Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Heteroannulation of bicyclobutane derivatives via Au-catalyzed hydration to enol ethers and intramolecular cyclization giving spirocyclobutanes

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Contents

1. General Information

- ^{1}H, ^{13}C, NMR spectra were measured by JEOL ECS 300, JEOL JNM-ECS 400 or JEOL JNMLA 500 spectrometers.

  ^{1}H NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of tetramethylsilane (TMS) at 0 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (Hz).

  ^{13}C NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of triplet for $CDCl_3$ at 77 ppm, septet for acetone-d6 at 29.8 ppm, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (Hz).

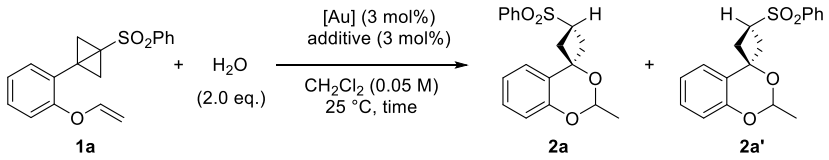
  C_6F_6 (singlet at -164.9 ppm) was used as an external standard for ^{19}F NMR.
- MALDI-MS spectra were obtained with JMS-S3000 (JEOL).
- Melting points were measured by BÜCHI B-545.
- Column chromatography on SiO_2 was performed with Kanto Chemical Silica Gel 60 (spherical, 63-210 μm or spherical, 40-50 μm).
- Commercially available organic and inorganic compounds were used without further purification.

2. Optimization of Reaction Conditions

General procedure for Table S1-S4

A flame dried test tube equipped with magnetic stirring bar was charged with Au catalyst (4.5 μmol, 3.0 mol%) and Ag additive (4.5 μmol, 3.0 mol%) under N_2 in glovebox. Dry solvent (3.0 mL, 0.05 M) and nucleophiles, H_2O or amines, (0.30 mmol, 2.0 eq. or specified eq.) was added to the mixture at ambient temperature. The solution was stirred at 25 ℃ for 5 min, and then the compound **1a** (0.15 mmol) was added. After specified reaction time, the mixture was filtered through short pad of silica gel. The yields of compounds **2** and **2'** were determined by ^{1}H NMR.

Table S1. Screening of Au catalysts and additives

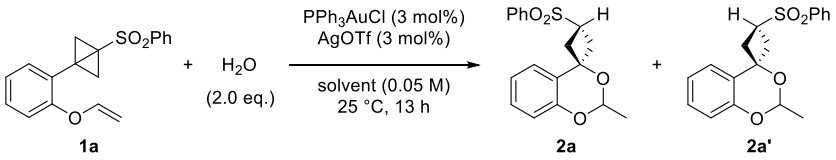


1a (0.15 mmol) **2a** **2a'**

entry	[Au]	additive	time	**2a**/**2a'** (%, *dr*)[a]
0	IPrAuNTf$_2$	-----	2 h	51%, (1.5 : 1)
1	IPrAuNTf$_2$	-----	13 h	67%, (1.3 : 1)
2	IPrAu(MeCN)BF$_4$	-----	20 h	16%, (0.81 : 1)
3	IPrAuCl	AgNTf$_2$	13 h	56%, (0.79 : 1)
4	XPhosAuCl	AgOTf	〃	83%, (0.90 : 1)
5	PPh$_3$AuCl	AgOTf	〃	86%, (0.90 : 1)
6	PPh$_3$AuCl	AgOAc	〃	N.D.[b]
7	PPh$_3$AuCl	AgBF$_4$	〃	34%, (0.51 : 1)
8	PPh$_3$AuCl	AgSbF$_6$	〃	54%, (0.80 : 1)
9	PPh$_3$AuCl	AgNTf$_2$	〃	76%, (0.80 : 1)
10[c]	-----	AgOTf	〃	57%, (0.67 : 1)
11	-----	-----	〃	N.R.[d]

[a] NMR yield using 1,3,5-trimethoxybenzene as an internal standard material.
[b] not detected. [c] used 6 mol% of additive. [d] no reaction.

Table S2. Screening of solvents

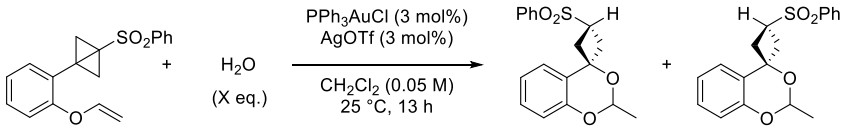


1a (0.15 mmol) **2a** **2a'**

entry	solvent	**2a**/**2a'** (%, *dr*)[a]
0	CH_2Cl_2	86%, (0.90 : 1)
1	$CHCl_3$	79%, (0.81 : 1)
2	PhMe	59%, (0.54 : 1)
3	THF	N.D.[b]
4	1,4-dioxane	52%, (0.55 : 1)
5	$MeNO_2$	85%, (1.16 : 1)
6	EtOAc	73%, (0.48 : 1)
7	MeCN	17%, (0.32 : 1)
8	DMF	N.R.[c]

[a] NMR yield using 1,3,5-trimethoxybenzene as an internal standard material.
[b] not detected. [c] no reaction.

Table S3. Screening of H_2O loading

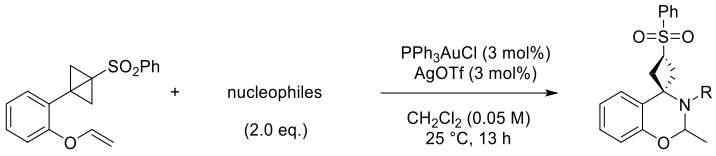


1a (0.15 mmol) **2a** **2a'**

entry	X	**2a**/**2a'** (%, *dr*)*
0	2	86%, (0.90 : 1)
1	1	78%, (0.92 : 1)
2	5	45%, (0.59 : 1)
3	10	10%, (0.45 : 1)

*NMR yield using 1,3,5-trimethoxybenzene as an internal standard material.

Table S4. Screening nucleophiles

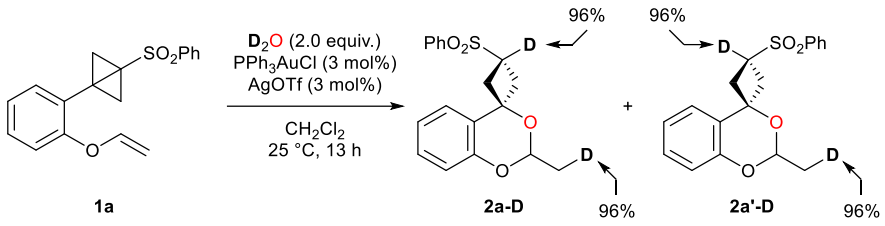


1a (0.15 mmol) desired products R : Boc, Ts, Ms, Ph

entry	nucleophiles	results
1	H_2N-Boc	complex mixture
2	H_2N-Ts	complex mixture
3	H_2N-Ms	complex mixture
4	aniline	complex mixture

3. Mechanistic Studies

3-1. Reaction using deuterium oxide

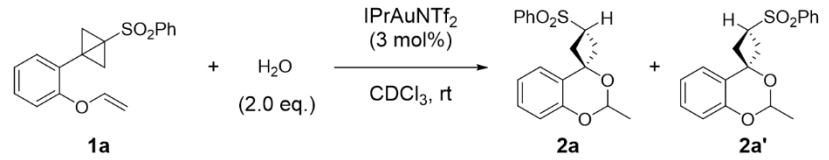


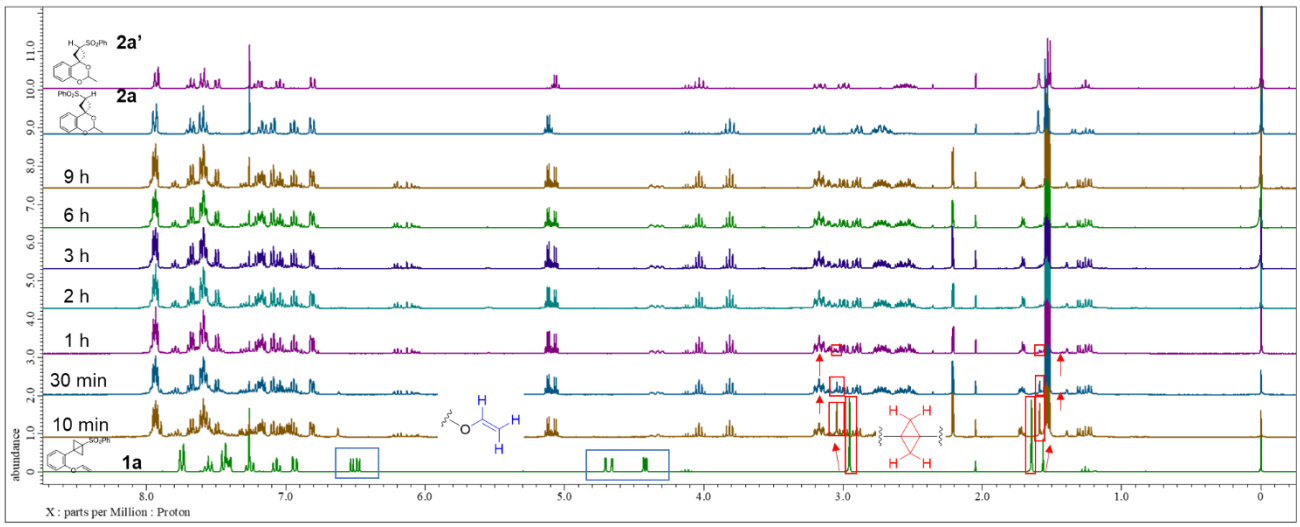
77% NMR yield (*dr* 1 : 1)

A flame dried test tube equipped with magnetic stirring bar was charged with PPh_3AuCl (2.2 mg, 4.5 μmol, 3.0 mol%) and AgOTf (1.2 mg, 4.5 μmol, 3.0 mol%) under N_2 in glovebox. Dry CH_2Cl_2 (3.0 mL, 0.05 M) and D_2O (5.4 μL, 0.30 mmol, 2.0 eq.) was added to the mixture at ambient temperature. The solution was stirred at 25 °C for 5 min, and then the compound **1a** (0.15 mmol) was added. After 13 h, the mixture was filtered through short pad of silica gel. The obtained residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 10:1) to give compounds **2a-D** and **2a'-D**.

3-2. NMR time course experiment

We conducted a time-course experiment using NMR to collect any information to shed light on the reaction mechanism. A solution of **1a** (0.15 mmol), H_2O (0.30 mmol) and 3 mol% of $IPrAuNTf_2$ in $CDCl_3$ was filled in a NMR tube. We recorded the ^1H NMR spectra of this reaction mixture at 10 and 30 min and at 1, 2, 3, 6, and 9 h after the start of the reaction and compared each spectrum with those of standard samples of **1a**, **2a** and **2a'**.



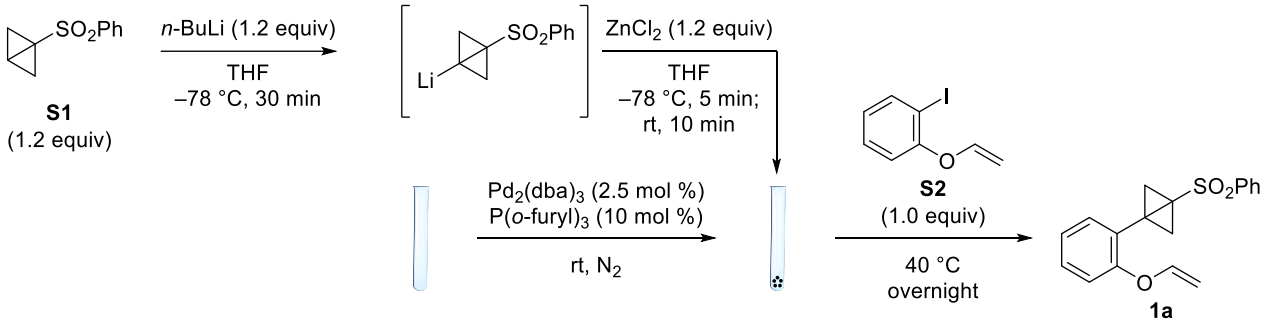


4. Experimental procedure

4-1. Preparation of starting materials

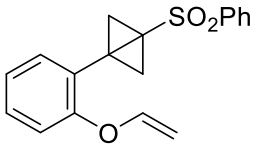
General procedure of Negishi coupling of bicyclobutane and aryl iodide

Starting materials **1** were prepared through slightly modified procedure of reported method reported by Anderson et al.[1]



Under a nitrogen atmosphere, to a solution of BCB **S1** (1.00 g, 5.15 mmol, 1.2 eq.) in THF (10.3 mL, 0.50 M) was added *n*-BuLi (1.6 M in hexane, 3.43 mL, 1.2 eq.) dropwise at –78 °C. The mixture was stirred for 30 min, then a solution of $ZnCl_2$ (1.0 M in THF, 5.2 mL, 1.2 eq.) was added, and the reaction was stirred for 5 min at –78 °C before bringing to rt, and stirred for a further 10 min. The solution of organozinc was transferred *via* cannula to a vial containing $Pd_2(dba)_3$ (98.2 mg, 107 µmol, 2.5 mol%), P(*o*-furyl)$_3$ (99.6 mg, 429 µmol, 10 mol%) and aryl iodide **S2** (1.06 g, 4.29 mmol, 1.0 eq.). The reaction mixture was stirred overnight at 40 °C, then it was filtrated through celite using EtOAc. The filtrate was washed with water, extracted with EtOAc, washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude mixture was purified by flash chromatography (hexane/EtOAc = 10:1 and toluene/hexane/EtOAc = 20:1:1) to give **1a** as pale yellow solid (818 mg, 2.62 mmol, 61% yield).

1-(phenylsulfonyl)-3-(2-(vinyloxy)phenyl)bicyclo[1.1.0]butane (**1a**)



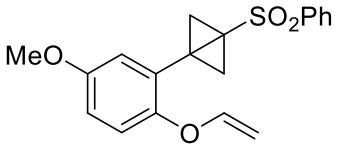
61% yield, pale yellow solid, m.p. 57.0 – 58.0 °C

^{1}H NMR (301 MHz, Acetone-d_6) δ 7.76 – 7.74 (m, 2H), 7.66 (tt, J = 7.3, 1.4 Hz, 1H), 7.56-7.52 (m, 2H), 7.45 (dd, J = 7.8, 1.4 Hz, 1H), 7.33-7.28 (m, 1H), 7.08 (td, J = 7.6, 1.1 Hz, 1H), 7.01 (dd, J = 8.0, 1.1 Hz, 1H), 6.66 (dd, J = 13.6, 6.0 Hz, 1H), 4.66 (dd, J = 13.6, 1.5 Hz, 1H), 4.43 (dd, J = 6.0, 1.5 Hz, 1H), 2.94 (s, 2H), 1.66 (s, 2H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 155.9, 148.3, 140.7, 132.9, 129.8, 128.8, 128.7, 127.3, 123.4, 120.6, 117.3, 95.2, 37.7, 33.8, 28.1.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{16}O_3NaS$ ([M+Na]$^+$): 335.0712, found 335.0710.

1-(5-methoxy-2-(vinyloxy)phenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane (**1b)**



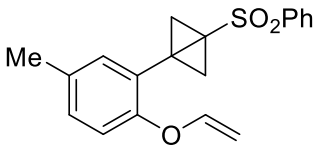
69% yield, white solid, m.p. 69.7 – 70.3 ℃

^{1}H NMR (400 MHz, Acetone-d_6) δ 7.78-7.75 (m, 2H), 7.69-7.64 (m, 1H), 7.58-7.54 (m, 2H), 7.01 (d, J = 3.1 Hz, 1H), 6.94 (d, J = 8.8 Hz, 1H), 6.86 (dd, J = 8.8, 3.1 Hz, 1H), 6.58 (dd, J = 13.7, 6.1 Hz, 1H), 4.50 (dd, J = 13.7, 1.7 Hz, 1H), 4.31 (dd, J = 6.1, 1.7 Hz, 1H), 3.76 (s, 3H), 2.91 (s, 2H), 1.66 (s, 2H).

^{13}C{^{1}H} NMR (101 MHz, Acetone-d_6) δ 155.7, 150.1, 149.7, 141.5, 133.3, 129.2, 127.2, 122.5, 119.5, 114.7, 114.0, 92.8, 55.1, 37.3, 34.0, 27.4.

sHRMS (MALDI) *m*/*z* calcd for $C_{19}H_{18}O_4NaS$ ([M+Na]$^+$): 365.0818, found 365.0818.

1-(5-methyl-2-(vinyloxy)phenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane **(1c)**



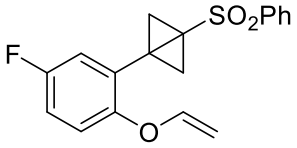
36% yield, colorless oil.

^{1}H NMR (400 MHz, Acetone-d_6) δ 7.74-7.72 (m, 2H), 7.67-7.63 (m, 1H), 7.55-7.51 (m, 2H), 7.19 (d, J = 1.8 Hz, 1H), 7.10 (dd, J = 8.2, 1.8 Hz, 1H), 6.89 (d, J = 8.2 Hz, 1H), 6.61 (dd, J = 13.6, 6.2 Hz, 1H), 4.60 (dd, J = 13.6, 1.5 Hz, 1H), 4.37 (dd, J = 6.2, 1.5 Hz, 1H), 2.93 (s, 2H), 1.63 (s, 2H).

^{13}C{^{1}H} NMR (101 MHz, Acetone-d_6) δ 154.6, 149.9, 142.1, 134.0, 133.5, 131.1, 130.1, 129.9, 128.0, 121.4, 118.2, 94.6, 37.7, 34.6, 28.3, 20.6.

HRMS (MALDI) *m*/*z* calcd for $C_{19}H_{18}O_3NaS$ ([M+Na]$^+$): 349.0869, found 349.0869.

1-(5-fluoro-2-(vinyloxy)phenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane **(1d)**



61% yield, colorless oil.

^{1}H NMR (301 MHz, $CDCl_3$) δ 7.79-7.76 (m, 2H), 7.61-7.55 (m, 1H), 7.49-7.44 (m, 2H), 7.10 (dd, J = 9.0, 2.7 Hz, 1H), 6.98-6.87 (m, 2H), 6.47 (dd, J = 13.8, 6.2 Hz, 1H), 4.63 (dd, J = 13.8, 1.9 Hz, 1H), 4.40 (dd, J = 6.2, 1.9 Hz, 1H), 2.95 (s, 2H), 1.66 (s, 2H).

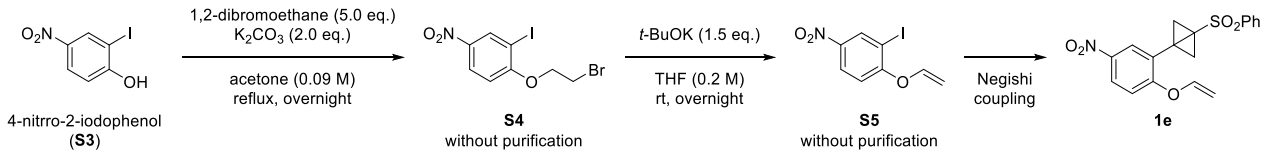
^{13}C{^{1}H} NMR (76 MHz, Acetone-d_6) δ 159.1 (d, $J_{C\text{-}F}$ = 239.9 Hz, aromatic C_α-F), 152.9 (d, $J_{C\text{-}F}$ = 2.2 Hz, aromatic C_δ), 150.0, 142.1, 134.1, 130.1, 127.9, 124.4 (d, $J_{C\text{-}F}$ = 8.7 Hz, aromatic C_γ), 120.1 (d, $J_{C\text{-}F}$ = 8.7 Hz, aromatic C_γ), 116.9 (d, $J_{C\text{-}F}$ = 24.6 Hz, aromatic C_β), 115.9 (d, $J_{C\text{-}F}$ = 23.1 Hz, aromatic C_β), 95.0, 38.3, 35.3, 27.6.

^{19}F NMR (283 MHz, Acetone-*d*6) δ -121.3.

HRMS (MALDI) *m*/*z* calcd for $C_{18}H_{15}FO_3NaS$ ([M+Na]$^+$):353.0618, found 353.0613.

1-(5-nitro-2-(vinyloxy)phenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane **(1e)**

Compound **S3** was prepared through a reported method.[2]



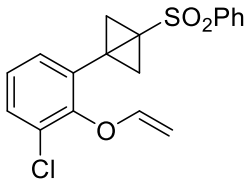
To a solution of 4-nitro-2-iodophenol (**S3**, 375 mg, 2.90 mmol, 1.0 eq.) and 1,2-dibromoethane (734 µL, 8.52 mmol, 5.0 eq.) in acetone (18.9 mL, 0.09 M) was added K_2CO_3 (471 mg, 3.41 mmol, 2.0 eq.). The resulting mixture was stirred under reflux conditions for overnight. The reaction was quenched with water and extracted with CH_2Cl_2. The organic layer was washed with brine, dried over Na_2SO_4, and concentrated to get crude compound **S4**. To a solution of crude compound in THF (14.5 mL, 0.20 M) was added *t*-BuOK (390 mg, 3.48 mmol, 1.5 eq.) portionwise. The resulting mixture was stirred at room temperature for overnight. The reaction was quenched with sat. NH_4Cl aq. and extracted with EtOAc, dried over Na_2SO_4, and concentrated to get crude compound **S5**, which was used in next step without purification. Under a nitrogen atmosphere, to a solution of BCB **S1** (676 mg, 3.48 mmol, 1.2 eq.) in THF (10.3 mL, 0.50 M) was added *n*-BuLi (1.6 M in hexane, 2.17 mL, 1.2 eq.) dropwise at –78 °C. The mixture was stirred for 30 min, then a solution of $ZnCl_2$ (1.0 M in THF, 3.48 mL, 1.2 eq.) was added, and the reaction was stirred for 5 min at –78 °C before bringing to rt, and stirred for a further 10 min. The solution of organozinc was transferred *via* cannula to a vial containing $Pd_2(dba)_3$ (72.5 µmol, 66.4 mg, 2.5 mol%), P(*o*-furyl)$_3$ (67.3 mg, 67.3 µmol, 10 mol%) and aryl iodide **S5** (1.0 eq.). The reaction mixture was stirred overnight at 40 °C, then it was filtrated through celite using EtOAc. The filtrate was washed with water, extracted with EtOAc, washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude mixture was purified by flash chromatography (hexane/EtOAc = 10:1 and toluene/hexane/EtOAc = 20:1:1) to give the BCB derivative **1e** as colorless oil (139 mg, 377 µmol, 3 steps: 13% yield).

