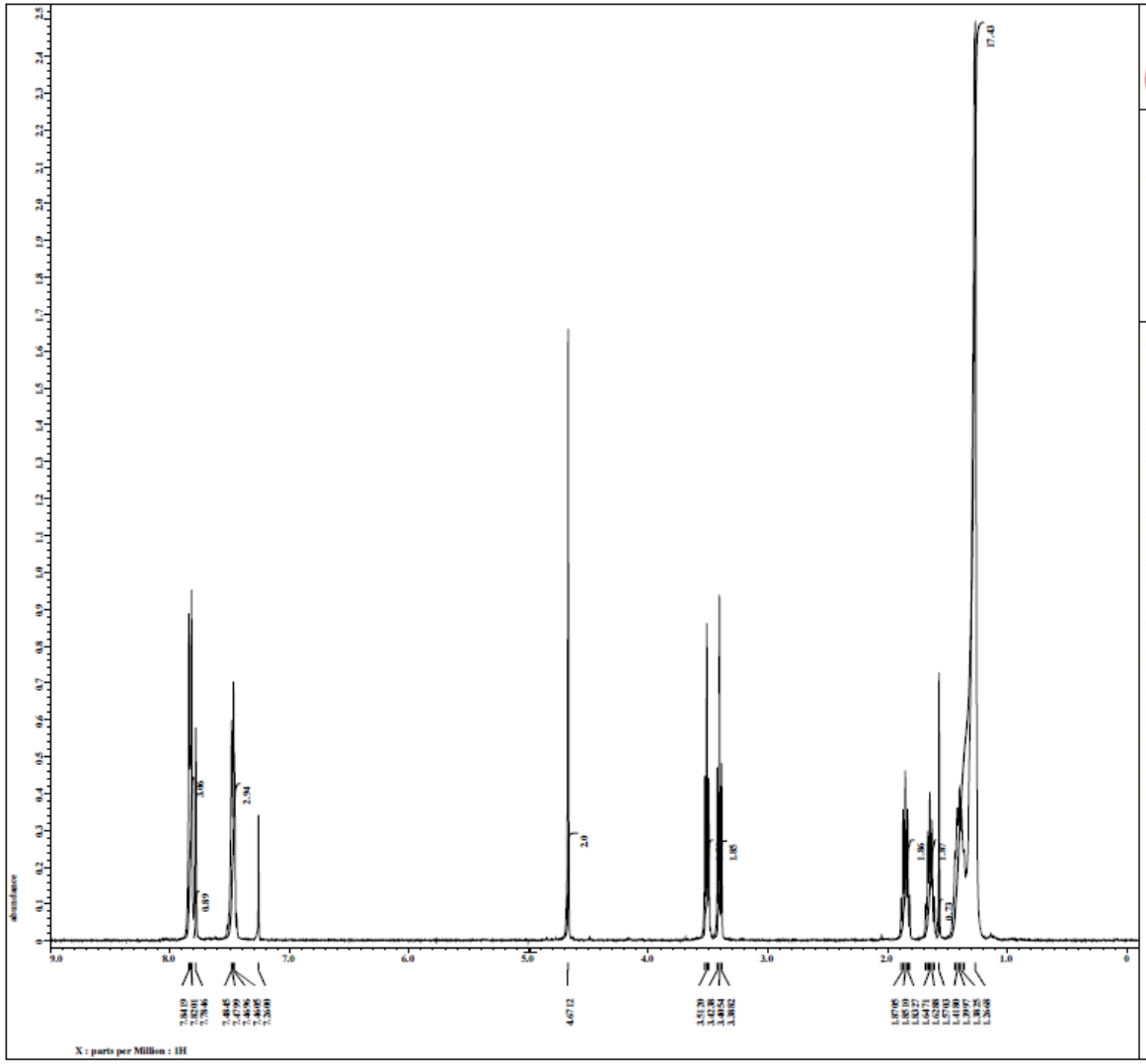
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.75817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 22
Total_scans       = 22

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 58
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 25.2[dC]
```



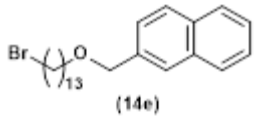
(13e)



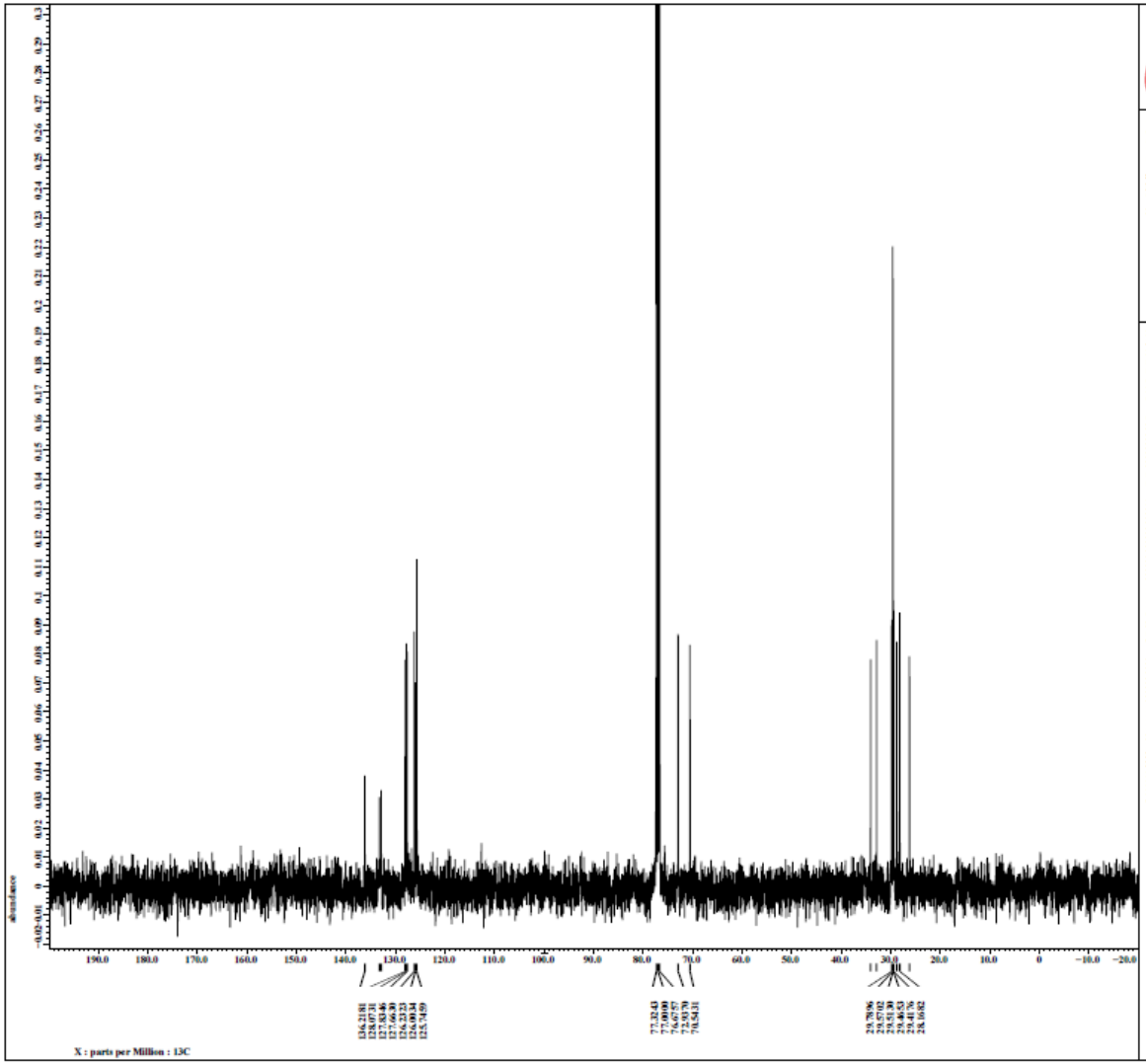
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180823_Br_C13_Naph-1.jdf

Filename	= 180823_Br_C13_Naph-4.
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-AUG-2018 13:08:59
Revision_time	= 24-AUG-2018 13:38:34
Current_time	= 24-AUG-2018 13:39:05
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [PPM]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 24.8[dC]



(14e)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

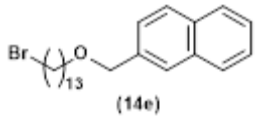
Derived from: 180823_13C_C13_Naph-1.jdf
```

```
Filename          = 180823_13C_C13_Naph-4
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 24-AUG-2018 13:14:47
Revision_time     = 24-AUG-2018 13:42:16
Current_time      = 24-AUG-2018 13:42:45

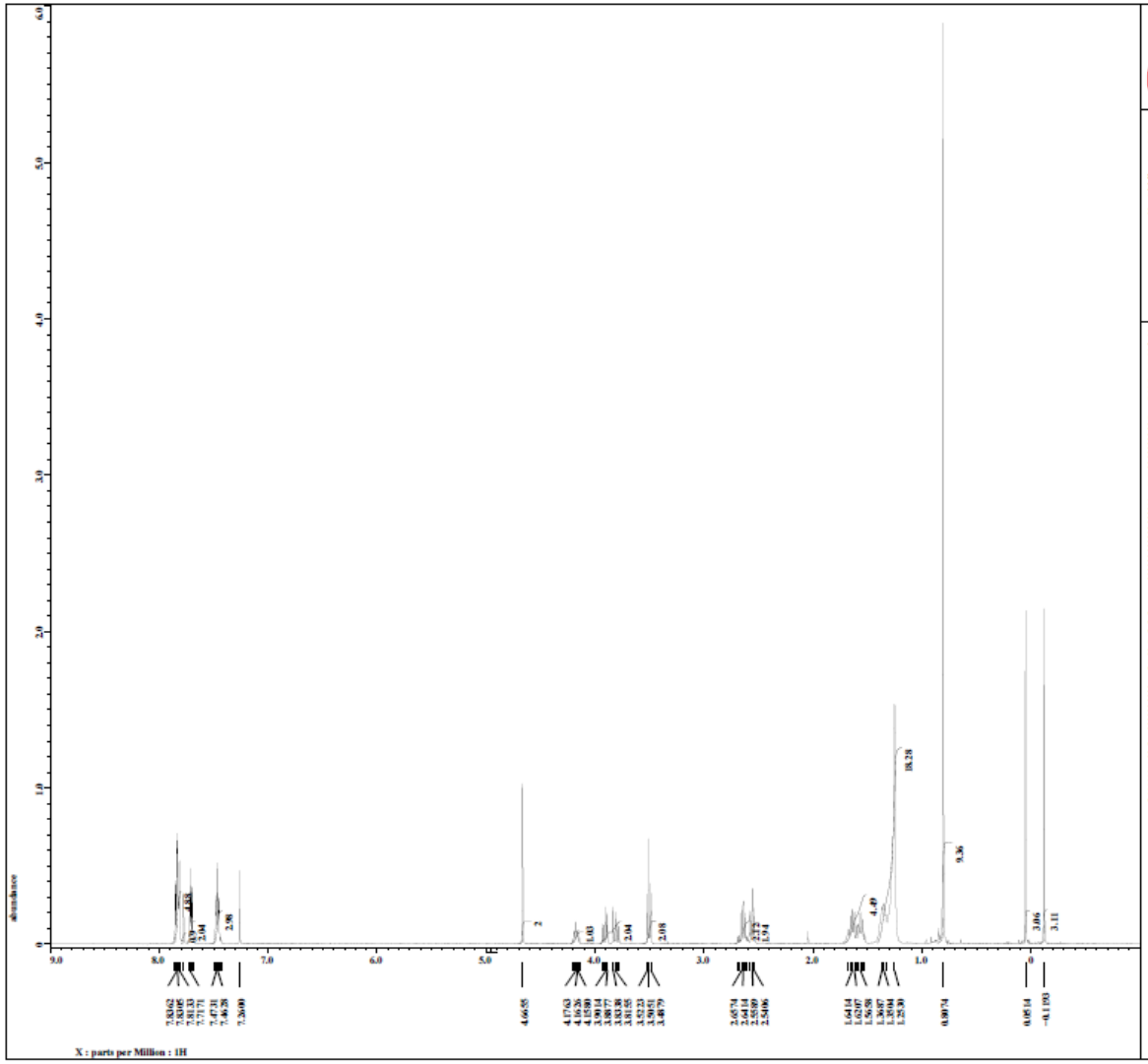
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92958861[Hz]
X_sweep           = 30.75817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 92
Total_scans       = 92

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 25[dC]
```



(14e)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

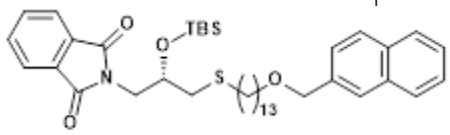
Derived from: 191016_phth_TBS_C13_NAP-4.
```

```
Filename          = 191016_phth_TBS_C13_N
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 16-OCT-2019 19:29:25
Revision_time     = 16-OCT-2019 20:10:20
Current_time      = 16-OCT-2019 20:11:05

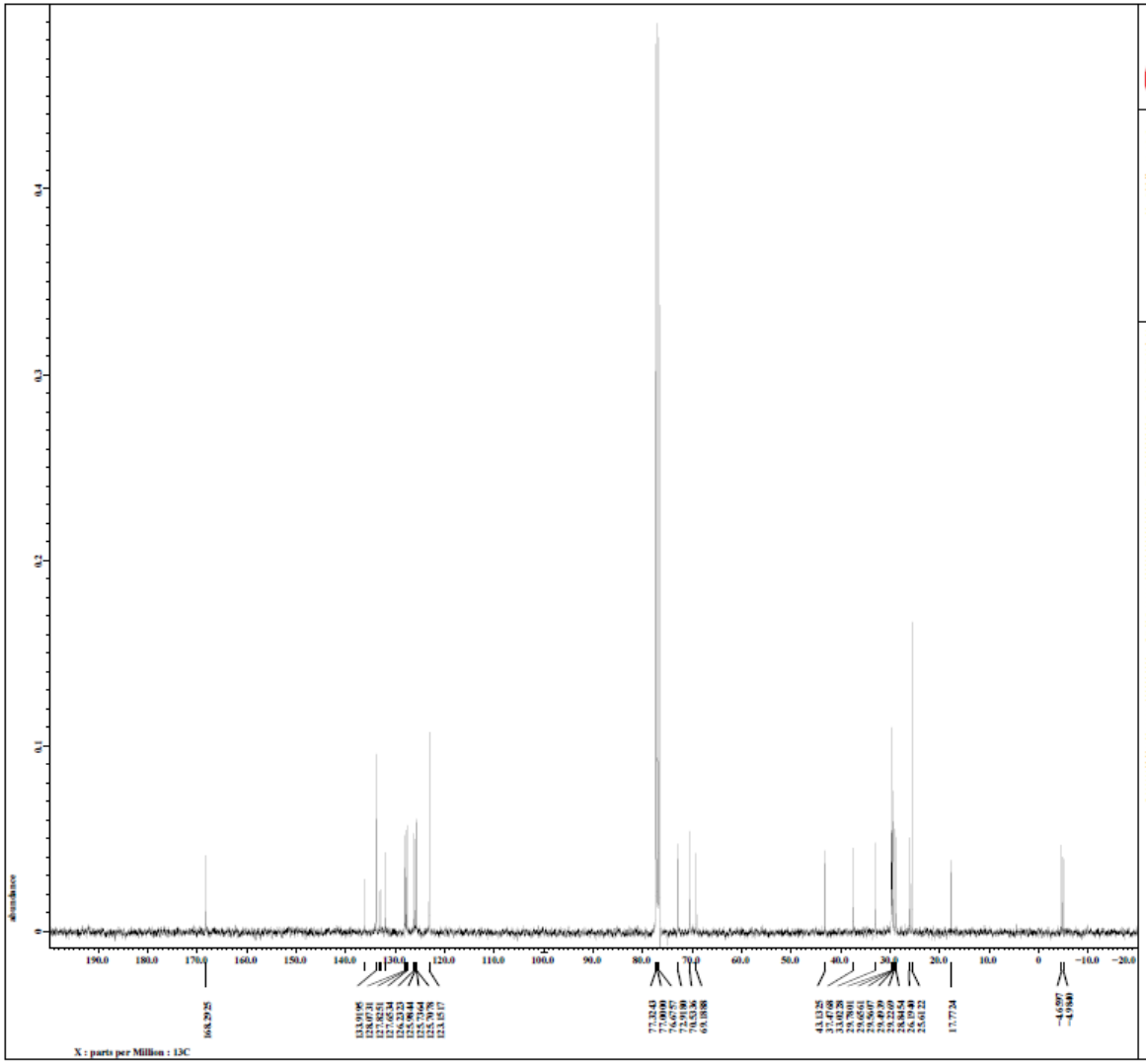
Comment           = single pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = 1
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dumb_preset       = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 21.7[dC]
```



(19e)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

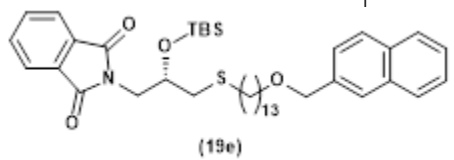
Derived from: 191016_13C_phth_TBS_C13_MA
```

```
Filename          = 191016_13C_phth_TBS_C
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 16-OCT-2019 20:10:23
Revision_time     = 16-OCT-2019 20:47:19
Current_time      = 16-OCT-2019 20:49:16

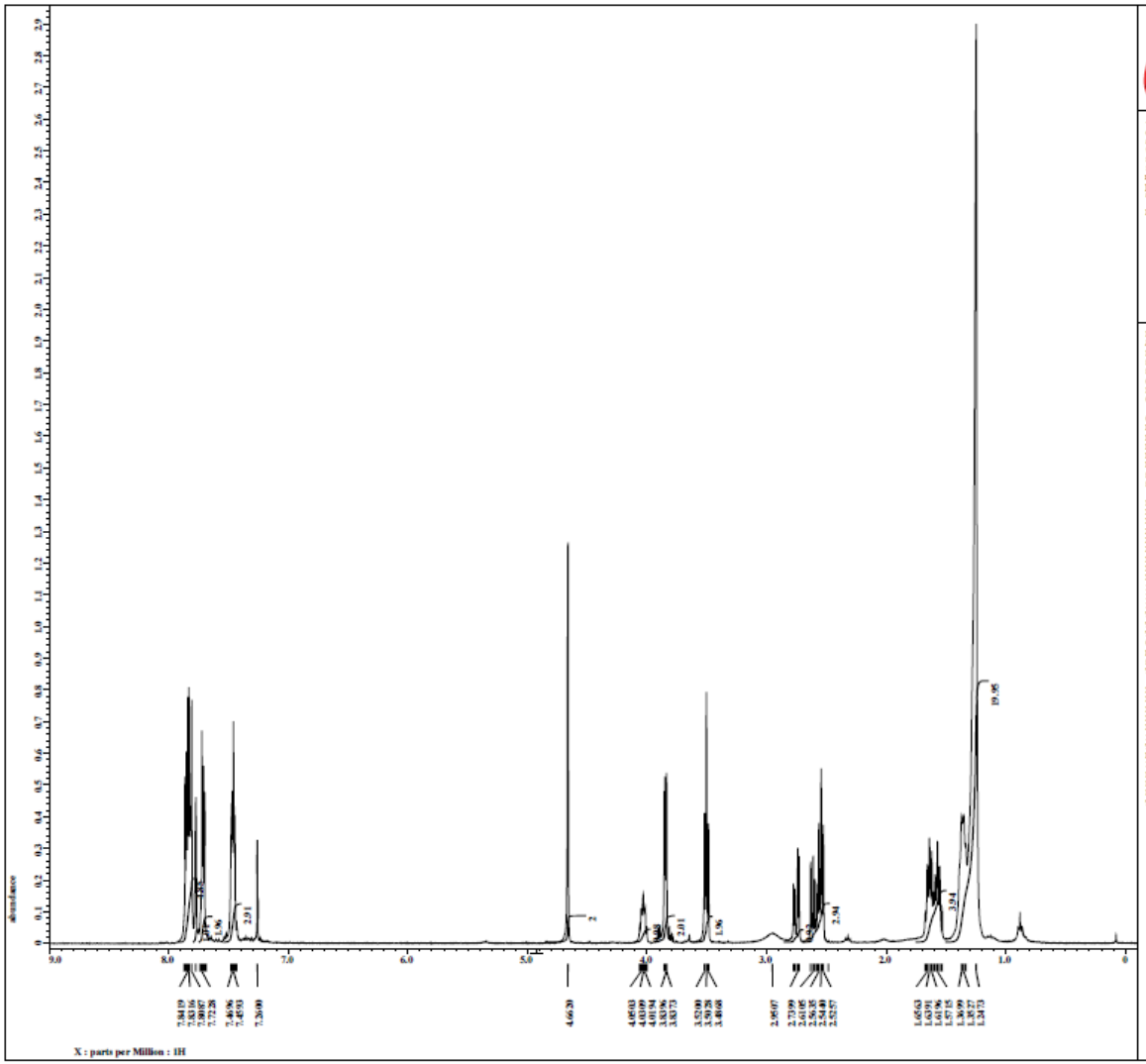
Comment           = single_pulse_decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = NCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T]  (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92258061[Hz]
X_sweep           = 30.76817724[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 777
Total_scans       = 777

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 34
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.2[dC]
```



(19e)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
TRUE
auto_reference : 5[%] : TRUE

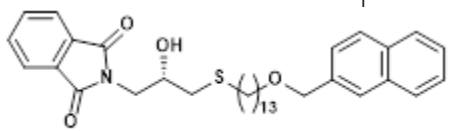
Derived from: 180731_2_phth_OH_C13_Nap-1
```

```
Filename          = 180731_2_phth_OH_C13_
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 31-JUL-2018 14:52:39
Revision_time     = 31-JUL-2018 15:17:01
Current_time      = 31-JUL-2018 15:17:37

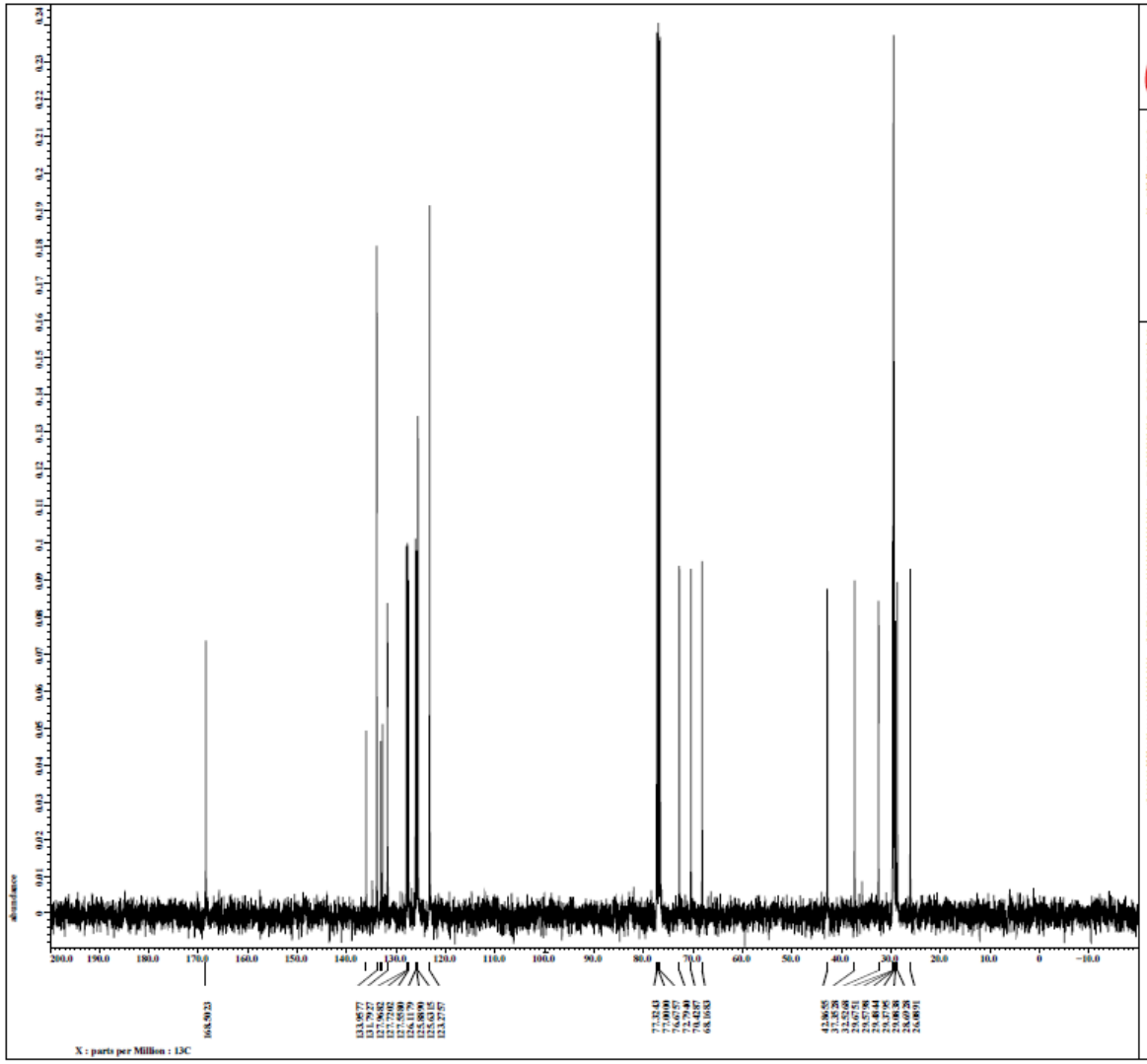
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35234116[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Delta_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.2[dC]
```



(20e)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

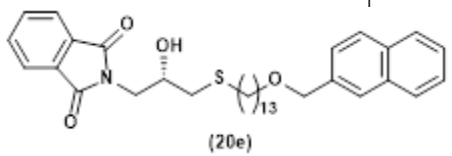
Derived from: 180721_13C_phth_OH_C13_Nap
```

```
Filename            = 180721_13C_phth_OH_C1
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = 1
Solvent             = CHLOROFORM-D
Creation_time       = 31-JUL-2018 14:42:49
Revision_time       = 31-JUL-2018 15:03:26
Current_time        = 31-JUL-2018 15:05:19

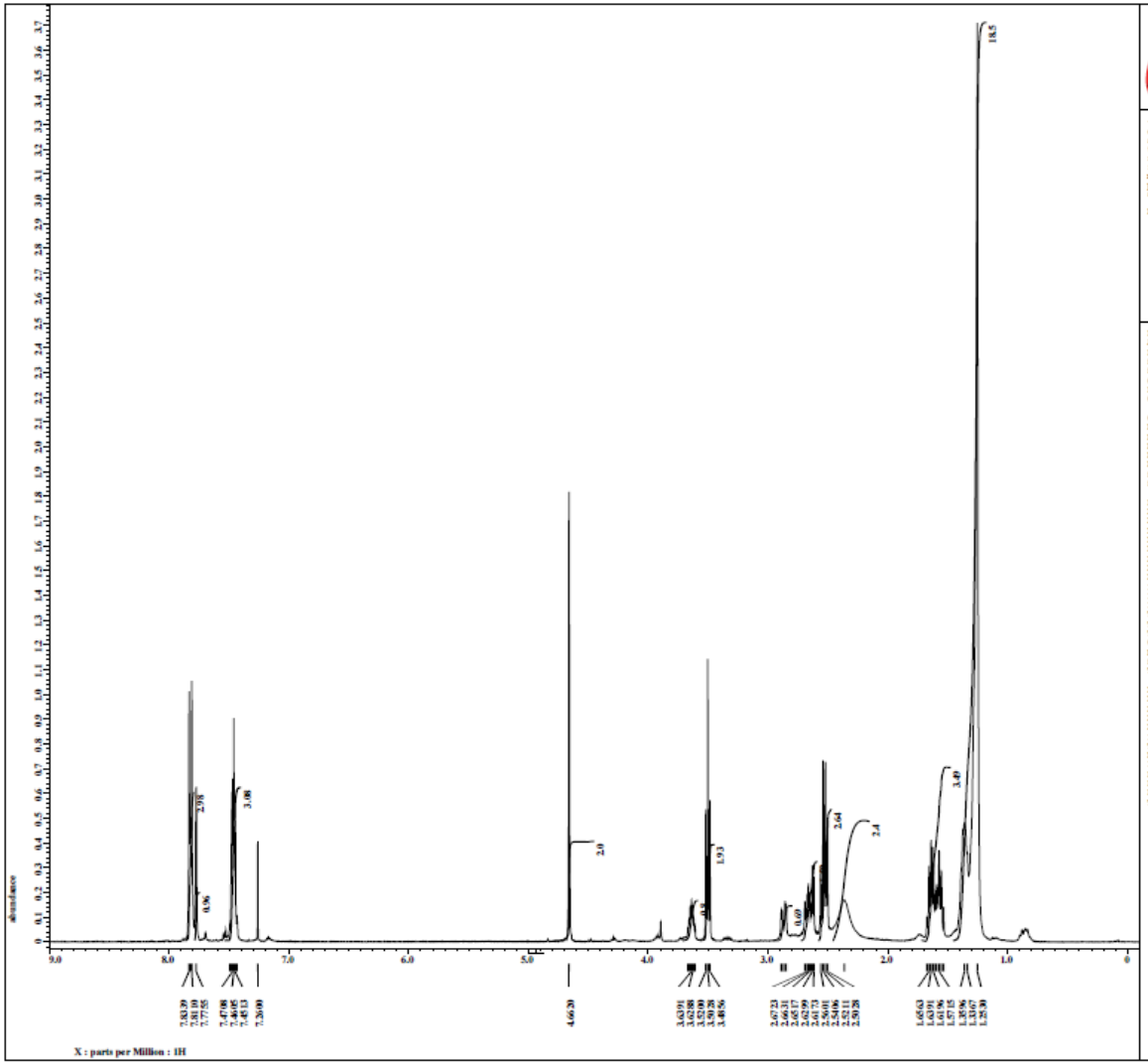
Comment             = single pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCX 400P
Spectrometer        = DELTA2_NMR

Field_strength      = 9.20197068[T] (390[MH
X_acq_duration      = 1.06430464[s]
X_domain            = 13C
X_freq              = 98.51479726[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.92358061[Hz]
X_sweep             = 30.78817724[kHz]
Irr_domain          = 1H
Irr_freq            = 391.78655441[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 55
Total_scans         = 55

X_90_width          = 9.25[us]
X_acq_time          = 1.06430464[s]
X_angle             = 30[deg]
X_atn               = 4[dB]
X_pulse             = 3.08333333[us]
Irr_atn_dec         = 17.0[dB]
Irr_atn_noe         = 17.0[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 48
Relaxation_delay    = 2[s]
Repetition_time     = 3.06430464[s]
Temp_get            = 22.4[dC]
```



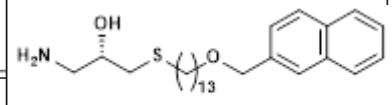
(20e)