^{1}H NMR (500 MHz, Acetone-d_6) δ 8.24 (d, J = 2.8 Hz, 1H), 8.19 (dd, J = 9.0, 2.8 Hz, 1H), 7.80-7.78 (m, 2H), 7.68-7.64 (m, 1H), 7.58-7.54 (m, 2H), 7.27 (d, J = 9.0 Hz, 1H), 6.88 (dd, J = 13.3, 5.9 Hz, 1H), 5.01 (dd, J = 13.3, 1.7 Hz, 1H), 4.74 (dd, J = 5.9, 1.7 Hz, 1H), 3.12 (s, 2H), 1.79 (s, 2H).

^{13}C{^{1}H} NMR (76 MHz, Acetone-d_6) δ 160.5, 146.6, 142.9, 141.1, 133.5, 129.3, 127.2, 125.6, 124.4, 122.2, 115.6, 98.6, 37.5, 34.6, 26.9.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{15}NO_5NaS$ ([M+Na]$^+$): 380.0563, found 380.0566.

1-(3-chloro-2-(vinyloxy)phenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane **(1f)**



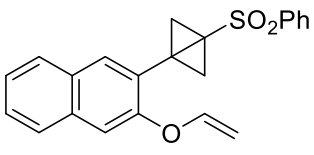
53% yield, colorless oil.

^{1}H NMR (301 MHz, $CDCl_3$) δ 7.78-7.75 (m, 2H), 7.63-7.57 (m, 1H), 7.50-7.45 (m, 3H), 7.36 (dd, J = 8.0, 1.5 Hz, 1H), 7.13 (t, J = 8.0 Hz, 1H), 6.47 (dd, J = 13.9, 6.4 Hz, 1H), 4.27-4.18 (m, 2H), 2.87 (s, 2H), 1.67 (s, 2H).

^{13}C{^{1}H} NMR (76 MHz, Acetone-d_6) δ 151.4, 151.2, 142.1, 134.2, 130.5, 130.1, 128.9, 128.7, 128.0, 127.8, 126.8, 91.6, 38.7, 35.9, 27.3.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{15}O_3NaSCl$ ([M+Na]$^+$): 369.0323, found 369.0326.

2-(3-(phenylsulfonyl)bicyclo[1.1.0]butan-1-yl)-3-(vinyloxy)naphthalene (**1g**)



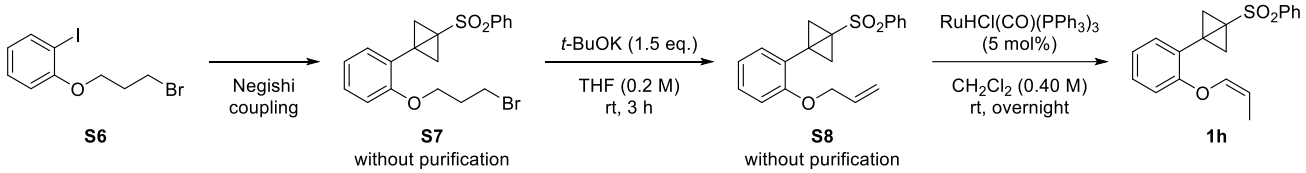
42% yield, Light brown solid, m.p. 82.2-83.2 °C.

^{1}H NMR (400 MHz, Acetone-d_6) δ 7.95 (s, 1H), 7.83 (d, J = 8.2 Hz, 1H), 7.80 (d, J = 8.7 Hz, 1H) 7.60-7.55 (m, 2H), 7.60-7.55(m, 1H), 7.50-7.40 (m, 5H), 6.83 (dd, J = 13.7, 6.0 Hz, 1H), 4.87 (dd, J = 13.7, 1.4 Hz, 1H), 4.57 (dd, J = 6.0, 1.4 Hz, 1H), 3.12 (s, 2H), 1.71 (s, 2H).

$^{13}C\{^1H\}$ NMR (101 MHz, Acetone-d_6) δ 154.8, 148.7, 142.1, 134.5, 133.9, 130.9, 130.6, 129.9, 128.1, 128.1, 127.9, 127.5, 125.8, 122.2, 112.2, 96.6, 37.8, 34.9, 30.6.

HRMS (MALDI) *m/z* calcd for $C_{22}H_{18}O_3NaS$ ([M+Na]$^+$): 385.0869, found 385.0871.

(Z)-1-(phenylsulfonyl)-3-(2-(prop-1-en-1-yloxy)phenyl)bicyclo[1.1.0]butane (**1h**)



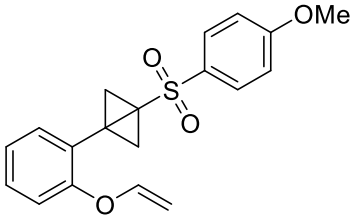
Under a nitrogen atmosphere, to a solution of BCB **S1** (820 mg, 4.22 mmol, 1.2 eq.) in THF (10.3 mL, 0.50 M) was added *n*-BuLi (1.6 M in hexane, 2.64 mL, 1.2 eq.) dropwise at –78 °C. The mixture was stirred for 30 min, then a solution of $ZnCl_2$ (1.0 M in THF, 4.22 mL, 1.2 eq.) was added, and the reaction was stirred for 5 min at –78 °C before bringing to rt, and stirred for a further 10 min. The solution of organozinc was transferred *via* cannula to a vial containing $Pd_2(dba)_3$ (80.4 mg, 88.0 µmol, 2.5 mol%), P(*o*-furyl)$_3$ (81.7 mg, 352 µmol, 10 mol%) and 1-(3-bromopropoxy)-2-iodobenzene **S6** (1.20 g, 3.52 mmol, 1.0 eq.). The reaction mixture was stirred overnight at 40 °C, then it was filtrated through celite using EtOAc. The filtrate was washed with water, extracted with EtOAc, washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The mixture was filtered through a short pad of silica gel to get crude compound **S8** which was used in next step without purification. To a solution of crude compound **S8** in THF (17.6 mL, 0.20 M) was added *t*-BuOK (474 mg, 4.22 mmol, 1.5 eq.) portionwise. The resulting mixture was stirred at room temperature for overnight. The reaction was quenched with water and sat. NH_4Cl aq. and extracted with EtOAc, dried over Na_2SO_4, and concentrated to get crude compound **S8** which was used in next step without purification. Then to a solution of crude compound **S8** in CH_2Cl_2 (17.5 mL, 0.20 M) was added $RuHCl(CO)(PPh_3)_3$ (166 mg, 0.175 mmol, 5 mol%). The resulting mixture was stirred at room temperature for overnight. The reaction mixture was filtered and combined filtrate was concentrated. The residue was purified by a silica gel column chromatography (hexane/EtOAc = 10:1 and toluene/hexane/EtOAc = 20:1:1) to get compound **1h** as colorless oil (200 mg, 613 µmol, 3 steps: 40%).

^1H NMR (400 MHz, Acetone-d_6) δ 7.74-7.70 (m, 2H), 7.65-7.60 (m, 1H), 7.53-7.49 (m, 2H), 7.40-7.38 (m, 1H), 7.28-7.24 (m, 1H), 7.01-6.95 (m, 2H), 6.36 (dq, J = 6.5, 1.7 Hz, 1H), 4.91-4.84 (m, 1H), 2.97 (s, 2H), 1.68 (dd, J = 6.5, 1.7 Hz, 3H), 1.65 (s, 2H).

$^{13}C\{^1H\}$ NMR (101 MHz, Acetone-d_6) δ 157.6, 142.2, 142.0, 133.9, 130.8, 130.0, 129.6, 127.9, 123.2, 120.9, 116.3, 107.6, 37.9, 34.4, 28.6, 9.6.

HRMS (MALDI) *m/z* calcd for $C_{19}H_{18}O_3NaS$ ([M+Na]$^+$): 349.0869, found 349.0867.

1-((4-methoxyphenyl)sulfonyl)-3-(2-(vinyloxy)phenyl)bicyclo[1.1.0]butane (**1i**)



65% yield, colorless oil.

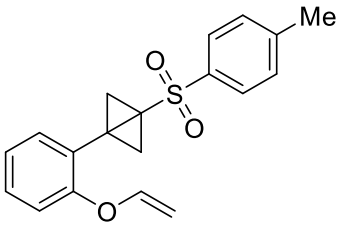
^1H NMR (400 MHz, Acetone-d_6) δ 7.64-7.60 (m, 2H), 7.42-7.40 (m, 1H), 7.32-7.27 (m, 1H), 7.06 (td, J = 7.7, 1.2 Hz, 1H), 7.02-6.98 (m, 3H), 6.64 (dd, J = 13.5, 6.2 Hz, 1H), 4.65 (dd, J = 13.5, 1.6 Hz, 1H), 4.42 (dd, J = 6.2, 1.6 Hz, 1H), 3.87

(s, 3H), 2.89 (s, 2H), 1.61 (s, 2H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 163.2, 155.9, 148.3, 132.2, 129.8, 129.6, 128.6, 123.4, 120.9, 117.3, 114.0, 95.2, 55.6, 37.4, 34.5, 27.5.

HRMS (MALDI) *m/z* calcd for $C_{19}H_{18}O_4NaS$ ($[M+Na]^+$): 365.0818, found 365.0819.

1-tosyl-3-(2-(vinyloxy)phenyl)bicyclo[1.1.0]butane **(1j)**



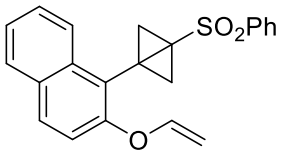
45% yield, colorless oil.

^1H NMR (400 MHz, Acetone-d_6) δ 7.60 (d, J = 8.2 Hz, 2H), 7.44-7.42 (m, 1H), 7.33-7.27 (m, 3H), 7.09-7.05 (m, 1H), 7.00 (d, J = 8.2 Hz, 1H), 6.64 (dd, J = 13.7, 6.1 Hz, 1H), 4.65 (dd, J = 13.7, 1.5 Hz, 1H), 4.42 (dd, J = 6.1, 1.5 Hz, 1H), 2.91 (s, 2H), 2.40 (s, 3H), 1.62 (s, 2H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 150.2, 141.8, 133.3, 133.0, 129.9, 129.1, 129.0, 127.6, 127.3, 127.1, 125.8, 91.4, 38.4, 38.2, 23.0, 12.6.

HRMS (MALDI) *m/z* calcd for $C_{19}H_{18}O_3NaS$ ($[M+Na]^+$): 349.0869, found 349.0866.

1-(3-(phenylsulfonyl)bicyclo[1.1.0]butan-1-yl)-2-(vinyloxy)naphthalene (**1k**)



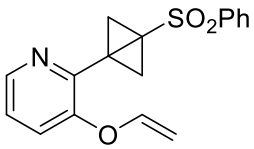
57% yield, white solid, m.p. 70.7 – 72.7 °C.

^1H NMR (301 MHz, $CDCl_3$) δ 8.21 (d, J = 8.6 Hz, 1H), 7.94-7.90 (m, 2H), 7.82 (d, J = 8.6 Hz, 2H), 7.59-7.46 (m, 4H), 7.43-7.38 (m, 1H), 7.31-7.28 (m, 1H), 6.76 (dd, J = 13.9, 6.0 Hz, 1H), 4.96 (dd, J = 13.9, 1.5 Hz, 1H), 4.60 (dd, J = 6.0, 1.5 Hz, 1H), 2.85 (s, 2H), 1.70 (s, 2H).

$^{13}C\{^1H\}$ NMR (76 MHz, Acetone-d_6) δ 152.8, 148.3, 143.0, 135.1, 133.1, 130.6, 130.0, 129.3, 128.7, 127.2, 127.0, 124.5, 124.2, 116.7, 114.1, 95.8, 37.6, 34.8, 28.1.

HRMS (MALDI) *m/z* calcd for $C_{22}H_{18}O_3NaS$ ($[M+Na]^+$): 385.0869, found 385.0868.

2-(3-(phenylsulfonyl)bicyclo[1.1.0]butan-1-yl)-3-(vinyloxy)pyridine (**1l**)



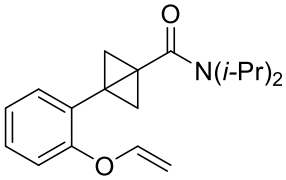
77% yield, brown oil.

^1H NMR (301 MHz, Acetone-d_6) δ 8.20-8.18 (m, 1H), 7.66-7.61 (m, 3H), 7.51-7.46 (m, 2H), 7.37 (d, J = 8.3, 1H), 7.28-7.24 (m, 1H), 6.64 (dd, J = 13.7, 5.9 Hz, 1H), 4.75 (dd, J = 13.7, 1.7 Hz, 1H), 4.53 (dd, J = 5.9, 1.7 Hz, 1H), 3.28 (s, 2H), 1.78 (s, 2H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 152.5, 147.5, 143.9, 142.0, 140.6, 133.0, 128.9, 127.3, 124.1, 122.8, 97.1, 38.3, 35.9, 29.3.

HRMS (MALDI) *m/z* calcd for $C_{17}H_{16}NO_3S$ ($[M+H]^+$): 314.0845, found 314.0849.

N,*N*-diisopropyl-3-(2-(vinyloxy)phenyl)bicyclo[1.1.0]butane-1-carboxamide (**1m**)



83% yield, yellow oil.

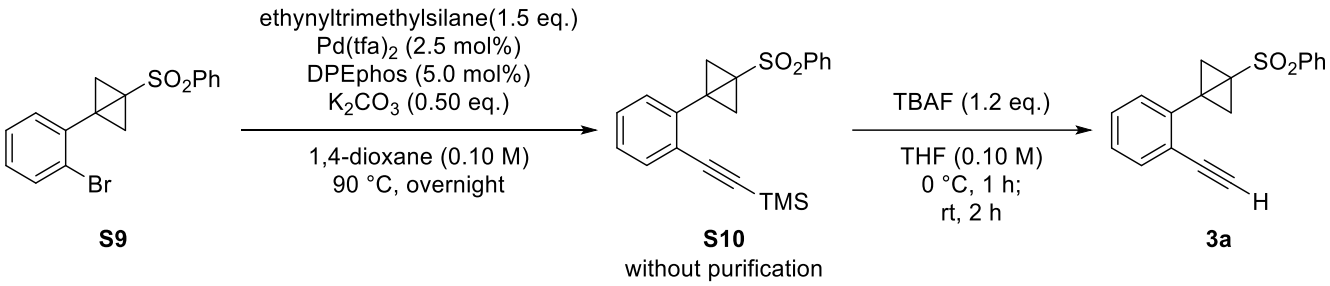
^1H NMR (500 MHz, Acetone-d_6) δ 7.34 (dd, J = 7.7, 1.4 Hz, 1H), 7.19-7.15 (m, 1H), 7.03-7.00 (m, 1H), 6.92-6.91 (m, 1H), 6.62 (dd, J = 13.7, 6.0 Hz, 1H), 4.81 (br, 1H), 4.65 (dd, J = 13.7, 1.7 Hz, 1H), 4.41 (dd, J = 6.0, 1.7 Hz, 1H), 3.39 (br, 1H), 2.71 (s, 2H), 1.33

(s, 2H), 1.23 (d, J = 6.9 Hz, 12H).

^{13}C{^{1}H} NMR (126 MHz, Acetone-d_6) δ 167.0, 156.5, 149.8, 131.8, 128.2, 125.9, 123.8, 117.3, 95.1, 49.9, 46.2, 37.8, 25.5, 21.4, 21.1, 20.1.

HRMS (MALDI) m/z calcd for $C_{19}H_{26}NO_2$ ([M+H]$^+$): 300.1958, found 300.1956.

1-(2-ethynylphenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane (**3a**)



S9 **S10** without purification **3a**

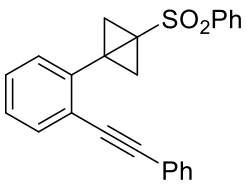
A flame dried test tube equipped with magnetic stirring bar was charged with the compound **S9** (460 mg, 1.32 mmol, 1.0 eq.), K_2CO_3 (91 mg, 0.66 mmol, 0.50 eq.), DPEphos (35.5 mg, 66 µmol, 5 mol%) and $Pd(tfa)_2$ (10.9 mg, 33 µmol, 2.5 mol%) under N_2 and dry 1,4-dioxane (13 mL, 0.10 M) was added. To a solution added ethynyltrimethylsilane (270 µL, 1.98 mmol, 1.5 eq.), then the mixture was stirred at 90 ℃ overnight. After completion of the reaction, the mixture was filtered and, concentrated to afford crude compound **S10**. To a solution of crude compound **S10** in THF (13.2 mL, 0.10 M) added TBAF (1.56 mL, 1.58 mmol, 1.0 M in THF solution, 1.2 eq.). The resulting mixture was stirred at 0 ℃ for 1 h followed by further stirring for 2 h at room temperature. The reaction mixture was quenched with water and extracted with CH_2Cl_2. The combined organic layer dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 10:1) to give compound **3a** as yellow oil (160 mg, 2 steps : 41%).

^{1}H NMR (301 MHz, Acetone-d_6) δ 7.86-7.82 (m, 2H), 7.73-7.67 (m, 1H), 7.64-7.57 (m, 3H), 7.50 (dd, J = 7.6, 1.6 Hz, 1H), 7.38 (td, J = 7.6, 1.6 Hz, 1H), 7.31 (td, J = 7.6, 1.6 Hz, 1H), 3.83 (s, 1H), 3.00 (s, 2H), 1.79 (s, 2H).

^{13}C{^{1}H} NMR (76 MHz, Acetone-d_6) δ 142.6, 134.7, 134.2, 134.2, 130.1, 129.5, 128.8, 128.2, 127.9, 124.1, 83.5, 83.0, 39.2, 36.1, 30.4.

HRMS (MALDI) m/z calcd for $C_{18}H_{14}O_2NaS$ ([M+Na]$^+$): 317.0607, found 317.0600.

1-(2-(phenylethynyl)phenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane (**3b**)



A flame dried test tube equipped with magnetic stirring bar was charged with a compound **S9** (348 mg, 1.0 mmol, 1.0 eq.), TEA (560 µL, 4.0 mmol, 4.0 eq.), CuI (38 mg, 0.20 mmol, 20 mol%) and $(PPh_3)_2PdCl_2$ (70 mg, 0.10 mmol, 10 mol%) under N_2 and dry DMF (10 mL, 0.10 M) was added. To the solution added ethynylbenzene (164 µL, 1.5 mmol, 1.5 eq.), then the mixture was stirred at 90 ℃ overnight. After completion of the reaction, the mixture was filtered and concentrated. The residue purified by flash column chromatography on silica gel (hexane/EtOAc = 10:1) to get **3b** as brown oil (209 mg, 564 µmol, 56%).

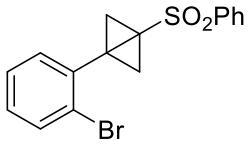
^{1}H NMR (400 MHz, Acetone-d_6) δ 7.85 (d, J = 8.2 Hz, 2H), 7.69-7.64 (m, 2H), 7.59-7.55 (m, 5H), 7.44-7.39 (m,

3H), 7.37-7.31 (m, 2H), 3.10 (s, 2H), 1.86 (s, 2H).

$^{13}C\{^{1}H\}$ NMR (101 MHz, Acetone-d_6) δ 142.4, 134.1, 134.0, 133.5, 132.0, 130.1, 129.9, 129.4, 129.2, 128.9, 128.2, 127.9, 124.7, 123.8, 94.0, 89.1, 39.0, 36.1, 30.7.

HRMS (MALDI) *m*/*z* calcd for $C_{24}H_{18}O_2NaS$ ($[M+Na]^+$): 393.0920, found 393.0920.

1-(2-bromophenyl)-3-(phenylsulfonyl)bicyclo[1.1.0]butane (**S9**)



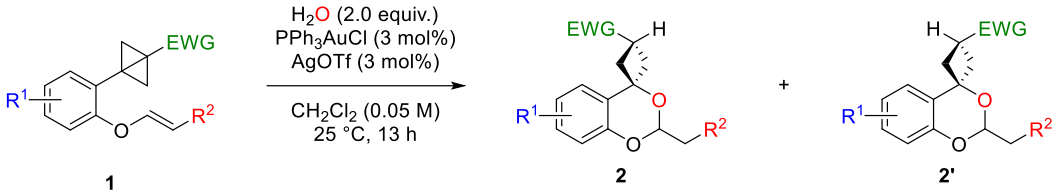
83% yield, pale yellow oil.

^{1}H NMR (301 MHz, Acetone-d_6) δ 7.96-7.93 (m, 2H), 7.82 (dd, J = 7.9, 1.7 Hz, 1H), 7.77-7.71 (m, 1H), 7.69-7.63 (m, 3H), 7.46-7.37 (m, 1H), 7.34-7.25 (m, 1H), 2.71 (s, 2H), 1.80 (s, 2H).

$^{13}C\{^{1}H\}$ NMR (101 MHz, Acetone-d_6) δ 142.2, 133.5, 133.3, 131.8, 130.0, 129.6, 129.5, 127.7, 127.1, 125.8, 39.5, 34.8, 31.8.

HRMS (MALDI) *m*/*z* calcd for $C_{16}H_{13}BrO_2NaS$ ($[M+Na]^+$): 370.9712, found 370.9712.

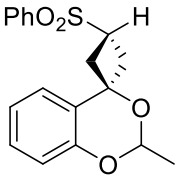
4-2. Heteroannulation of bicyclobutane derivatives via Au-catalyzed addition of water to enol ethers followed by intramolecular nucleophilic addition



General procedure

A flame dried test tube equipped with magnetic stirring bar was charged with PPh_3AuCl (2.2 mg, 4.5 μmol, 3.0 mol%) and AgOTf (1.2 mg, 4.5 μmol, 3.0 mol%) under N_2 in glovebox. Dry CH_2Cl_2 (3.0 mL, 0.05 M) and H_2O (5.4 μL, 0.30 mmol, 2.0 eq.) was added to the mixture at ambient temperature. The solution was stirred at 25 °C for 5 min, and then the compound **1** (0.15 mmol) was added. After 13 h, the mixture was filtered through short pad of silica gel. The obtained residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 10:1) to give compounds **2** and **2'**.

(3'r,4r)-2-methyl-3'-(phenylsulfonyl)spiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2a**)



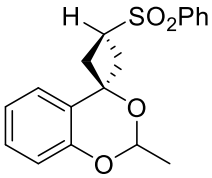
40% yield, pale yellow solid, m.p. 149.5-150.3 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.92 (m, 2H), 7.70-7.66 (m, 1H), 7.61-7.57 (m, 2H), 7.49 (dd, J = 7.8, 1.4 Hz, 1H), 7.22-7.17 (m, 1H), 7.04 (td, J = 7.6, 1.1 Hz, 1H), 6.81 (dd, J = 8.0, 1.1 Hz, 1H), 5.06 (q, J = 5.1 Hz, 1H), 4.07-3.99 (m, 1H), 3.16 (dd, J = 13.1, 8.9 Hz, 1H), 2.99 (dd, J = 13.1, 8.9 Hz, 1H), 2.62-2.48 (m, 2H), 1.52 (d, J = 5.1 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.2, 138.0, 133.9, 129.4, 129.0, 128.2, 126.3, 125.6, 121.9, 116.3, 93.1, 75.3, 50.9, 39.5, 37.1, 20.7.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{18}O_4NaS$ ($[M+Na]^+$): 353.0818, found 353.0820.

(3's,4s)-2-methyl-3'-(phenylsulfonyl)spiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2a'**)



45% yield, pale yellow solid, m.p. 139.1-141.1 °C.

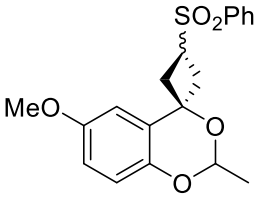
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.71-7.66 (m, 1H), 7.62-7.57 (m, 2H), 7.19-7.15 (m, 1H), 7.09 (dd, J = 7.8, 1.3 Hz, 1H), 6.94 (td, J = 7.6, 1.3 Hz, 1H), 6.81 (dd, J = 8.2 Hz, 1H), 5.11 (q, J = 5.0 Hz, 1H), 3.85-3.77 (m, 1H), 3.20-3.15 (m, 1H), 2.93-2.88 (m, 1H), 2.78-2.66 (m, 2H), 1.54 (d, J = 5.0 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 151.9, 137.9, 133.9, 129.4, 128.8, 128.4, 126.4, 123.3, 121.6, 116.8, 92.7, 72.1, 49.6, 40.4, 37.3, 20.7.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{18}O_4NaS$ ($[M+Na]^+$): 353.0818, found 353.0817.

6-methoxy-2-methyl-3'-(phenylsulfonyl)spiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2b, 2b'**) (diastereomixture)

60% yield (**2b**:**2b'** = 0.80:1), colorless oil.



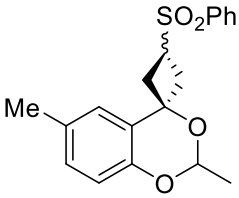
^{1}H NMR (301 MHz, $CDCl_3$) δ 7.91-7.88 (m, 2H), 7.68-7.61 (m, 1H), 7.58-7.52 (m, 2H), 7.07 (d, J = 2.4 Hz, 0.53H), 6.76-6.68 (m, 2H), 6.60 (m, 0.41H), 5.02 (q, J = 4.7 Hz, 0.45H), 4.97 (q, J = 5.0 Hz, 0.58H), 4.00 (quin., J = 8.6 Hz, 0.63H), 3.79 (m, 2.3H), 3.71 (s, 1.2H), 3.13 (dd, J = 13.2, 8.6 Hz, 1H), 2.97 (dd, J = 13.2, 8.6 Hz, 0.55H), 2.85 (dd, J = 12.0, 8.6 Hz, 0.45H), 2.75-2.44 (m, 2H), 1.49-1.46 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 154.3, 154.1, 146.1, 145.8, 137.8, 137.7, 133.8, 133.8, 129.3, 128.2, 128.1, 127.0, 126.1, 117.2, 117.0, 115.5, 113.6, 110.2, 109.1, 93.0, 92.6, 75.1, 72.0, 55.7, 50.7, 49.5, 40.3, 39.4, 37.1, 37.0, 20.6, 20.5.

HRMS (MALDI) *m/z* calcd for $C_{19}H_{20}O_5NaS$ ([M+Na]$^+$): 383.0924, found 383.0925.

2,6-dimethyl-3'-(phenylsulfonyl)spiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2c, 2c'**) (diastereomixture)

63% (**2c**:**2c'** = 1.2:1), colorless oil.



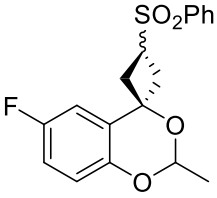
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.91 (m, 2H), 7.70-7.65 (m, 1H), 7.60-7.56 (m, 2H), 7.17 (d, J = 1.4 Hz, 0.45H), 6.99-6.94 (m, 1H), 6.85 (d, J = 1.8 Hz, 0.55H), 6.69 (dd, J = 8.2, 1.8 Hz, 1H), 5.06 (q, J = 5.0 Hz, 0.55H), 5.01 (q, J = 5.0 Hz, 0.45H), 4.06-3.97 (m, 0.45H), 3.85-3.76 (m, 0.55H), 3.17-3.10 (m, 1H), 2.97-2.85 (m, 1H), 2.75-2.64 (m, 1H), 2.58-2.44 (m, 1H), 2.29-2.38 (s, 1.4H), 2.20-2.29 (s, 1.7H), 1.52(m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 150.1, 149.7, 137.9, 137.9, 133.9, 133.9, 131.2, 130.9, 129.7, 129.5, 129.4, 128.4, 128.3, 126.3, 126.0, 125.2, 123.5, 116.5, 116.1, 93.1, 92.6, 75.3, 72.1, 51.1, 49.6, 40.4, 39.5, 37.2, 37.1, 20.8, 20.8, 20.72, 20.65.

HRMS (MALDI) *m/z* calcd for $C_{19}H_{20}O_4NaS$ ([M+Na]$^+$): 367.0975, found 37.0971.

6-fluoro-2-methyl-3'-(phenylsulfonyl)spiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2d, 2d'**) (diastereomixture)

92% yield (**2d**:**2d'** = 0.60:1), colorless oil.



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.91 (m, 1H), 7.70-7.67 (m, 1H), 7.61-7.57 (m, 1H), 7.19 (dd, J = 8.9, 3.0 Hz, 0.62H), 6.92-6.85 (m, 1H), 6.81-6.78 (m, 0.38H), 6.78-6.73 (m, 1H), 5.06 (q, J = 5.2 Hz, 0.38H), 5.01 (q, J = 5.2 Hz, 0.62H), 4.07-3.98 (m, 0.62H), 3.82-3.73 (m, 0.38H), 3.18 (dd, J = 13.3, 9.2 Hz, 0.38H), 3.09 (dd, J = 13.3, 8.7 Hz, 0.62H), 2.98-2.88 (m, 1H), 2.76-2.48 (m, 2H), 1.53 (d, J = 5.0 Hz, 1.1H), 1.51 (d, J = 5.5 Hz, 1.9H).

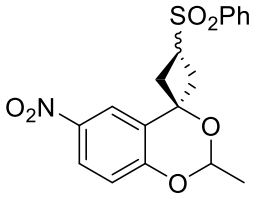
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 157.48 (d, $J_{C\text{-}F}$ = 241 Hz), 157.32 (d, $J_{C\text{-}F}$ = 241 Hz), 148.3, 148.0, 137.8, 137.7, 134.0, 129.4, 128.3, 128.2, 127.2 (d, $J_{C\text{-}F}$ = 6.7 Hz), 126.6 (d, $J_{C\text{-}F}$ = 6.7 Hz), 118.0 (d, $J_{C\text{-}F}$ = 7.7 Hz), 117.5 (d, $J_{C\text{-}F}$ = 7.7 Hz), 116.2 (d, $J_{C\text{-}F}$ = 23 Hz), 115.9 (d, $J_{C\text{-}F}$ = 23 Hz), 112.5 (d, $J_{C\text{-}F}$ = 24 Hz), 109.7 (d, $J_{C\text{-}F}$ = 24 Hz), 93.3, 92.9, 74.9, 72.0, 50.7, 49.4, 40.2, 39.4, 37.12, 37.09, 20.60, 20.54.

^{19}F NMR (283 MHz, $CDCl_3$) δ -123.642, -124.378.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{17}O_4FNaS$ ([M+Na]$^+$): 371.0724, found 371.0725.

2-methyl-6-nitro-3'-(phenylsulfonyl)spiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2e, 2e'**) (diastereomixture)

12% yield (**2e**/**2e'** = 0.36:1), colorless oil.



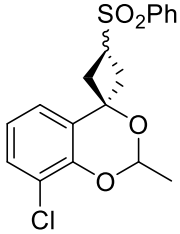
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, J = 2.7 Hz, 0.75H), 8.10-8.06 (m, 1H), 8.02 (d, J = 2.7 Hz, 0.25H), 7.96-7.93 (m, 2H), 7.73-7.68 (m, 1H), 7.63-7.59 (m, 2H), 6.92-6.88 (m, 1H), 5.21 (q, J = 5.0 Hz, 0.25H), 5.13 (q, J = 5.2 Hz, 0.70H), 4.10-4.01 (m, 0.72H), 3.94-3.85 (m, 0.22H), 3.27-3.21 (m, 0.23H), 3.16 (dd, J = 13.3, 8.7 Hz, 0.74H), 3.00-2.93 (m, 1H), 2.78-2.55 (m, 2H), 1.60 (d, J = 5.0 Hz, 0.70H), 1.57 (d, J = 5.0 Hz, 2.2H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 157.3, 157.2, 142.1, 141.7, 137.6, 137.3, 134.2, 129.6, 129.5, 128.4, 126.6, 125.8, 124.9, 122.6, 119.8, 117.6, 117.2, 93.9, 93.6, 75.1, 72.2, 50.6, 48.9, 39.7, 39.3, 36.9, 36.7, 20.5, 20.4.

HRMS (MALDI) *m*/*z* calcd for $C_{18}H_{17}NO_6NaS$ ([M+Na]$^+$): 398.0669, found 398.0666.

8-chloro-2-methyl-3'-(phenylsulfonyl)spiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2f, 2f'**) (diastereomixture)

50% yield (**2f**:**2f'** = 0.67:1), colorless oil.



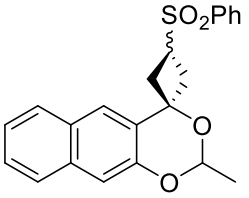
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.89 (m, 2H), 7.69-7.65 (m, 1H), 7.60-7.55 (m, 2H), 7.43 (dd, J = 7.8, 1.4 Hz, 0.60H), 7.27-7.22 (m, 1H), 7.00-6.94 (m, 1H), 6.86 (t, J = 8.0 Hz, 0.40H), 5.15 (q, J = 5.2 Hz, 0.40H), 5.08 (q, J = 5.2 Hz, 0.6H), 4.06-3.97 (m, 0.6H), 3.83-3.74 (m, 0.4H), 3.20-3.12 (m, 1H), 3.01-2.87 (m, 1H), 2.75-2.48 (m, 2H), 1.59 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 148.3, 148.0, 137.9, 137.7, 134.0, 134.0, 129.4, 129.4, 129.3, 128.4, 128.2, 127.9, 127.3, 124.8, 121.9, 121.6, 121.6, 121.1, 93.8, 93.5, 75.1, 72.1, 50.8, 49.4, 40.2, 39.5, 37.1, 20.6, 20.5.

HRMS (MALDI) *m*/*z* calcd for $C_{18}H_{17}O_4NaSCl$ ([M+Na]$^+$): 387.0428, found 387.0424.

2'-methyl-3-(phenylsulfonyl)spiro[cyclobutane-1,4'-naphtho[2,3-*d*][1,3]dioxine] (**2g, 2g'**) (diastereomixture)

48% yield (**2g**:**2g'** = 0.55:1), colorless oil.



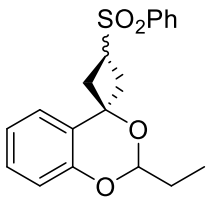
^{1}H NMR (301 MHz, $CDCl_3$) δ 7.99-7.96 (m, 2.7H), 7.93 (s, 0.7H), 7.73-7.57 (m, 4.7H), 7.45-7.29 (m, 2.1H), 7.20 (s, 1H), 5.23 (q, J = 5.2 Hz, 0.33H), 5.16 (q, J = 5.0 Hz, 0.69H), 4.14-4.05 (m, 0.68H), 4.02-3.94 (m, 0.34H), 3.35-3.23 (m, 1H), 3.10-2.95 (m, 1H), 2.90-2.60 (m, 2H), 1.61-1.57 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 150.3, 150.0, 137.9, 137.8, 134.0, 133.9, 133.8, 129.4, 129.1, 128.8, 128.4, 128.3, 128.0, 127.9, 127.4, 127.1, 126.7, 126.6, 126.6, 126.4, 126.0, 124.3, 122.4, 111.7, 111.3, 93.3, 92.9, 75.7, 72.5, 51.0, 49.7, 40.7, 40.2, 37.8, 37.6, 20.9, 20.8.

HRMS (MALDI) *m*/*z* calcd for $C_{22}H_{20}O_4NaS$ ([M+Na]$^+$): 403.0975, found 403.0973.

2-ethyl-3'-(phenylsulfonyl)spiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2h, 2h'**) (diastereomixture)

63% (**2h**:**2h'** = 0.89:1), colorless oil.



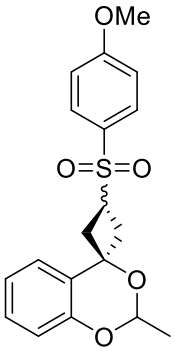
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.92 (m, 2H), 7.70-7.65 (m, 1H), 7.61-7.56 (m, 2H), 7.49-7.47 (m, 0.57H), 7.27-7.23 (m, 2H), 7.09 (dd, J = 7.8, 1.8 Hz, 0.45H), 7.0-7.01 (m, 1H), 6.95-6.92 (m, 0.45H), 6.83-6.80 (m, 1H), 4.88 (t, J = 5.0 Hz, 0.47H), 4.83 (t, J = 5.0 Hz, 0.53H),

4.06-3.98 (m, 0.52H), 3.87-3.78 (m, 0.43H), 3.16 (dd, J = 13.3, 9.2 Hz, 1H), 3.00-2.97 (m, 0.55H), 2.88-2.83 (m, 0.46H), 2.78-2.66 (m, 1H), 2.62-2.47 (m, 1H), 1.89-1.77 (m, 2H), 1.07-1.01 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, Acetone-d_6) δ 152.5, 152.2, 138.1, 138.0, 134.0, 134.0, 129.5, 129.1, 129.0, 128.9, 128.5, 128.3, 126.7, 126.4, 126.0, 125.4, 123.4, 121.9, 121.6, 116.9, 116.5, 97.0, 96.5, 75.4, 72.3, 51.1, 49.7, 40.5, 39.5, 37.4, 37.3, 27.52, 27.50, 21.6, 8.0, 7.9.

HRMS (MALDI) m/z calcd for $C_{19}H_{20}O_4NaS$ ([M+Na]$^+$): 367.0975, found 367.0974.

3'-((4-methoxyphenyl)sulfonyl)-2-methylspiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2i, 2i'**) (diastereomixture)



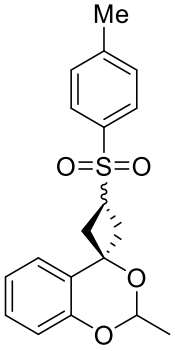
quant. (**2i**:**2i'** = 0.78:1), colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.82 (m, 2H), 7.48 (dd, J = 7.8, 1.4 Hz, 058H), 7.21-7.14 (m, 1H), 7.09 (dd, J = 7.8, 1.4 Hz, 0.40H), 7.04-7.01 (m, 3H), 6.96-6.91 (m, 0.38H), 6.81-6.76 (m, 1H), 5.11 (q, J = 5.2 Hz, 0.46H), 5.06 (q, J = 5.2 Hz, 0.59H), 4.03-3.95 (m, 0.53H), 3.88 (s, 3H), 3.82-3.74 (m, 0.41H), 3.16-3.10 (m, 1H), 2.96 (dd, J = 13.1, 8.9 Hz, 0.62H), 2.86 (dd, J = 12.4, 8.7 Hz, 0.40H), 2.76-2.64 (m, 0.88H), 2.61-2.47 (m, 1.2H), 1.54-1.51 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, Acetone-d_6) δ 163.9, 163.9, 152.2, 151.9, 130.5, 130.4, 129.4, 129.3, 128.9, 128.8, 126.5, 126.4, 125.7, 123.3, 121.8, 121.6, 116.7, 116.2, 114.6, 93.1, 92.7, 75.2, 72.1, 55.7, 51.1, 49.8, 40.4, 39.6, 37.3, 37.1, 20.71, 20.65.

HRMS (MALDI) m/z calcd for $C_{19}H_{20}O_5NaS$ ([M+Na]$^+$): 383.0924, found 383.0921.

2-methyl-3'-tosylspiro[benzo[*d*][1,3]dioxine-4,1'-cyclobutane] (**2j, 2j'**) (diastereomixture)



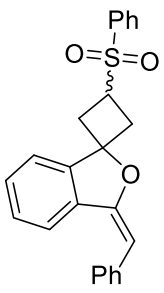
72% yield (**2j**:**2j'** = 0.87:1), colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.79 (m, 2H), 7.49 (dd, J = 7.8, 1.4 Hz, 0.52H), 7.37 (dd, J = 8.2, 2.7 Hz, 2H), 7.21-7.14 (m, 1H), 7.09 (dd, J = 8.0, 1.6 Hz, 0.40H), 7.06-7.02 (m, 0.58H), 6.96-6.92 (m, 0.42H), 6.82-6.79 (m, 1H), 5.11 (q, J = 5.0 Hz, 0.41H), 5.06 (q, J = 5.0 Hz, 0.52H), 4.05-3.96 (m, 0.59H), 3.83-3.74 (m, 0.40H), 3.18-3.12 (m,1H), 3.00-2.95 (m,0.60H), 2.91-2.86 (m, 0.44H), 2.76-2.65 (m, 1H), 2.61-2.48 (m, 1H), 2.46 (s, 3H), 1.54 (d, J = 5.0 Hz, 1.2H), 1.52 (d, J = 5.0 Hz, 1.6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.2, 151.9, 144.9, 144.9, 135.0, 134.9, 130.0, 128.9, 128.8, 128.4, 128.2, 126.4, 126.4, 125.7, 123.3, 121.9, 121.6, 116.7, 116.3, 93.1, 92.7, 75.2, 72.1, 51.0, 49.6, 40.4, 39.5, 37.3, 37.1, 21.6, 20.71, 20.65.

HRMS (MALDI) m/z calcd for $C_{19}H_{20}O_4NaS$ ([M+Na]$^+$): 367.0975, found 367.0975.

A heteroannulation was performed using **3b** following the procedure for **2**, giving **4b** in 65% yield as a *E*/*Z* mixture (1/0.9). The compound data of major *E* isomer was shown below.



(*E*)-3'-benzylidene-3-(phenylsulfonyl)-3'H-spiro[cyclobutane-1,1'-isobenzofuran] ((*E*)-**4b**)

34% yield, brown oil. (major product)

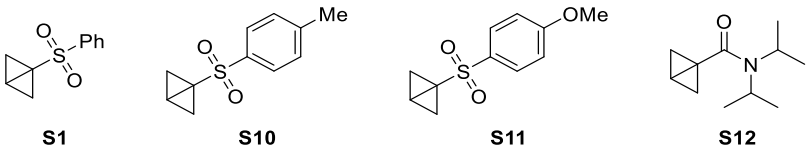
^{1}H NMR (301 MHz, Acetone-d_6) δ 8.03-7.99 (m, 2H), 7.83-7.65 (m, 7H), 7.45-7.41 (m, 2H), 7.37-7.33 (m, 2H), 7.19-7.14 (m, 1H), 6.10 (s, 1H), 4.37-4.29 (m, 1H), 3.31–3.25 (m, 2H), 2.77-2.73 (m,

2H).