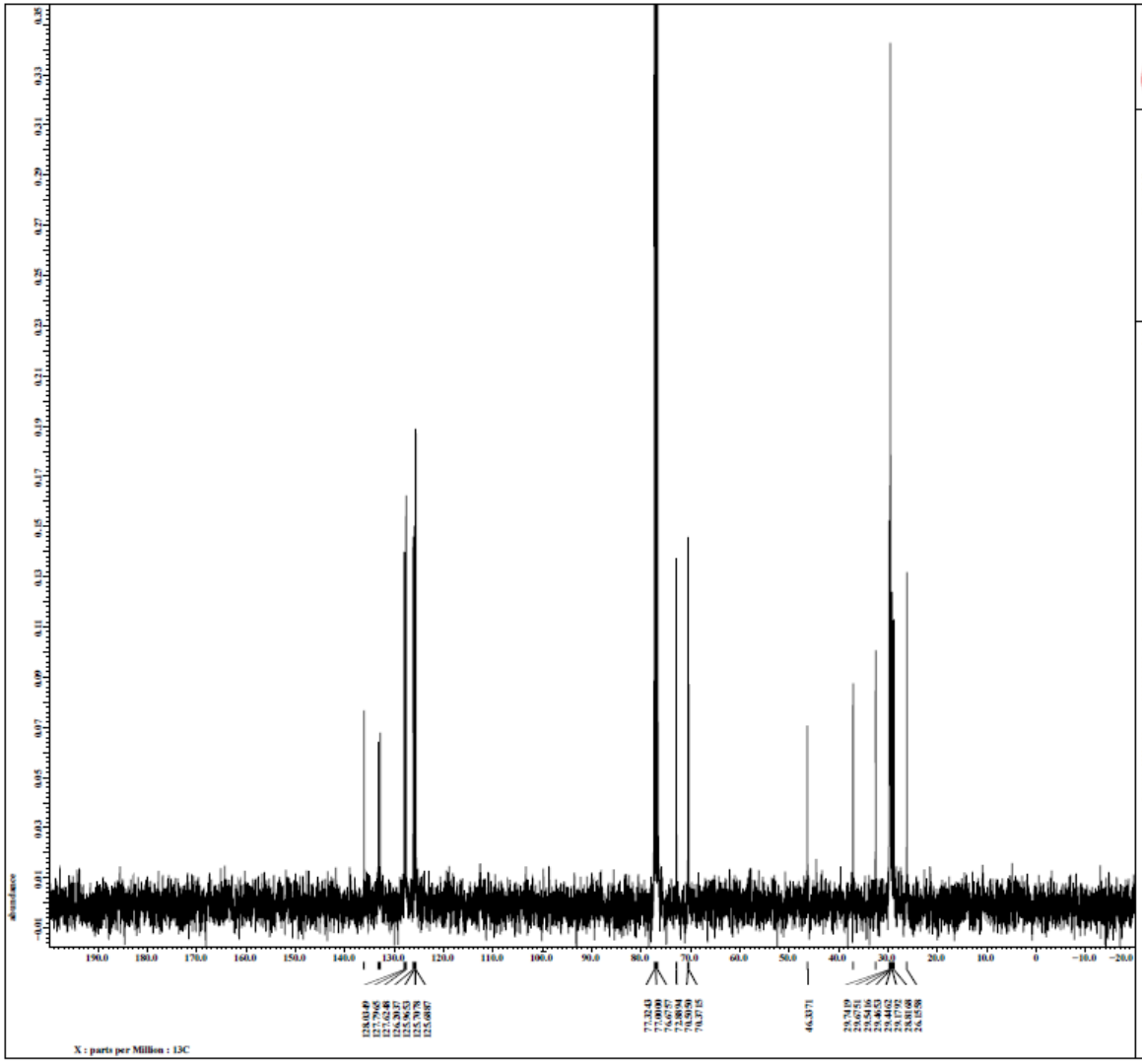
```
----- PROCESSING PARAMETERS -----
dc balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180801_phth_OH_C13_Nap-1.j
```

Filename	= 180801_phth_OH_C13_Na
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 1-AUG-2018 09:44:27
Revision_time	= 1-AUG-2018 10:07:16
Current_time	= 1-AUG-2018 10:07:49
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 131107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (399[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44878791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 7
Total_scans	= 7
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 26
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 22.6[dC]



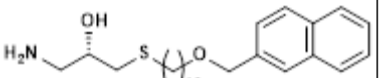
(21e)



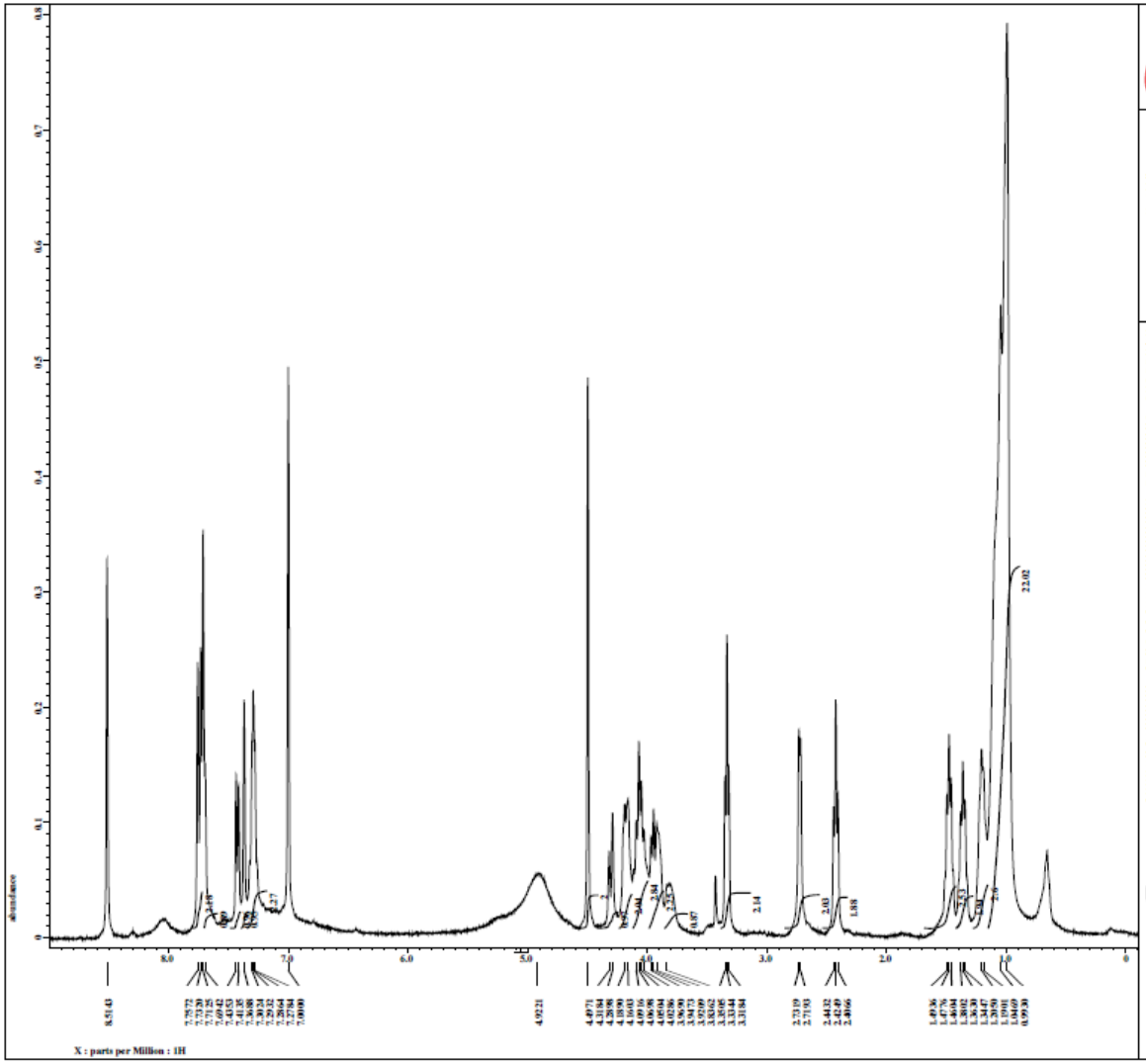
```
----- PROCESSING PARAMETERS -----
dc balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 160801_13C_phth_OH_C13_Nap
```

Filename	= 160801_13C_phth_OH_C1
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 1-AUG-2016 09:50:55
Revision_time	= 1-AUG-2016 10:11:52
Current_time	= 1-AUG-2016 10:12:21
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06420464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.76817724[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 102
Total_scans	= 102
X_90_width	= 9.25[us]
X_acq_time	= 1.06420464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 3.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 58
Relaxation_delay	= 2[s]
Repetition_time	= 3.06420464[s]
Temp_get	= 22.9[dC]



(21e)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

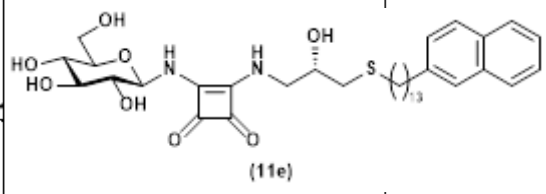
Derived from: GlcN_DES_C13_Naph-1.jdf
```

```
Filename          = GlcN_DES_C13_Naph-4.j
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 31-AUG-2018 13:02:00
Revision_time     = 31-AUG-2018 13:34:31
Current_time      = 31-AUG-2018 13:35:00

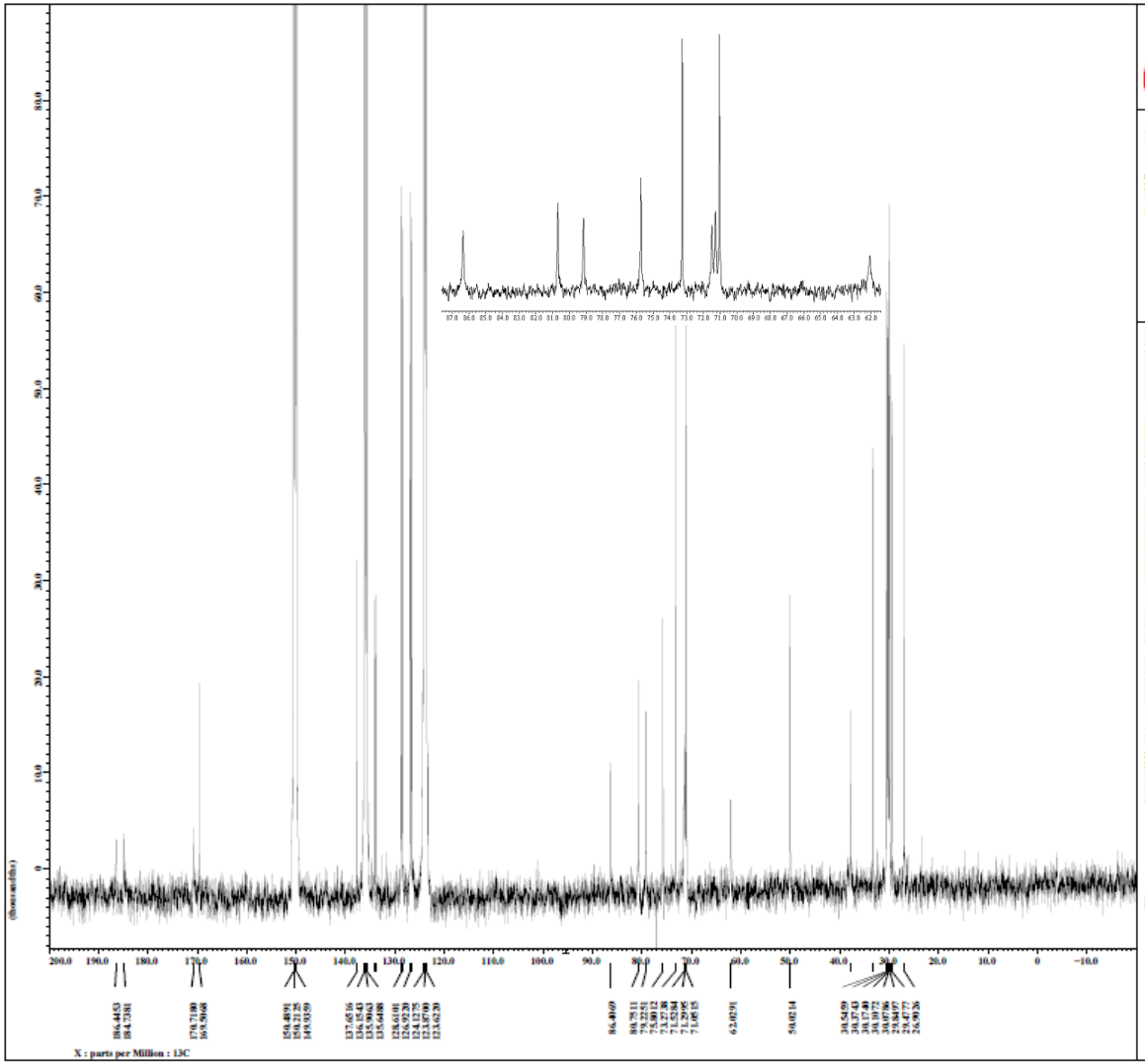
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 131107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 24
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.4[dC]
```



(11e)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

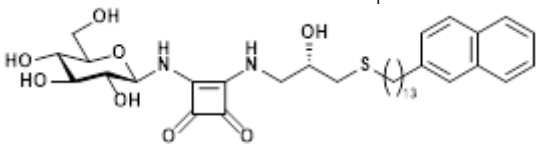
Derived from: 190612_13C_GlcNDRS_C10_NAP
```

```
Filename          = 190612_13C_GlcNDRS_C1
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 12-JUN-2019 14:20:54
Revision_time     = 12-JUN-2019 14:55:06
Current_time      = 12-JUN-2019 14:55:48

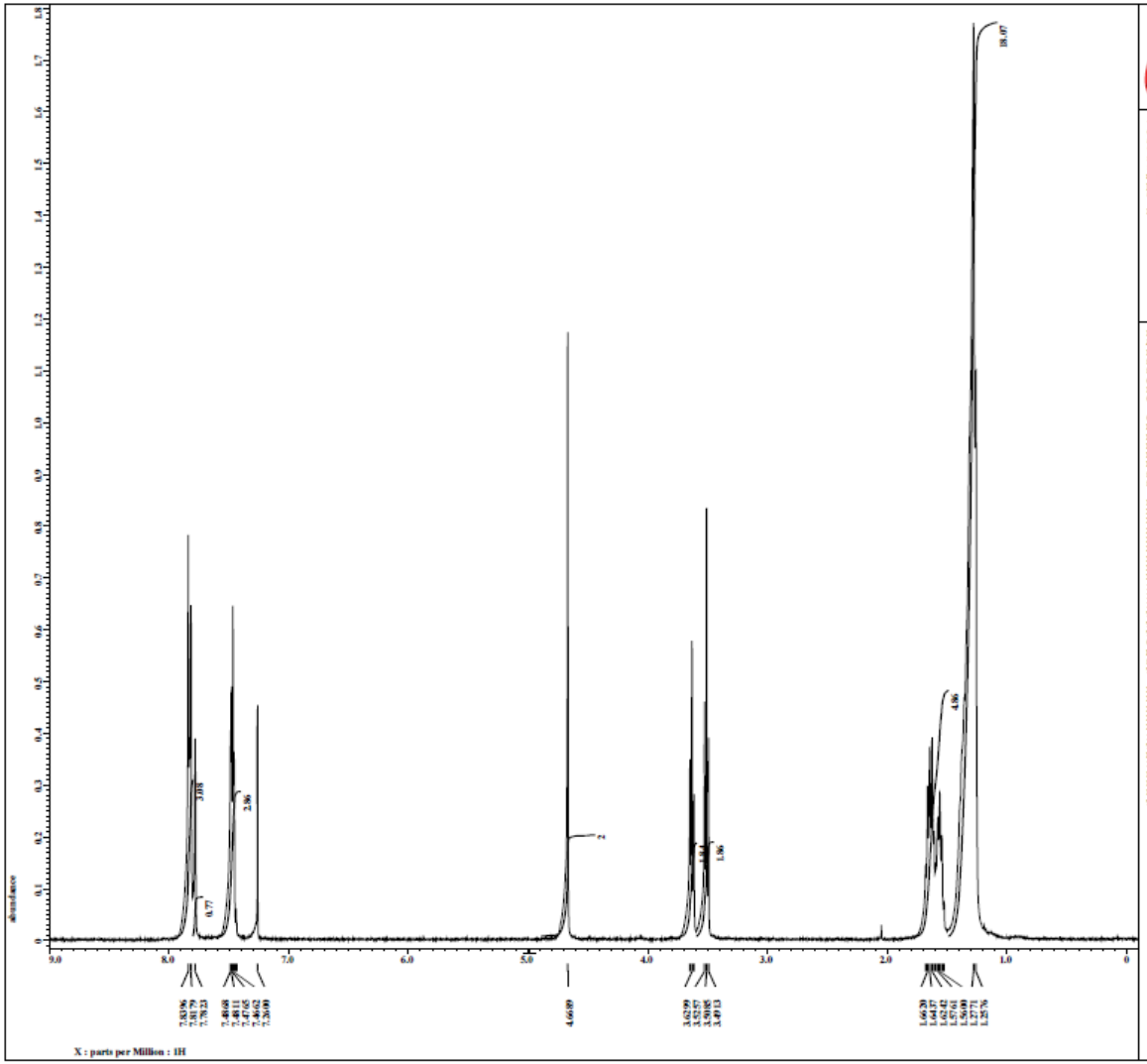
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1322
Total_scans       = 1322

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 21.9[dC]
```



(11e)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
expp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

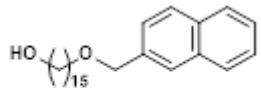
Derived from: 180823_HO_C15_Naph-1.jdf
```

```
Filename          = 180823_HO_C15_Naph-4.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 23-AUG-2018 15:39:39
Revision_time     = 23-AUG-2018 16:07:43
Current_time      = 23-AUG-2018 16:08:10

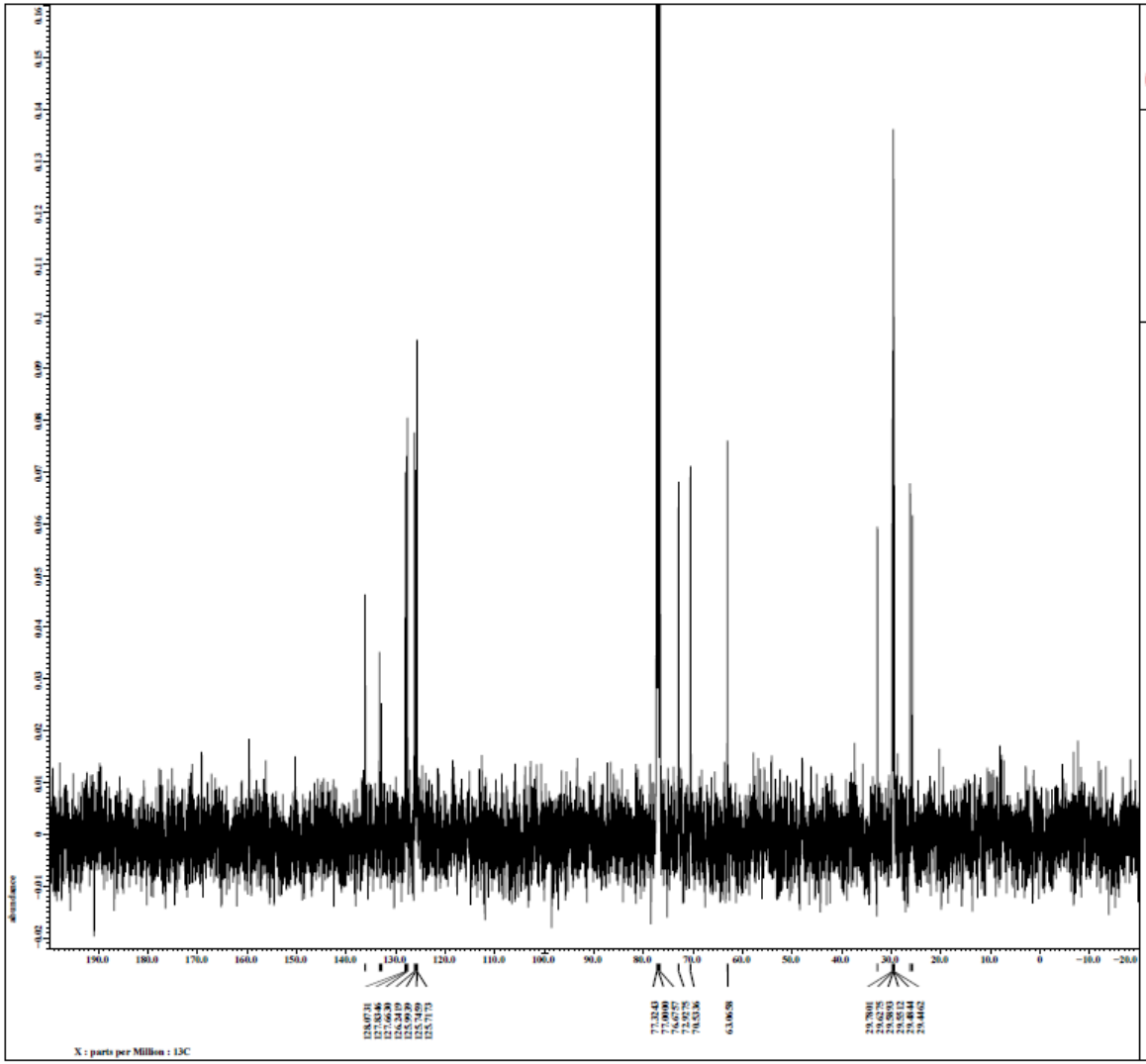
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 131107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 24.8[dC]
```



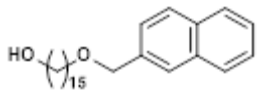
(13f)



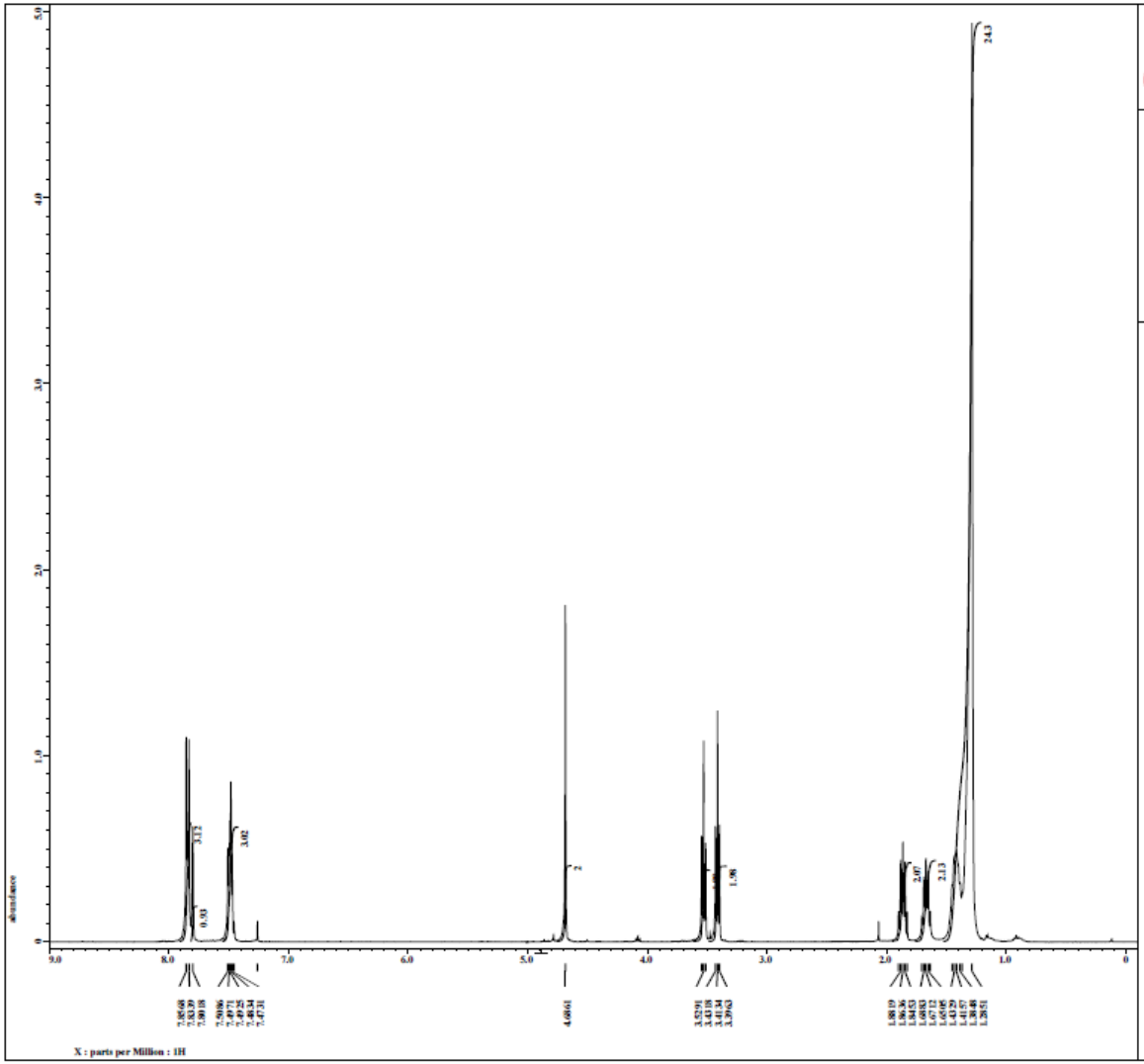
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180823_13C_HO_C15_Naph-1.j

Filename	= 180823_13C_HO_C15_Nap
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 23-AUG-2018 15:46:16
Revision_time	= 23-AUG-2018 16:14:35
Current_time	= 23-AUG-2018 16:15:15
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.75817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 107
Total_scans	= 107
X_90_width	= 9.25[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 2.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 58
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 25[dC]



(13f)




```
----- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

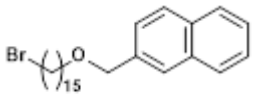
Derived from: 180823_Br_C15_Naph-1.jdf
```

```
Filename          = 180823_Br_C15_Naph-4.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 24-AUG-2018 19:07:48
Revision_time     = 24-AUG-2018 19:26:14
Current_time      = 24-AUG-2018 19:26:26

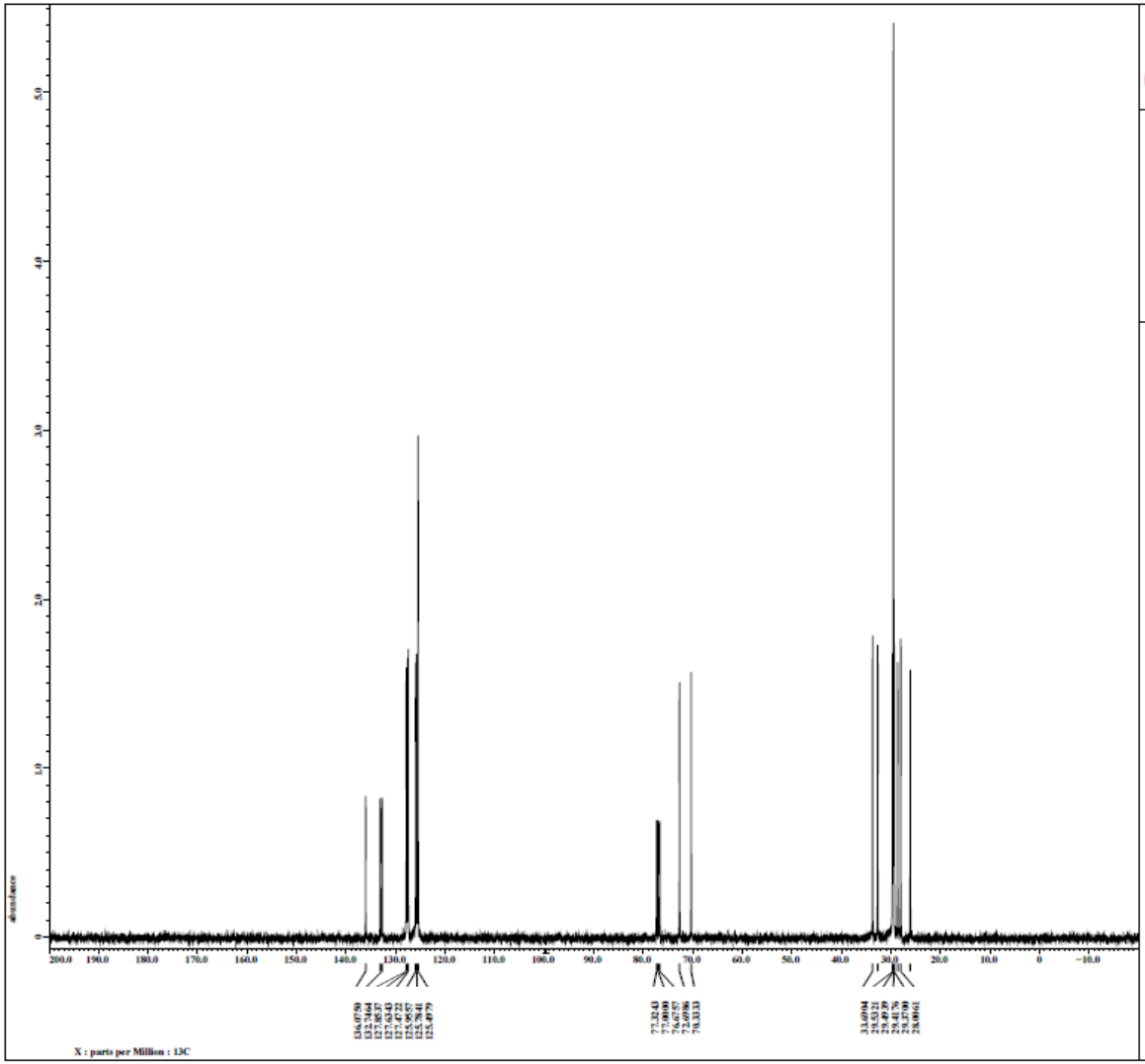
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [PPM]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 24.7[dC]
```



(14f)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

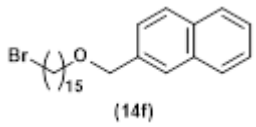
Derived from: 180718_13C_Br_C15_Nap-1.jd
```

```
Filename          = 180718_13C_Br_C15_Nap
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 19-JUL-2018 20:28:56
Revision_time     = 19-JUL-2018 20:51:22
Current_time      = 19-JUL-2018 20:53:06

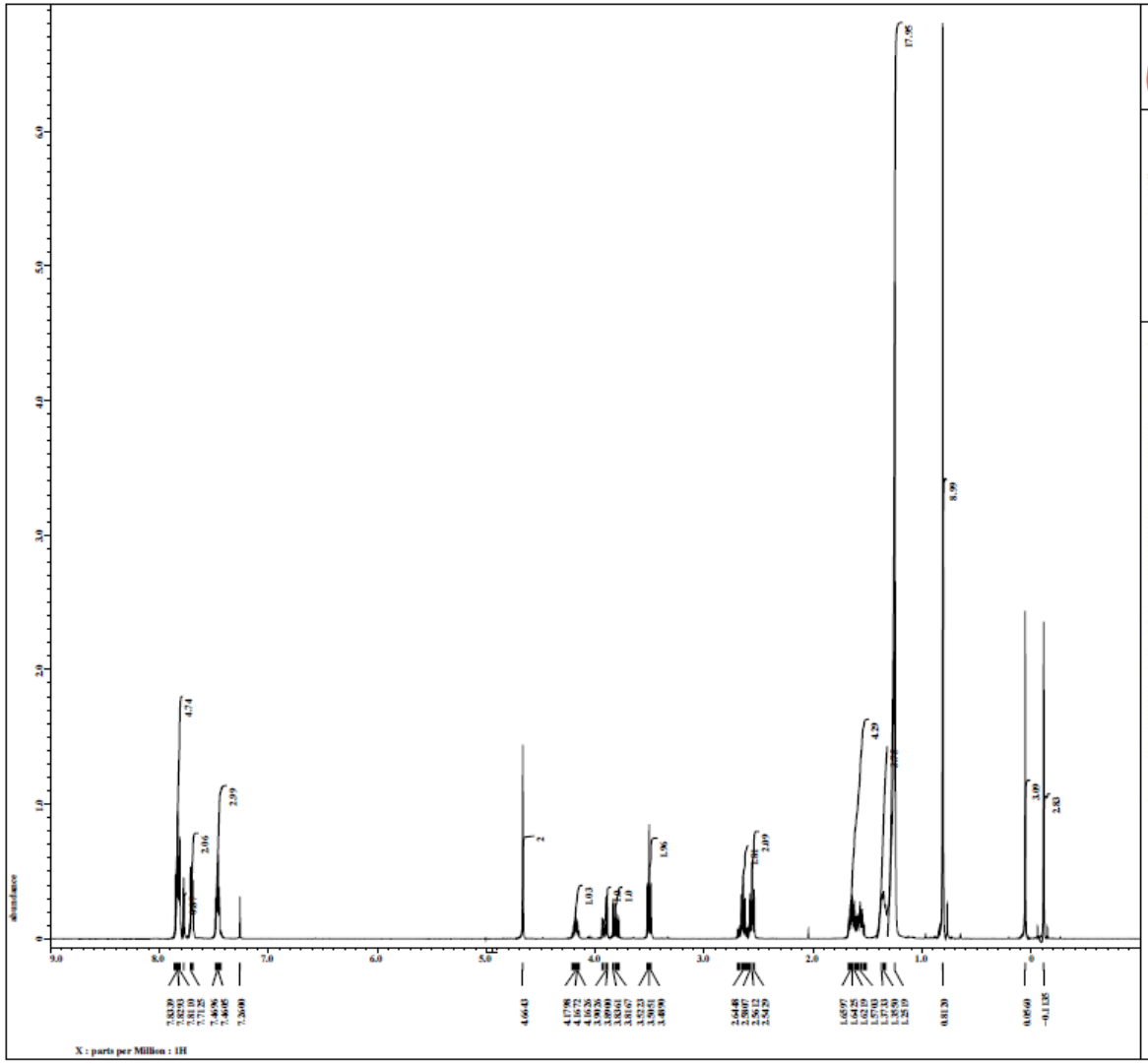
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK_400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.76817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 27
Total_scans       = 27

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.5[dB]
Irr_atn_noe       = 17.5[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.6[dC]
```