$^{13}C\{^1H\}$ NMR (101 MHz, Acetone-d_6) δ 153.8, 143.9, 138.6, 136.3, 134.0, 133.8, 129.7, 129.6, 129.0, 128.5, 128.3, 128.2, 125.6, 120.6, 119.7, 96.8, 83.9, 47.5, 38.3.

HRMS (MALDI) *m/z* calcd for $C_{24}H_{20}O_3NaS$ ([M+Na]$^+$): 411.1025, found 411.1027.

4-3. Preparation of BCB units

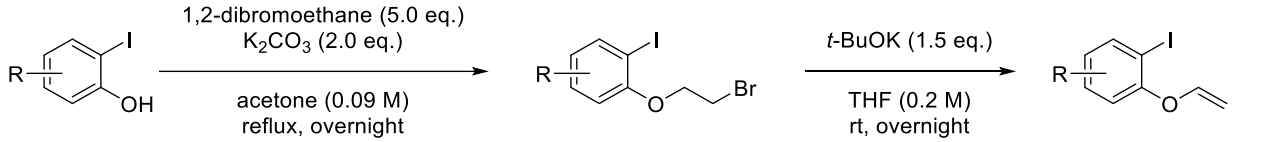


S1 **S10** **S11** **S12**

Compounds **S1**, **S10**, **S11** were prepared according to the literature procedure.[3]

Compound **S12** was prepared according to the literature procedure.[1]

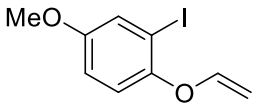
4-4. Preparation of Ar units



To a solution of 2-iodophenol derivative (1.0 eq.) and 1,2-dibromoethane (5.0 eq.) in acetone (0.09 M) was added K_2CO_3 (2.0 eq.). The resulting mixture was stirred under reflux conditions for overnight. The reaction was quenched with water and extracted with CH_2Cl_2. The organic layer was washed with brine, dried over Na_2SO_4, and concentrated. The crude product was purified by a silica gel column chromatography (hexane/EtOAc = 20:1) to give 2-iodophenol bromoethyl ether derivative as a colorless oil or color solid.

To a solution of 2-iodophenol bromoethyl ether derivative (1.0 eq.) in THF (0.20 M) was added *t*-BuOK (1.5 eq.) portionwise. The resulting mixture was stirred at room temperature for overnight. The reaction mixture was filtered and washed with CH_2Cl_2. The combined filtrate was concentrated, and the residue was purified by a silica gel column chromatography (hexane/EtOAc = 25:1) to give 2-iodophenol vinyl ether derivative (2 step yields) as oil or solid.

2-iodo-4-methoxy-1-(vinyloxy)benzene (**S13**)



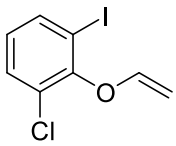
62% yield, colorless oil.

^1H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, J = 2.7 Hz, 1H), 6.93 (d, J = 9.0 Hz, 1H), 6.86 (dd, J = 9.0, 2.7 Hz, 1H), 6.53 (dd, J = 14.0, 6.2 Hz, 1H), 4.58 (dd, J = 14.0, 2.1 Hz, 1H), 4.39 (dd, J = 6.2, 2.1 Hz, 1H), 3.77 (s, 3H).

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 156.3, 149.6, 149.4, 124.2, 119.0, 115.2, 93.9, 88.2, 55.8.

HRMS (MALDI) *m/z* calcd for $C_9H_9O_2I$ ([M]$^+$): 275.9642, found 275.9641.

1-chloro-3-iodo-2-(vinyloxy)benzene **(S14)**



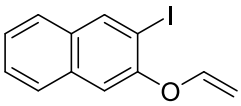
58% yield, colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (dd, J = 8.0, 1.6 Hz, 1H), 7.41 (dd, J = 8.2, 1.4 Hz, 1H), 6.88 (t, J = 8.0 Hz, 1H), 6.56 (dd, J = 14.0, 6.3 Hz, 1H), 4.35 (dd, J = 6.3, 2.7 Hz, 1H), 4.24 (dd, J = 14.2, 2.7 Hz, 1H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 150.9, 148.7, 138.1, 130.9, 127.9, 127.5, 92.1, 91.6.

HRMS (MALDI) *m*/*z* calcd for C_8H_7OClI ([M+H]$^+$): 280.9225, found 280.9231.

2-iodo-3-(vinyloxy)naphthalene **(S15)**



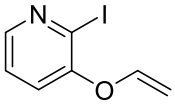
65% yield, pale yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.36 (s, 1H), 7.71 (t, J = 8.9 Hz, 2H), 7.48 (t, J = 7.4 Hz, 1H), 7.41 (t, J = 8.0 Hz, 1H), 7.30 (s, 1H), 6.71 (dd, J = 13.6, 5.8 Hz, 1H), 4.93 (dd, J = 13.6, 1.9 Hz, 1H), 4.62 (dd, J = 5.8, 1.9 Hz, 1H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 152.9, 147.9, 139.6, 134.0, 131.7, 127.2, 127.0, 126.7, 125.4, 112.0, 97.0, 87.8.;

HRMS (MALDI) *m*/*z* calcd for $C_{12}H_{10}OI$ ([M+H]$^+$): 296.9771, found 296.9764.

2-iodo-3-(vinyloxy)pyridine **(S16)**

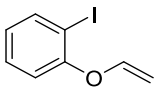


46% yield, brown oil.

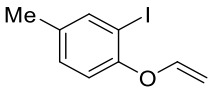
^{1}H NMR (301 MHz, Acetone-d_6) δ 8.12 (dd, J = 4.3, 1.9 Hz, 1H), 7.45-7.37 (m, 2H), 6.81 (dd, J = 13.7, 6.0 Hz, 1H), 4.83 (dd, J = 13.7, 2.0 Hz, 1H), 4.64 (dd, J = 6.0, 2.0 Hz, 1H).

^{13}C{^{1}H} NMR (101 MHz, Acetone-d_6) δ 153.1, 147.6, 145.4, 124.2, 123.2, 111.9, 96.7.

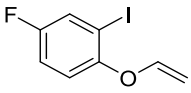
HRMS (MALDI) *m*/*z* calcd for C_7H_7NOI ([M+H]$^+$):247.9567, found 247.9563.



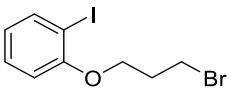
S2



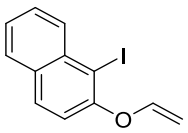
S17



S18



S19



S20

The compounds **S2**, **S17**, **S18** were prepared according to the literature procedure.[1]

The compound **S19** was prepared according to the literature procedure.[4]

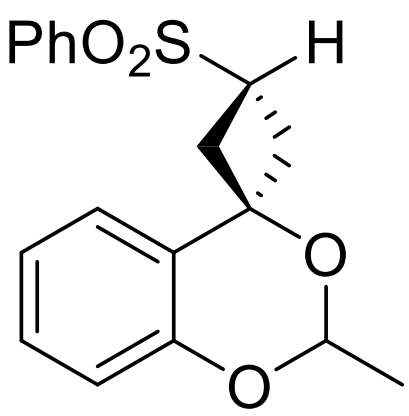
The compound **S20** was prepared according to the literature procedure.[5]

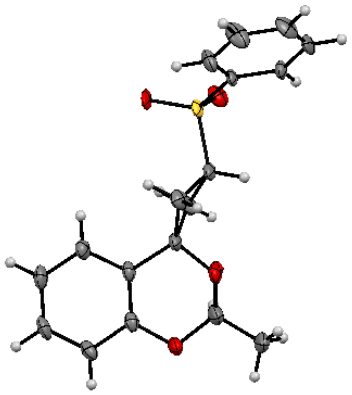
5. References

1. R. E. McNamee, A. L. Thompson, E. A. Anderson, *J. Am. Chem. Soc.* **2021**, *143*, 21246-21251.
2. R. V. Rozhkov, R. C. Larock, *J. Org. Chem.* **2010**, *75*, 4131-4134.
3. M. Jung, V. N. G. Lindsay, *J. Am. Chem. Soc.* **2022**, *144*, 4764-4769.
4. A. Martins, U. Marquardt, N. Kasravi, D. Alberico, M. Lautens, *J. Org. Chem.* **2006**, *71*, 4937-4942.
5. N. Sakiyama, K. Noguchi, K. Tanaka, *Angew. Chem. Int. Ed.* **2012**, *51*, 5976-5980.

6. X-ray Crystallographic Analysis

(3'r,4r)-2-methyl-3'-(phenylsulfonyl)spiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2a**)





CCDC number 2247662

```
Bond precision:   C-C = 0.0039 A          Wavelength=0.71075

Cell:              a=11.154(2)     b=8.1794(13)        c=17.502(3)
                   alpha=90        beta=96.951(4)      gamma=90
Temperature:       273 K

                  Calculated                  Reported
Volume            1585.0(5)                   1585.1(5)
Space group       P 21/n                      P 1 21/n 1
Hall group        -P 2yn                      -P 2yn
Moiety formula    C18 H18 O4 S                C18 H18 O4 S
Sum formula       C18 H18 O4 S                C18 H18 O4 S
Mr                330.38                      330.40
Dx,g cm-3         1.385                       1.384
Z                 4                           4
Mu (mm-1)         0.222                       0.222
F000              696.0                       696.0
F000'             696.83
h,k,lmax          14,10,22                    14,10,22
Nref              3645                        3639
Tmin,Tmax         0.961,0.978                 0.344,0.978
Tmin'             0.875

Correction method= # Reported T Limits: Tmin=0.344 Tmax=0.978
AbsCorr = MULTI-SCAN

Data completeness= 0.998           Theta(max)= 27.514

R(reflections)= 0.0695( 2734)                wR2(reflections)=
                                             0.1891( 3639)
S = 1.018                  Npar= 218
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

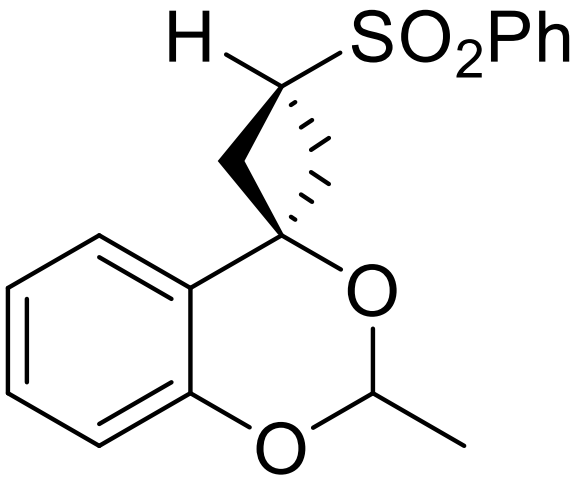
```
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
           The relevant atom site should be identified.
PLAT097_ALERT_2_C Large Reported Max.  (Positive) Residual Density       1.40 eA-3
PLAT213_ALERT_2_C Atom O4              has ADP max/min Ratio .....        3.4 prolat
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor ....        2.7 Note
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.689 Check
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.91Ang From O3      .       0.55 eA-3
PLAT975_ALERT_2_C Check Calcd Resid. Dens.  0.97Ang From O1      .       0.49 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H1A       .      -0.31 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H1B       .      -0.36 eA-3
PLAT977_ALERT_2_C Check Negative Difference Density on H1C       .      -0.51 eA-3
```

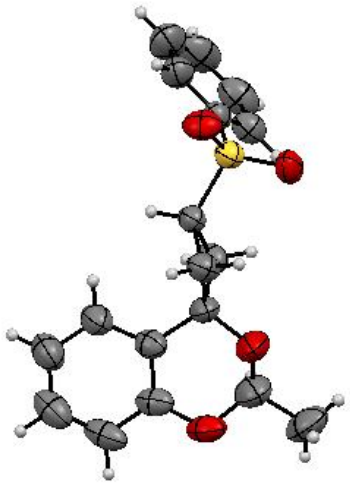
Alert level G

```
CHEMS02_ALERT_1_G  Please check that you have entered the correct
           _publ_requested_category classification of your compound;
           FI or CI or EI for inorganic; FM or CM or EM for metal-organic;
           FO or CO or EO for organic.
           From the CIF: _publ_requested_category   CHOOSE FI FM FO CI CM CO or A
           From the CIF: _chemical_formula_sum :C18 H18 O4 S1
PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite          4 Note
PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ...          2 Report
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT  Unusually Large       0.13 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records          1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records          1 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)        273 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)        273 Check
PLAT230_ALERT_2_G Hirshfeld Test Diff for    O2A      --C2       .        6.0 s.u.
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd  1  )         4% Note
PLAT793_ALERT_4_G Model has Chirality at C2          (Centro SPGR)          R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............        10 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          2 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        4.2 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          3 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
  10 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  16 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
  13 ALERT type 2 Indicator that the structure model may be wrong or deficient
   5 ALERT type 3 Indicator that the structure quality may be low
   4 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

(3's,4s)-2-methyl-3'-(phenylsulfonyl)spiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2a'**)





CCDC number 2247661

Bond precision: C-C = 0.0055 A Wavelength=1.54187

Cell: a=8.3144(3) b=9.8167(4) c=20.8013(7)
alpha=90 beta=99.087(7) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	1676.49(11)	1676.48(11)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C18 H18 O4 S	C18 H18 O4 S
Sum formula	C18 H18 O4 S	C18 H18 O4 S
Mr	330.38	330.40
Dx,g cm-3	1.309	1.309
Z	4	4
Mu (mm-1)	1.865	1.866
F000	696.0	696.0
F000'	699.36	
h,k,lmax	10,11,25	9,11,24
Nref	3067	3027
Tmin,Tmax	0.671,0.756	0.513,0.756
Tmin'	0.411	

Correction method= # Reported T Limits: Tmin=0.513 Tmax=0.756
AbsCorr = MULTI-SCAN

Data completeness= 0.987 Theta(max)= 68.194

R(reflections)= 0.0569(2023) wR2(reflections)= 0.1610(3027)

S = 1.026 Npar= 208

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

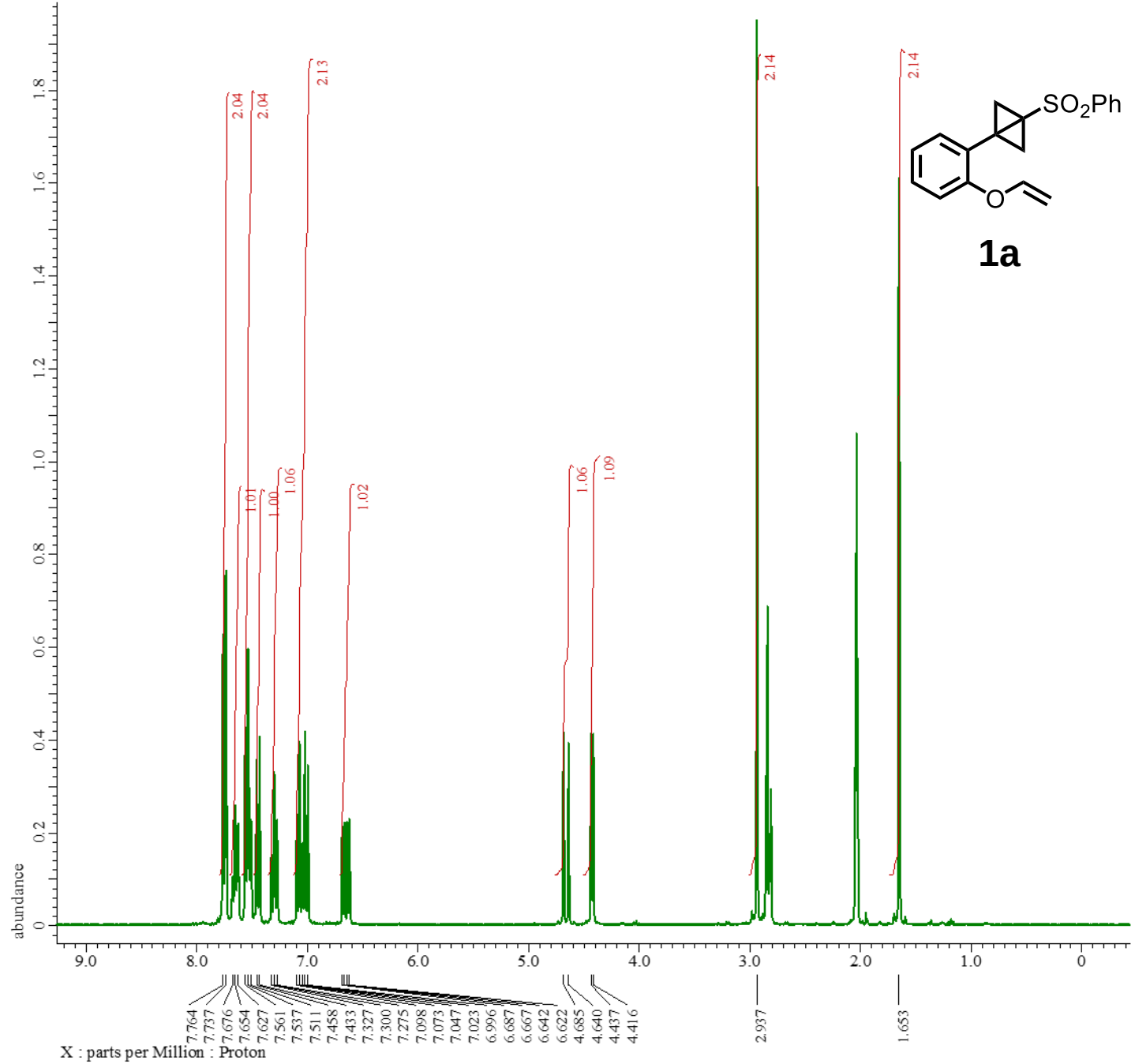
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of O4 Check
PLAT340_ALERT_3_C Low Bond Precision on C–C Bonds ..... 0.0055 Ang.

Alert level G

CHEMS02_ALERT_1_G Please check that you have entered the correct
_publ_requested_category classification of your compound;
FI or CI or EI for inorganic; FM or CM or EM for metal-organic;
FO or CO or EO for organic.
From the CIF: _publ_requested_category CHOOSE FI FM FO CI CM CO or A
From the CIF: _chemical_formula_sum :C18 H18 O4 S1
PLAT793_ALERT_4_G Model has Chirality at C18 (Centro SPGR) R Verify
PLAT882_ALERT_1_G No Datum for _diffrn_reflns_av_unetI/netI ..... Please Do !
PLAT986_ALERT_1_G No non-zero f' Anomalous Scattering Values Found Please Check

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
4 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
1 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

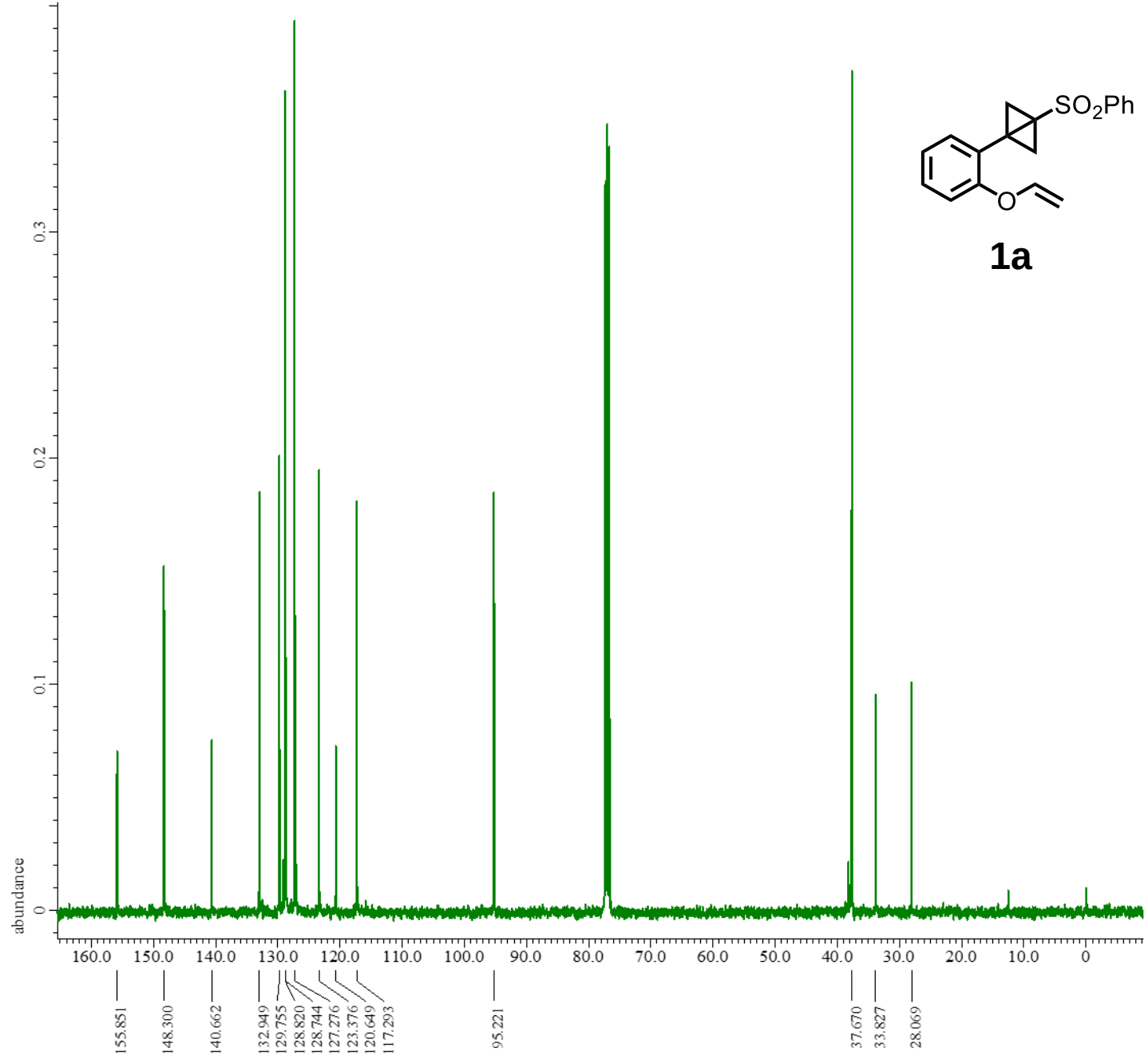


1a

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 1 normal sub Acetone-d6_P
Author              = delta
Experiment          = proton.jxp
Sample_Id           = normal sub recrystallizat
Solvent             = ACETONE-D6
Actual_Start_Time   = 20-FEB-2023 16:45:15
Revision_Time       = 15-MAR-2023 15:36:16

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 40
Temp_Get            = 19.3[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

[1] H NMR spectrum of **1a** (301 MHz, Acetone-d_6)

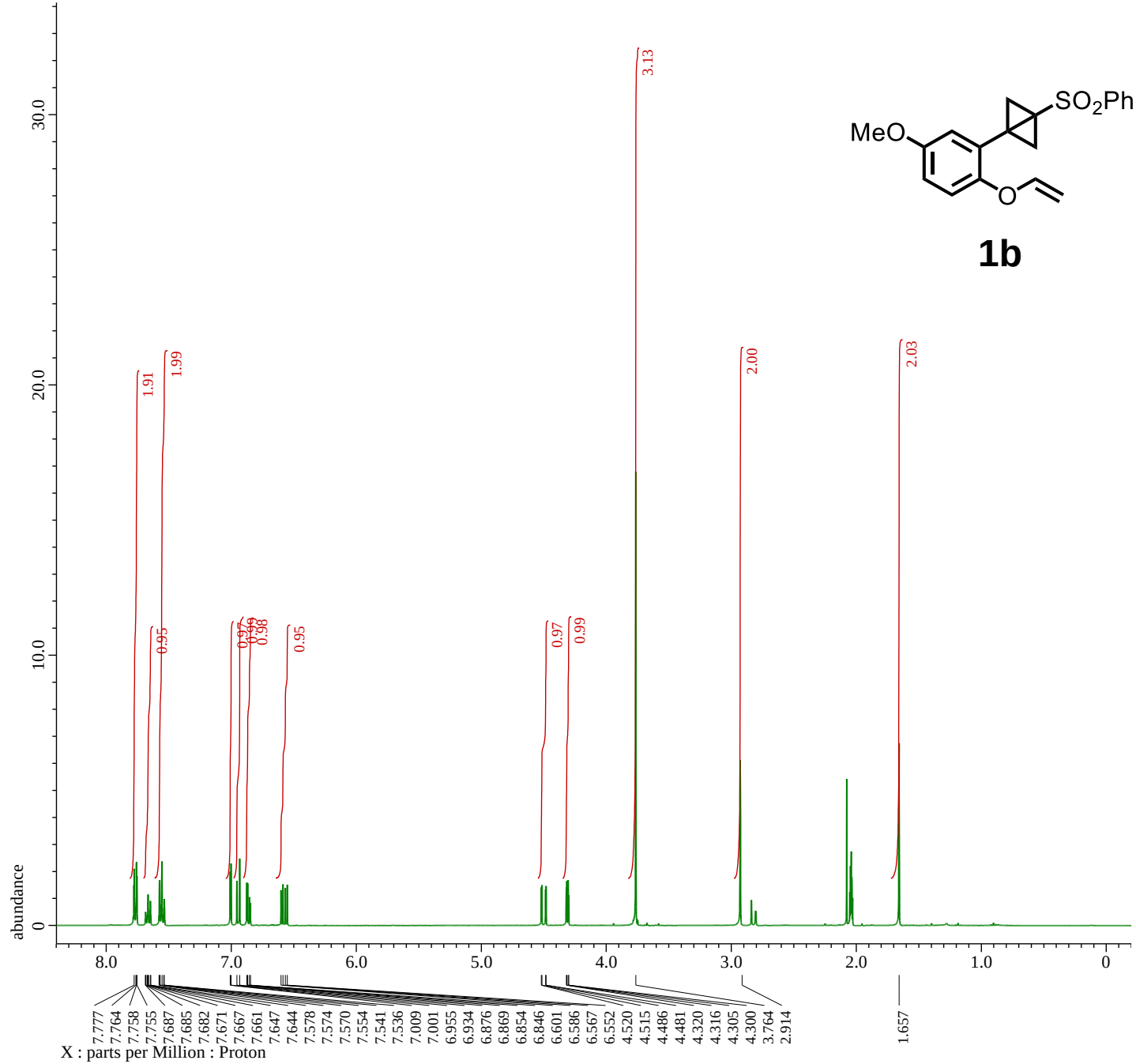


1a

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

```
Filename            = 1 normal CDCl3_Carbon-1-2
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = BCB derivatives normal CD
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 28-APR-2022 21:04:59
Revision_Time       =  4-JAN-2023 12:39:39

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 400
Total_Scans         = 400

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 8.6[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 2.86666667[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1a** (101 MHz, $CDCl_3$)

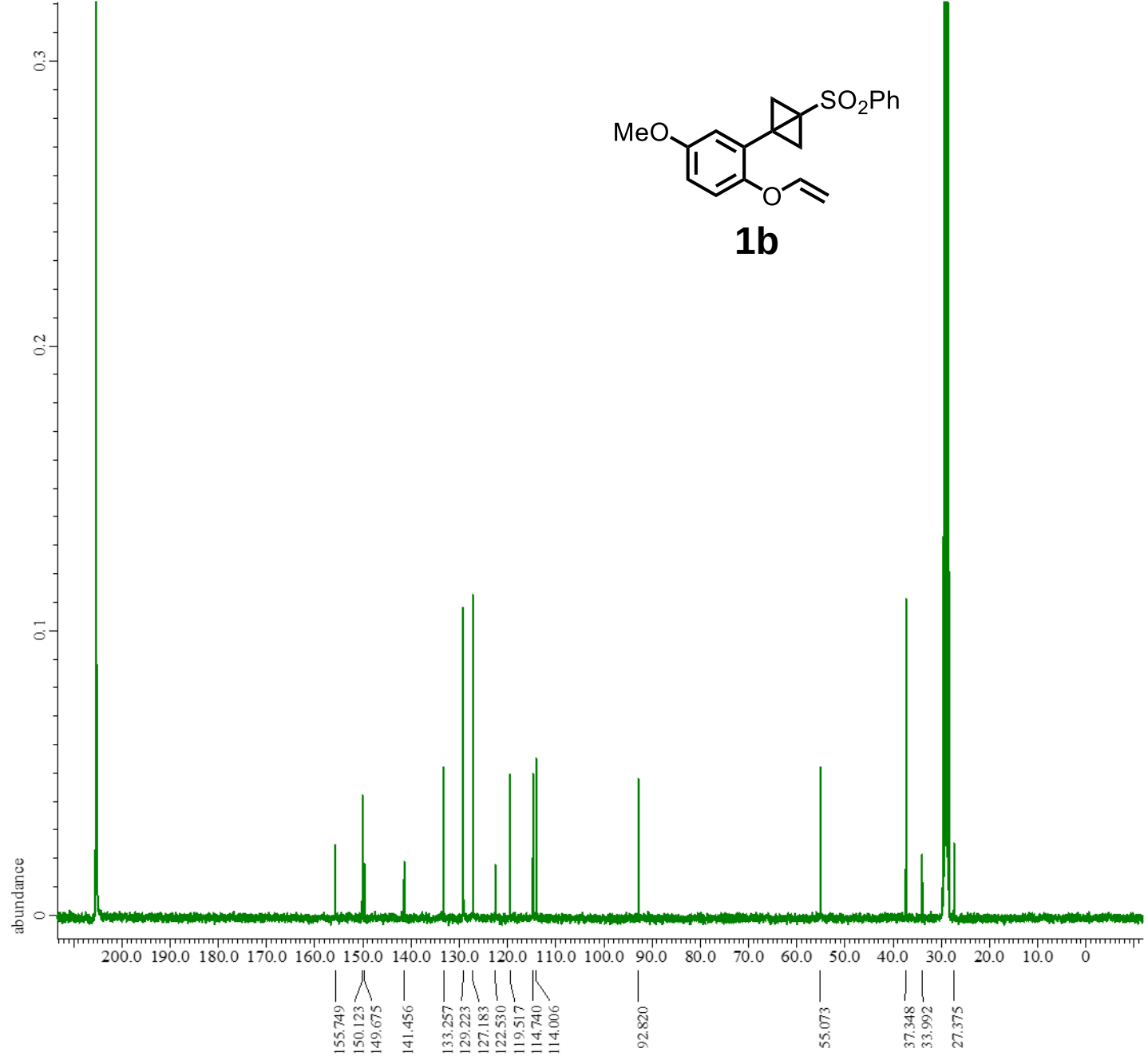


1b

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 2 4-OMe Acetone-d6_Proton
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210578 Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   =  7-NOV-2022 13:59:16
Revision_Time       = 30-DEC-2022 20:22:49

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 40
Temp_Get            = 20.1[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

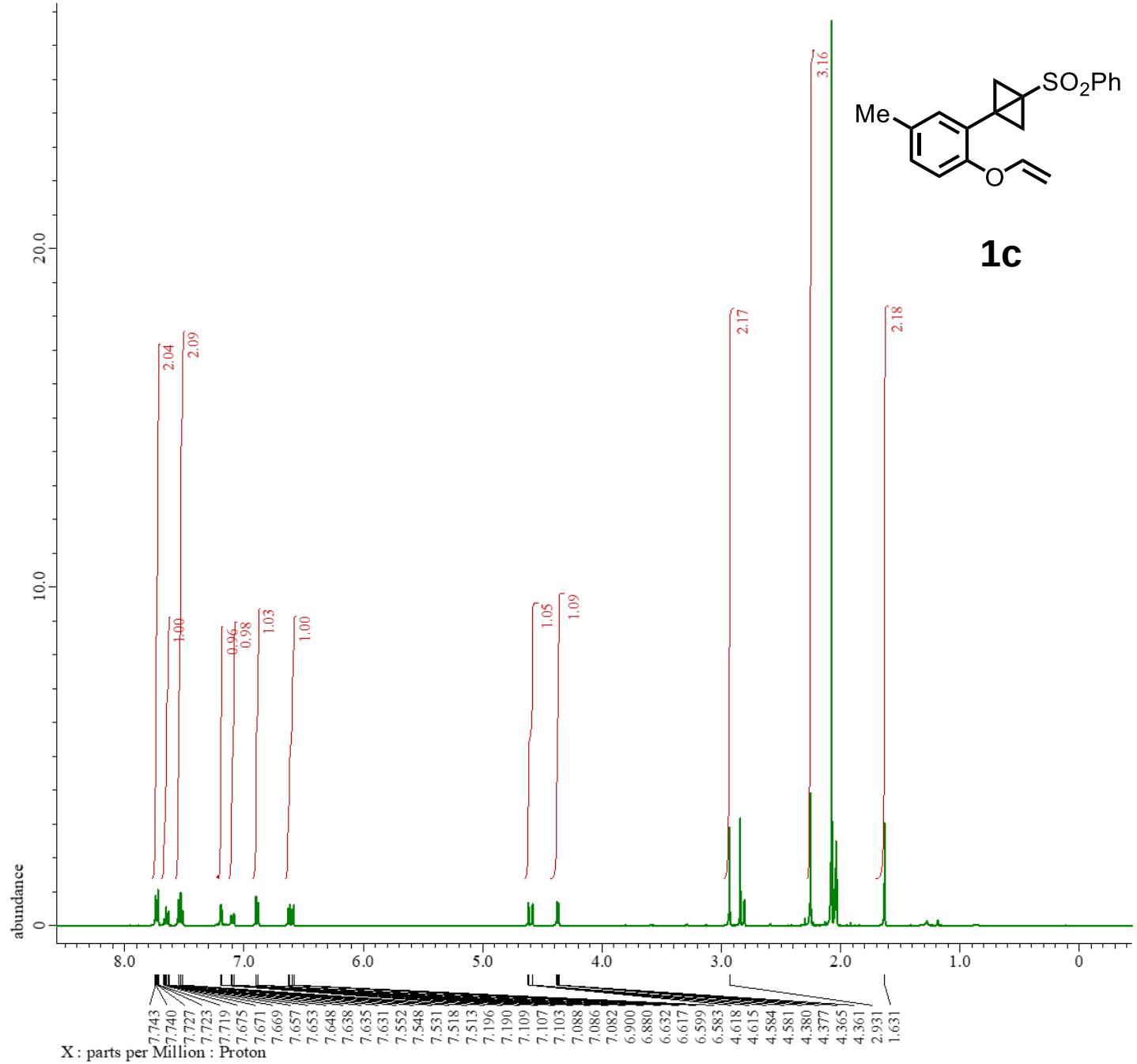
[1] H NMR spectrum of **1b** (400 MHz, Acetone-d_6)




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

```
Filename            = 2 4-OMe Acetone-d6_Carbon
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210578 Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   =  7-NOV-2022 15:19:28
Revision_Time       = 17-MAR-2023 15:47:40

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.3[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1b** (101 MHz, Acetone-d_6)

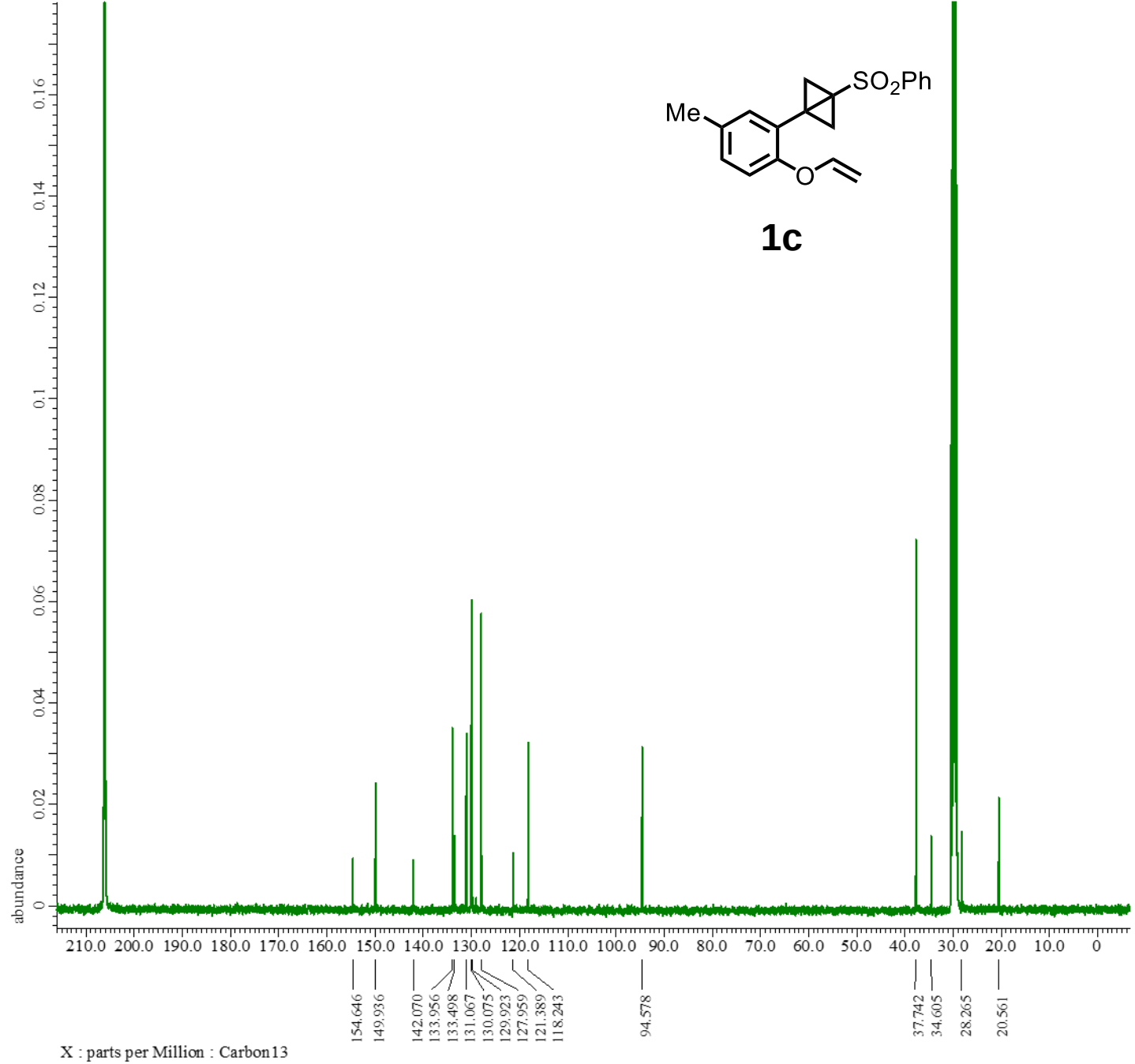



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 5 4-Me Acetone-d6_Proton-
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 4-Me Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 29-NOV-2022 22:33:25
Revision_Time       = 15-MAR-2023 18:55:51

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 40
Temp_Get            = 19.9[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

7.743 7.740 7.727 7.723 7.719 7.675 7.671 7.669 7.657 7.653 7.648 7.638 7.635 7.631 7.552 7.548 7.531 7.518 7.513 7.196 7.190 7.109 7.107 7.103 7.088 7.086 7.082 6.900 6.880 6.632 6.617 6.599 6.583 4.618 4.615 4.584 4.581 4.380 4.377 4.365 4.361 2.931 1.631

[1] H NMR spectrum of **1c** (400 MHz, Acetone-d_6)