(14f)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

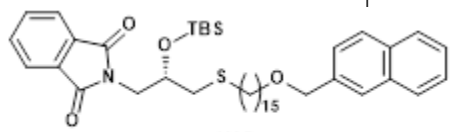
Derived from: 180720_phth_TBS_C15_Nap-1.
```

```
Filename          = 180720_phth_TBS_C15_N
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 20-JUL-2018 16:06:20
Revision_time     = 20-JUL-2018 17:30:05
Current_time      = 20-JUL-2018 17:30:51

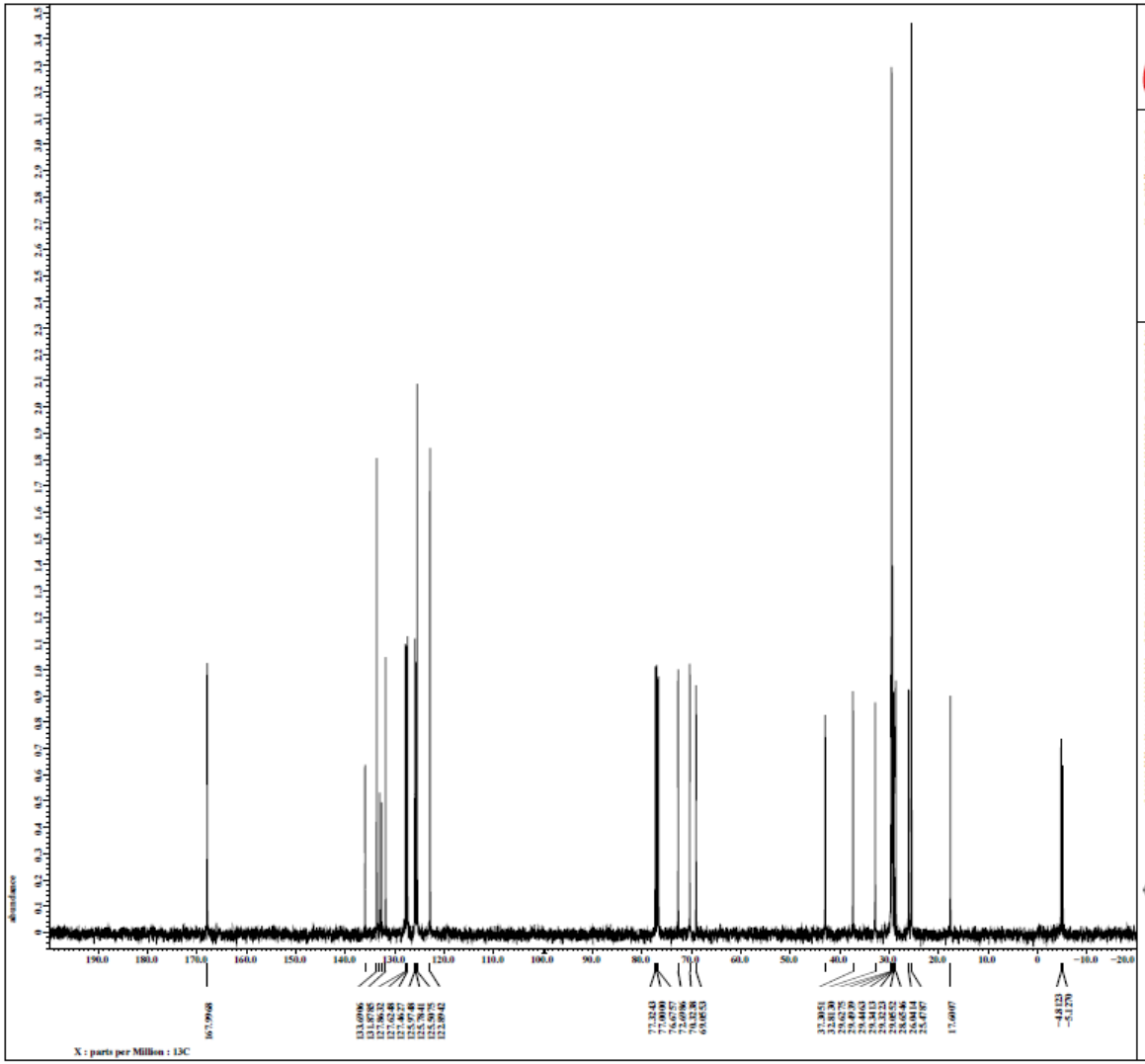
Comment           = single pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.7[dC]
```



(19f)



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
auto_reference : 5[%] : TRUE
auto_reference : 5[%] : TRUE

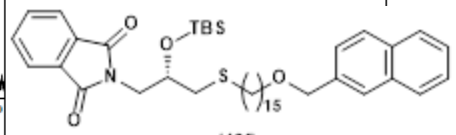
Derived from: 180720_13C_phth_TBS_C15_Na
```

```
Filename          = 180720_13C_phth_TBS_C
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 20-JUL-2018 16:10:05
Revision_time     = 20-JUL-2018 17:25:15
Current_time      = 20-JUL-2018 17:25:50

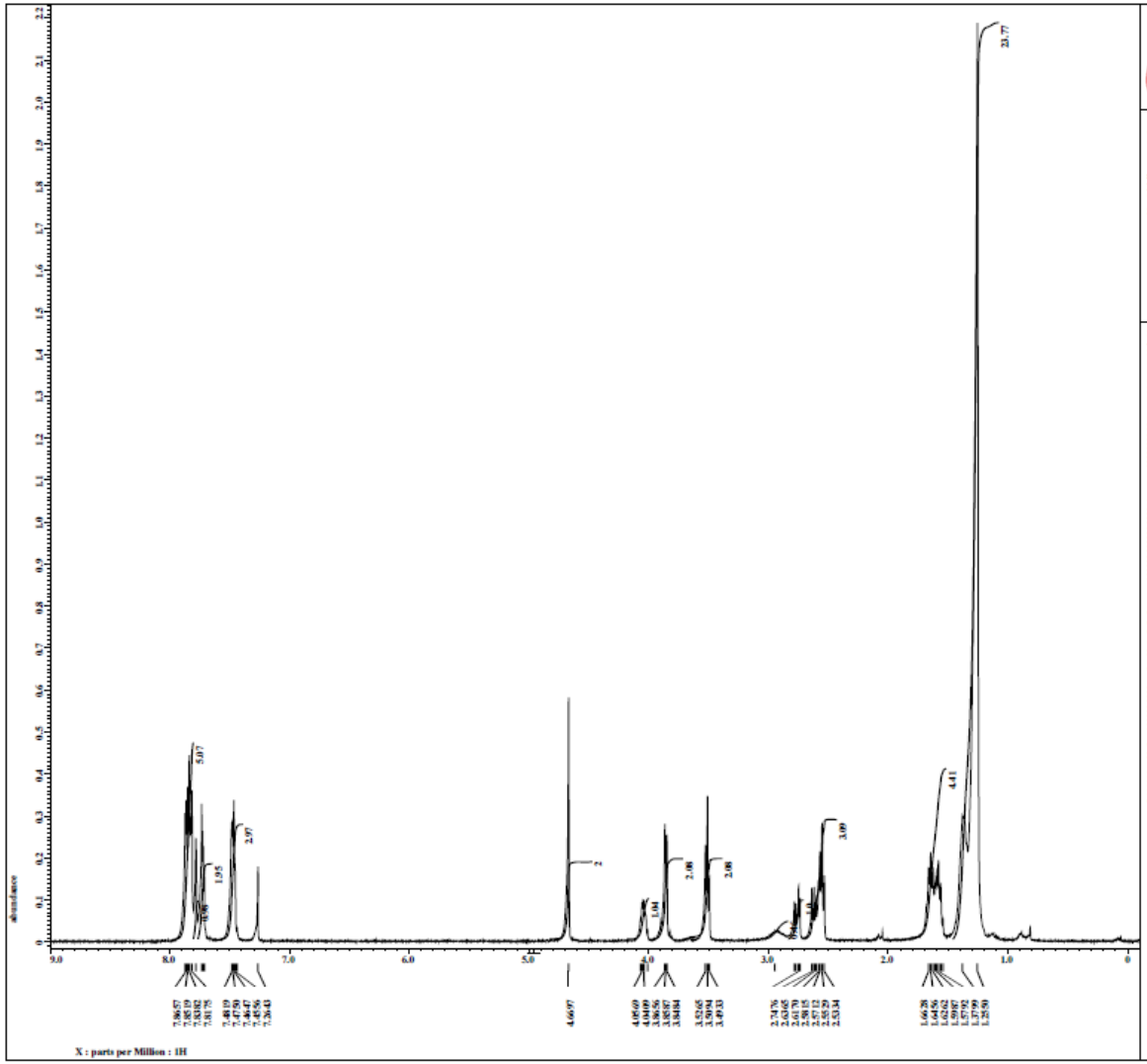
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 25
Total_scans       = 25

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22[dC]
```



(19f)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid : 0[%] : 60[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

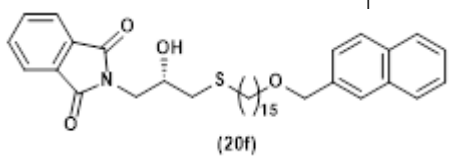
Derived from: 180723_phth_OH_C15_Nap-1.j
```

```
Filename          = 180723_phth_OH_C15_Na
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 23-JUL-2018 14:09:52
Revision_time     = 23-JUL-2018 14:23:15
Current_time      = 23-JUL-2018 14:23:49

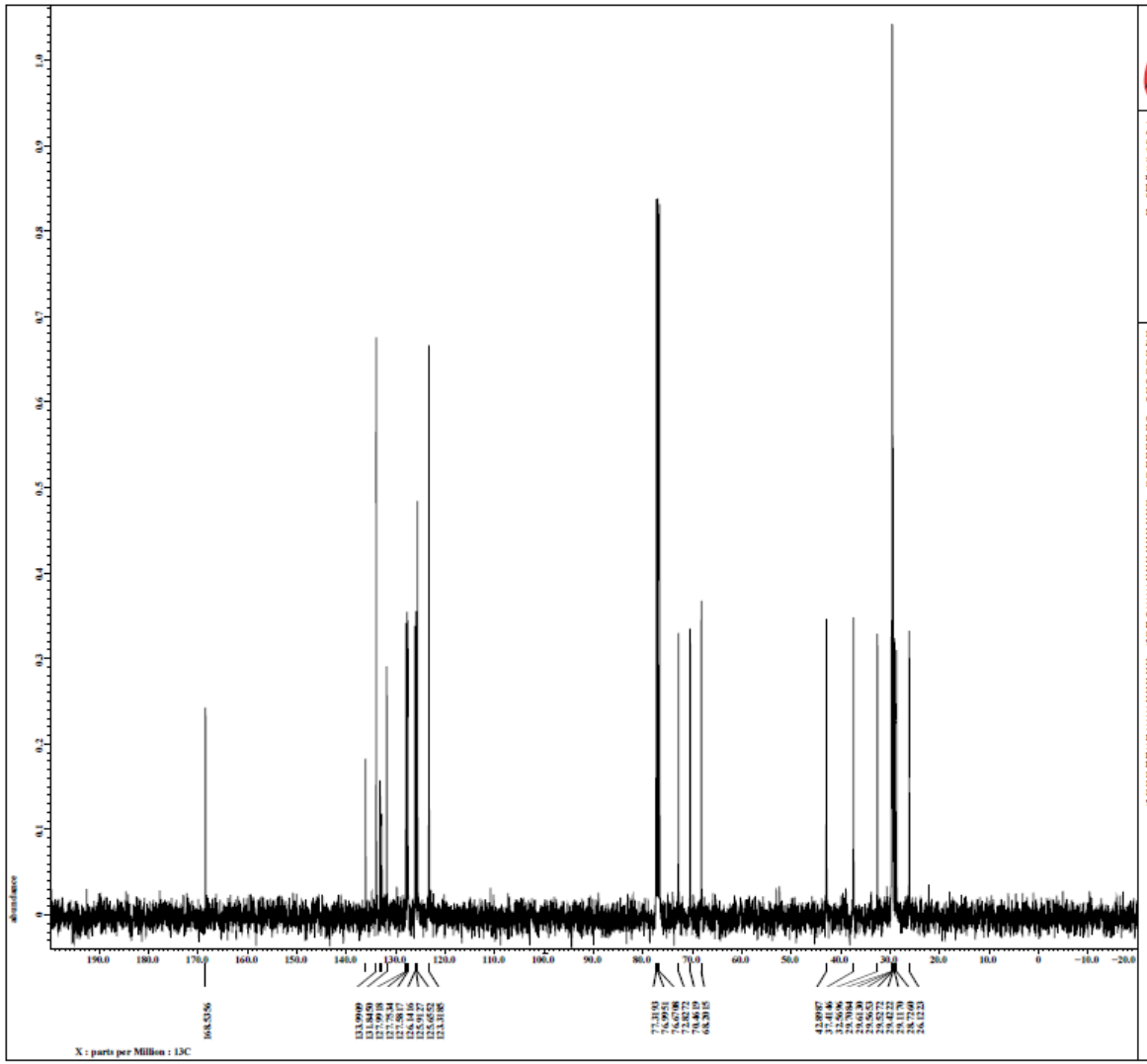
Comment           = single pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = 1
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35234118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5
Total_scans       = 5

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Decim_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 24.2[dC]
```



(20f)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

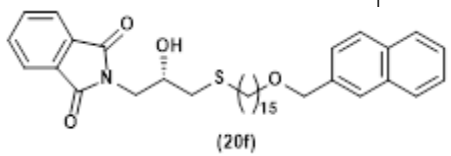
Derived from: 180723_13C_phth_OH_C15_Nap
```

```
Filename            = 180723_13C_phth_OH_C1
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = 1
Solvent             = CHLOROFORM-D
Creation_time       = 23-JUL-2018 14:15:11
Revision_time       = 23-JUL-2018 14:27:53
Current_time        = 23-JUL-2018 14:28:25

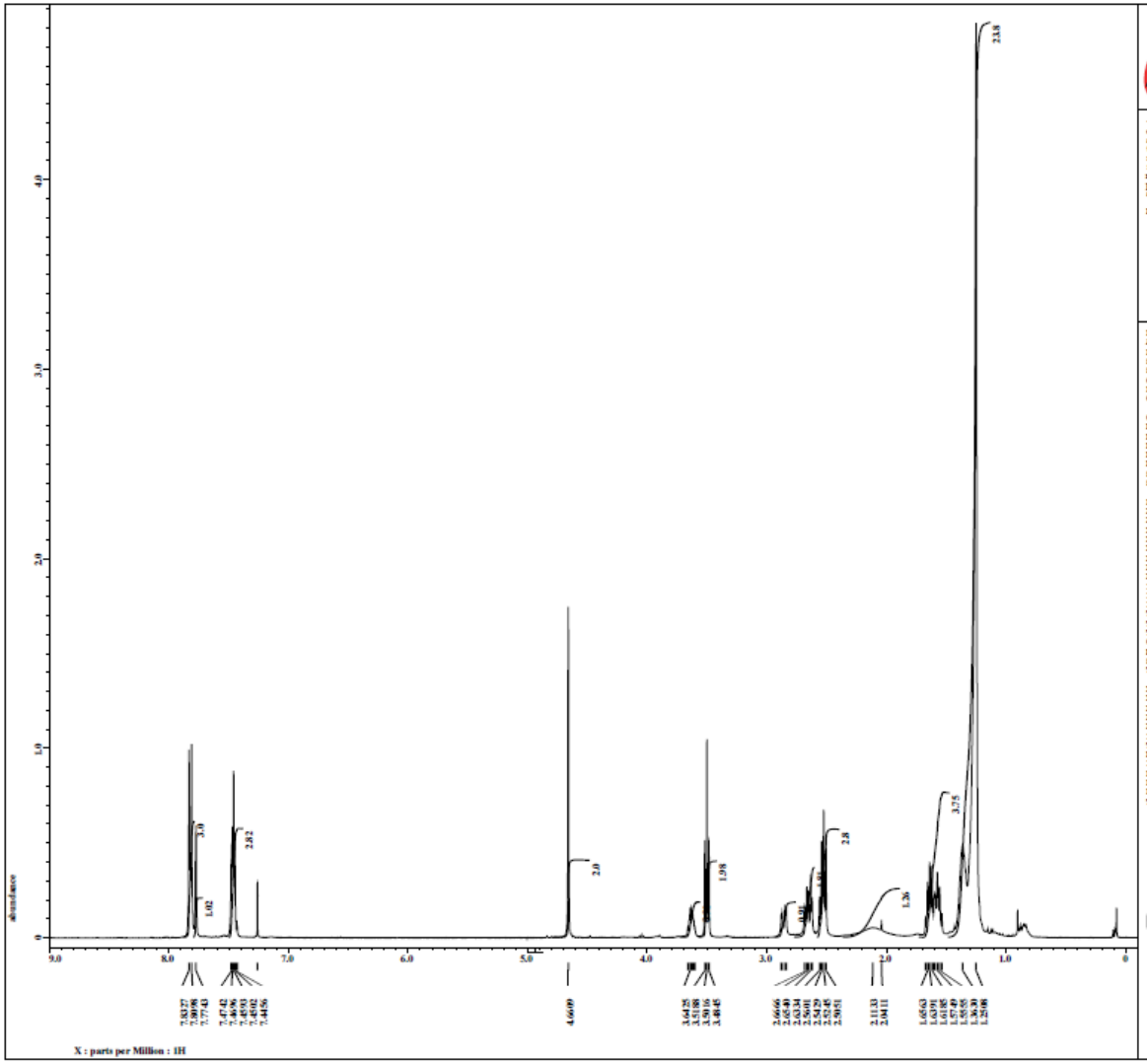
Comment             = single_pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCX 400P
Spectrometer        = DELTA2_NMR

Field_strength      = 9.20197068[T] (390[MH
X_acq_duration      = 1.06430464[s]
X_domain            = 13C
X_freq              = 98.51479726[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.92958061[Hz]
X_sweep             = 30.78817724[kHz]
Irr_domain          = 1H
Irr_freq            = 391.78655441[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 32
Total_scans         = 32

X_90_width          = 9.25[us]
X_acq_time          = 1.06430464[s]
X_angle             = 30[deg]
X_atn               = 4[dB]
X_pulse             = 3.08333333[us]
Irr_atn_dec         = 17.8[dB]
Irr_atn_noe         = 17.8[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 60
Relaxation_delay    = 2[s]
Repetition_time     = 3.06430464[s]
Temp_get            = 23.9[dC]
```



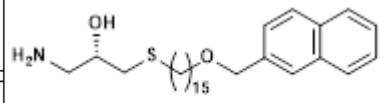
(20f)



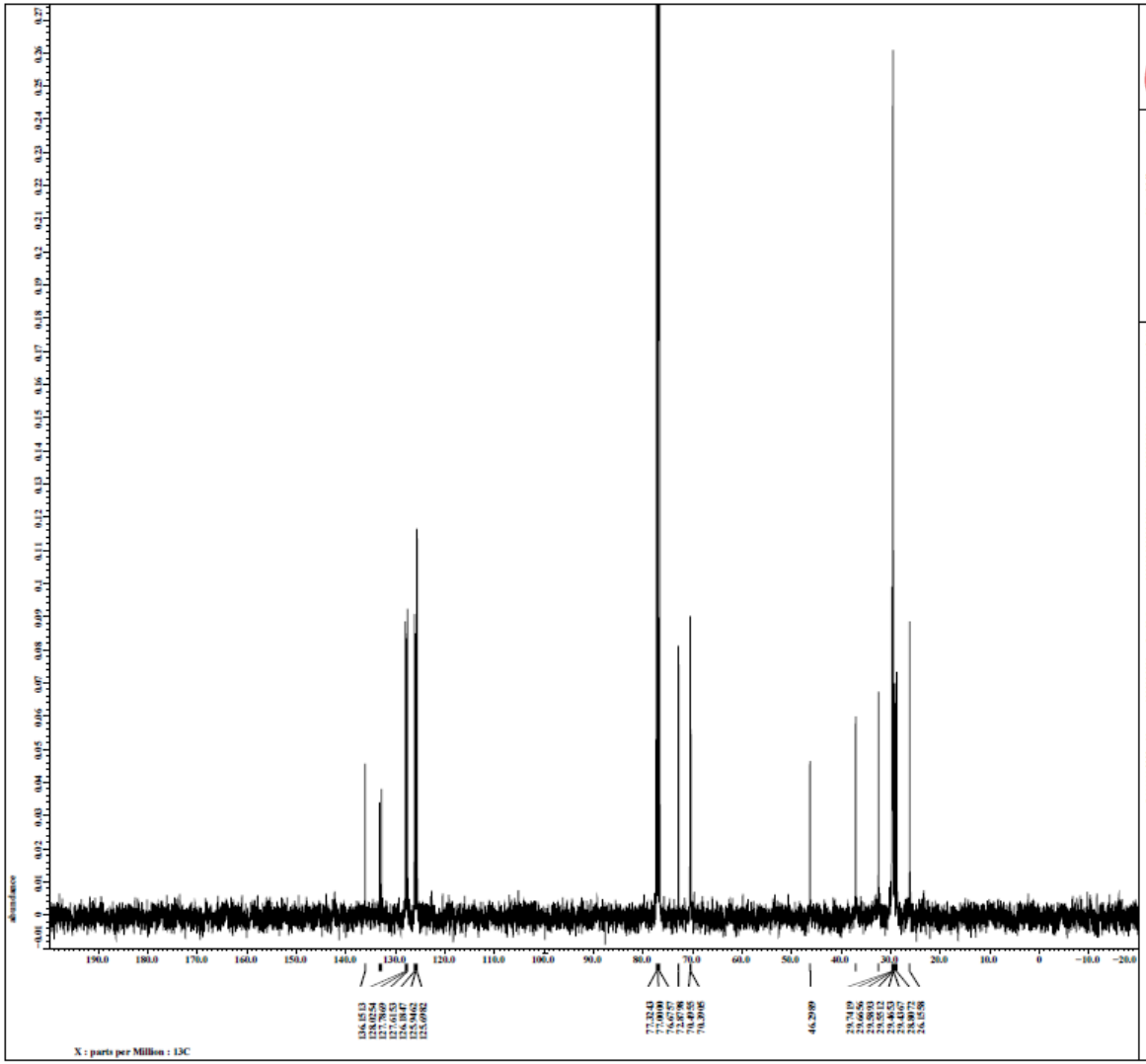
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180724_HZN_OK_C15_Nap-1.jd

Filename	= 180724_HZN_OK_C15_Nap
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-JUL-2018 12:24:11
Revision_time	= 24-JUL-2018 12:57:36
Current_time	= 24-JUL-2018 12:58:09
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 12107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= 1
Site	= NCH_400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44870791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Delta_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 34
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 22.9[dC]



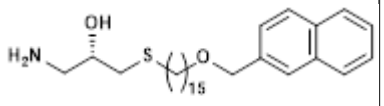
(21f)



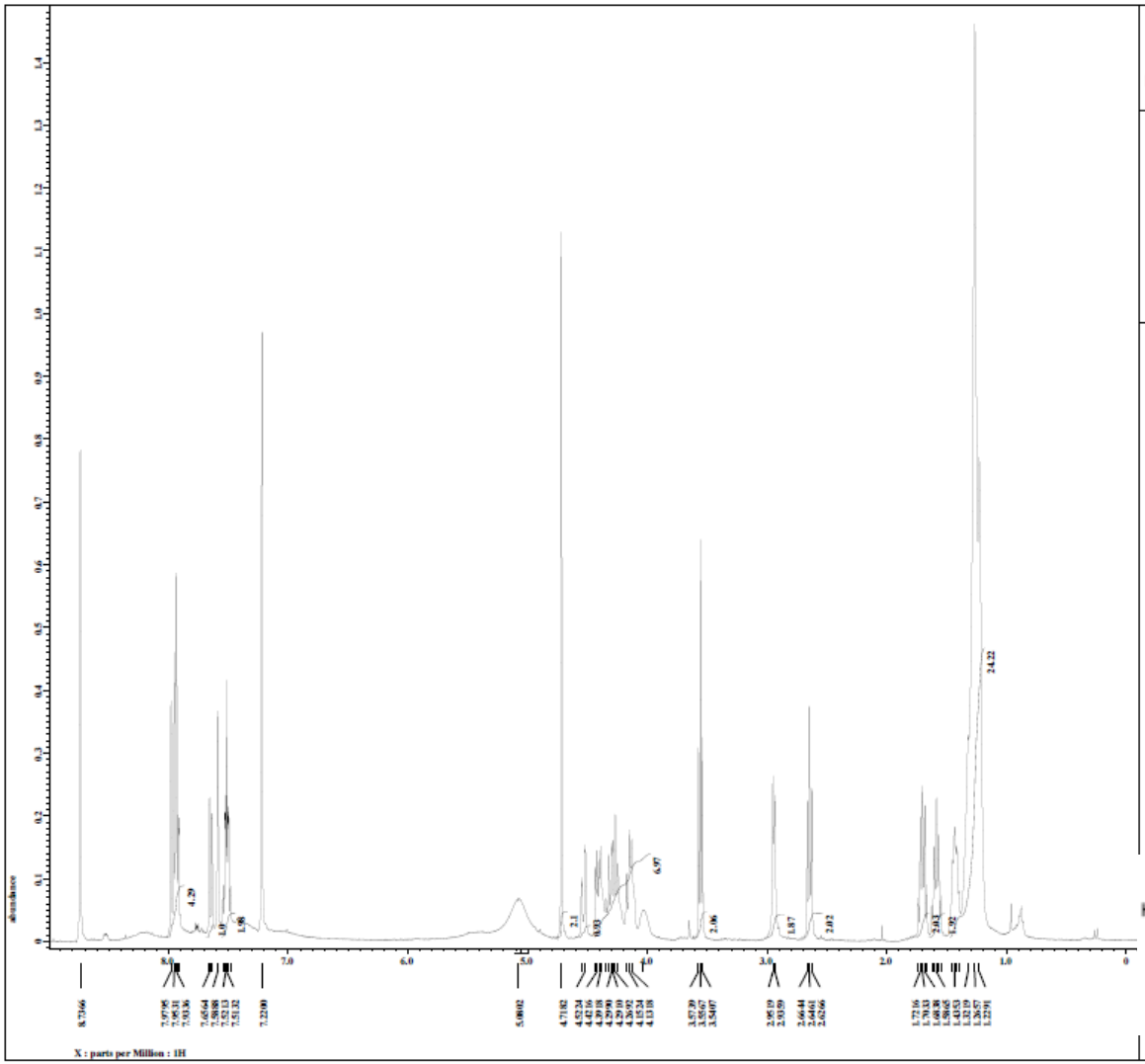
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 180724_13C_H2N_OH_C15_Nap-

Filename	= 180724_13C_H2N_OH_C15
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-JUL-2018 12:30:20
Revision_time	= 24-JUL-2018 12:51:36
Current_time	= 24-JUL-2018 12:52:12
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06420464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.92358061[Hz]
X_sweep	= 30.78817724[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 95
Total_scans	= 95
X_90_width	= 9.25[us]
X_acq_time	= 1.06420464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 3.08333333[us]
Irr_atn_dec	= 17.0[dB]
Irr_atn_noe	= 17.0[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06420464[s]
Temp_get	= 22.2[dC]



(21f)



JEOL

```
---- 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
auto_reference : 5[%] : TRUE

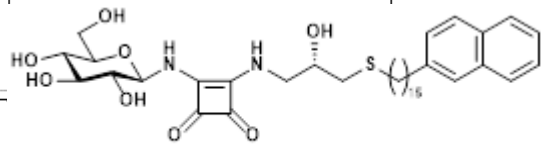
Derived from: 180731_GlcN_DNS_C15_Nap-1.
```

```
Filename          = 180731_GlcN_DNS_C15_N
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 31-JUL-2018 11:23:00
Revision_time     =  4-JUN-2019 16:19:32
Current_time      =  4-JUN-2019 16:20:12

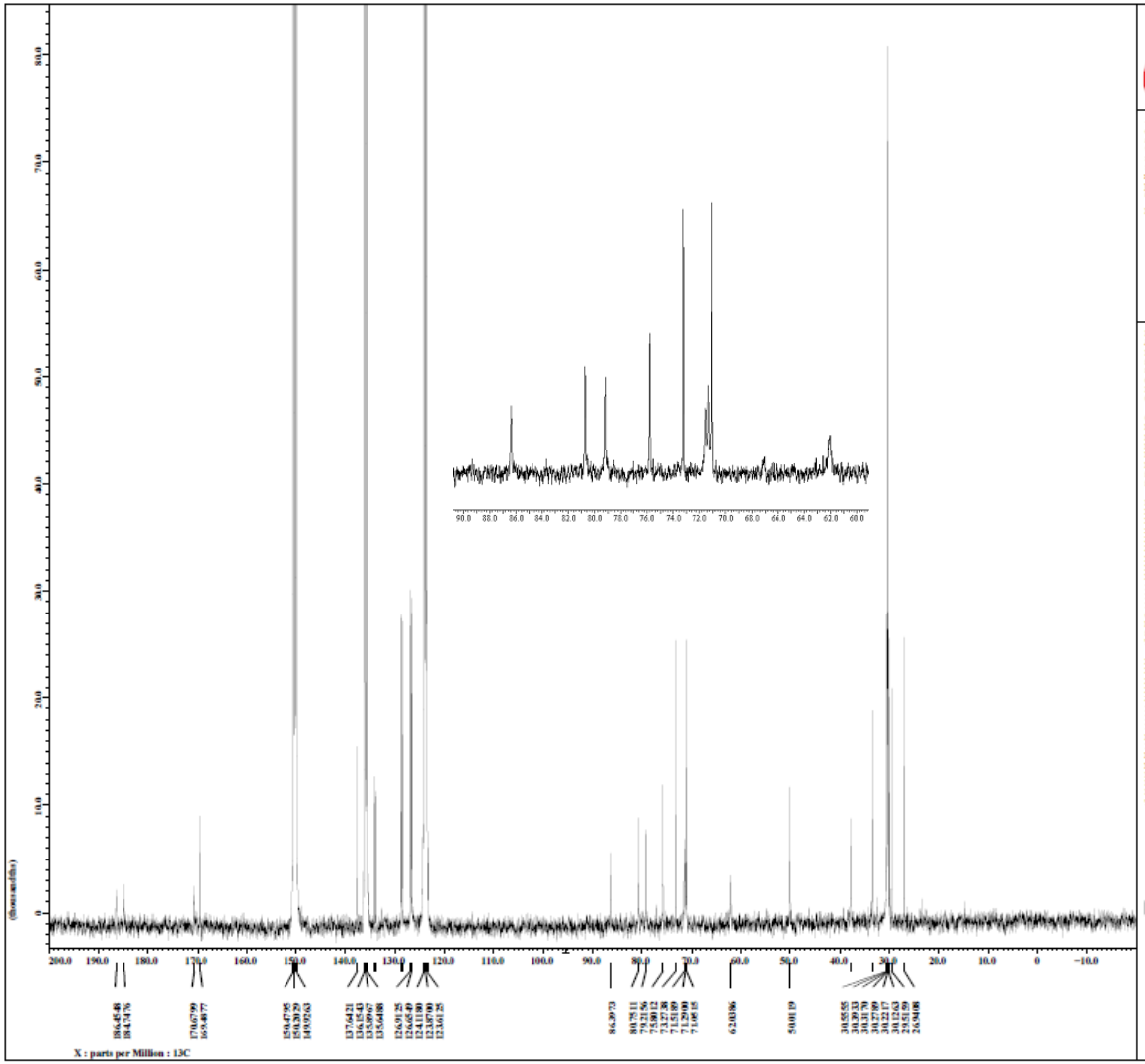
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.6[dC]
```