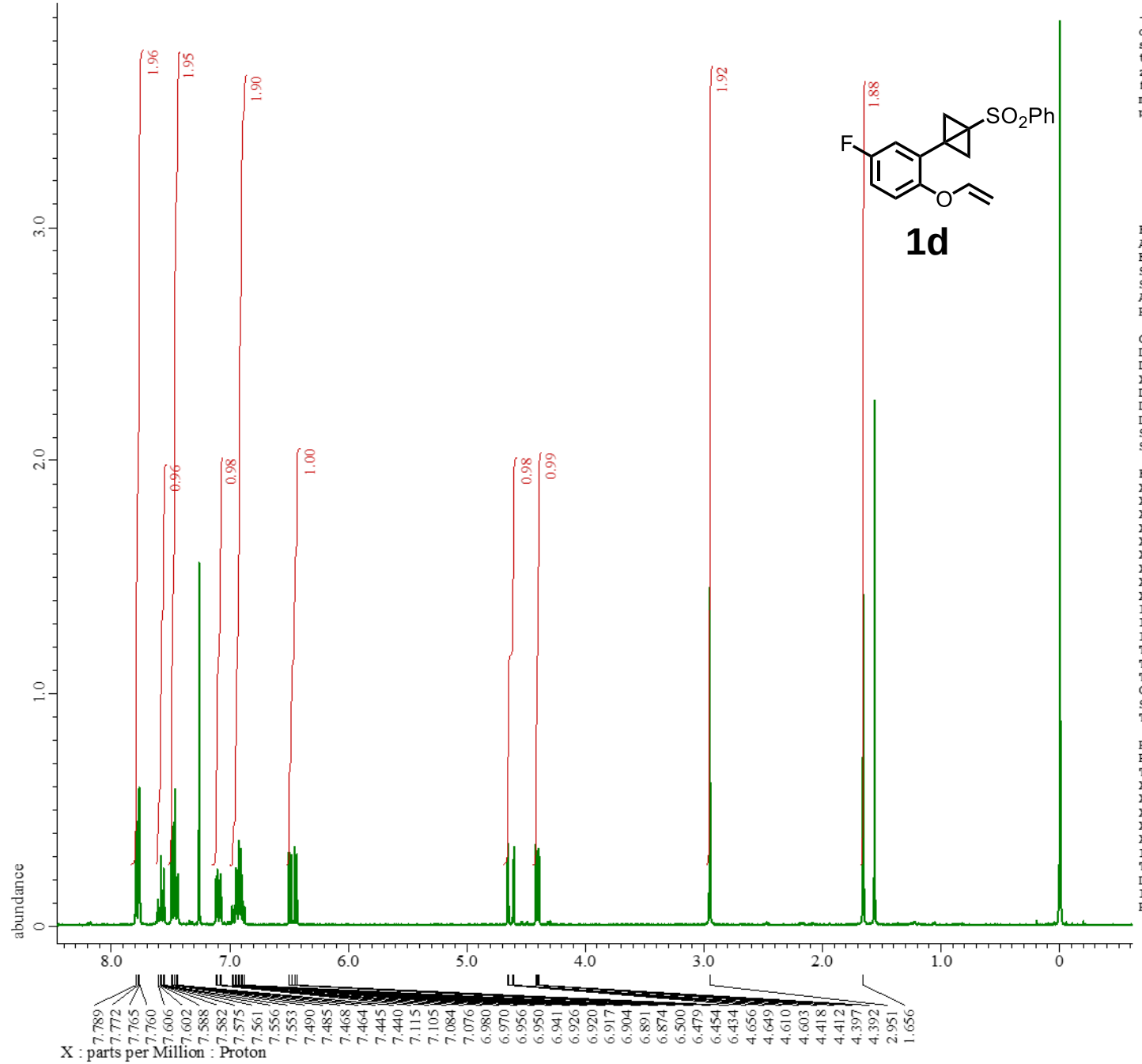


1c

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

```
Filename            = 5 4-Me Acetone-d6_Carbon-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 4-Me Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   =  2-DEC-2022 03:07:04
Revision_Time       = 17-MAR-2023 15:45:54

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 3450
Total_Scans         = 3450

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1c** (101 MHz, Acetone-d_6)

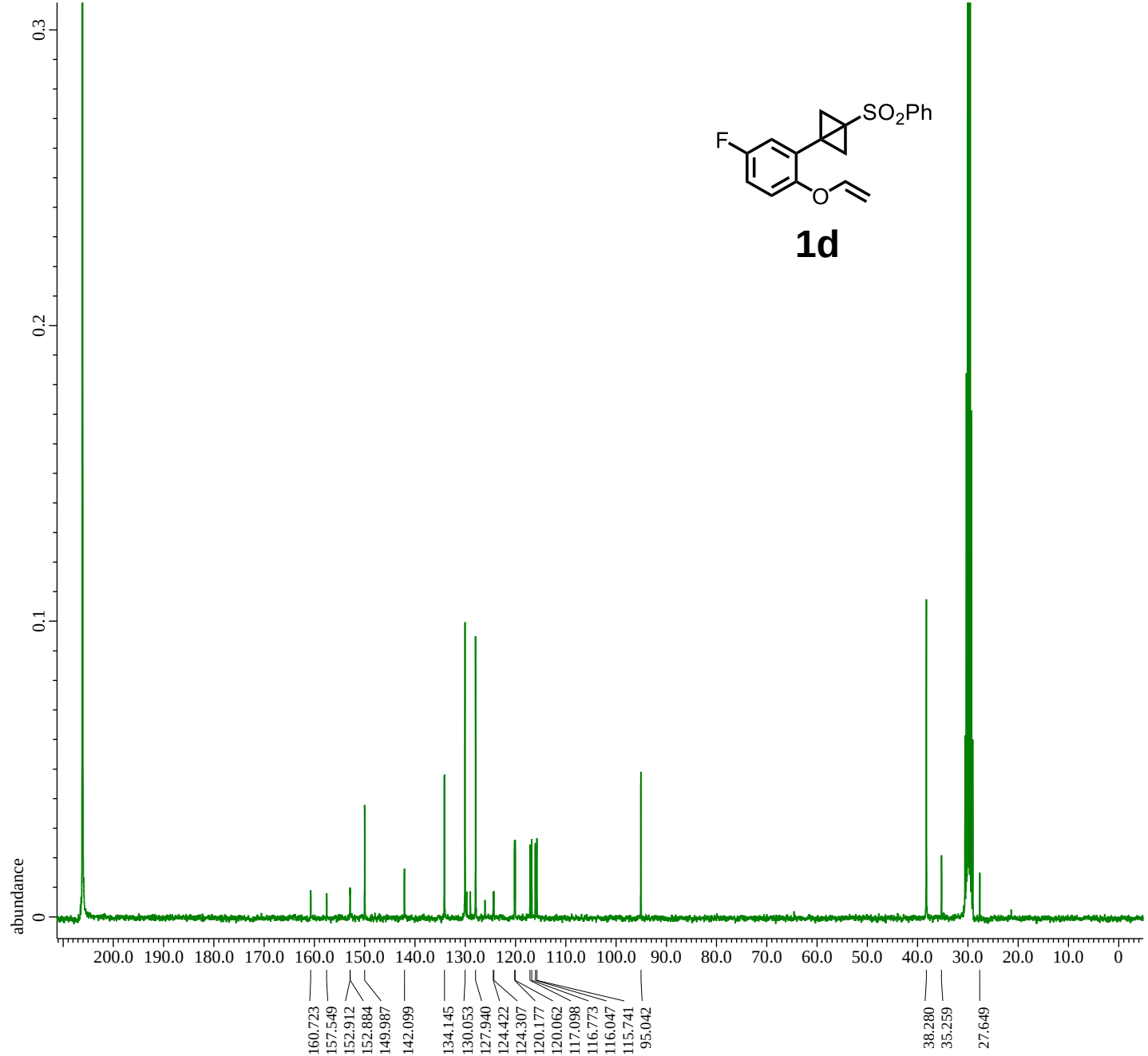


1d

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 4 4-F CDCl3_Proton-1-2.jd
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210381 column 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 24-MAY-2022 12:20:16
Revision_Time       =  4-JAN-2023 13:39:55

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 46
Temp_Get            = 19.8[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

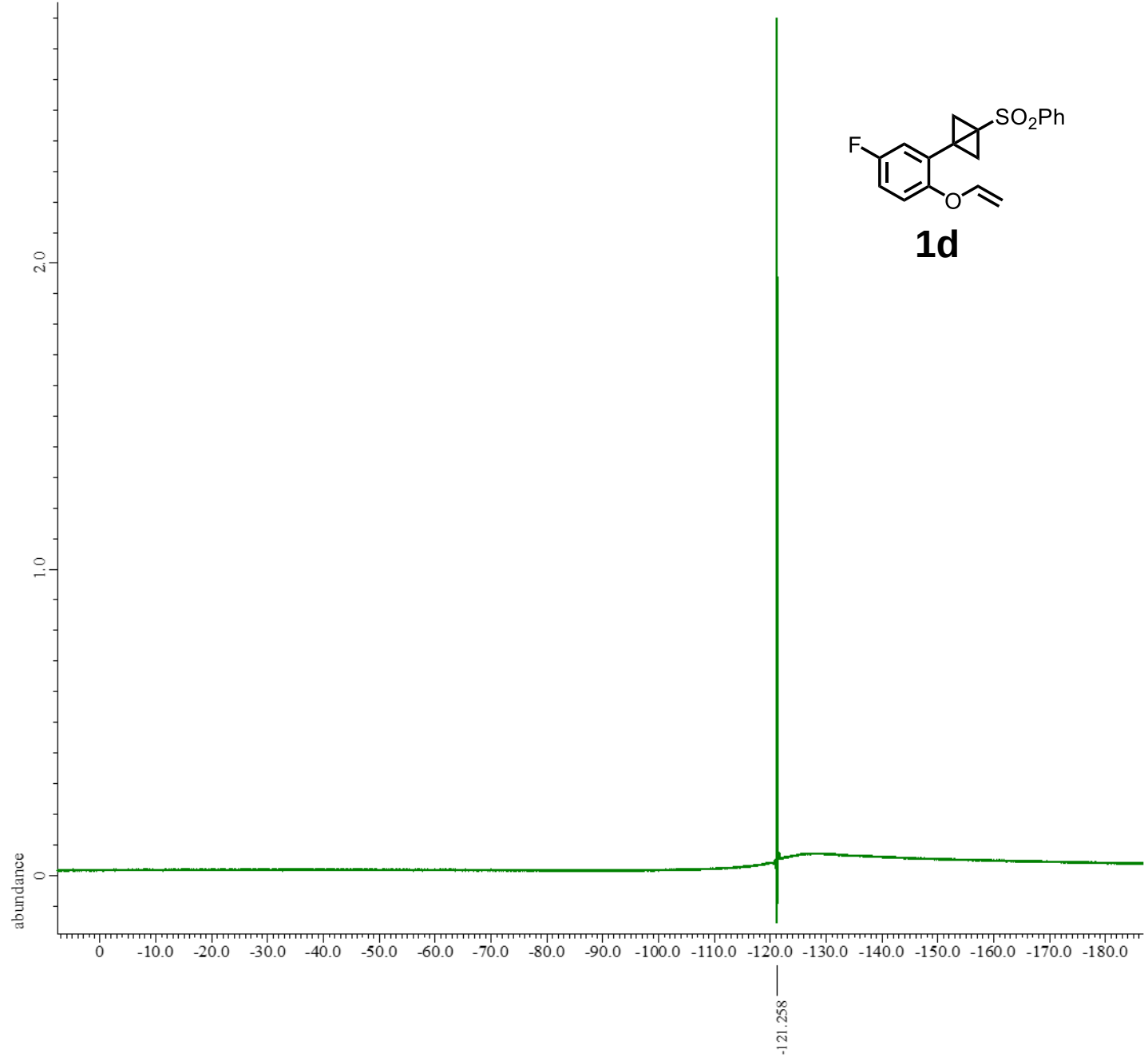
[1] H NMR spectrum of **1d** (301 MHz, $CDCl_3$)



1d

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

Filename           = 4 4-F Acetone-d6_Carbon-1
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = 210581 Acetone-d6
Solvent            = ACETONE-D6
Actual_Start_Time  = 29-NOV-2022 18:05:13
Revision_Time      = 15-MAR-2023 19:16:30

Comment            = single pulse decoupled ga
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = Carbon
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECS 300
Spectrometer       = DELTA2_NMR

Field_Strength     = 7.0586013[T] (300[MHz])
X_Acq_Duration     = 1.38412032[s]
X_Domain           = 13C
X_Freq             = 75.56823426[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.72248054[Hz]
X_Sweep            = 23.67424242[kHz]
X_Sweep_Clipped    = 18.93939394[kHz]
Irr_Domain         = Proton
Irr_Freq           = 300.52965592[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1024
Total_Scans        = 1024

Relaxation_Delay   = 2[s]
Recvr_Gain         = 50
Temp_Get           = 20.8[dC]
X_90_Width         = 11.4[us]
X_Acq_Time         = 1.38412032[s]
X_Angle            = 30[deg]
X_Atn              = 5.4[dB]
X_Pulse            = 3.8[us]
Irr_Atn_Dec        = 21.6[dB]
Irr_Atn_Noe        = 21.6[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.118[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.38412032[s]
```

^{13}C NMR spectrum of **1d** (76 MHz, Acetone-d_6)

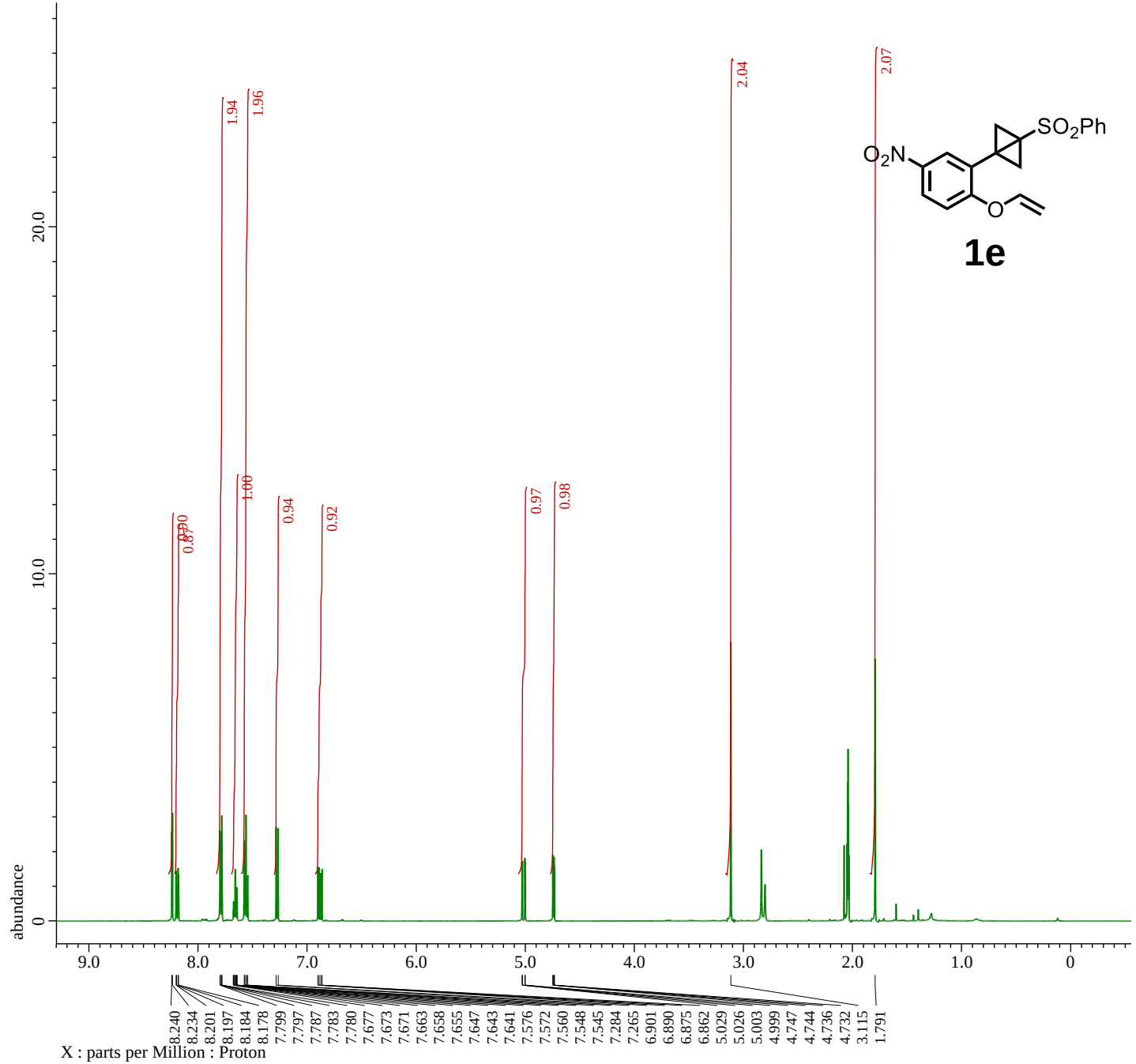


1d

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 4_4-F Acetone-d6_single_p
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = 210581 Acetone-d6
Solvent           = ACETONE-D6
Actual_Start_Time = 29-NOV-2022 17:56:45
Revision_Time     = 17-MAR-2023 15:50:34

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Fluori
Dim_Title         = Fluorine19
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 300
Spectrometer      = DELTA2_NMR

Field_Strength    = 7.0586013[T] (300[MHz])
X_Acq_Duration    = 0.15368192[s]
X_Domain          = 19F
X_Freq            = 282.78036857[MHz]
X_Offset          = -80[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 6.5069463[Hz]
X_Sweep           = 106.6098081[kHz]
X_Sweep_Clipped   = 85.28784648[kHz]
Irr_Domain        = Fluorine19
Irr_Freq          = 282.78036857[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Fluorine19
Tri_Freq          = 282.78036857[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 64
Total_Scans       = 64

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 20.3[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.15368192[s]
X_Angle           = 45[deg]
X_Atn             = 2.8[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 5.15368192[s]
```

^{19}F NMR spectrum of **1d** (283 MHz, Acetone-d_6)



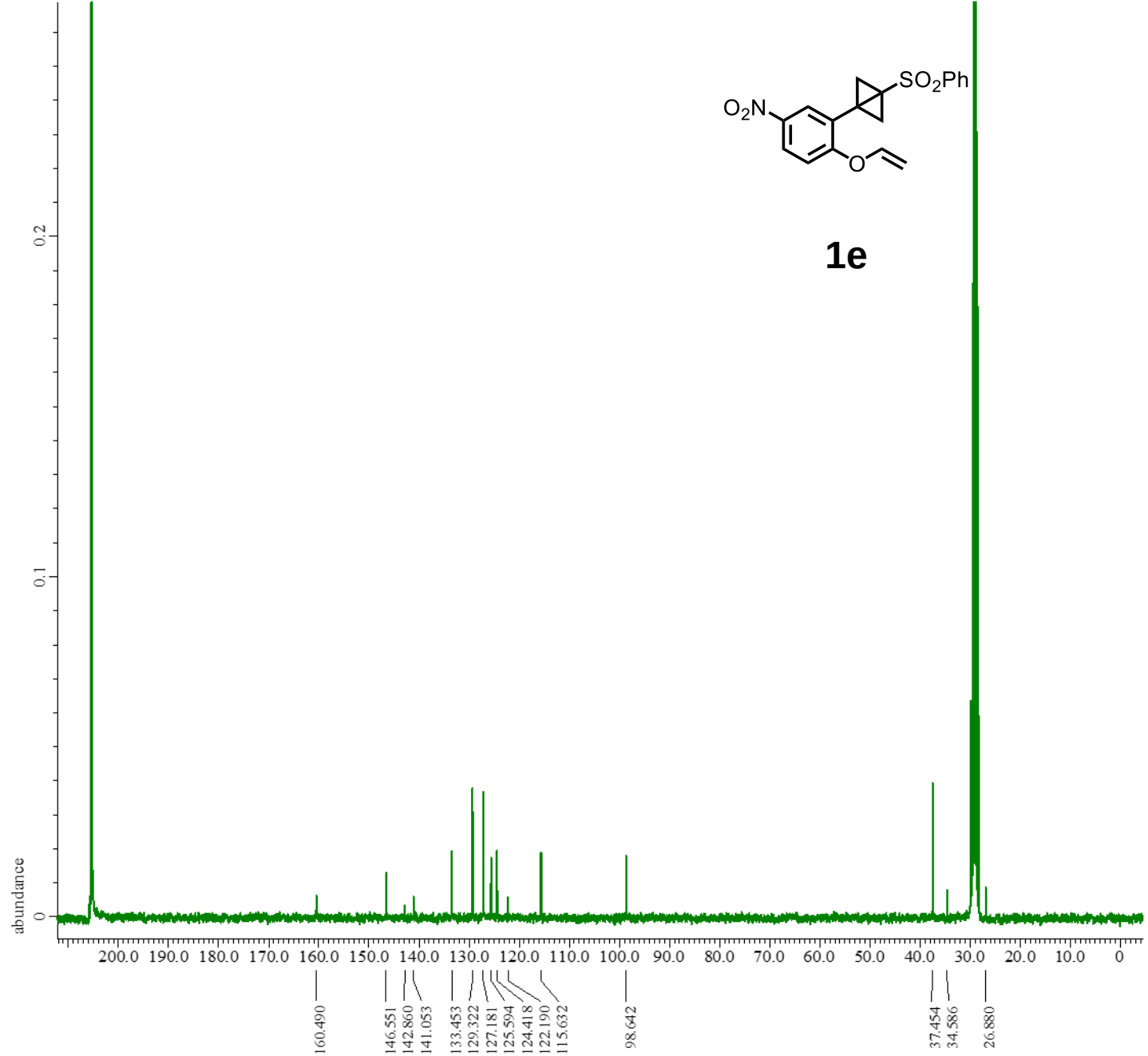
1e

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 3 4-NO2 Acetone-d6_Proton
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210636 column Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 28-NOV-2022 22:44:55
Revision_Time       = 15-MAR-2023 19:40:38

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 50
Temp_Get            = 20.2[dC]
X_90_Width          = 12.9[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.6[dB]
X_Pulse             = 6.45[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

8.240 8.234 8.201 8.197 8.184 8.178 7.799 7.797 7.787 7.783 7.780 7.677 7.673 7.671 7.663 7.658 7.655 7.647 7.643 7.641 7.576 7.572 7.560 7.548 7.545 7.284 7.265 6.901 6.890 6.875 6.862 5.029 5.026 5.003 4.999 4.747 4.744 4.736 4.732 3.115 1.791

[1] H NMR spectrum of **1e** (500 MHz, Acetone-d_6)

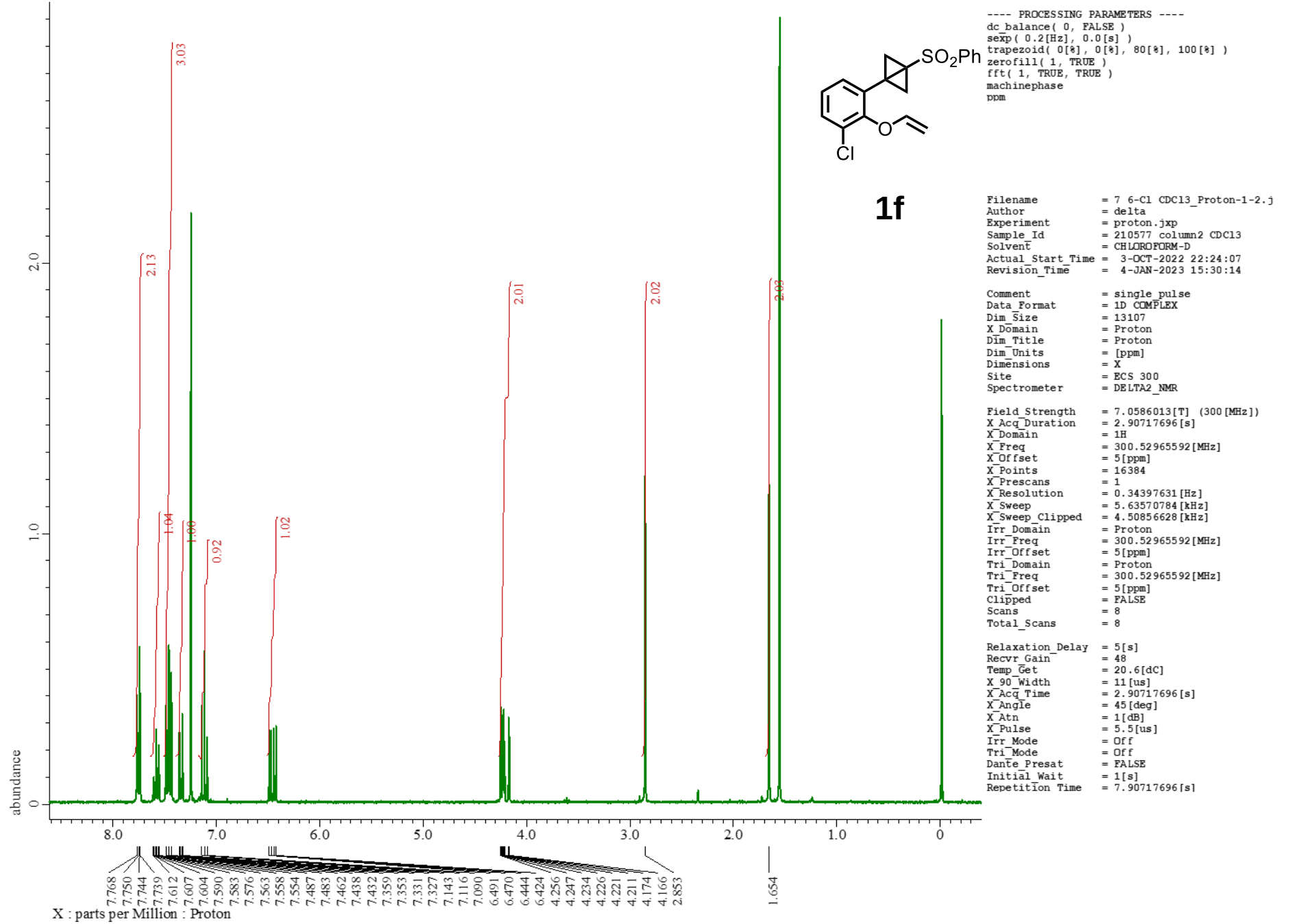


1e

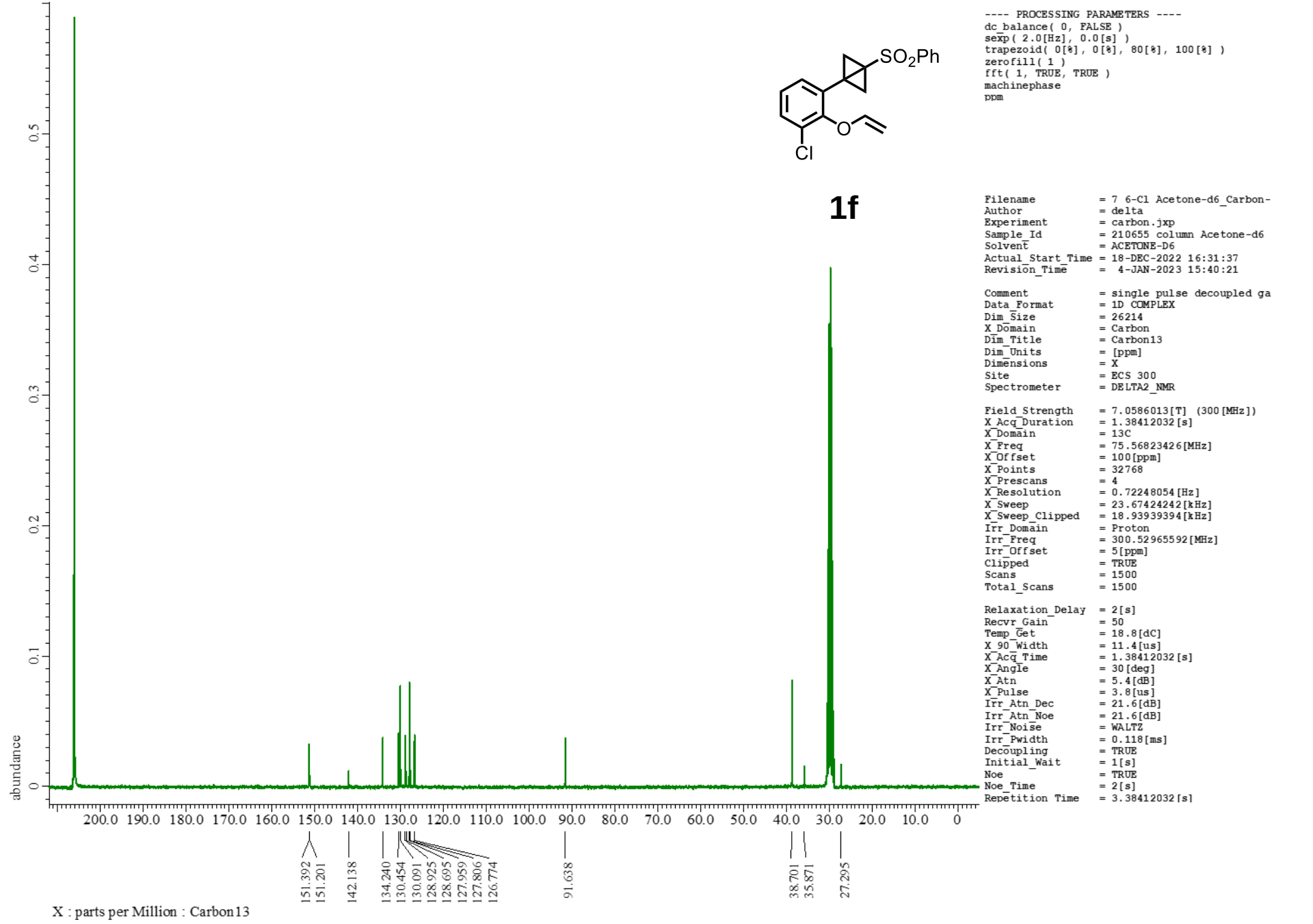
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 3 4-NO2 Acetone-d6_Carbon
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210636 column Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 29-NOV-2022 10:05:34
Revision_Time       =  4-JAN-2023 15:48:19

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 1.38412032[s]
X_Domain            = 13C
X_Freq              = 75.56823426[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.72248054[Hz]
X_Sweep             = 23.67424242[kHz]
X_Sweep_Clipped     = 18.93939394[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 928
Total_Scans         = 928

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 21[dC]
X_90_Width          = 11.4[us]
X_Acq_Time          = 1.38412032[s]
X_Angle             = 30[deg]
X_Atn               = 5.4[dB]
X_Pulse             = 3.8[us]
Irr_Atn_Dec         = 21.6[dB]
Irr_Atn_Noe         = 21.6[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.118[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.38412032[s]
```

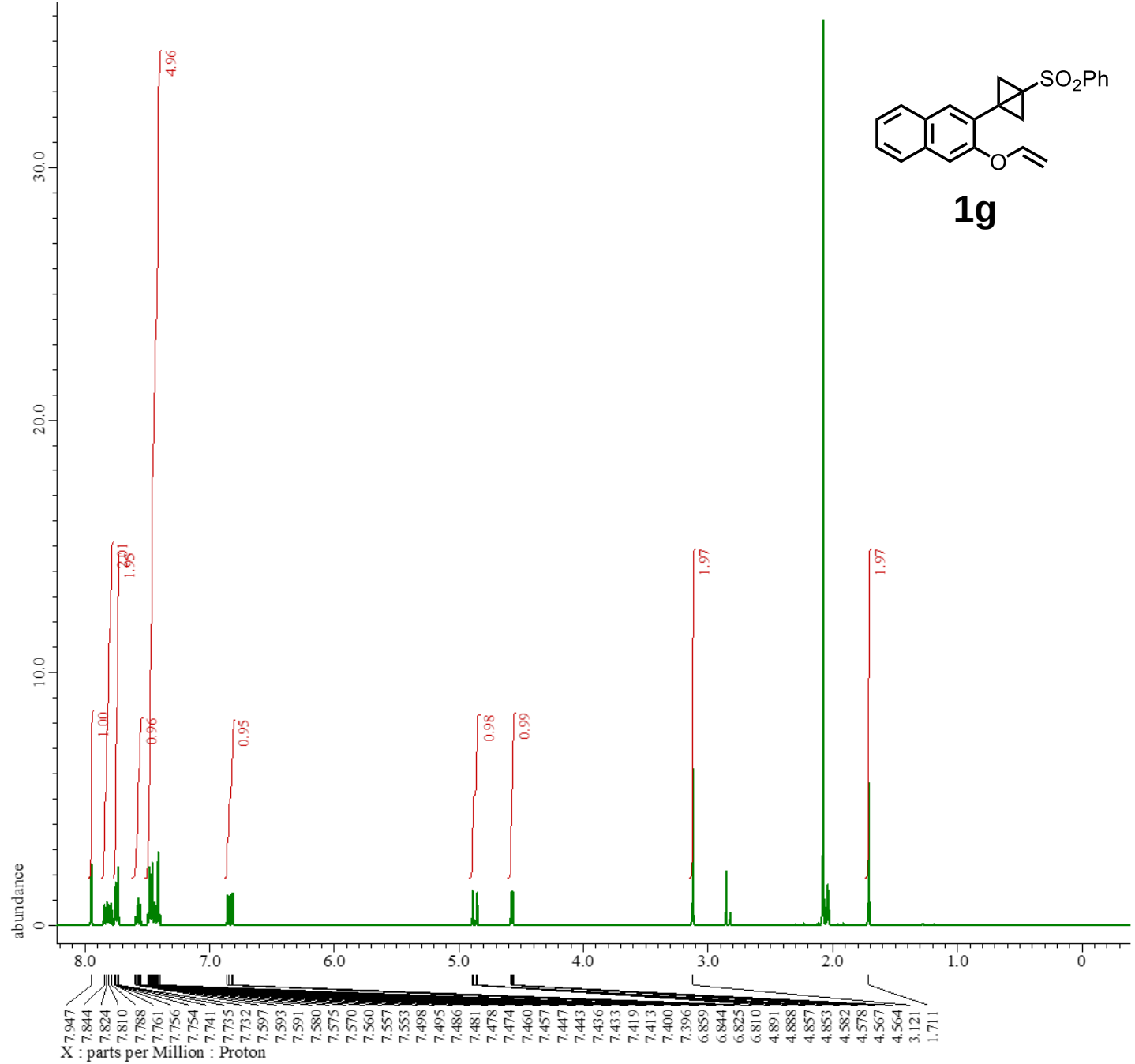
^{13}C NMR spectrum of **1e** (76 MHz, Acetone-d_6)



[1] H NMR spectrum of **1f** (301 MHz, Acetone-d_6)



^{13}C NMR spectrum of **1f** (76 MHz, Acetone-d_6)

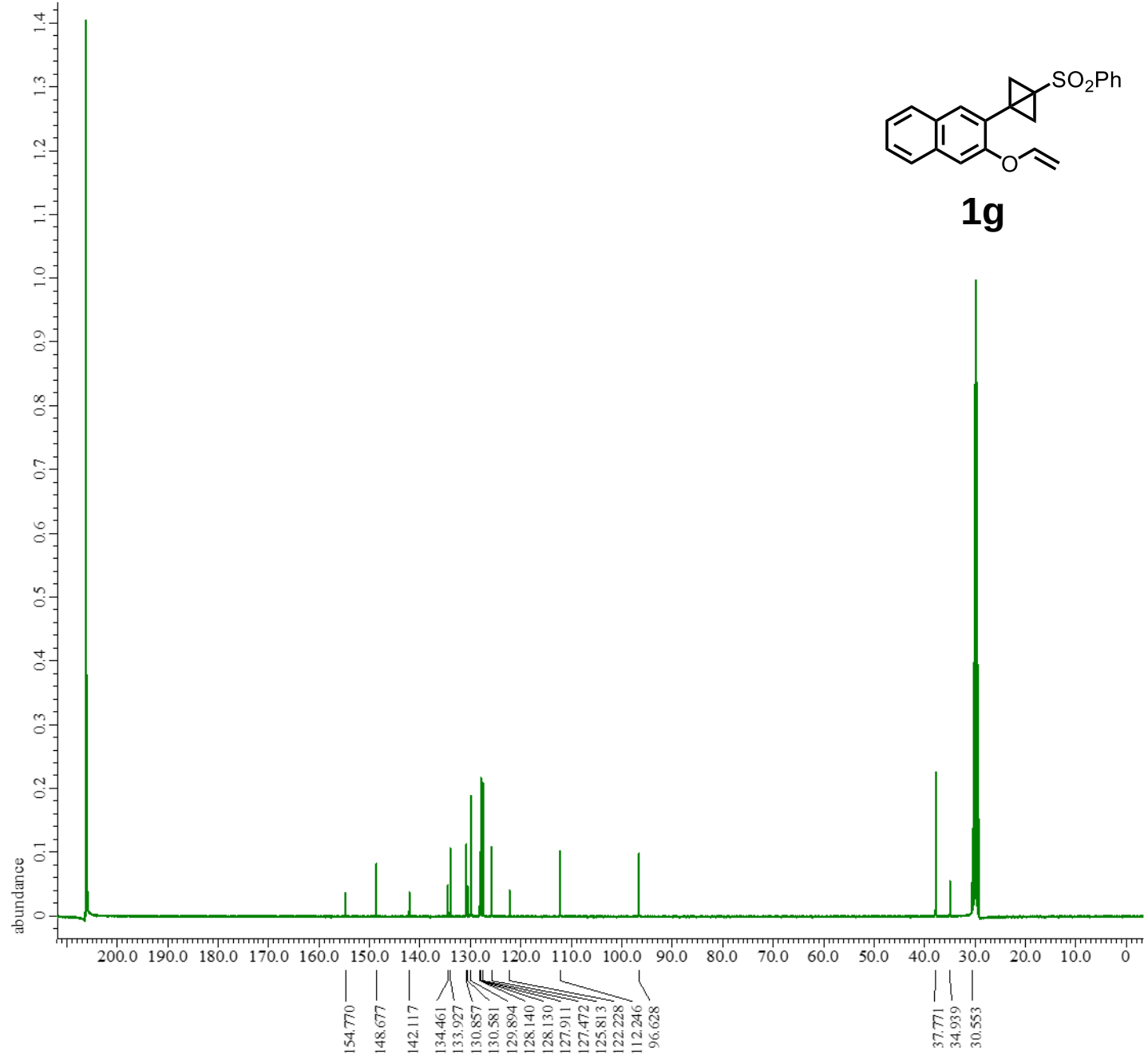


1g

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 6 2,3-naphthyl Acetone-d6
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 2,3-naphthyl Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 29-NOV-2022 22:25:34
Revision_Time       =  4-JAN-2023 16:46:55

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 36
Temp_Get            = 19.6[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **1g** (400 MHz, Acetone-d_6)