(11f)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

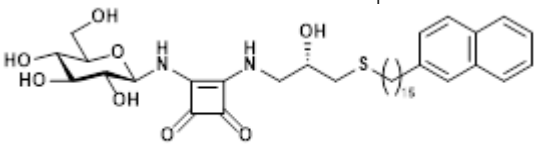
Derived from: 180731_13C_GlcN_DHS_C15_Na
```

```
Filename          = 180731_13C_GlcN_DHS_C
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 31-JUL-2018 12:29:06
Revision_time     =  4-JUN-2019 16:15:32
Current_time      =  4-JUN-2019 16:16:12

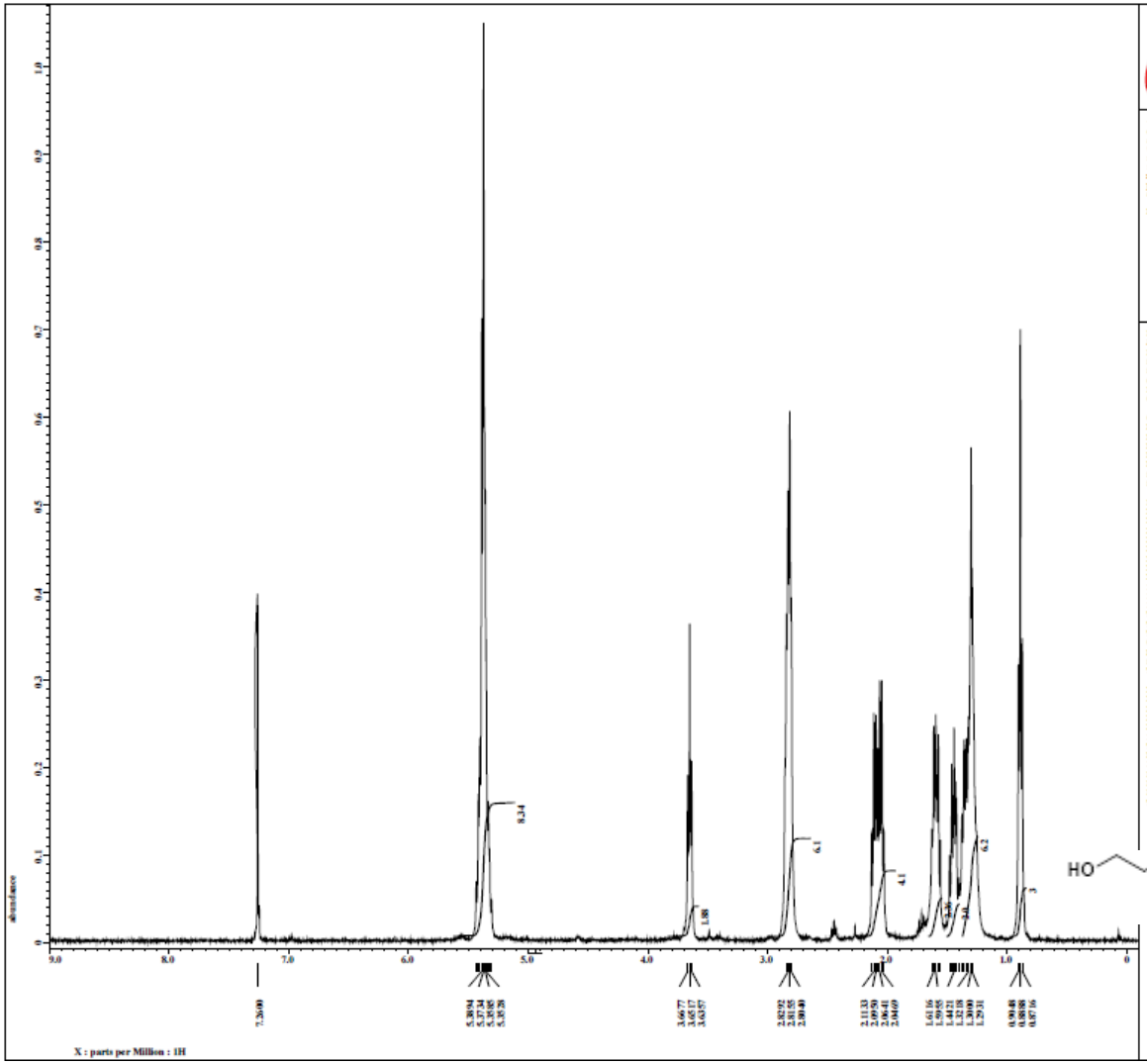
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1280
Total_scans       = 1280

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.0[dB]
Irr_atn_noe       = 17.0[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 46
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22[dC]
```



(11f)




```
---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 181109_cr_LAH_arachidonic_
```

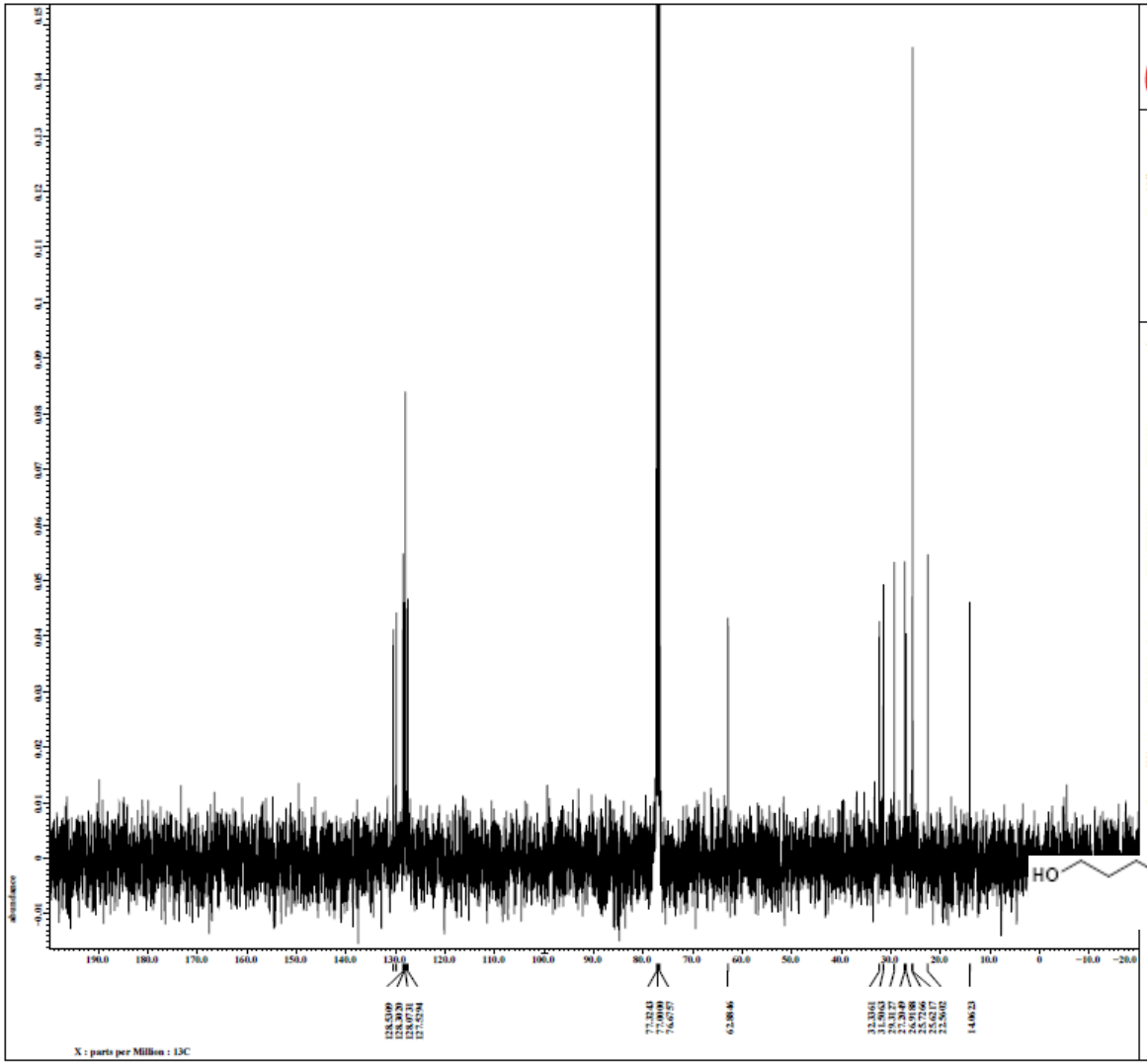
```
Filename          = 181109_cr_LAH_arachid
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     =  9-NOV-2018 15:27:39
Revision_time     = 22-APR-2019 17:27:55
Current_time      = 22-APR-2019 17:36:36

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.8[dC]
```

(16)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 181109_13C_cr_LAE_arachido
```

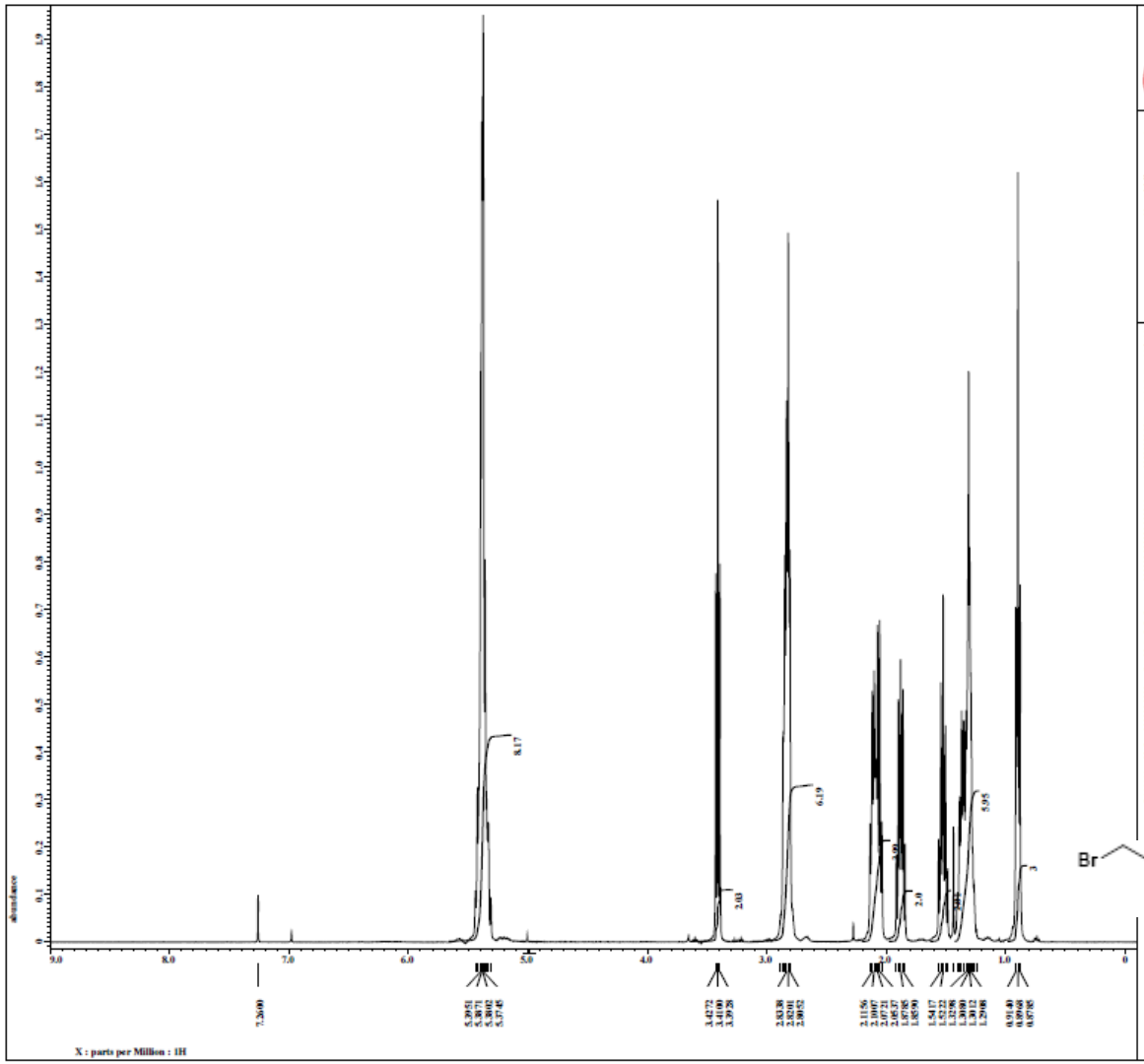
```
Filename            = 181109_13C_cr_LAE_ara
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = 1
Solvent             = CHLOROFORM-D
Creation_time       =  9-NOV-2018 15:22:40
Revision_time       =  9-NOV-2018 16:00:14
Current_time        =  9-NOV-2018 16:01:12

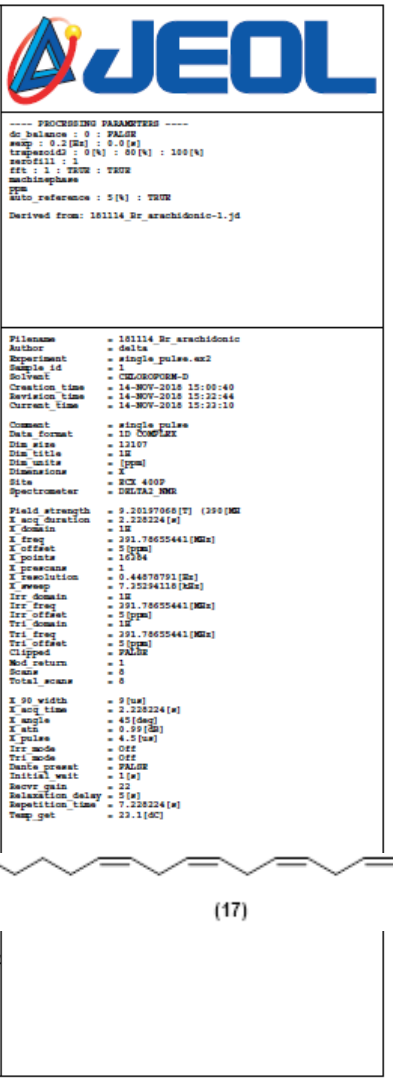
Comment             = single_pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCX 400P
Spectrometer        = DELTA2_NMR

Field_strength      = 9.20197068[T]  (390[MH
X_acq_duration      = 1.06430464[s]
X_domain            = 13C
X_freq              = 98.51479726[MHz]
X_offset            = 100[ppm]
X_points            = 27763
X_prescans          = 4
X_resolution        = 0.93958061[Hz]
X_sweep             = 29.75817734[kHz]
Irr_domain          = 1H
Irr_freq            = 391.78655441[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 79
Total_scans         = 79

X_90_width          = 9.25[us]
X_acq_time          = 1.06430464[s]
X_angle             = 30[deg]
X_atn               = 4[dB]
X_pulse             = 2.05333333[us]
Irr_atn_dec         = 17.5[dB]
Irr_atn_noe         = 17.5[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     = 3.06430464[s]
Temp_get            = 22.1[dC]
```

(16)





```
---- 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
auto_reference : 5[%] : TRUE

Derived from: 181114_Br_arachidonic-1.jd
```

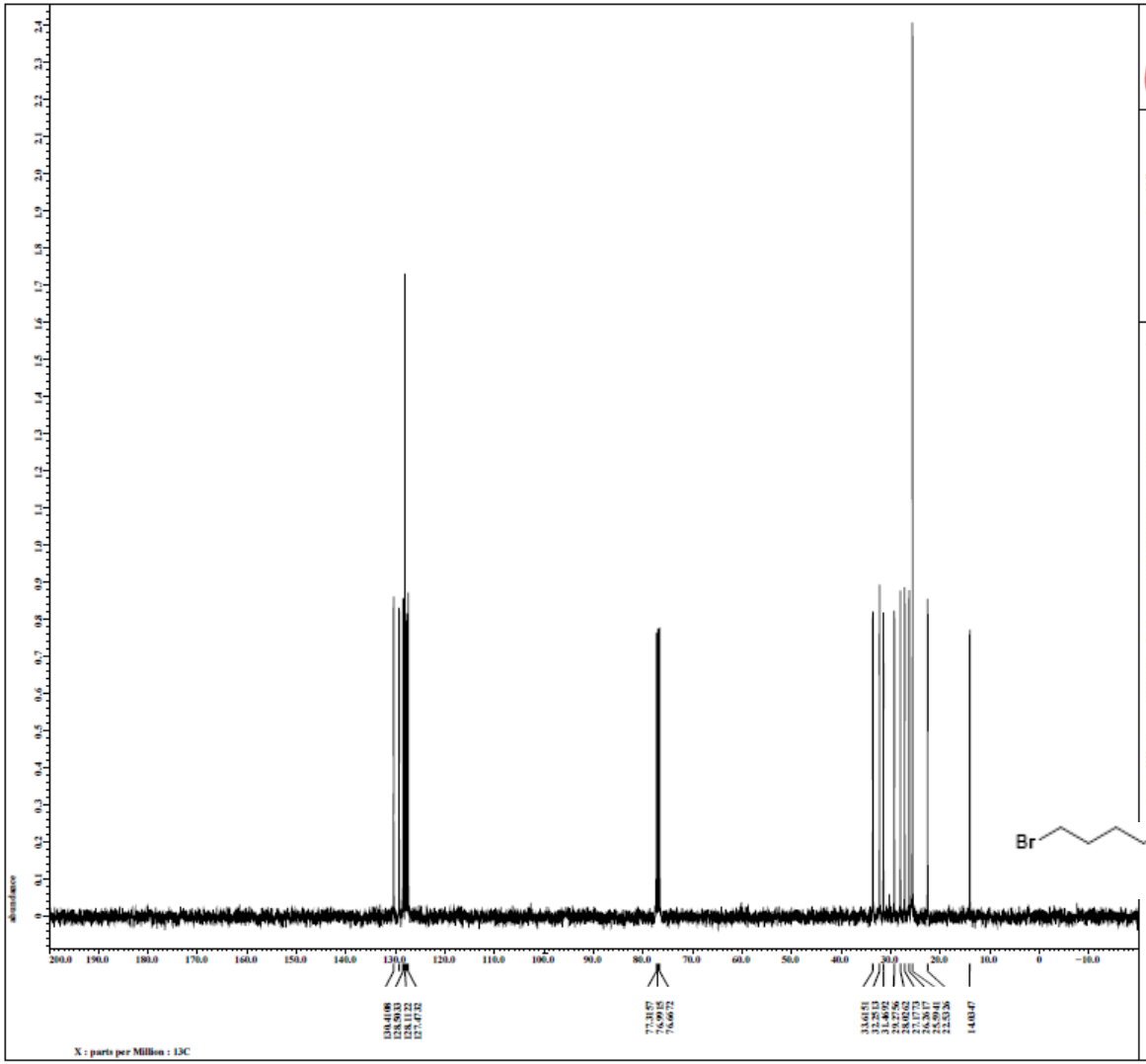
```
Filename          = 181114_Br_arachidonic
Author            = delta
Experiment        = single_pulse.exp2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 14-NOV-2018 15:00:40
Revision_time     = 14-NOV-2018 15:22:44
Current_time      = 14-NOV-2018 15:23:10

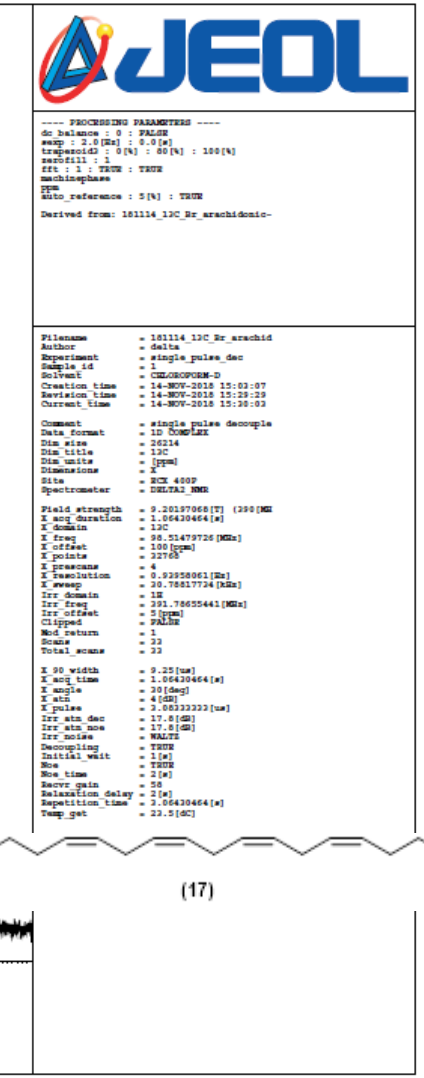
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 23.1[dC]
```


(17)





```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zero fill : 1
fft : 1 : TRUE : TRUE
machinephase
PPM
auto_reference : 5[%] : TRUE

Derived from: 161114_13C_Br_arachidonic-
```

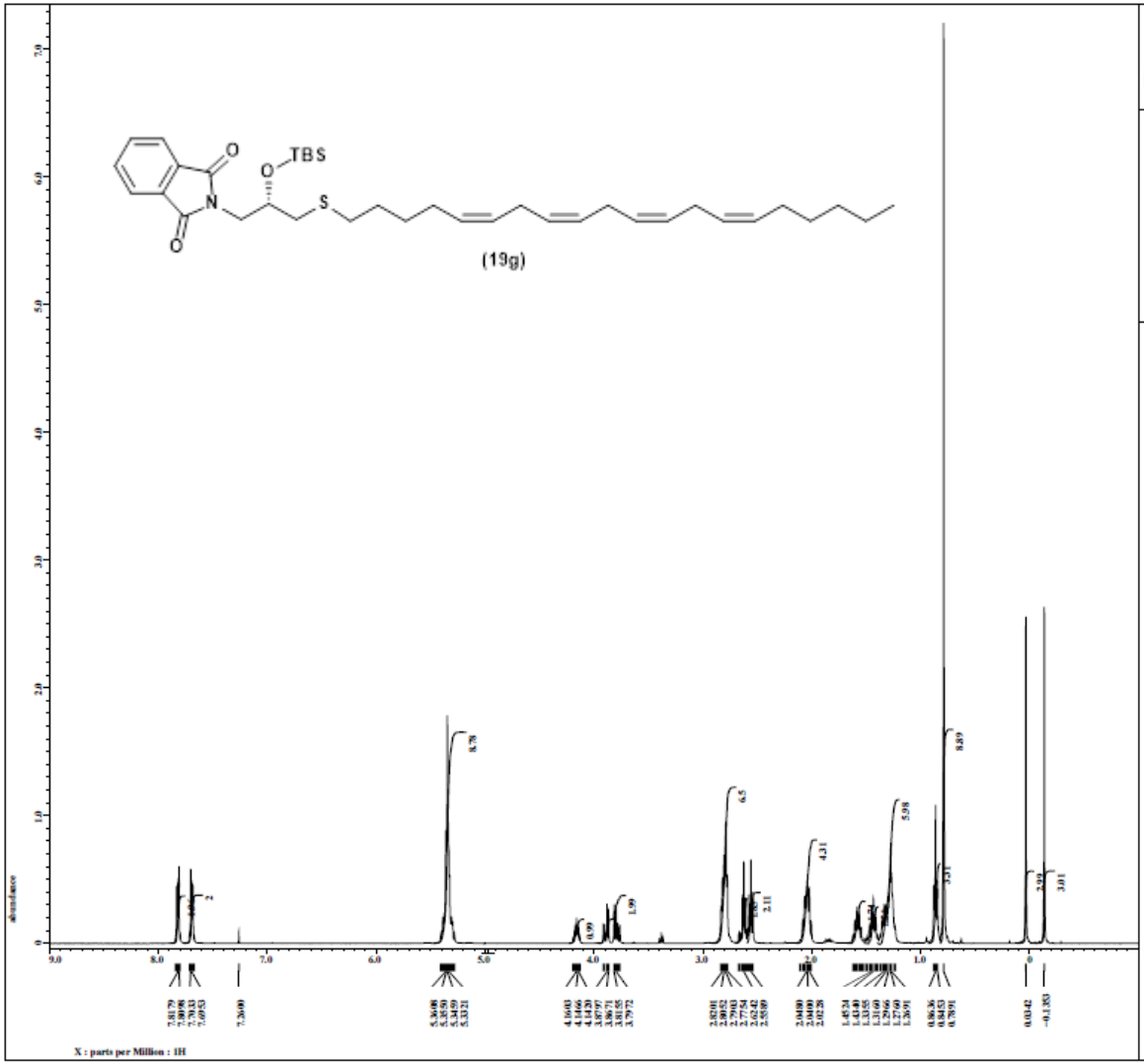
```
Filename            = 161114_13C_Br_arachid
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = 1
Solvent             = CHLOROFORM-D
Creation_time       = 14-NOV-2016 15:03:07
Revision_time       = 14-NOV-2016 15:29:29
Current_time        = 14-NOV-2016 15:30:03

Comment             = single pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCX 400P
Spectrometer        = DELTA2_NMR

Field_strength      = 9.20197068[T] (390[MH
X_acq_duration      = 1.06430464[s]
X_domain            = 13C
X_freq              = 98.51479726[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.92958061[Hz]
X_sweep             = 30.78817724[kHz]
Irr_domain          = 1H
Irr_freq            = 391.78655441[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 32
Total_scans         = 33

X_90_width          = 9.25[us]
X_acq_time          = 1.06430464[s]
X_angle             = 30[deg]
X_atn               = 4[dB]
X_pulse             = 3.08333333[us]
Irr_atn_dec         = 17.8[dB]
Irr_atn_noe         = 17.8[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 58
Relaxation_delay    = 2[s]
Repetition_time     = 3.06430464[s]
Temp_get            = 23.5[dC]
```


(17)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
TRUE
auto_reference : 5[%] : TRUE

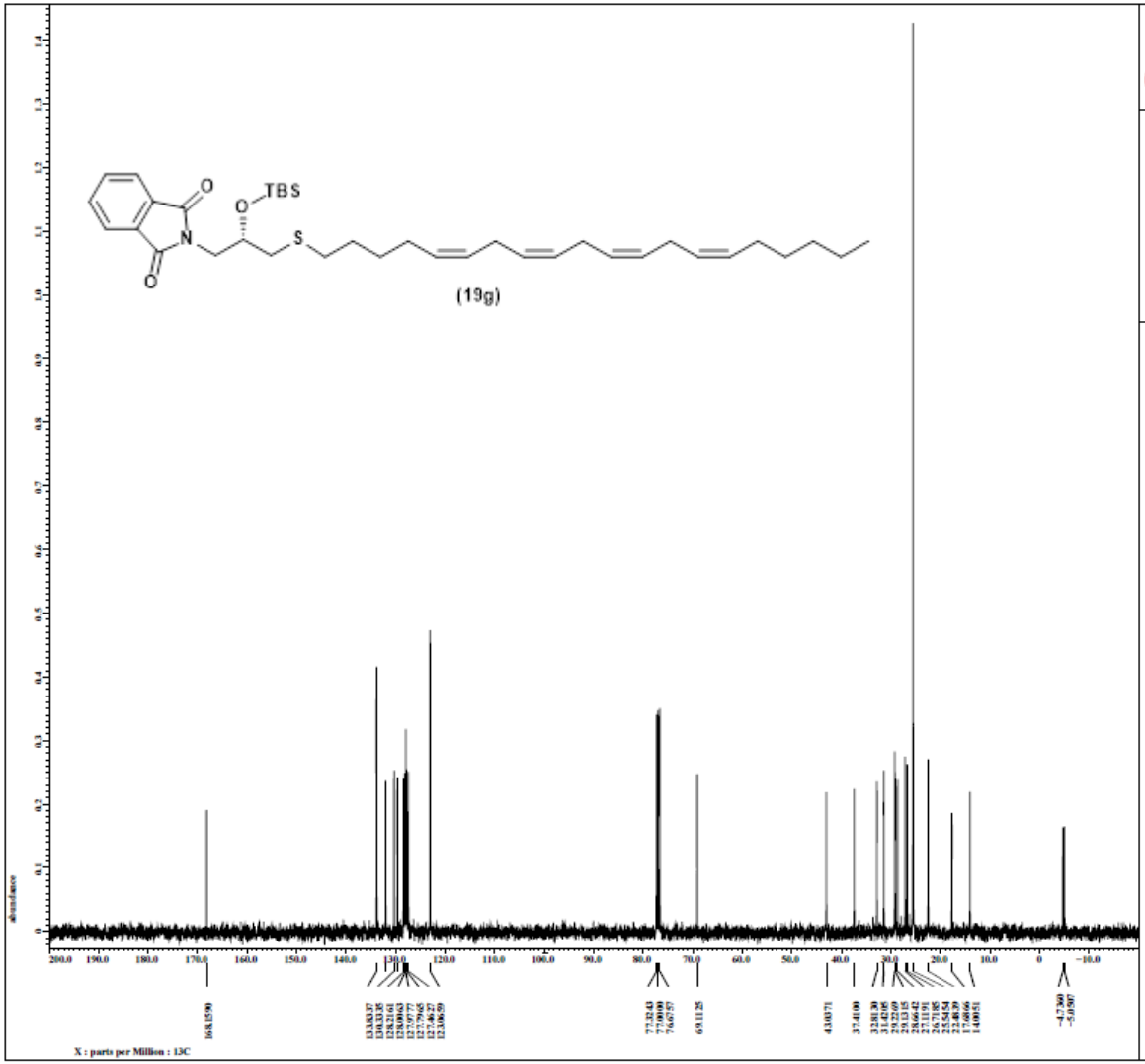
Derived from: 181115_phth_TBS_arachidoni
```

```
Filename          = 181115_phth_TBS_arach
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 15-NOV-2018 15:30:03
Revision_time     = 15-NOV-2018 16:13:20
Current_time      = 15-NOV-2018 16:13:45

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

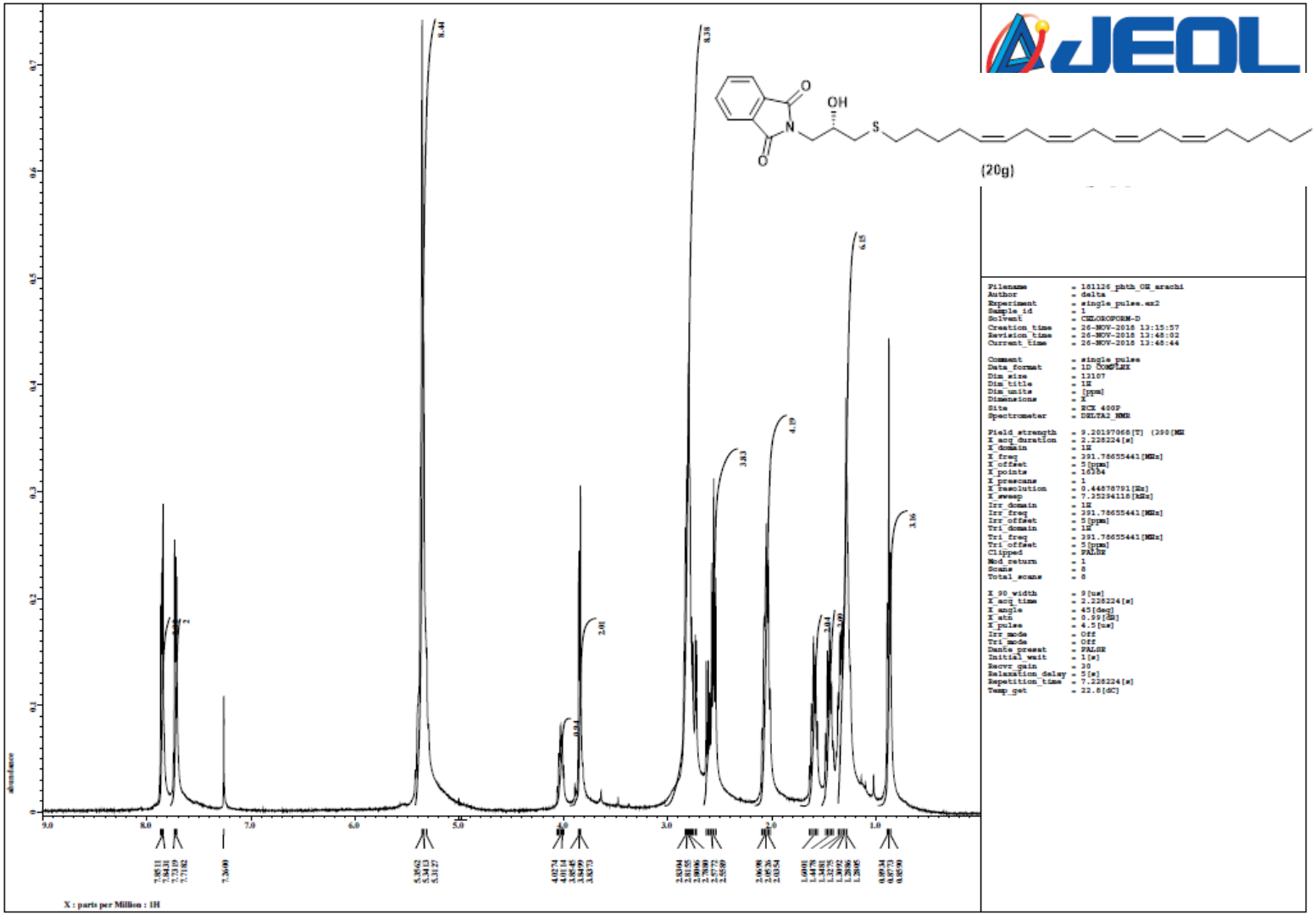
X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 20
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 23.1[dC]
```



---- PROCESSING PARAMETERS ----
dc balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 161115_13C_phth_TBS_arachi

Filename	= 161115_13C_phth_TBS_a
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 15-NOV-2018 15:21:27
Revision_time	= 15-NOV-2018 16:09:14
Current_time	= 15-NOV-2018 16:09:47
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78517734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 17
Total_scans	= 17
X_90_width	= 9.25[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 2.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 22.3[dC]

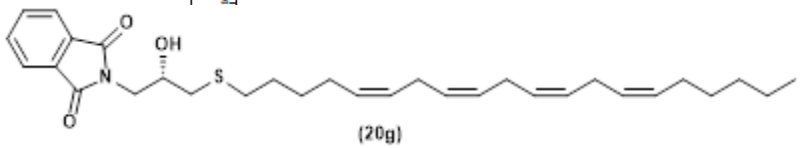



```
Filename          = 161126_phth_OH_arachi
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 26-NOV-2016 13:15:57
Revision_time     = 26-NOV-2016 13:48:02
Current_time      = 26-NOV-2016 13:48:44

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = 1
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T]  (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = PALEH
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.95[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Decim_presat      = PALEH
Initial_wait      = 1[s]
Recvr_gain        = 20
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.5[dC]
```



(20g)

JEOL

```
----- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

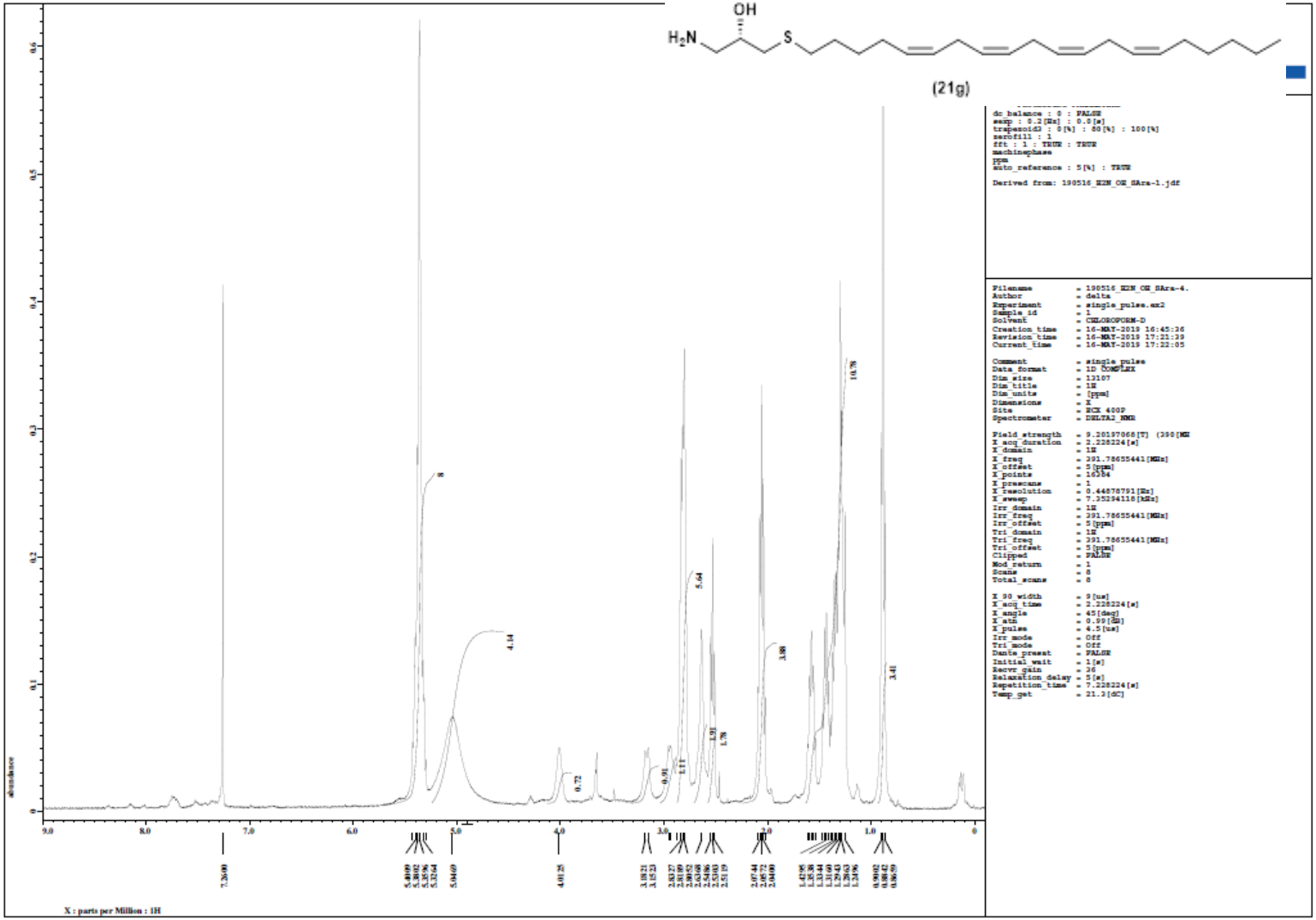
Derived from: 161126_13C_phth_OH_arachid
```

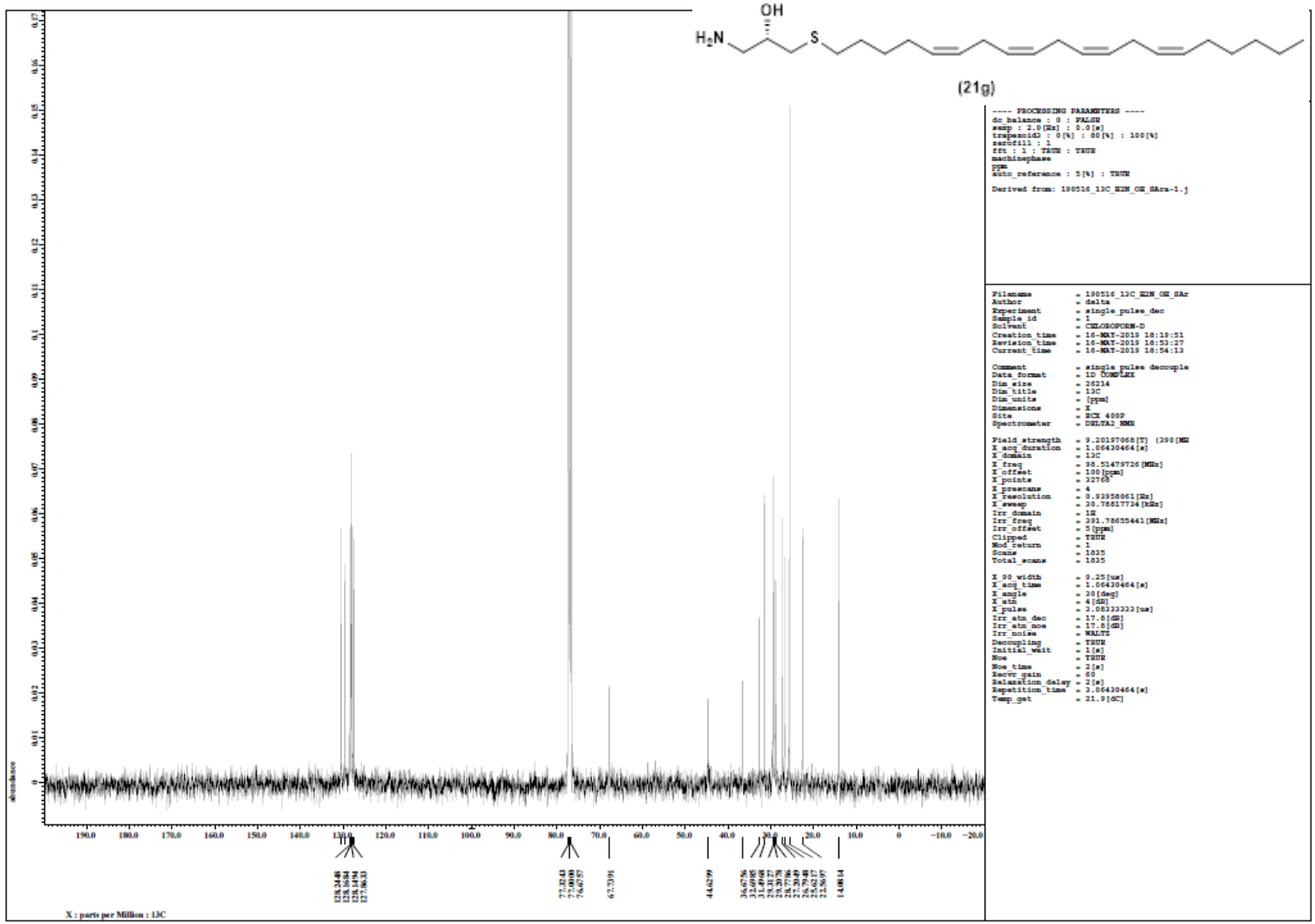
```
Filename          = 161126_13C_phth_OH_ar
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 26-NOV-2018 13:19:31
Revision_time     = 26-NOV-2018 13:50:35
Current_time      = 26-NOV-2018 13:51:20

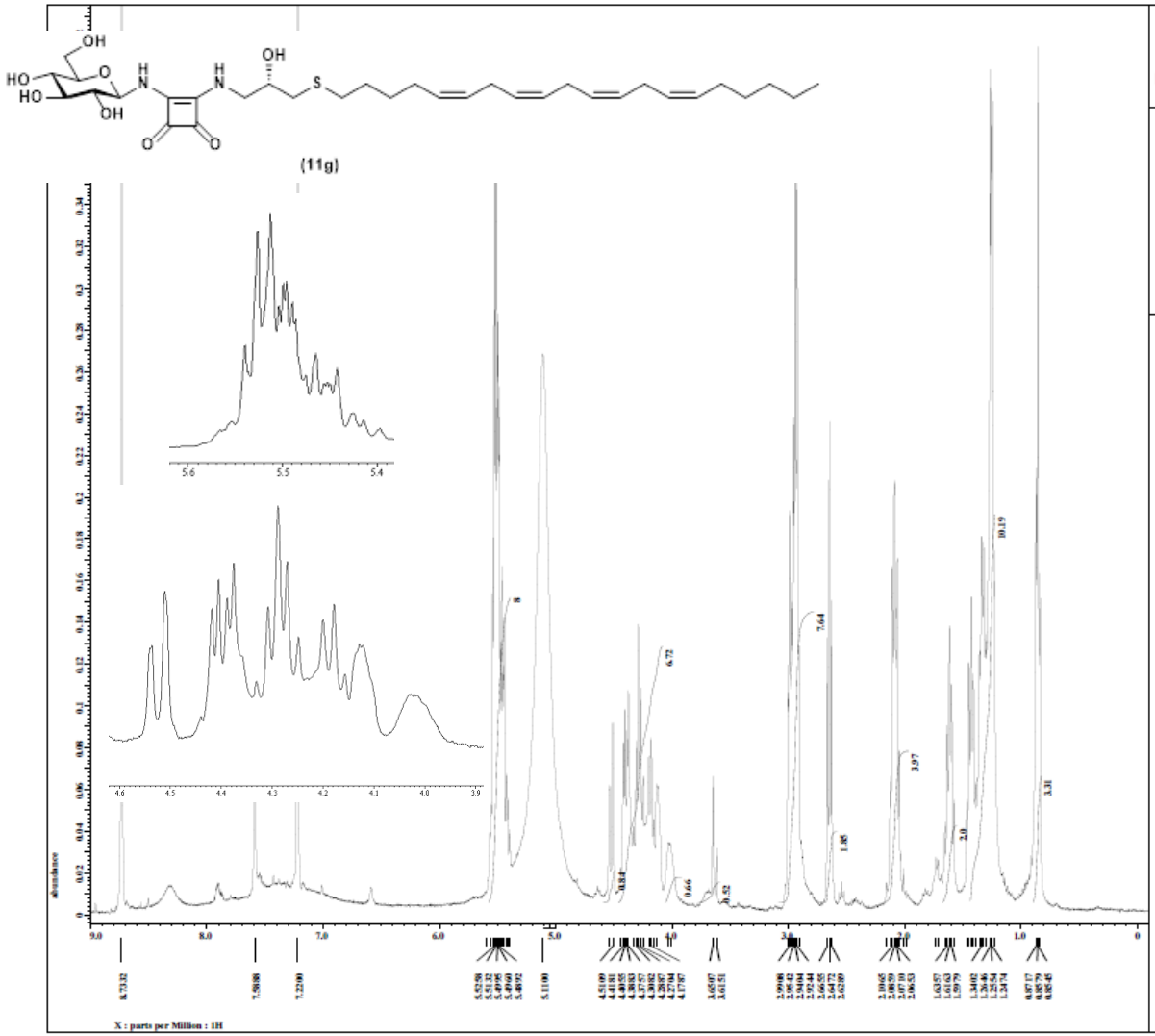
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32765
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 29.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 59
Total_scans       = 59

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06420464[s]
Temp_get          = 23.1[dC]
```


OH
H2N
S
(21g)
dc_balance : 0 : FALSE
expp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
Derived from: 190516_HZN_OH_SAra-1.jdf
Filename = 190516_HZN_OH_SAra-4.
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 16-MAY-2019 16:45:36
Revision_time = 16-MAY-2019 17:21:39
Current_time = 16-MAY-2019 17:22:05
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 131107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35234118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 26
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 21.2[dC]
8
4.34
0.72
0.91
1.11
1.91
1.78
5.64
3.88
10.78
3.41
X : parts per Million : 1H


OH
H2N
S
(21g)
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
Derived from: 190516_13C_H2N_OH_SAra-1.j
Filename = 190516_13C_H2N_OH_SAr
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 16-MAY-2019 18:19:51
Revision_time = 16-MAY-2019 18:53:27
Current_time = 16-MAY-2019 18:54:13
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 16214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = NCX 400F
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.92258061[Hz]
X_sweep = 30.75817724[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 1825
Total_scans = 1825
X_90_width = 9.25[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4[dB]
X_pulse = 3.08333333[us]
Irr_atn_dec = 17.8[dB]
Irr_atn_noe = 17.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 21.9[dC]
abundance
X : parts per Million : 13C



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

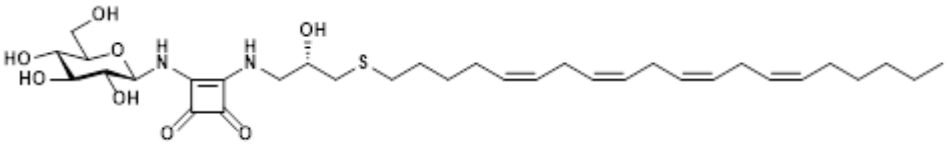
Derived from: 190921_H_GlcNDRS_OH_SAra-1
```

```
Filename          = 190921_H_GlcNDRS_OH_S
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 22-SEP-2019 16:33:31
Revision_time     = 26-SEP-2019 10:42:50
Current_time      = 26-SEP-2019 10:43:29

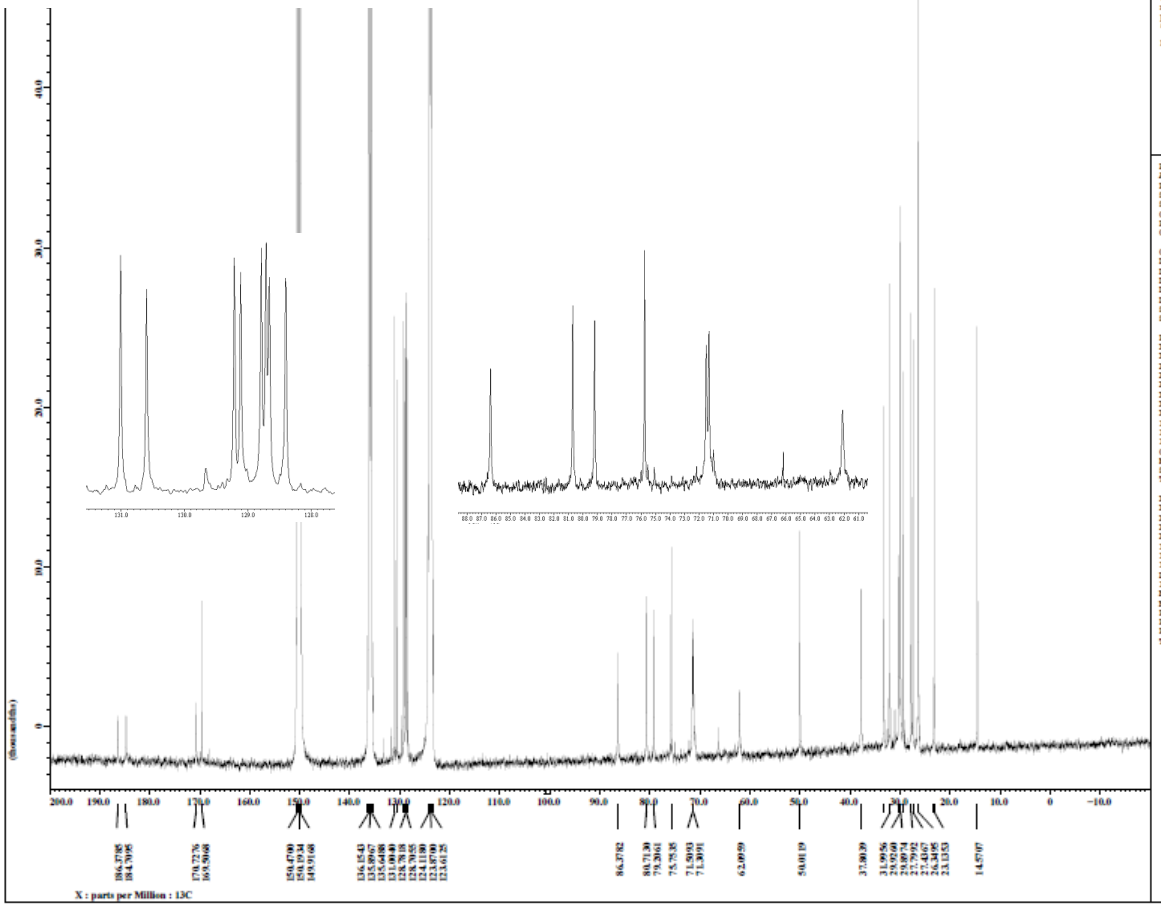
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 24
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.1[dC]
```



(11g)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
expp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
manichasphase
ppm
auto_reference : 5[%] : TRUE

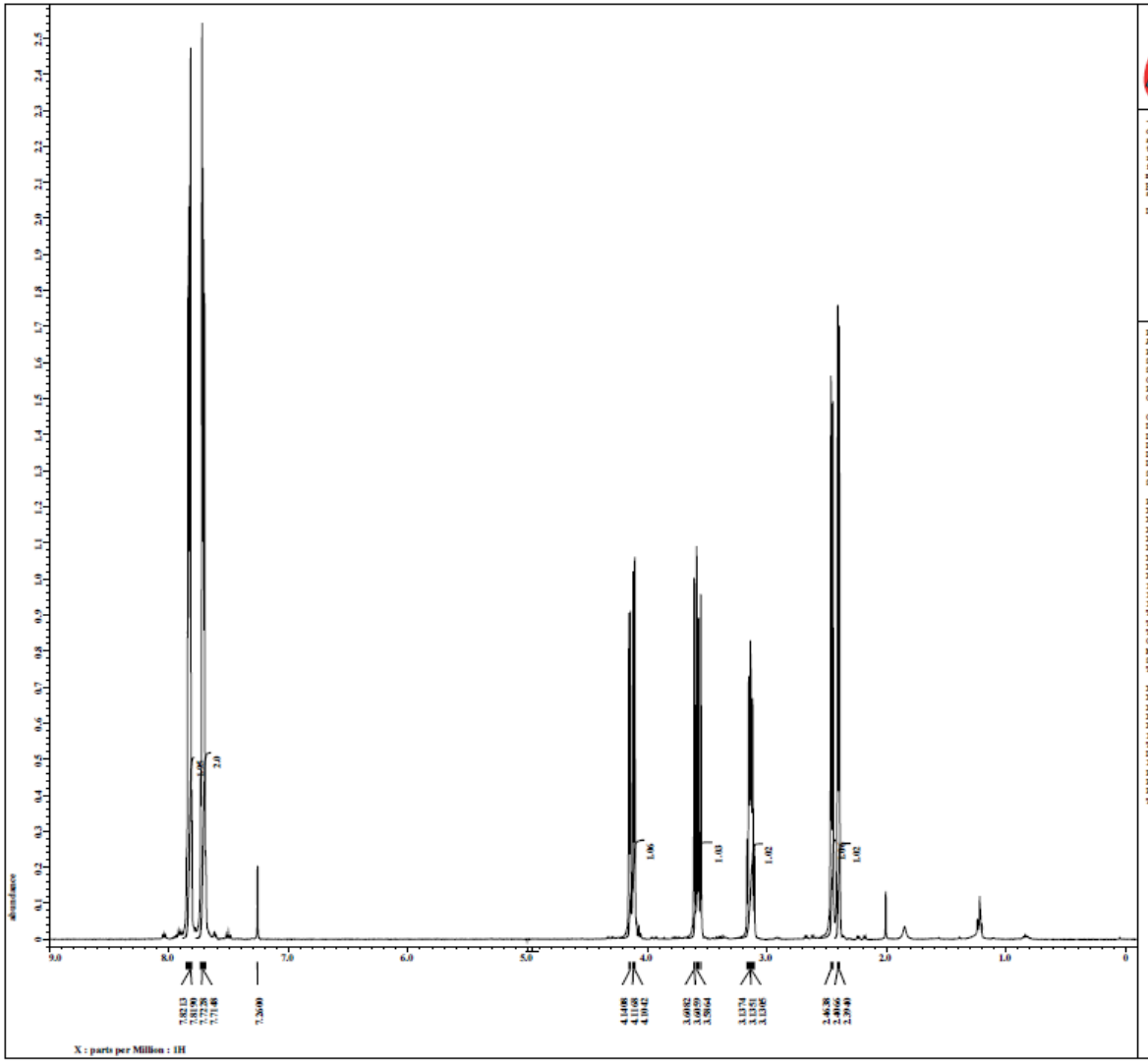
Derived from: 190921_13C-1.jdf
```

```
Filename          = 190921_13C-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = B#C13185
Solvent           = PYRIDINE-D5
Creation_time     = 23-SEP-2019 16:45:20
Revision_time     = 26-SEP-2019 10:31:21
Current_time      = 26-SEP-2019 10:32:55

Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 20.75817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 28400
Total_scans       = 28400

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 2.05223232[us]
Irr_atn_dec       = 17.0[dB]
Irr_atn_noe       = 17.0[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 52
Relaxation_delay  = 2[s]
Repetition_time   = 3.06420464[s]
Temp_get          = 22[dC]
```



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machine-phase
ppm
auto_reference : 5[%] : TRUE

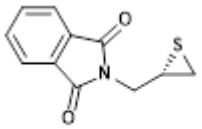
Derived from: 190122_phthN_thiirane-1.jd
```

```
Filename          = 190122_phthN_thiirane
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 22-JAN-2019 11:02:37
Revision_time     = 22-JAN-2019 11:26:54
Current_time      = 22-JAN-2019 11:29:20

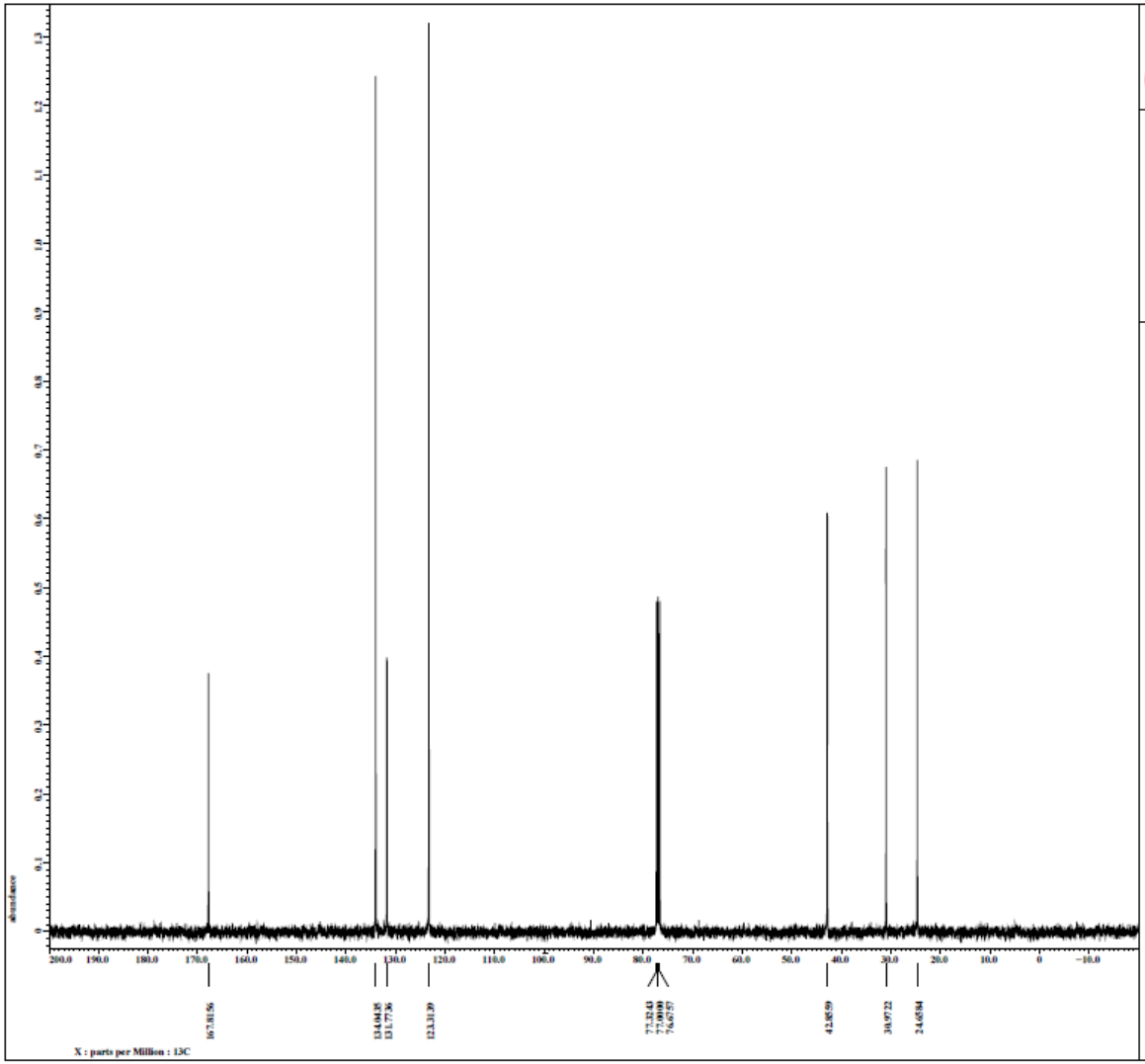
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 20
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.7[dC]
```



(23)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

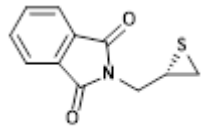
Derived from: 190122_13C_phthN_thiirane-
```

```
Filename          = 190122_13C_phthN_thii
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 22-JAN-2019 11:06:41
Revision_time     = 22-JAN-2019 11:31:15
Current_time      = 22-JAN-2019 11:31:47

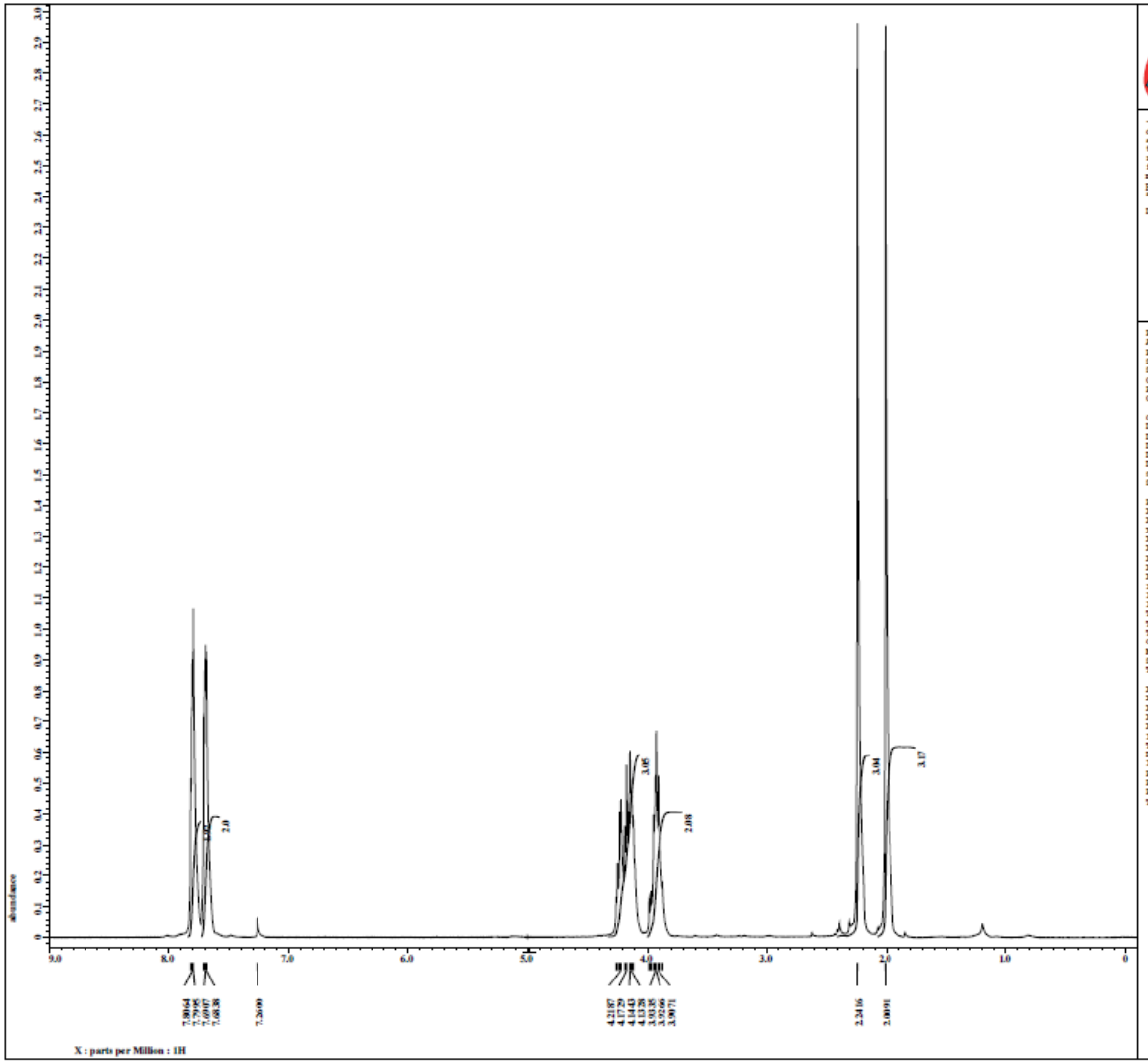
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK_400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.76817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 67
Total_scans       = 67

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.5[dB]
Irr_atn_noe       = 17.5[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   = 3.06430464[s]
Temp_get          = 22.1[dC]
```



(23)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

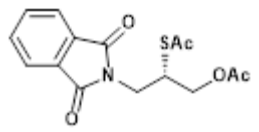
Derived from: 190123_phthN_SAc_OAc-1.jdf
```

```
Filename          = 190123_phthN_SAc_OAc-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 23-JAN-2019 16:14:16
Revision_time     = 23-JAN-2019 16:26:46
Current_time      = 23-JAN-2019 16:29:11

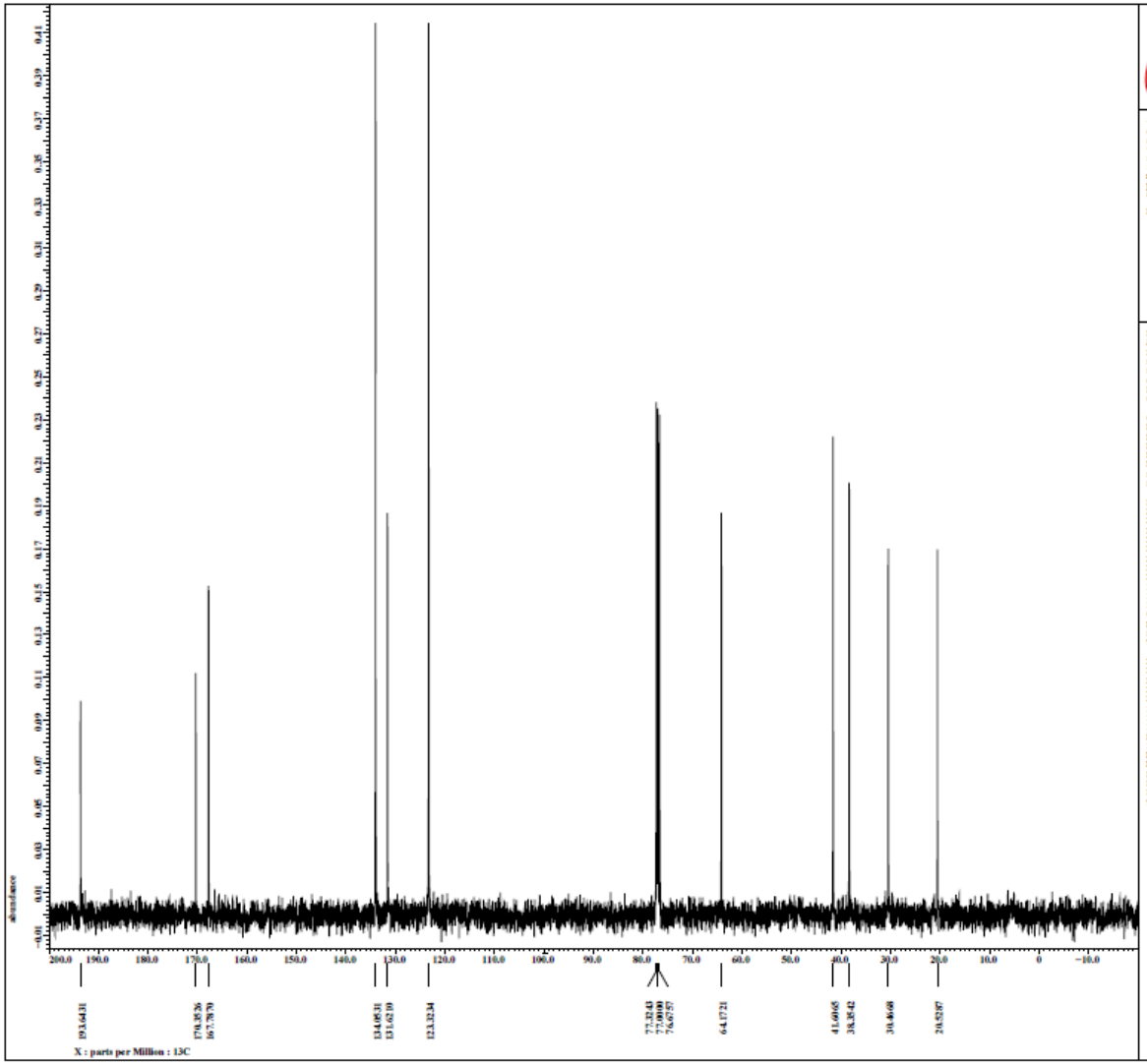
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = NCX 4000
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 7[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35234118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 7
Total_scans       = 7

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 28
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.1[dC]
```



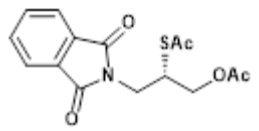
(24)



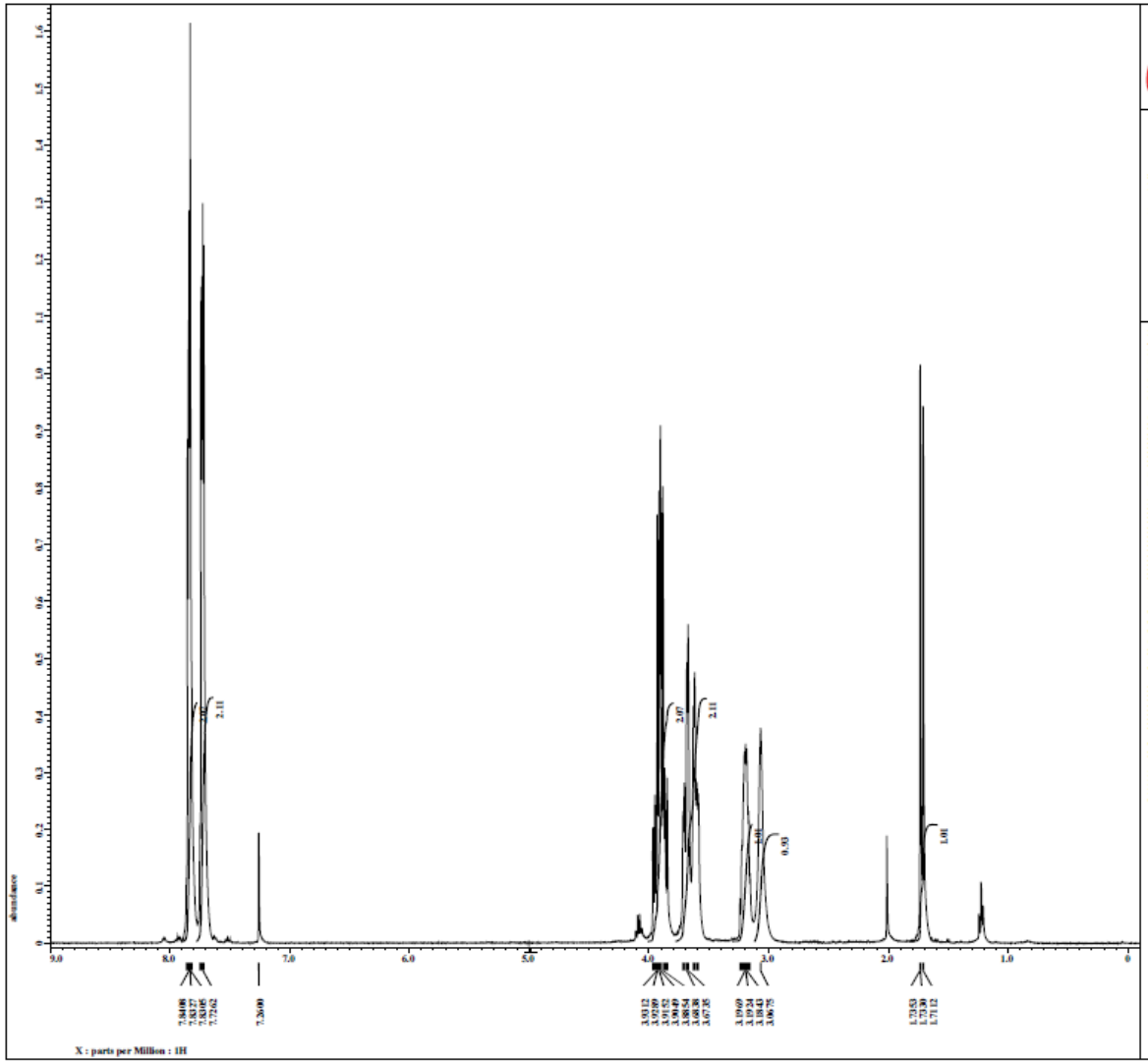
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190123_13C_phthN_SAc_OAc-1

Filename	= 190123_13C_phthN_SAc_
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 23-JAN-2019 16:16:54
Revision_time	= 23-JAN-2019 16:41:16
Current_time	= 23-JAN-2019 16:42:14
Comment	= single_pulse_decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= 1
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78617734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 41
Total_scans	= 41
X_90_width	= 9.25[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 2.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 3[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 22.5[dC]



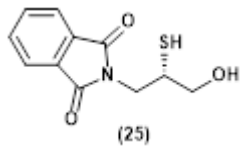
(24)



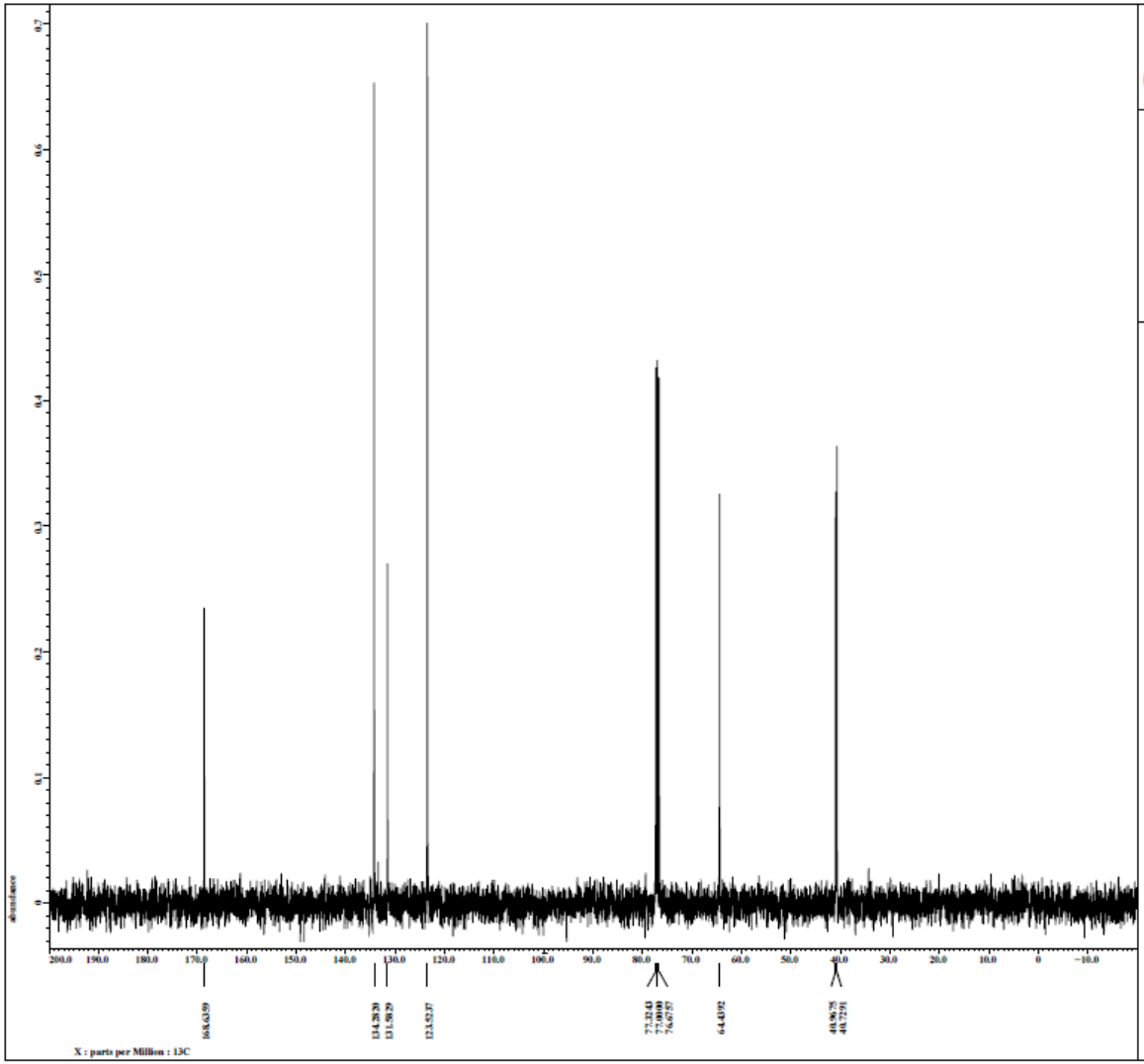
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190124_phthN_SH_OH-1.jdf

Filename	= 190124_phthN_SH_OH-4.
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 24-JAN-2019 11:09:42
Revision_time	= 24-JAN-2019 12:34:26
Current_time	= 24-JAN-2019 12:35:00
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 12107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK_400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44876791[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 22.2[dC]



(25)



JEOL

```
----- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

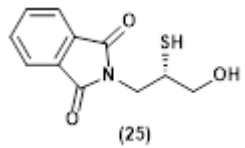
Derived from: 190124_13C_phthN_SH_OH-1.j
```

```
Filename          = 190124_13C_phthN_SH_O
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 24-JAN-2019 11:11:24
Revision_time     = 24-JAN-2019 12:26:22
Current_time      = 24-JAN-2019 12:27:05

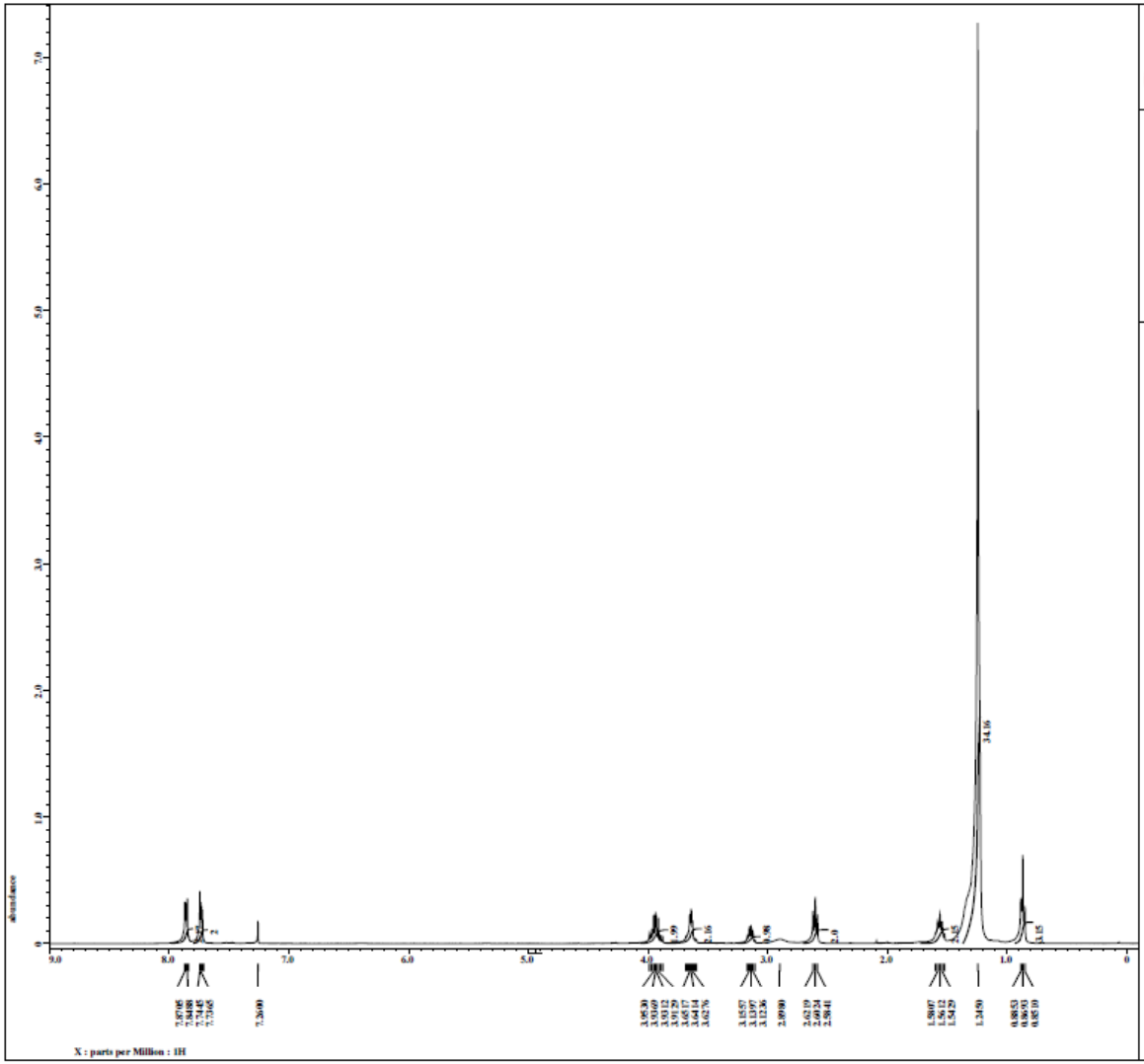
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32765
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 19
Total_scans       = 19

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06420464[s]
Temp_get          = 22.6[dC]
```



(25)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

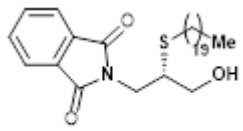
Derived from: 190128_3_phthN_SR_OH-1.jdf
```

```
Filename          = 190128_3_phthN_SR_OH-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 28-JAN-2019 16:59:55
Revision_time     = 28-JAN-2019 17:26:26
Current_time      = 28-JAN-2019 17:27:02

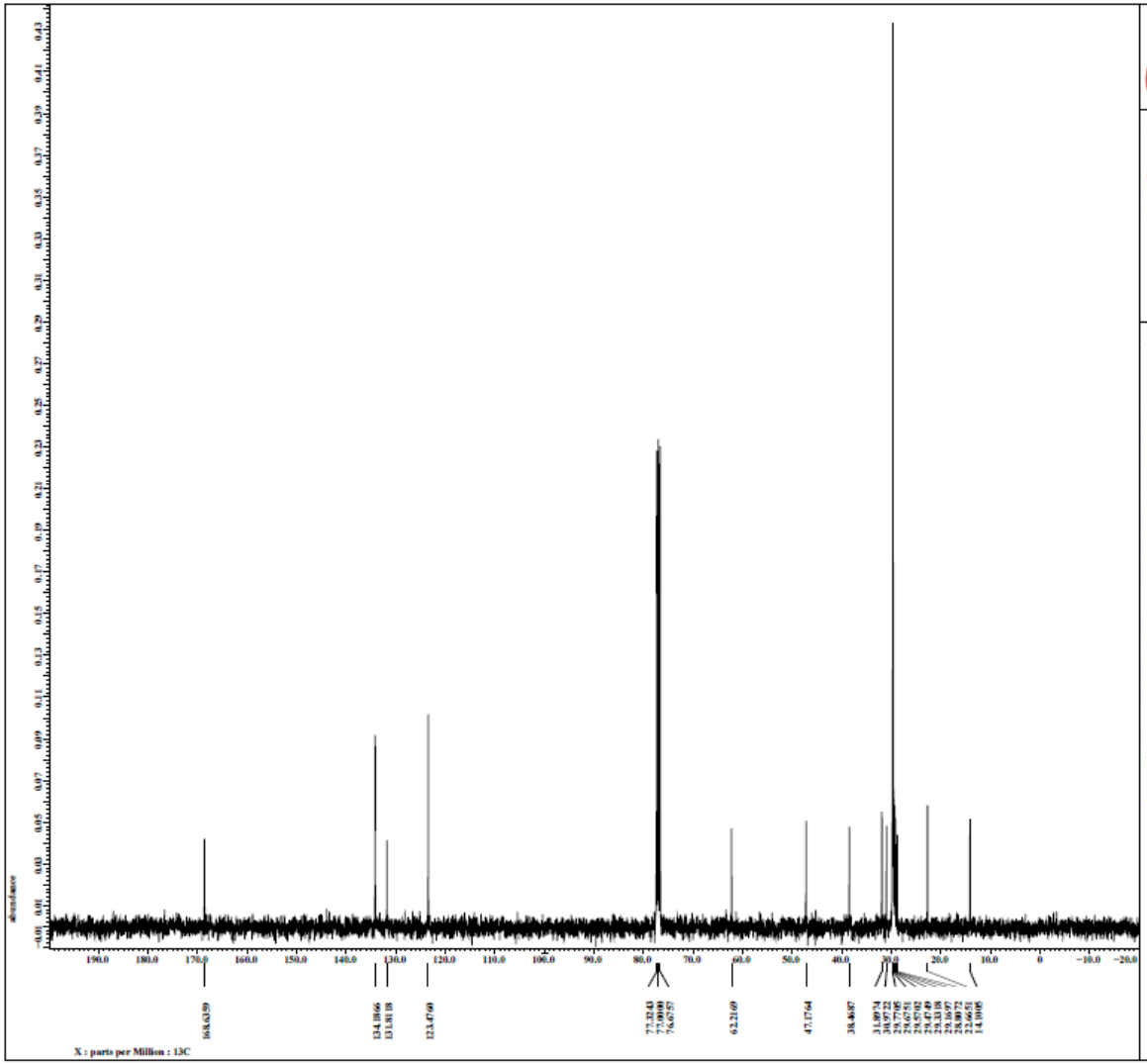
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[Hz]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Delta_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 20
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.2[dC]
```



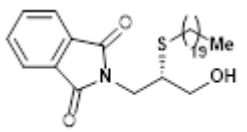
(26a)



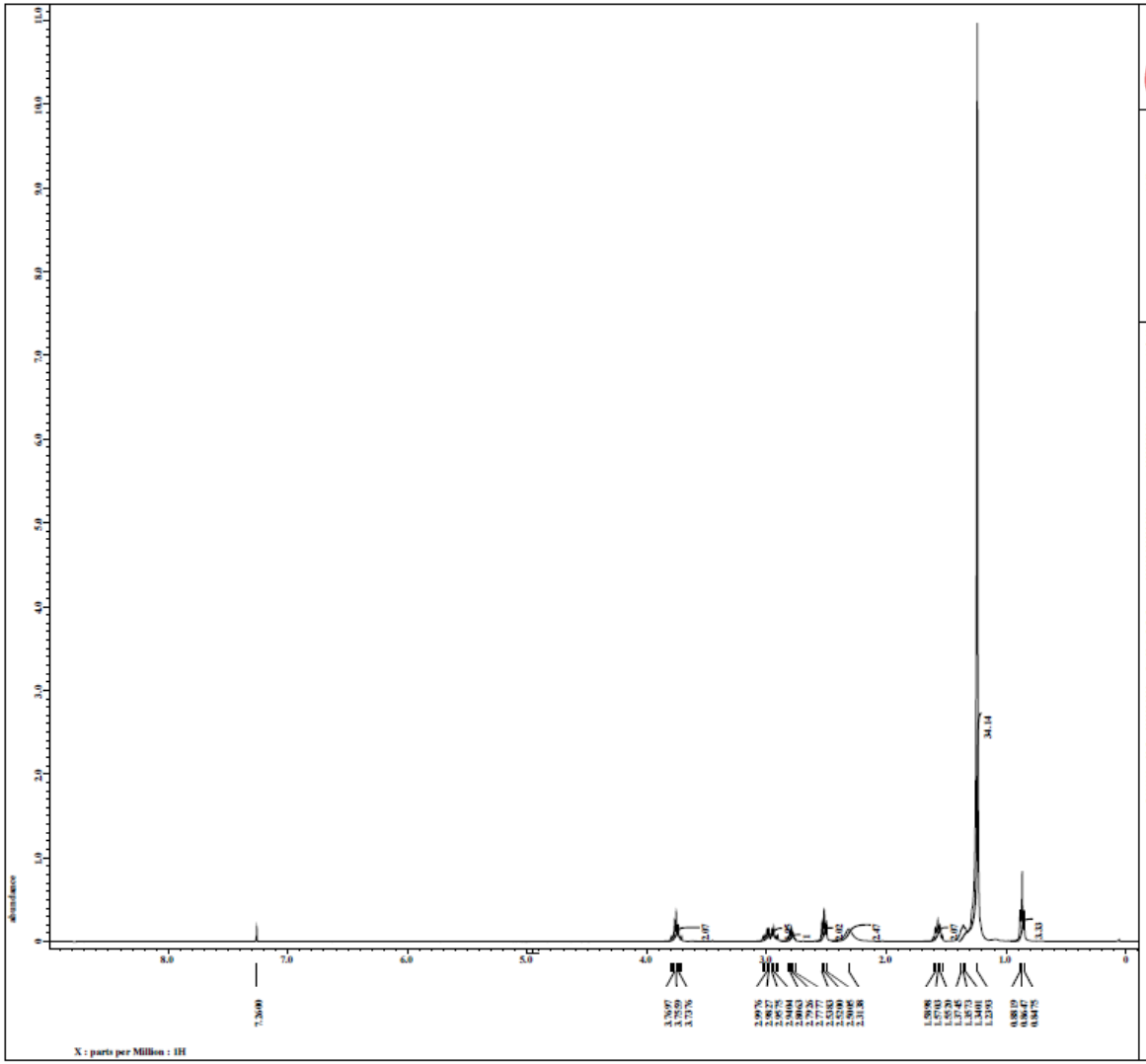
```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190128_13C_3_phthN_SR_CH-1
```

Filename	= 190128_13C_3_phthN_SR
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 28-JAN-2019 17:05:29
Revision_time	= 28-JAN-2019 17:30:40
Current_time	= 28-JAN-2019 17:31:11
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06420464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93858061[Hz]
X_sweep	= 30.75817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 01
Total_scans	= 01
X_90_width	= 9.25[us]
X_acq_time	= 1.06420464[s]
X_angle	= 30[deg]
X_atn	= 4[dB]
X_pulse	= 3.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06420464[s]
Temp_get	= 22.8[dC]



(26a)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
expp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

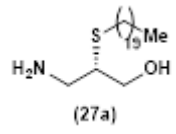
Derived from: 190129_HZN_SC20_OZ-1.jdf
```

```
Filename          = 190129_HZN_SC20_OZ-4.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 29-JAN-2019 16:05:42
Revision_time     = 29-JAN-2019 16:30:54
Current_time      = 29-JAN-2019 16:31:29

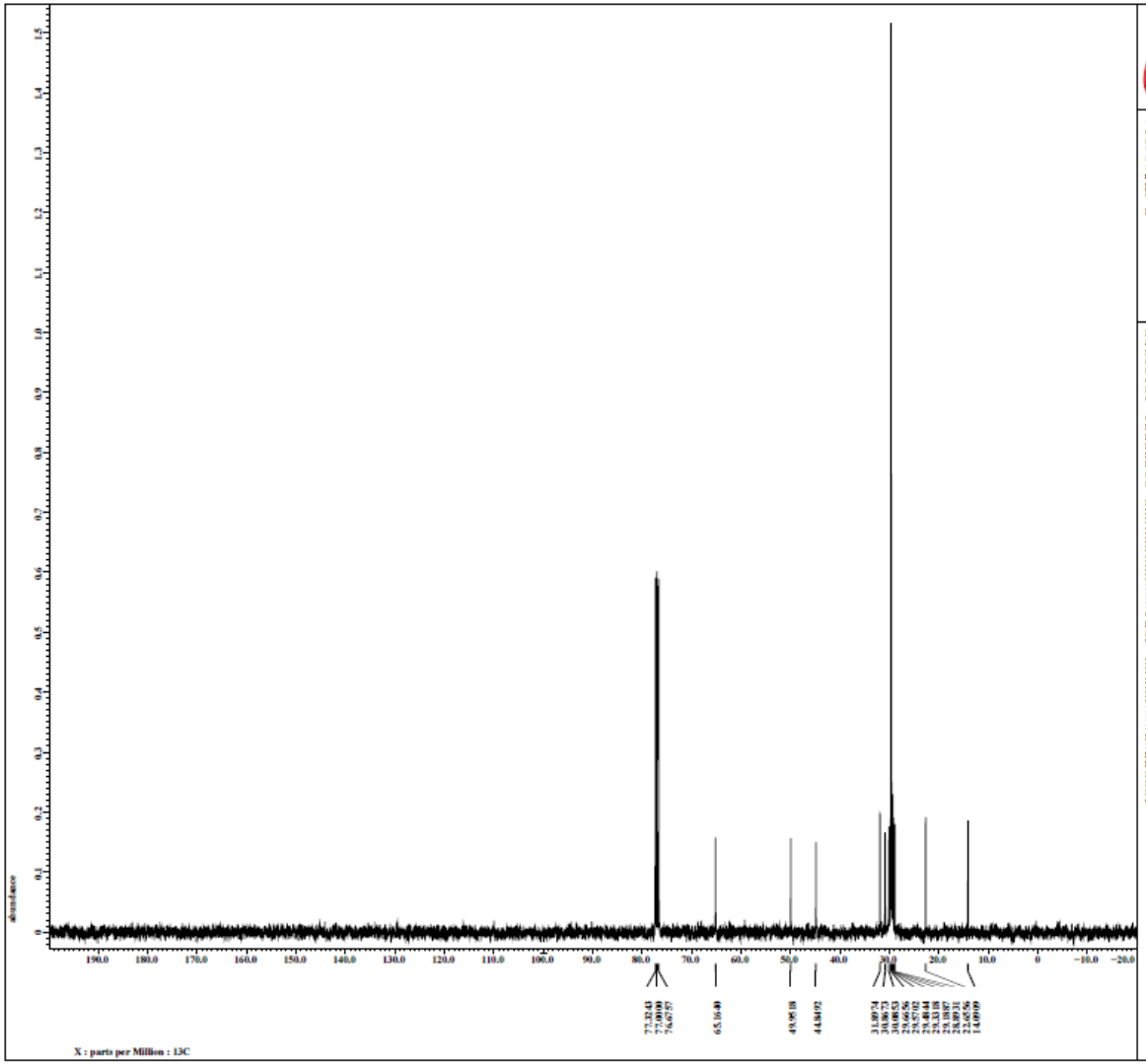
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 131072
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

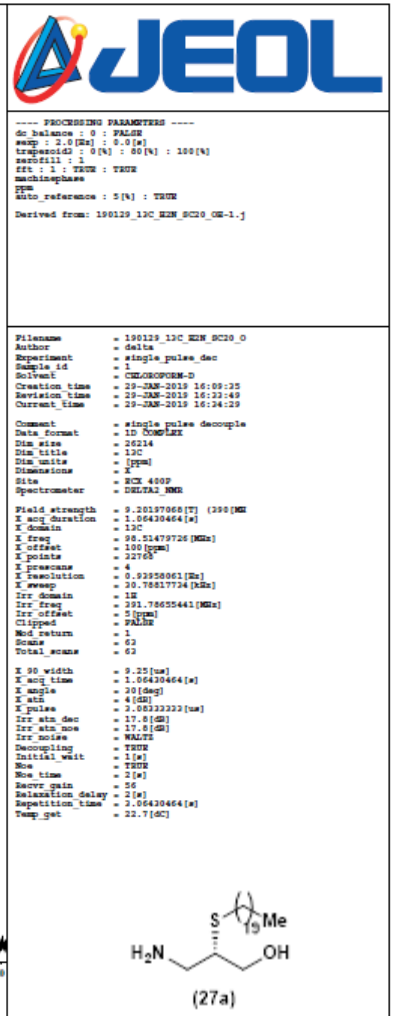
Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 7
Total_scans       = 7

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.3[dC]
```



(27a)





```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190129_13C_HZN_SC20_OH-1.j
```

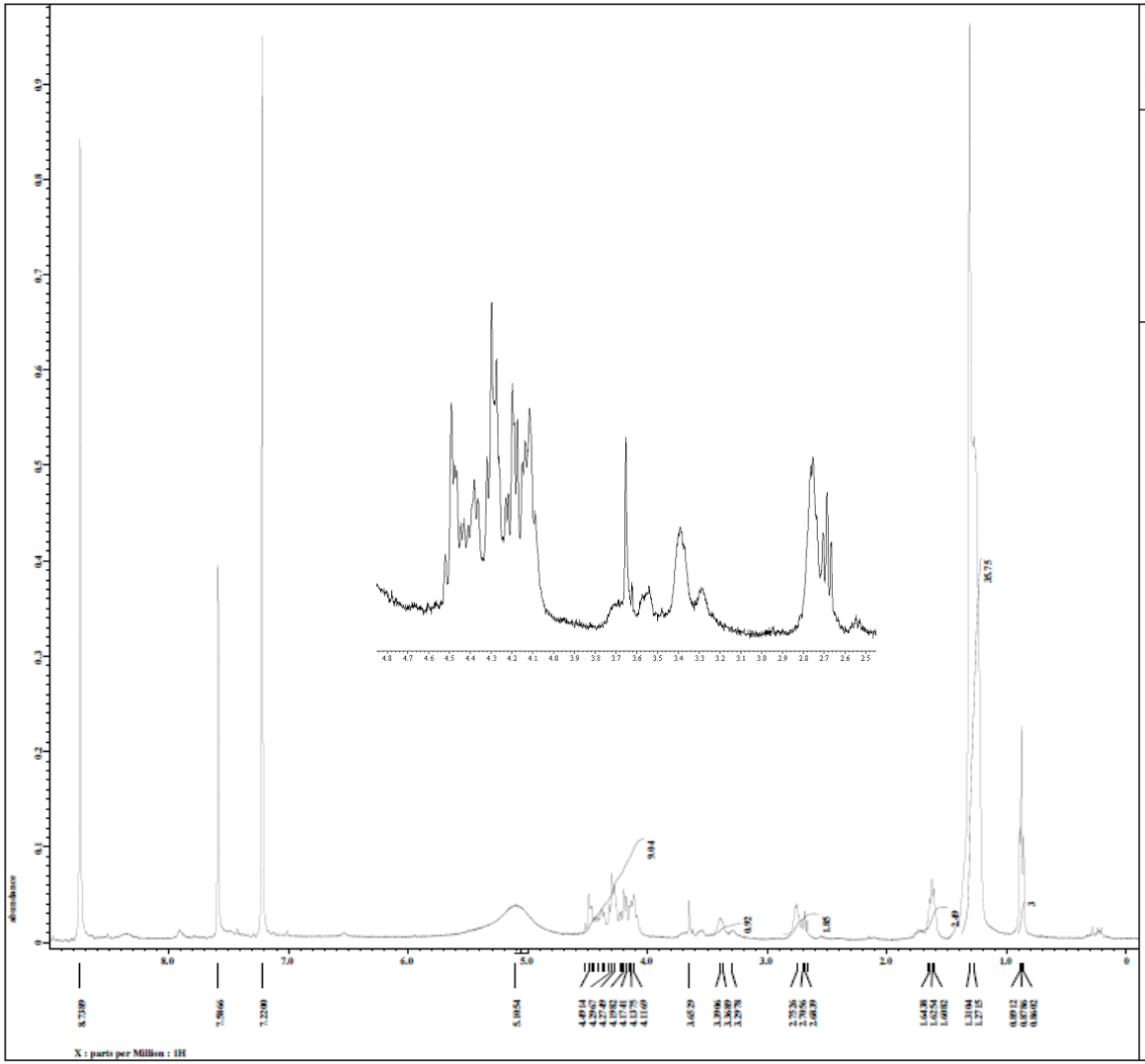
```
Filename          = 190129_13C_HZN_SC20_O
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 29-JAN-2019 16:09:25
Revision_time     = 29-JAN-2019 16:23:49
Current_time      = 29-JAN-2019 16:24:29

Comment           = single_pulse_decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.75817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 63
Total_scans       = 63

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 22.7[dC]
```

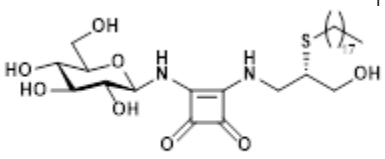

(27a)



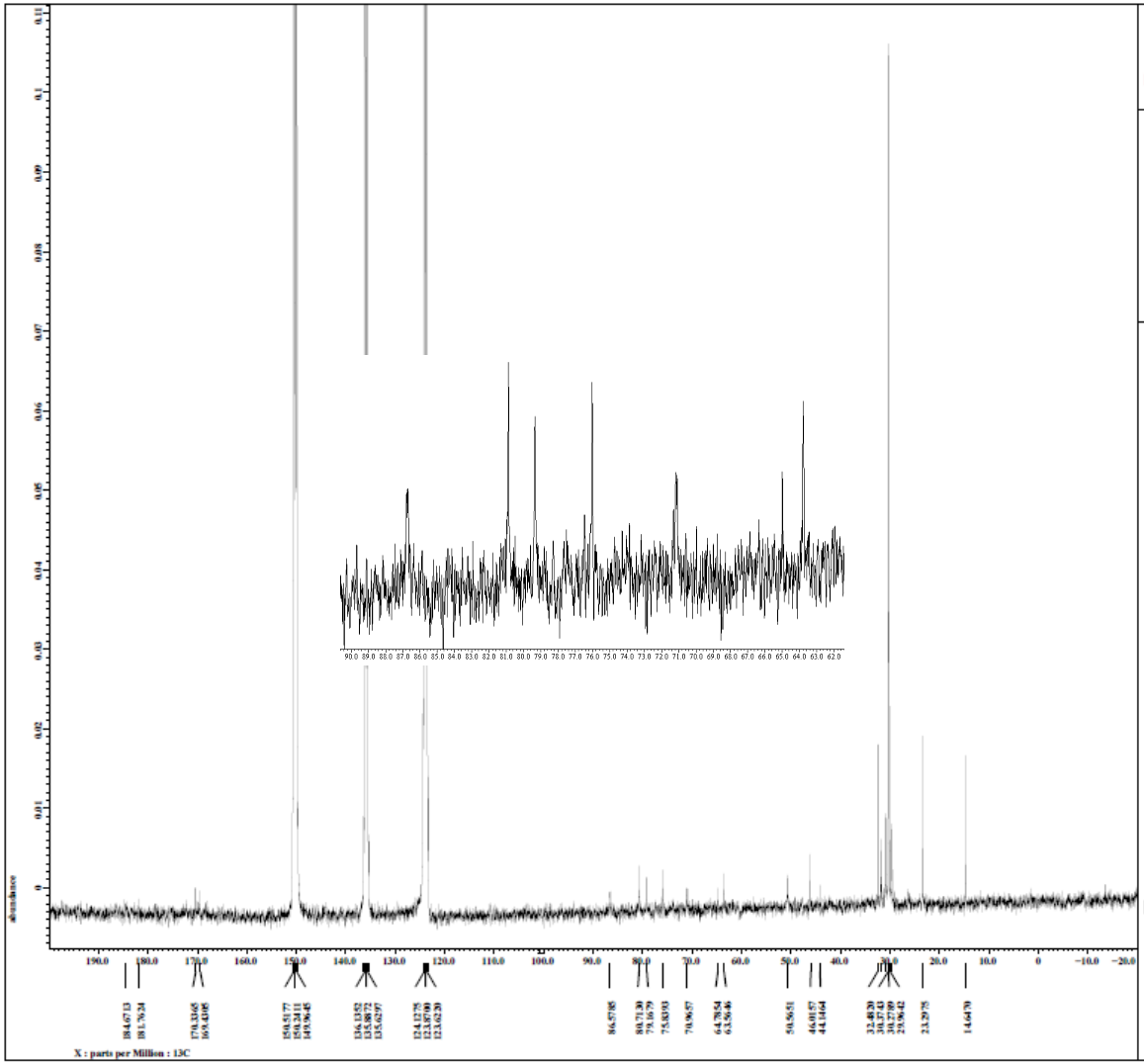
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

Derived from: 190131_GlcNDRS_SC20_OH-1.j

Filename	= 190131_GlcNDRS_SC20_O
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= 1
Solvent	= PYRIDINE-D5
Creation_time	= 31-JAN-2019 16:00:27
Revision_time	= 5-JUN-2019 14:13:39
Current_time	= 5-JUN-2019 14:14:09
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= RCK 400F
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197066[T] (390[MH
X_acq_duration	= 2.228224[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.44876791[Hz]
X_sweep	= 7.35234118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9[us]
X_acq_time	= 2.228224[s]
X_angle	= 45[deg]
X_atn	= 0.99[dB]
X_pulse	= 4.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_preset	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 26
Relaxation_delay	= 5[s]
Repetition_time	= 7.228224[s]
Temp_get	= 22.8[dC]



(11h)




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

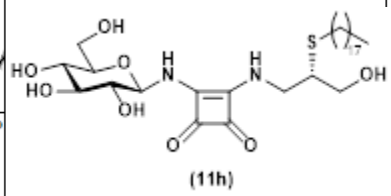
Derived from: 190201_13C_GlcNDRS_SC20_OH
```

```
Filename          = 190201_13C_GlcNDRS_SC
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     =  2-FEB-2019 02:26:49
Revision_time     =  5-JUN-2019 14:05:23
Current_time      =  5-JUN-2019 14:09:00

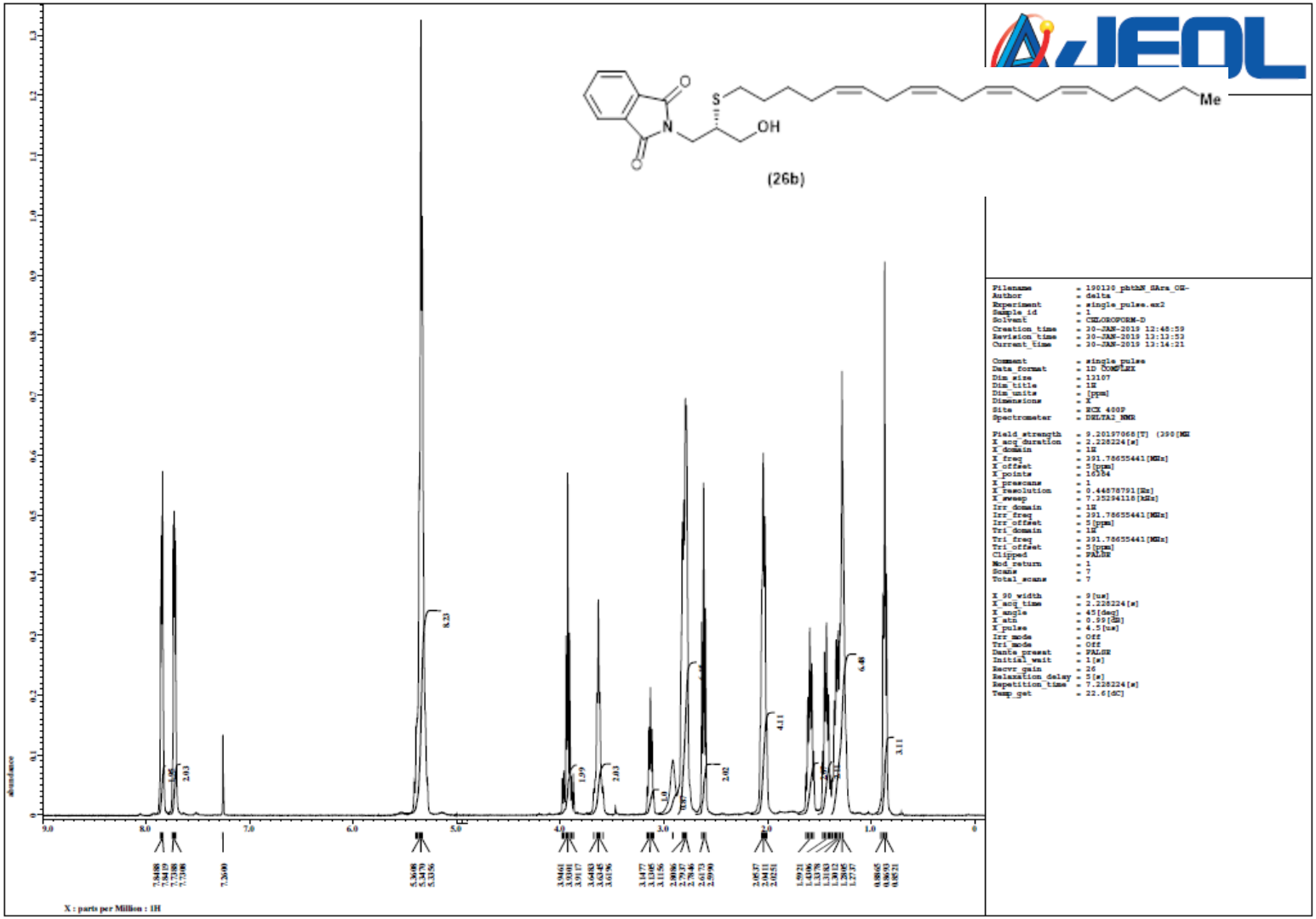
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 1.06420464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 7168
Total_scans       = 7168

X_90_width        = 9.25[us]
X_acq_time        = 1.06420464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.6[dB]
Irr_atn_noe       = 17.6[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   = 3.06420464[s]
Temp_get          = 22.7[dC]
```



(11h)

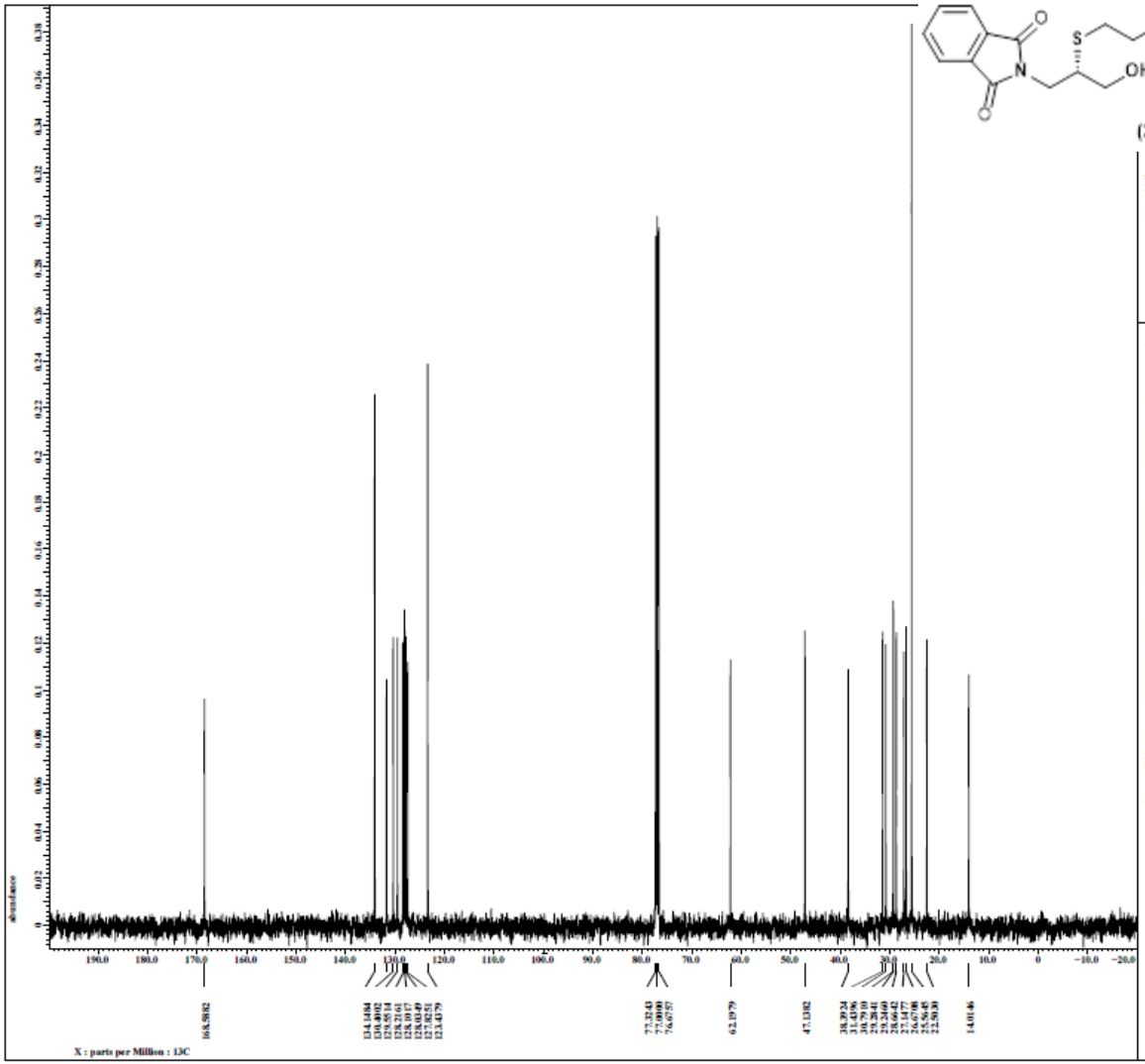


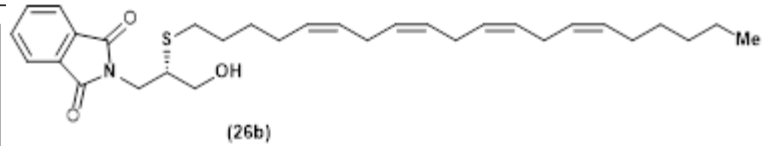

```
Filename          = 190130_phthN_SAra_OE-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = CHLOROFORM-D
Creation_time     = 30-JAN-2019 12:45:59
Revision_time     = 30-JAN-2019 13:13:52
Current_time      = 30-JAN-2019 13:14:21

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = 1
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.25294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 7
Total_scans       = 7

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.6[dC]
```



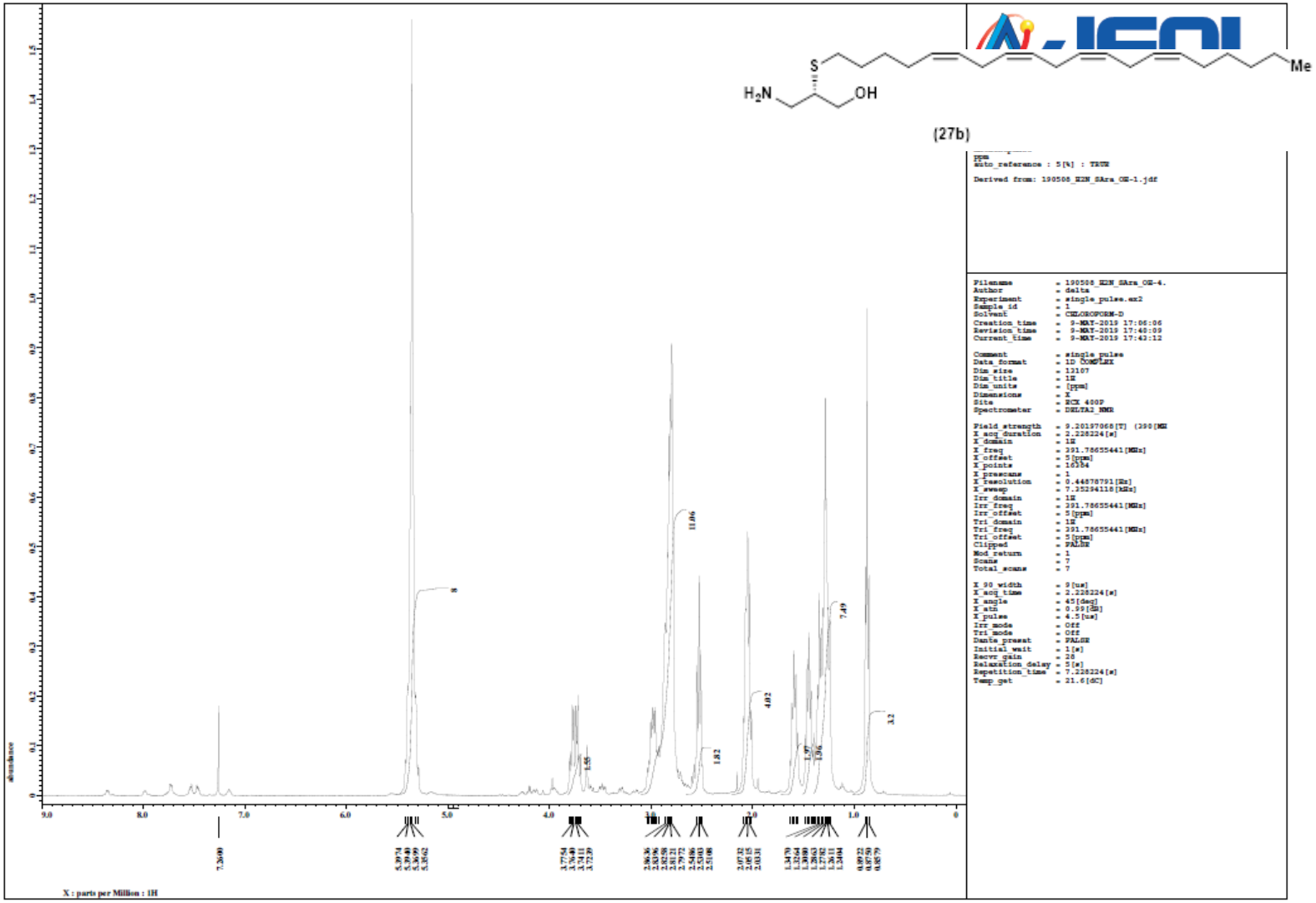


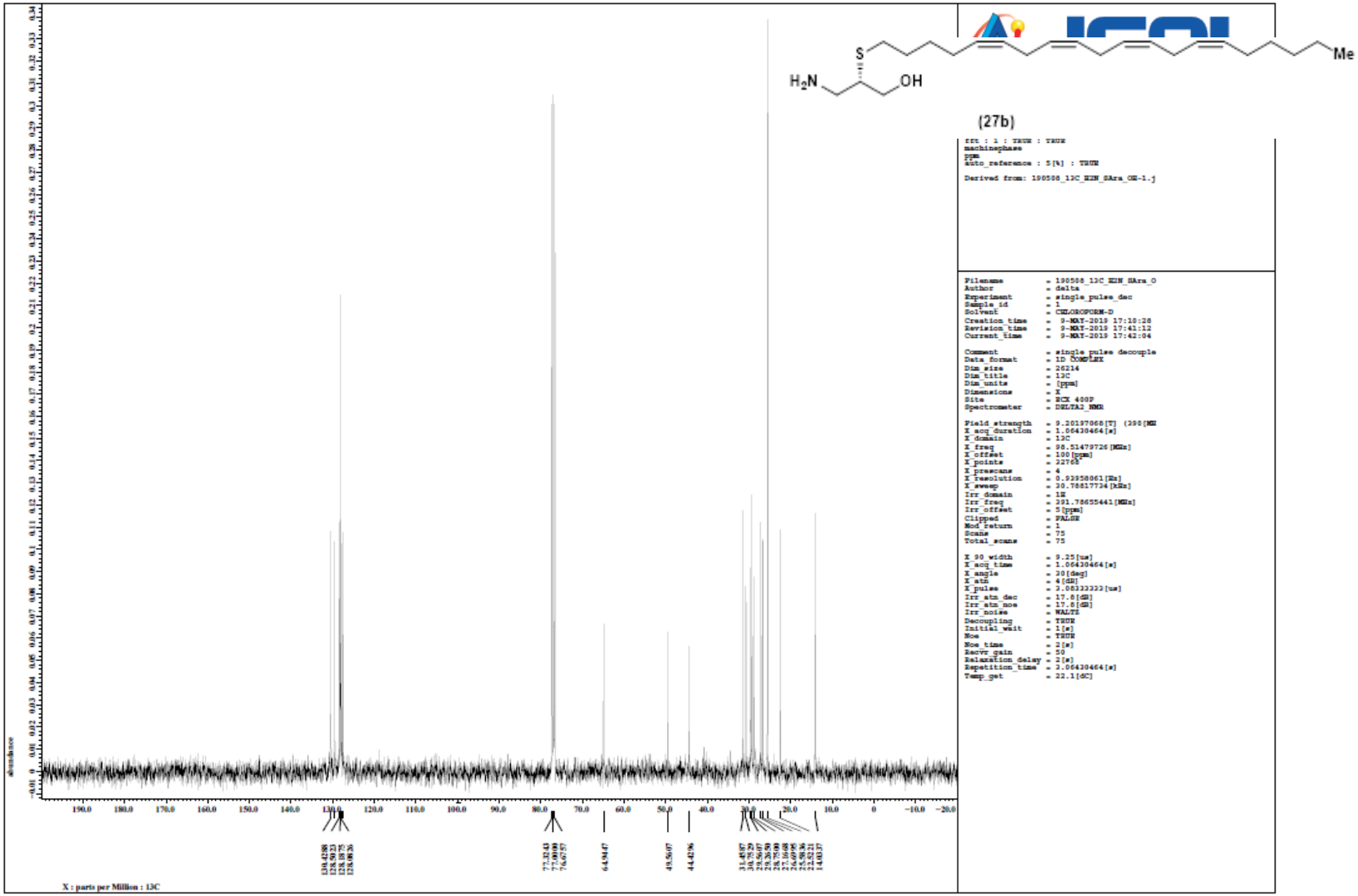
(26b)

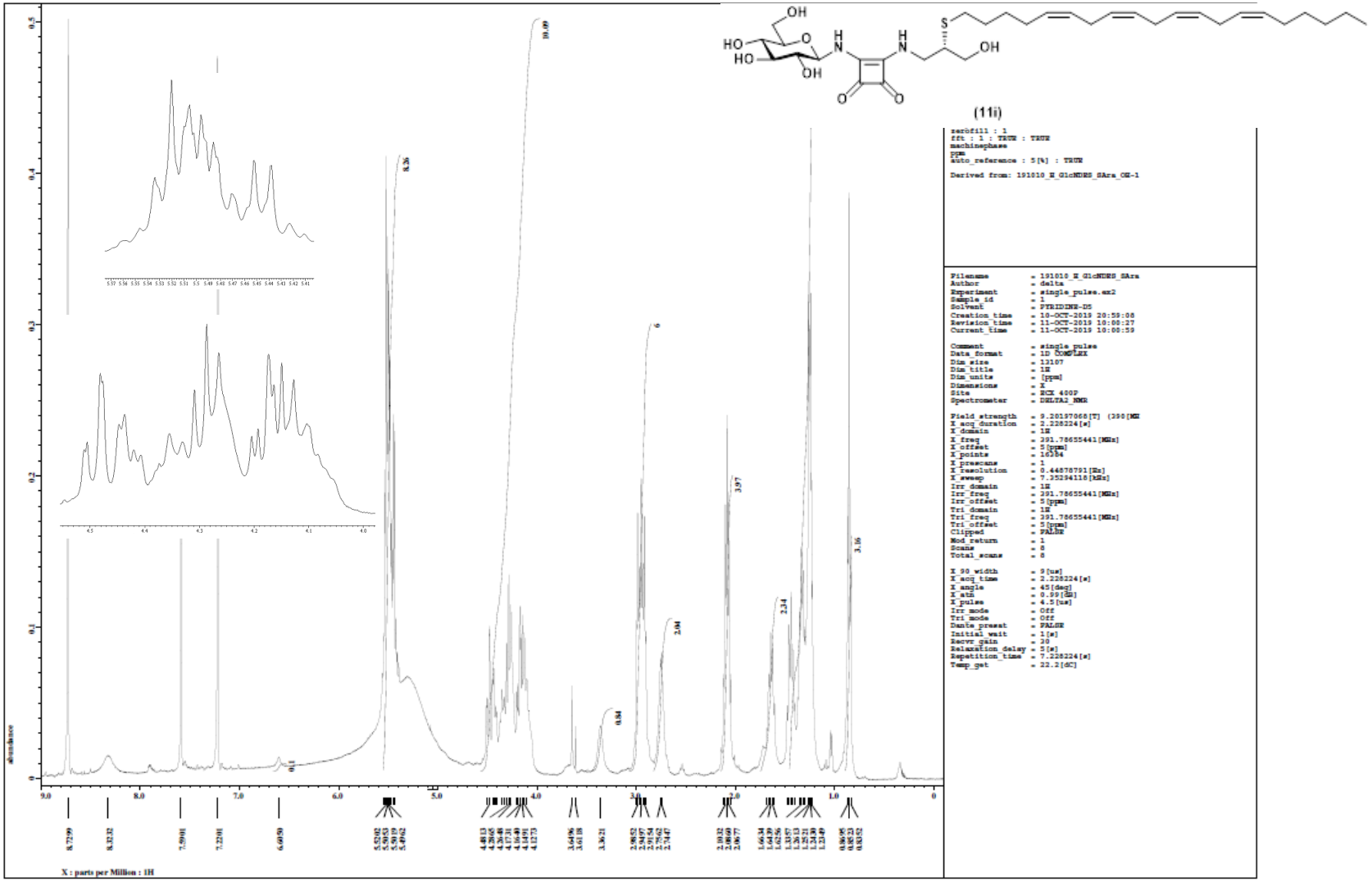
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

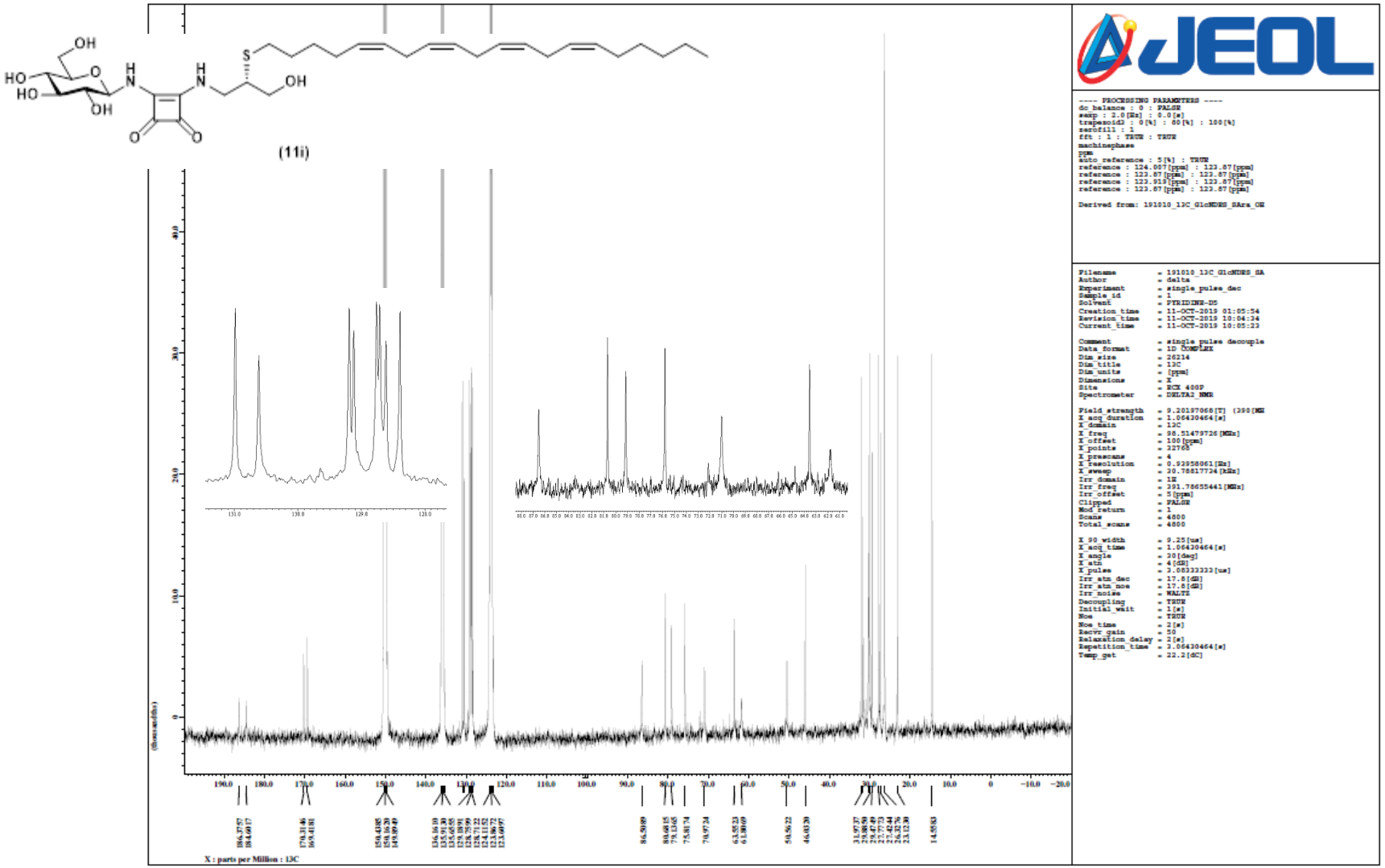
Derived from: 190130_13C_phthN_SAra_OH-1

Filename	= 190130_13C_phthN_SAra
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= 1
Solvent	= CHLOROFORM-D
Creation_time	= 30-JAN-2019 12:53:25
Revision_time	= 30-JAN-2019 13:17:03
Current_time	= 30-JAN-2019 13:17:52
Comment	= single_pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= RCX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32765
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 79
Total_scans	= 79
X_90_width	= 9.25[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 6[dB]
X_pulse	= 3.08333333[us]
Irr_atn_dec	= 17.8[dB]
Irr_atn_noe	= 17.8[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 22[dC]


S
H2N
OH
Me
(27b)
ppm
auto_reference : 5[%] : TRUE
Derived from: 190508_HZN_SAra_OH-1.jdf
Filename = 190508_HZN_SAra_OH-4.
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 9-MAY-2019 17:06:06
Revision_time = 9-MAY-2019 17:40:09
Current_time = 9-MAY-2019 17:43:12
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = NCX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 7
Total_scans = 7
X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 28
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 21.6[dC]
Abundance
X : parts per Million : 1H


S
H2N
OH
Me
(27b)
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
Derived from: 190508_13C_H2N_SAra_OH-1.j
Filename = 190508_13C_H2N_SAra_O
Author = delta
Experiment = single_pulse_dec
Sample_id = 1
Solvent = CHLOROFORM-D
Creation_time = 9-MAY-2019 17:10:28
Revision_time = 9-MAY-2019 17:41:12
Current_time = 9-MAY-2019 17:42:04
Comment = single_pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = RCE 400F
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.93958061[Hz]
X_sweep = 20.75817734[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 75
Total_scans = 75
X_90_width = 9.25[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4[dB]
X_pulse = 2.05333333[us]
Irr_atn_dec = 17.8[dB]
Irr_atn_noe = 17.8[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 22.1[dC]
X : parts per Million : 13C


OH
HO
HO
OH
O
H
N
H
N
S
OH
O
O
(11i)
9.09
8.36
6
3.97
2.84
2.34
3.16
0.84
0.41
abundance
X : parts per Million : 1H
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
Derived from: 191010_H_GlcNDH5_SAra_OH-1
Filename = 191010_H_GlcNDH5_SAra
Author = delta
Experiment = single_pulse.ex2
Sample_id = 1
Solvent = PYRIDINE-D5
Creation_time = 10-OCT-2019 20:59:06
Revision_time = 11-OCT-2019 10:00:27
Current_time = 11-OCT-2019 10:00:59
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 131107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = RCX 400P
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.228224[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.44878791[Hz]
X_sweep = 7.35294116[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 9[us]
X_acq_time = 2.228224[s]
X_angle = 45[deg]
X_atn = 0.99[dB]
X_pulse = 4.5[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Initial_wait = 1[s]
Recvr_gain = 20
Relaxation_delay = 5[s]
Repetition_time = 7.228224[s]
Temp_get = 22.2[dC]




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exmp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
reference : 124.007[ppm] : 123.87[ppm]
reference : 123.87[ppm] : 123.87[ppm]
reference : 123.919[ppm] : 123.87[ppm]
reference : 123.87[ppm] : 123.87[ppm]

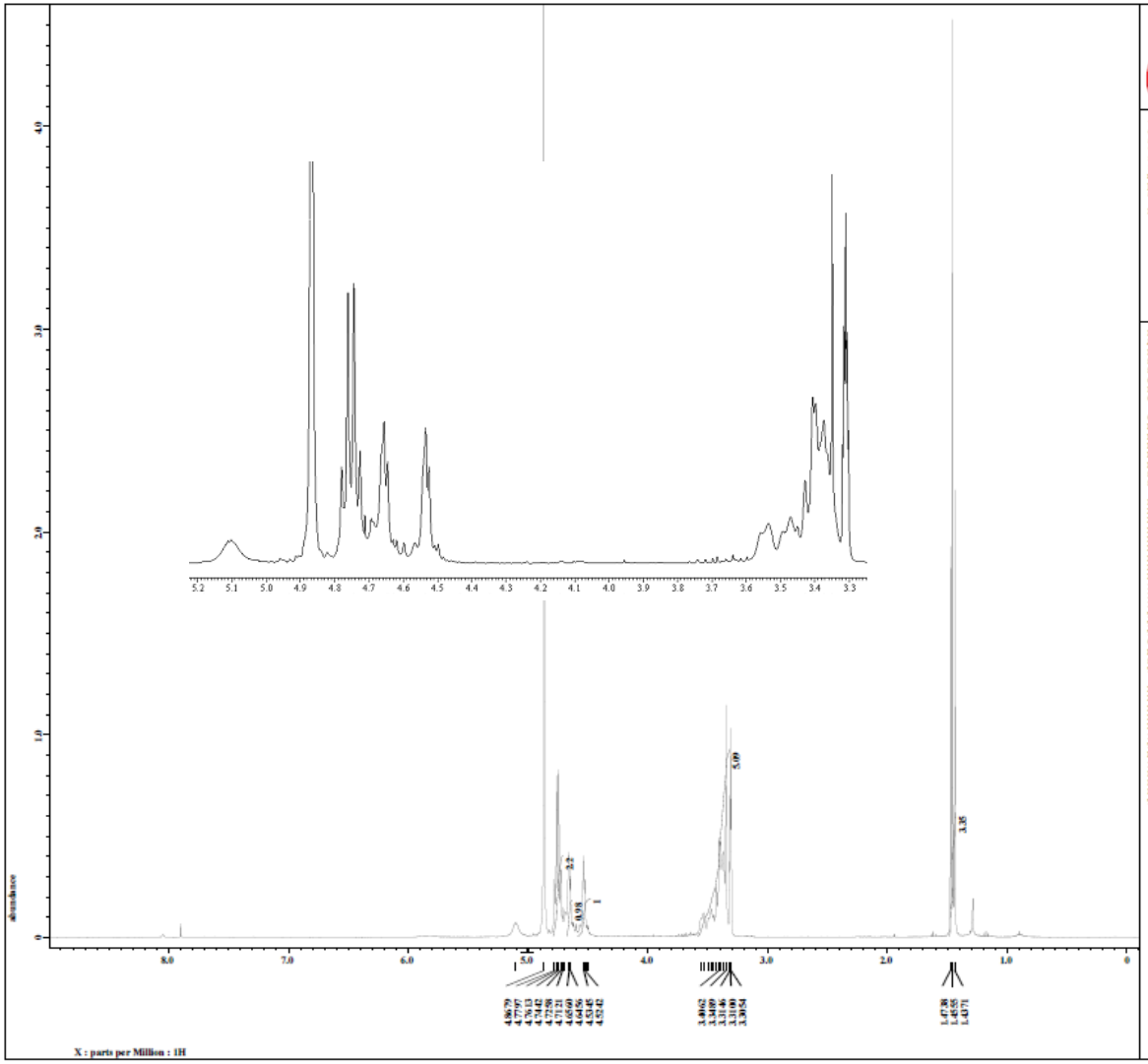
Derived from: 191010_13C_GlcNDRS_SAra_OH
```

```
Filename          = 191010_13C_GlcNDRS_SA
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 11-OCT-2019 01:05:54
Revision_time     = 11-OCT-2019 10:04:24
Current_time      = 11-OCT-2019 10:05:23

Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCK 400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78217734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 4800
Total_scans       = 4800

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 22.2[dC]
```



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ft
auto_reference : 5[%] : TRUE

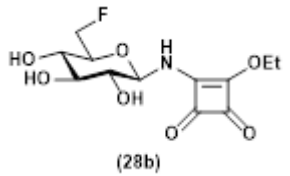
Derived from: 180406_H_6PGlc_DRS-1.jdf
```

```
Filename          = 180406_H_6PGlc_DRS-4.
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = METHANOL-D3
Creation_time     =  6-APR-2018 10:05:52
Revision_time     = 23-APR-2019 11:06:27
Current_time      = 23-APR-2019 11:07:12

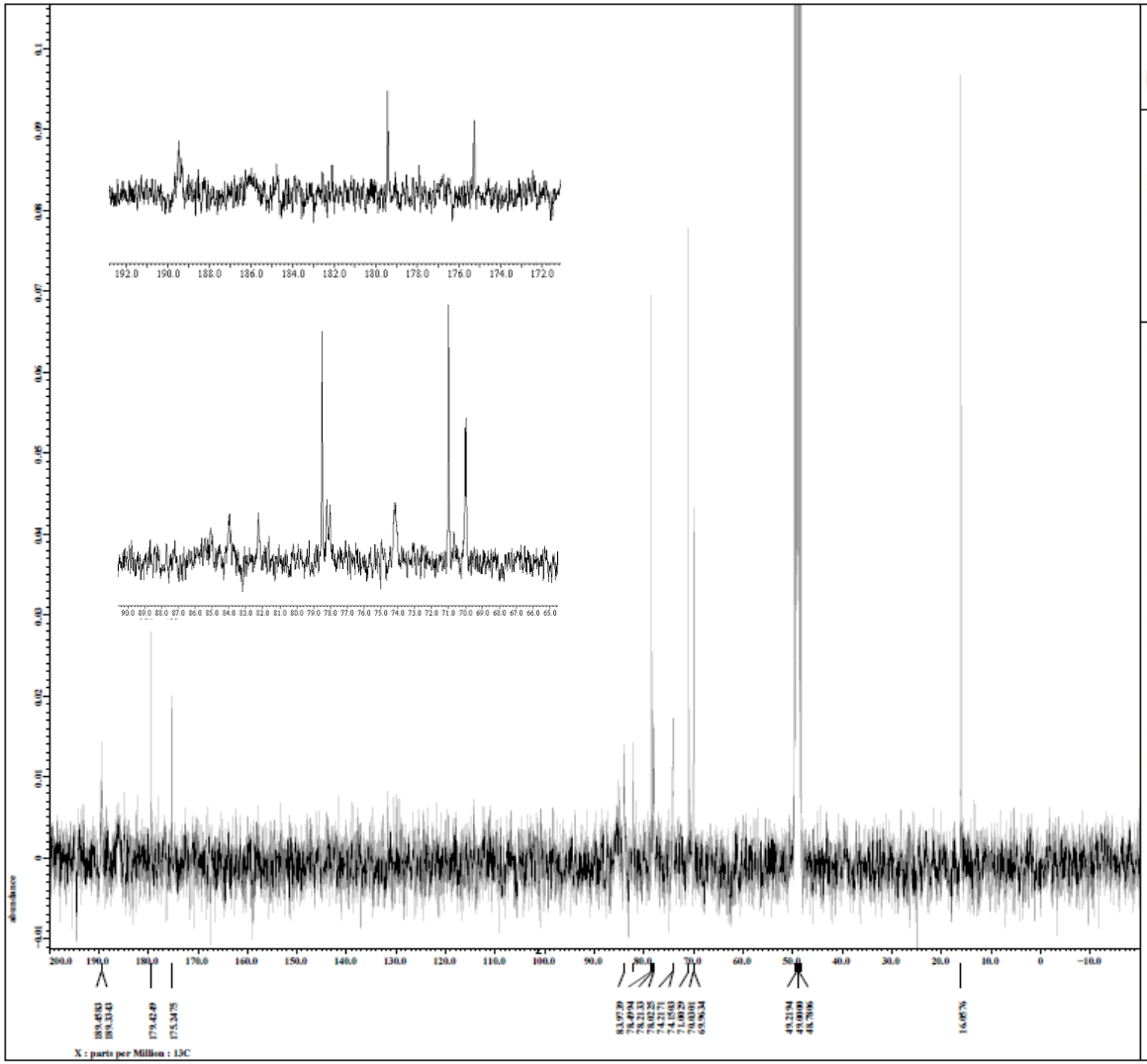
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = NCH 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T]  (399[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 402.3[dC]
```



(28b)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

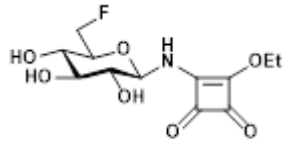
Derived from: 180406_13C_6PUlc_DHS-1.jdf
```

```
Filename          = 180406_13C_6PUlc_DHS-
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = METHANOL-D3
Creation_time     =  5-APR-2018 21:14:00
Revision_time     = 23-APR-2019 11:02:07
Current_time      = 23-APR-2019 11:02:38

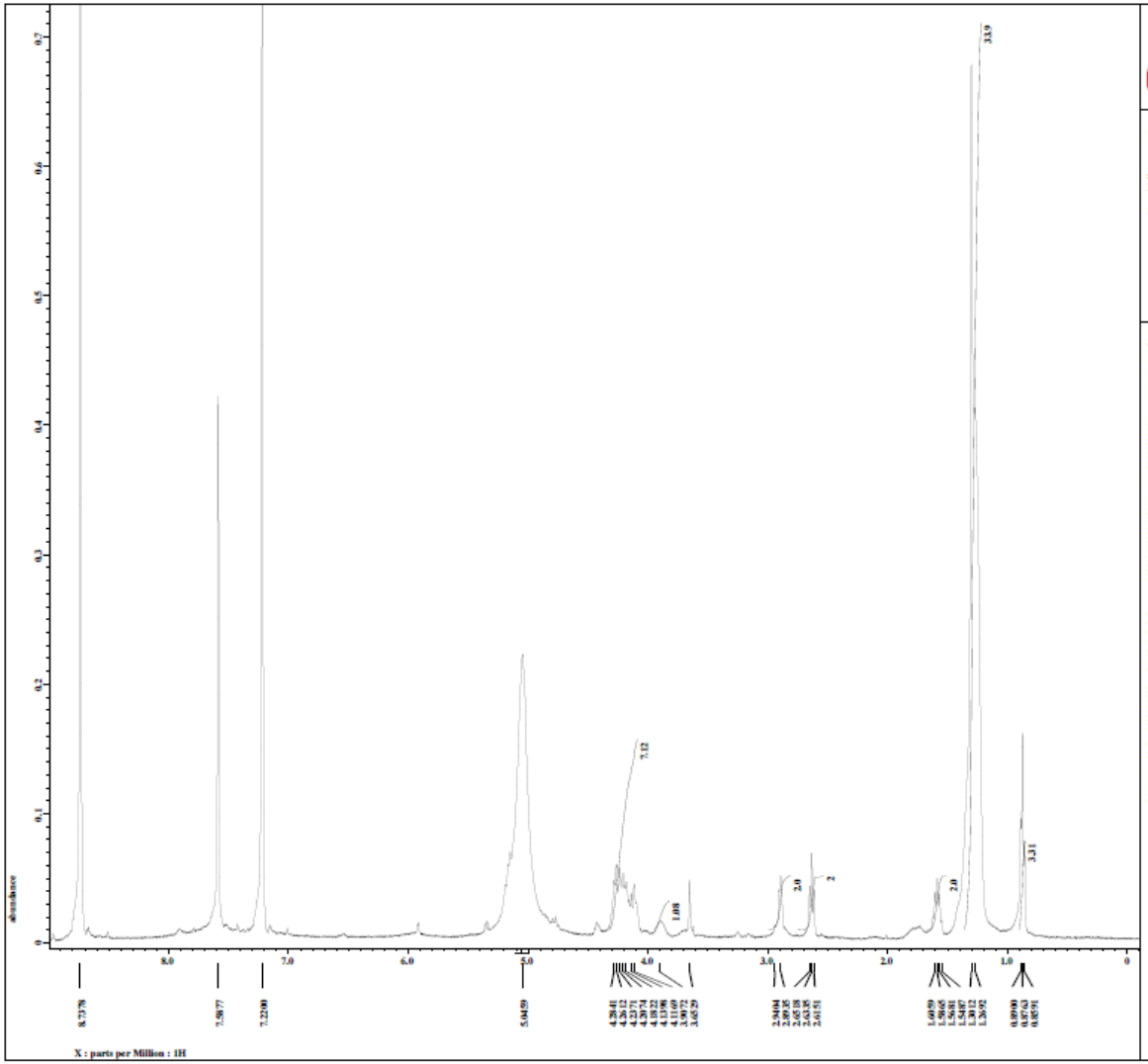
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400F
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.75617734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 65
Total_scans       = 65

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 401.9[dC]
```



(28b)



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 0.2[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

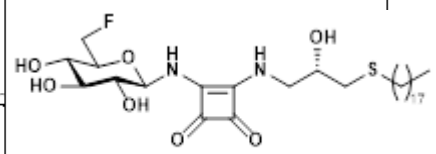
Derived from: 181104_6P_GlcN_DNS_C16-1.j
```

```
Filename          = 181104_6P_GlcN_DNS_C1
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     =  4-NOV-2018 20:47:03
Revision_time     =  7-MAY-2019 17:33:25
Current_time      =  7-MAY-2019 17:34:05

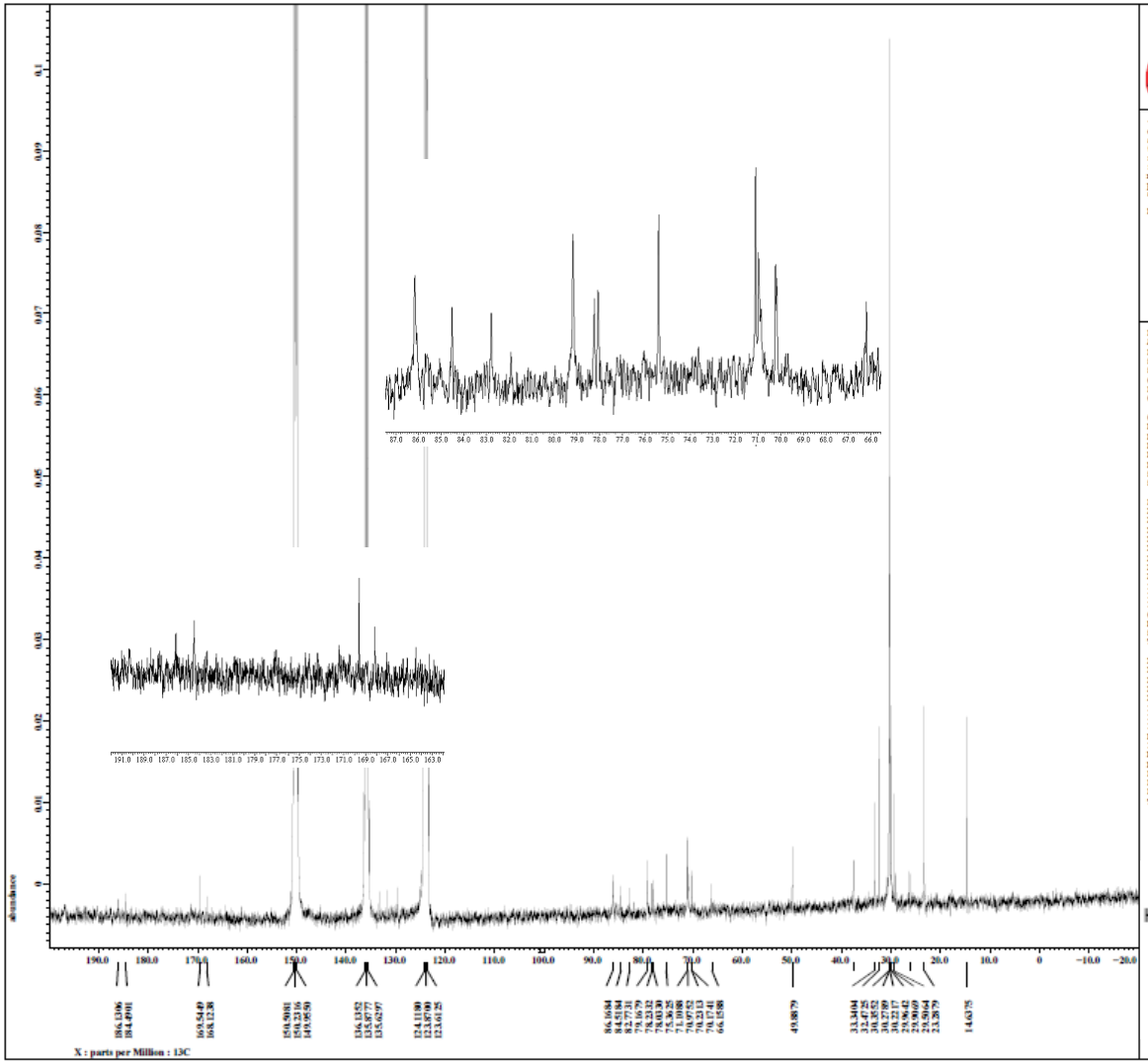
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44876791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 22.9[dC]
```



(11j)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

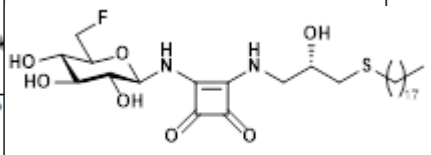
Derived from: 181104_13C_6P_GlcN_DRS_C16
```

```
Filename          = 181104_13C_6P_GlcN_DR
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     =  5-NOV-2018 08:24:51
Revision_time     =  7-MAY-2019 17:25:25
Current_time      =  7-MAY-2019 17:27:44

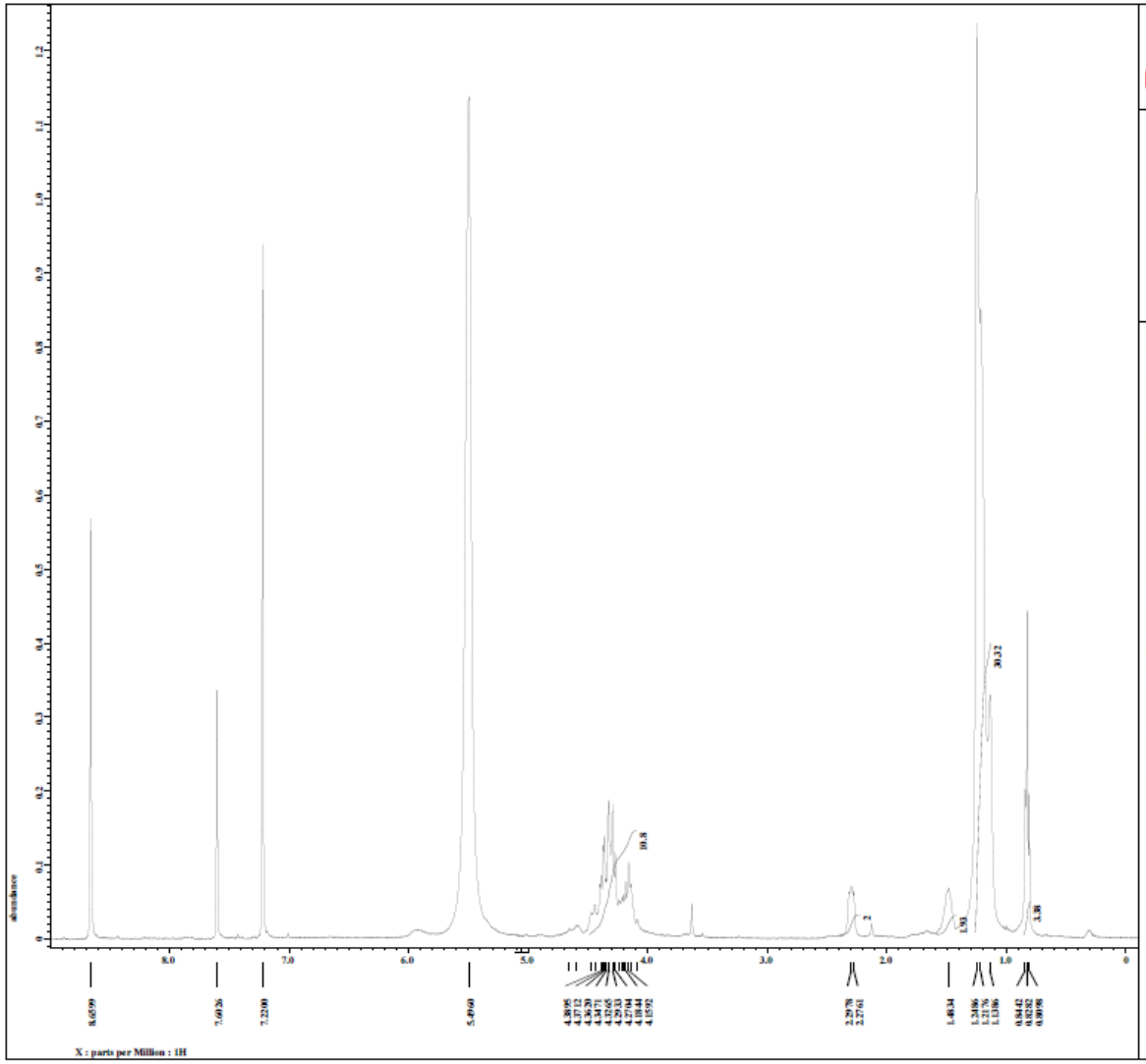
Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197066[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.92358061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 13440
Total_scans       = 13440

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atn_dec       = 17.8[dB]
Irr_atn_noe       = 17.8[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   = 3.06430464[s]
Temp_get          = 22.6[dC]
```



(11j)



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exsp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 50[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE
auto_reference : 5[%] : TRUE

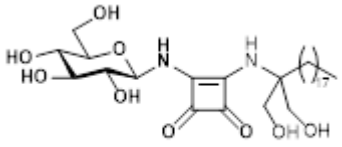
Derived from: GlcN_DRS_diol_C18-1.jdf
```

```
Filename          = GlcN_DRS_diol_C18-5.j
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 21-AUG-2018 09:45:49
Revision_time     =  5-JUN-2019 14:02:27
Current_time      =  5-JUN-2019 14:03:00

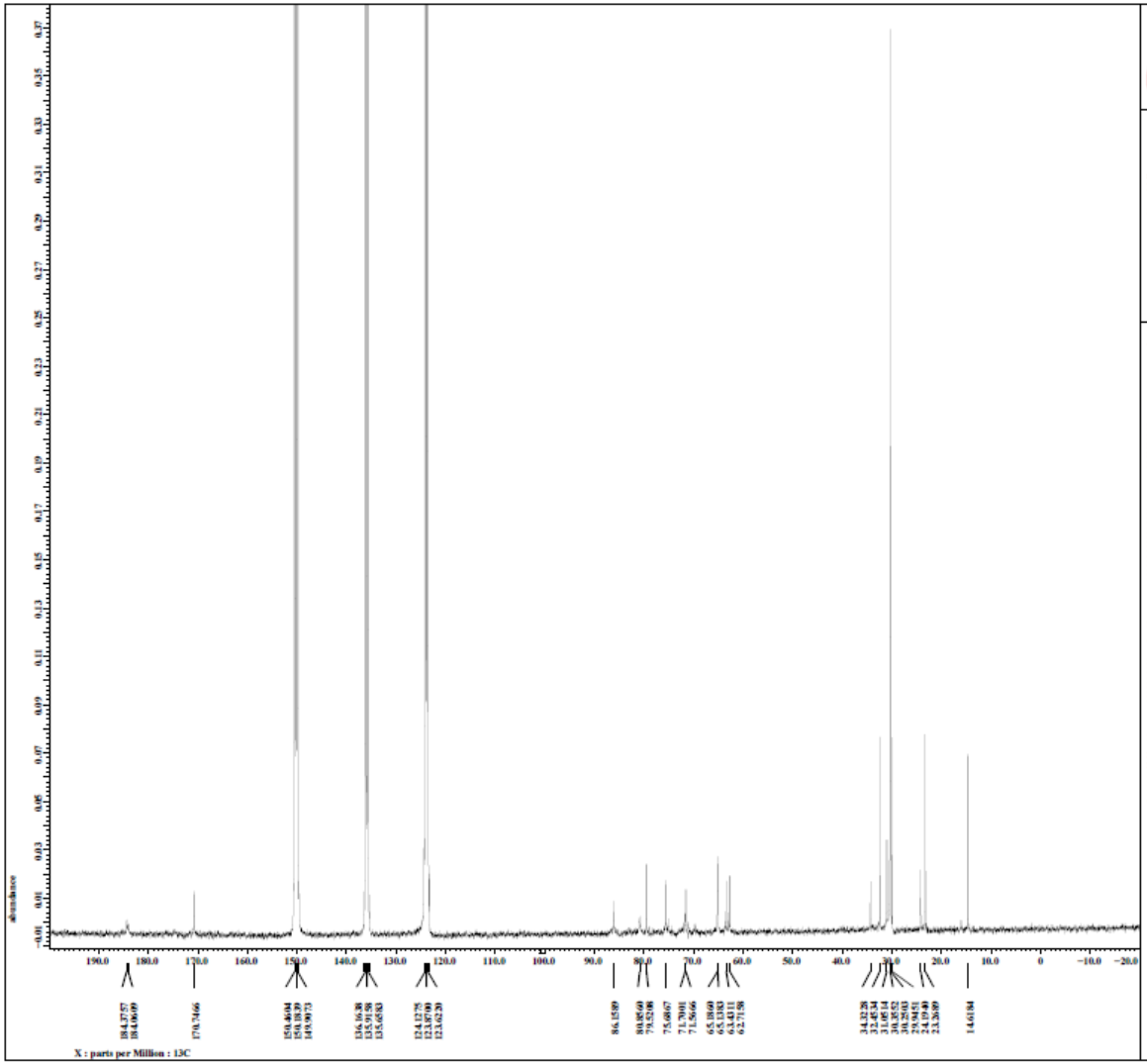
Comment           = single pulse
Data_format       = 1D COMPLEX
Dim_size          = 12107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.228224[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.44878791[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 9[us]
X_acq_time        = 2.228224[s]
X_angle           = 45[deg]
X_atn             = 0.99[dB]
X_pulse           = 4.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_preset      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 7.228224[s]
Temp_get          = 23.6[dC]
```



(11k)



JEOL

```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
expp : 2.0[Hz] : 0.0[s]
trapezoid2 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
auto_reference : 5[%] : TRUE

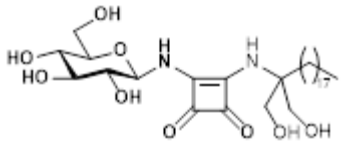
Derived from: 180817_13C_GlcN_DHS_HSN_d1
```

```
Filename          = 180817_13C_GlcN_DHS_H
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 1
Solvent           = PYRIDINE-D5
Creation_time     = 19-AUG-2018 02:24:26
Revision_time     =  5-JUN-2019 13:46:33
Current_time      =  5-JUN-2019 13:46:48

Comment           = single_pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = RCX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.75817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 7124
Total_scans       = 7124

X_90_width        = 9.25[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 3.08333333[us]
Irr_atm_dec       = 17.8[dB]
Irr_atm_noe       = 17.8[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 58
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 23[dC]
```



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