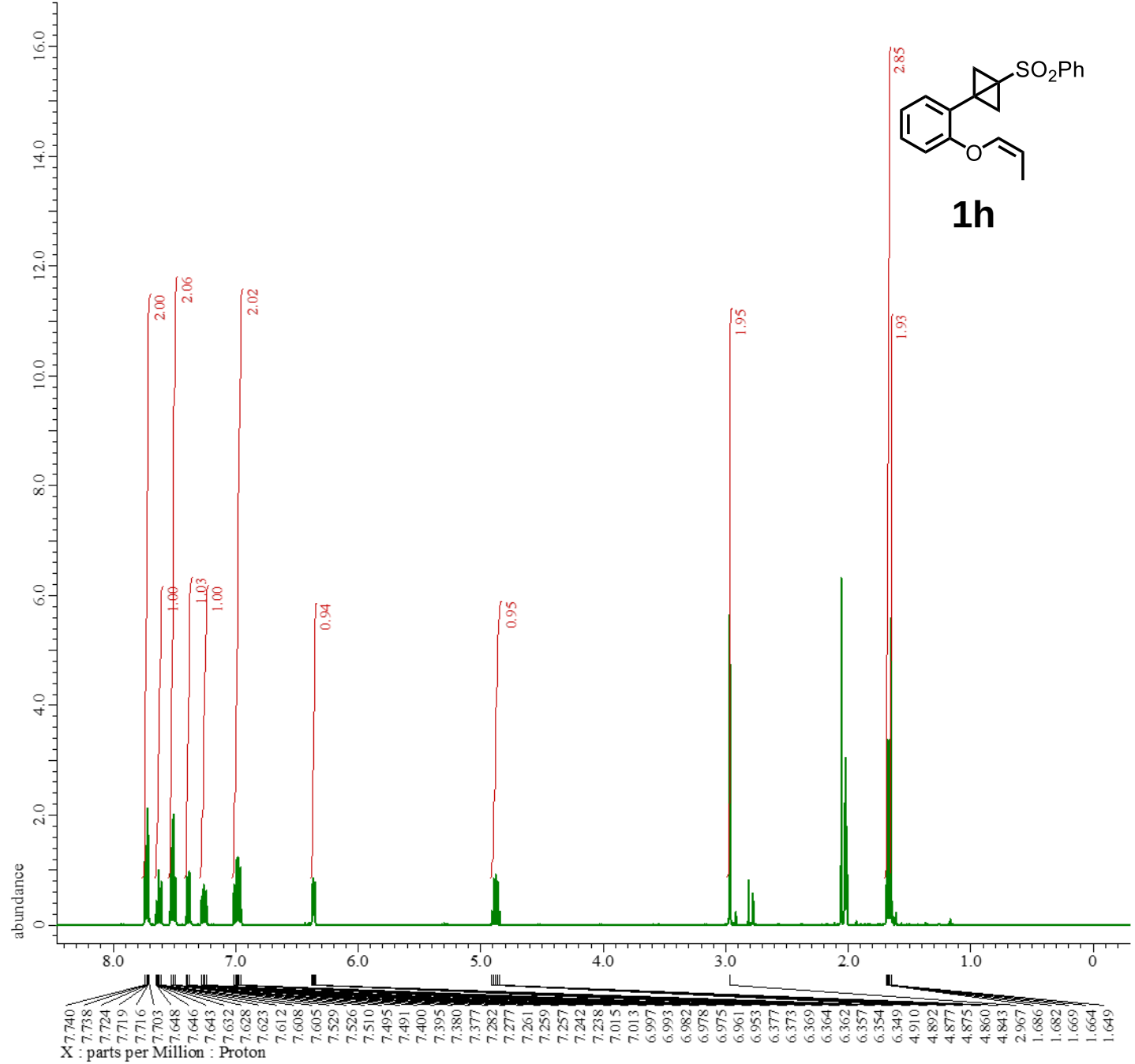


1g

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

```
Filename            = 6 2,3-naphthyl Acetone-d6
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 2,3-naphthyl Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   =  2-DEC-2022 00:06:27
Revision_Time       = 11-FEB-2023 18:39:20

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 3450
Total_Scans         = 3450

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1g** (101 MHz, Acetone-d_6)

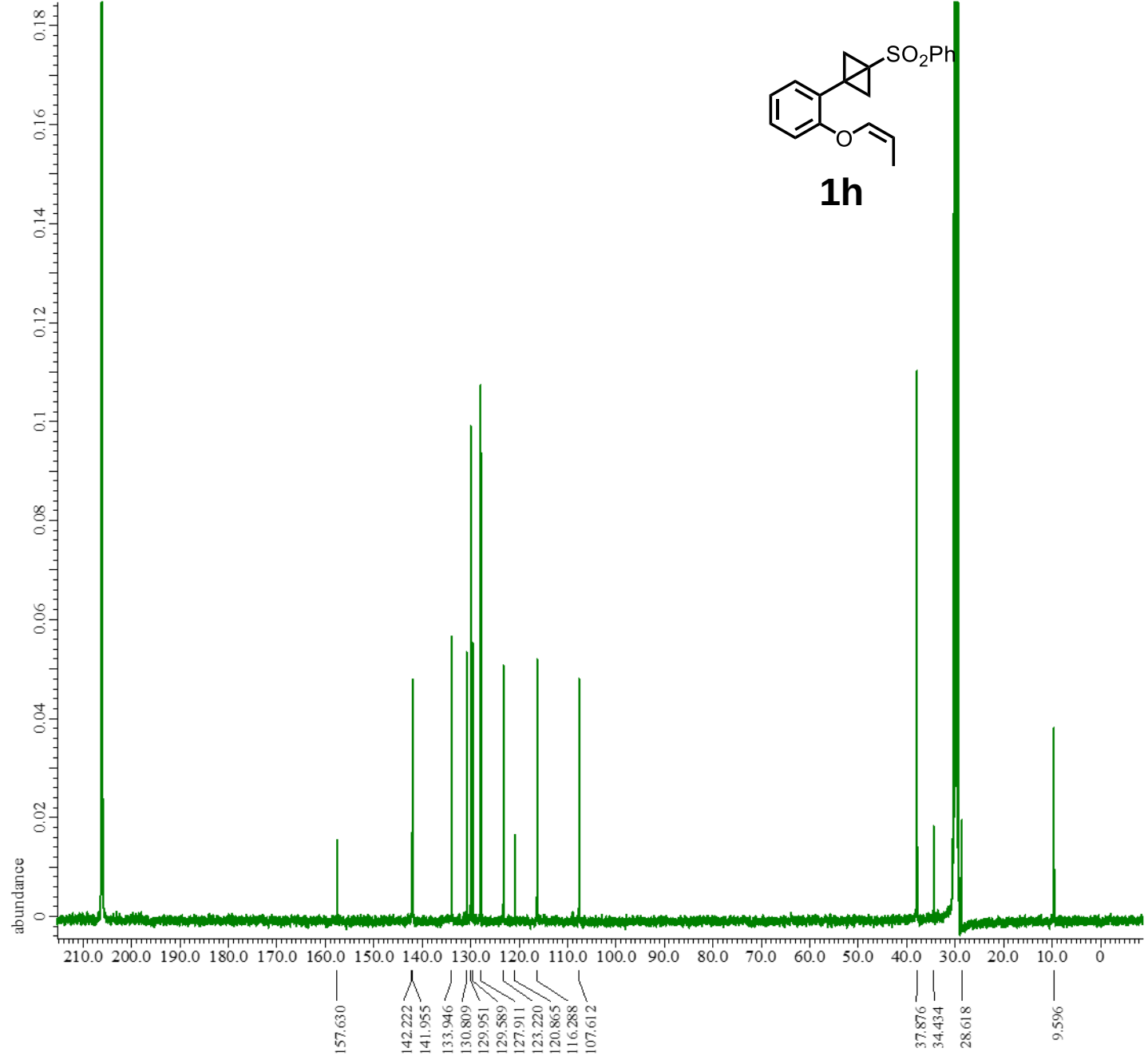


1h

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 8 O-propenyl Acetone-d6_P
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210674 column Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   =  6-JAN-2023 22:59:59
Revision_Time       = 15-MAR-2023 20:29:35

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 40
Temp_Get            = 20.6[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

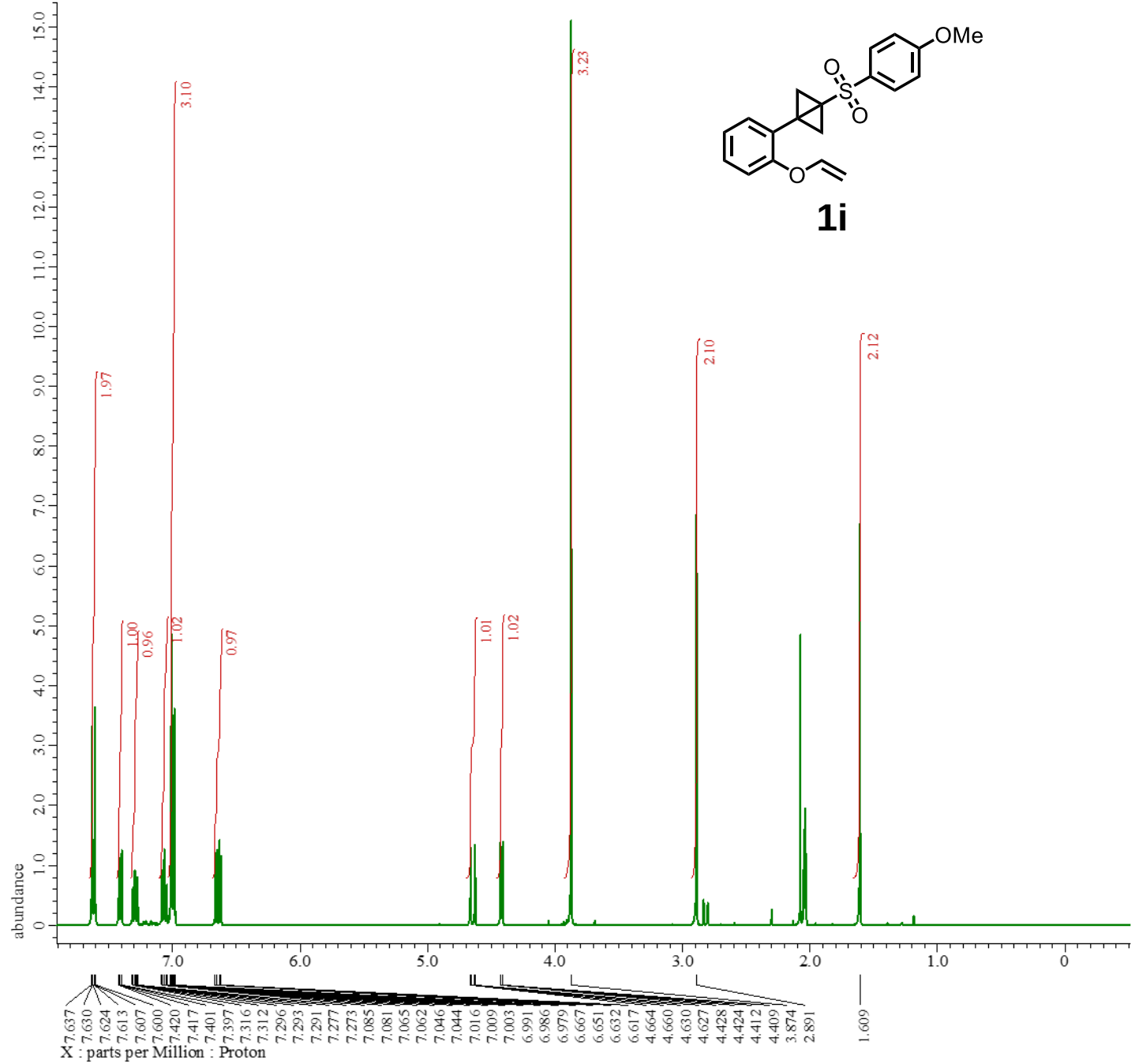
[1] H NMR spectrum of **1h** (400 MHz, Acetone-d_6)




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

```
Filename            = 8 O-propenyl Acetone-d6_C
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210674 column Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   =  7-JAN-2023 19:35:49
Revision_Time       = 17-MAR-2023 15:58:15

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 2300
Total_Scans         = 2300

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 21[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1h** (101 MHz, Acetone-d_6)

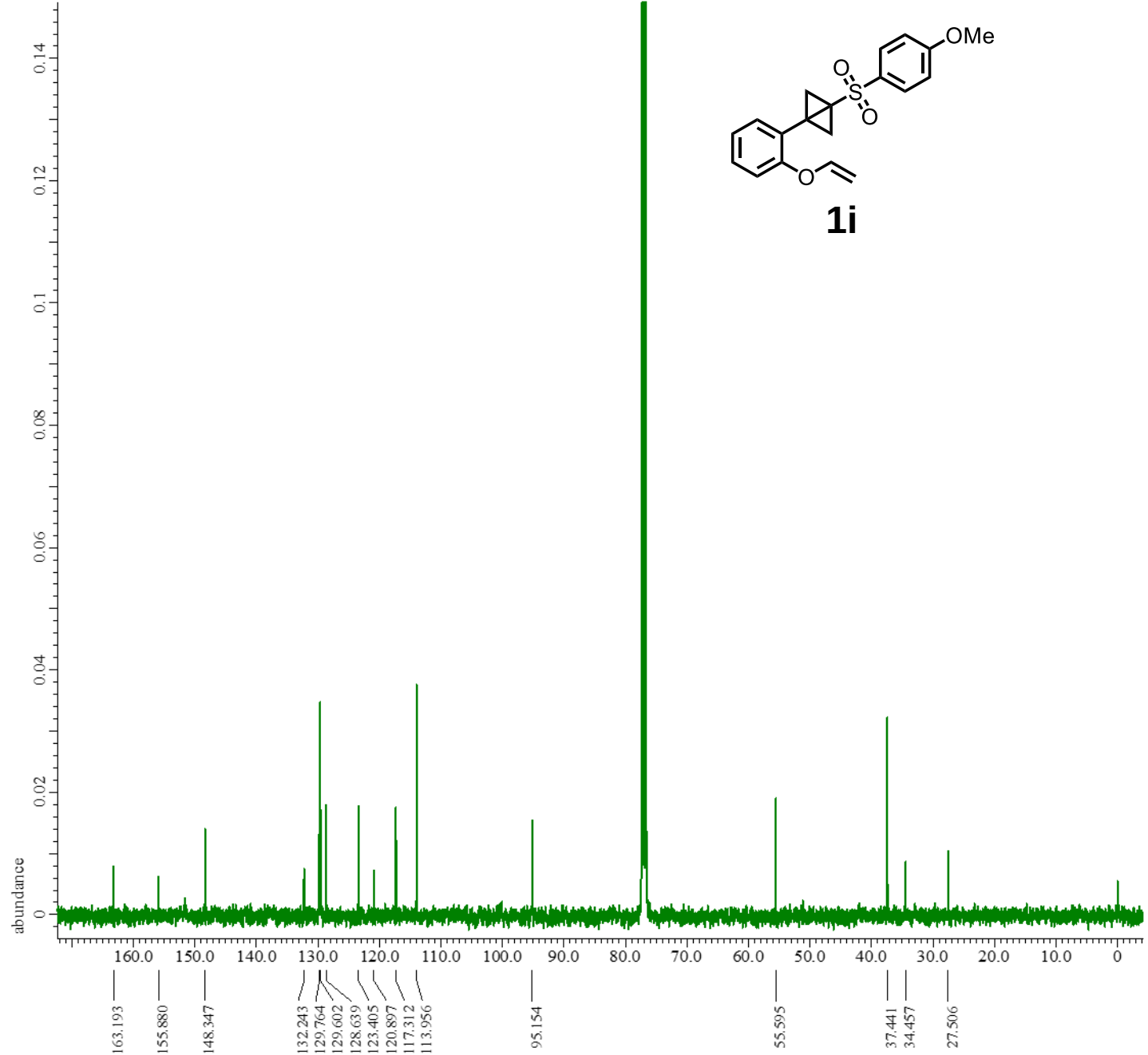



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 10 EWG 4-OMe Acetone-d6_P
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210662 column Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 19-DEC-2022 22:17:45
Revision_Time       = 15-MAR-2023 21:12:17

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 38
Temp_Get            = 20.3[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

7.637 7.630 7.624 7.613 7.607 7.600 7.420 7.417 7.401 7.397 7.316 7.312 7.296 7.293 7.291 7.277 7.273 7.085 7.081 7.065 7.062 7.046 7.044 7.016 7.009 7.003 6.991 6.986 6.979 6.667 6.651 6.632 6.617 4.664 4.660 4.630 4.627 4.428 4.424 4.412 4.409 3.874 2.891 1.609

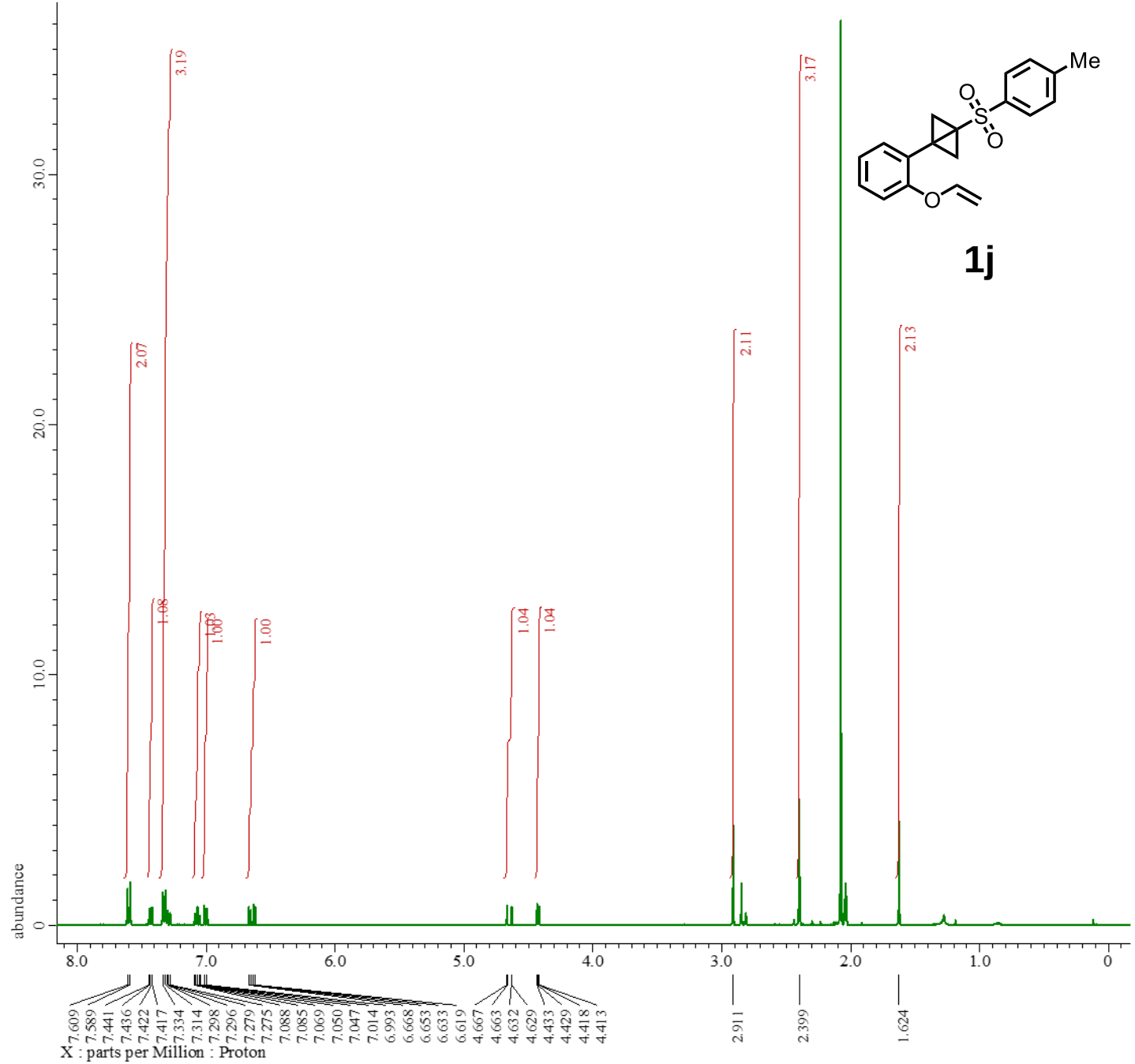
[1] H NMR spectrum of **1j** (400 MHz, Acetone-d_6)




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

Filename	= 10 EWG 4-MeOPh CDCl3_Carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 210545 column 2 CDCl3
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 23-SEP-2022 19:27:57
Revision_Time	= 4-JAN-2023 21:31:29
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1024
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 20.4[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 4.7[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 22.346[dB]
Irr_Atn_Noe	= 22.346[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]

^{13}C NMR spectrum of **1j** (101 MHz, $CDCl_3$)

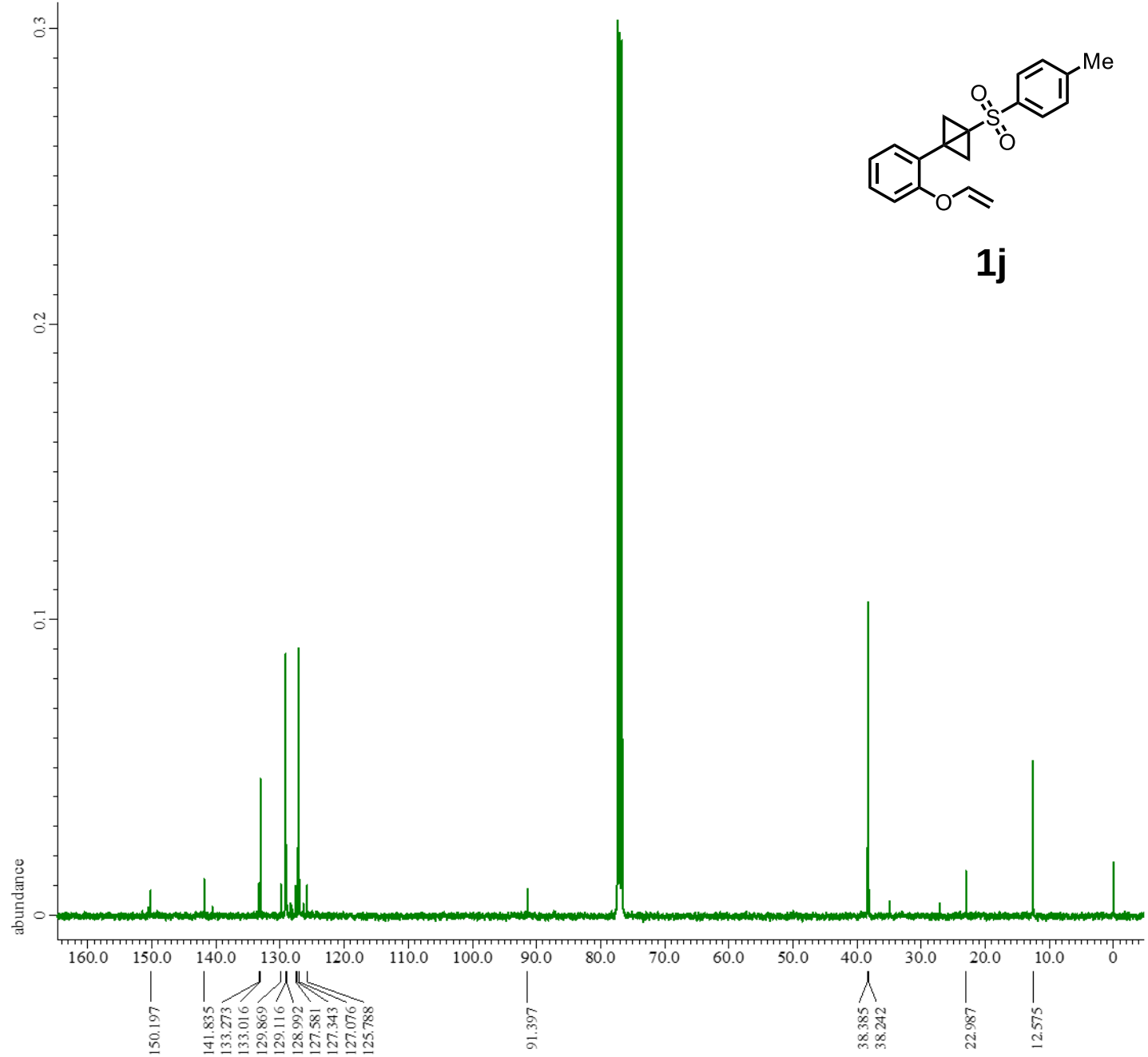


1j

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 9 EWG 4-Me Acetone-d6_Pro
Author              = delta
Experiment          = proton.jxp
Sample_Id           = EWG 4-Me Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 30-NOV-2022 09:52:05
Revision_Time       = 15-MAR-2023 22:09:18

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 36
Temp_Get            = 19.8[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **1g** (400 MHz, Acetone-d_6)

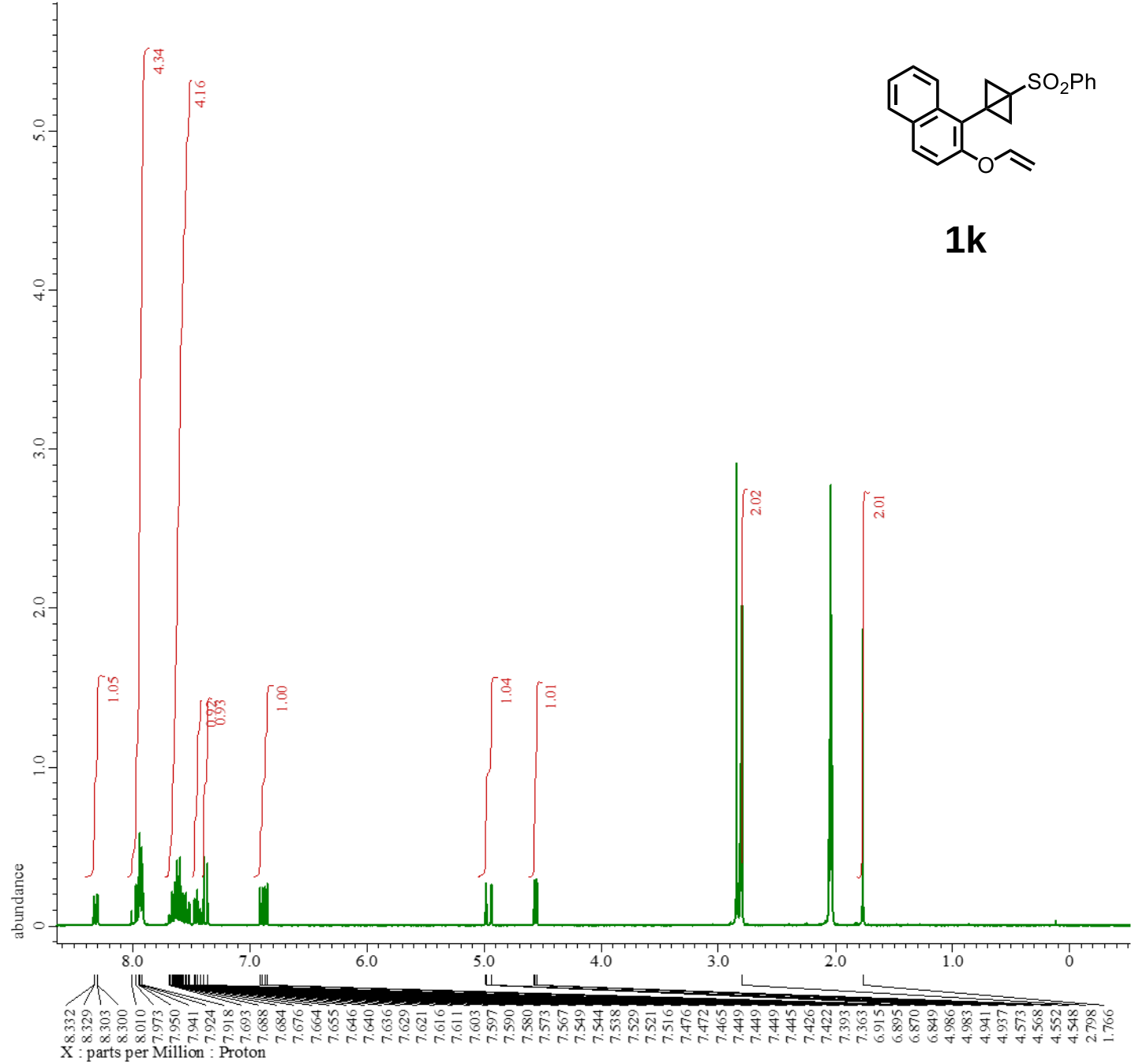


1j

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

```
Filename            = 9 EWG p-tolyl CDCl3_Carbo
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210549 GPC CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  1-OCT-2022 22:48:19
Revision_Time       =  4-JAN-2023 20:57:33

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1800
Total_Scans         = 1800

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.2[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1g** (101 MHz, $CDCl_3$)

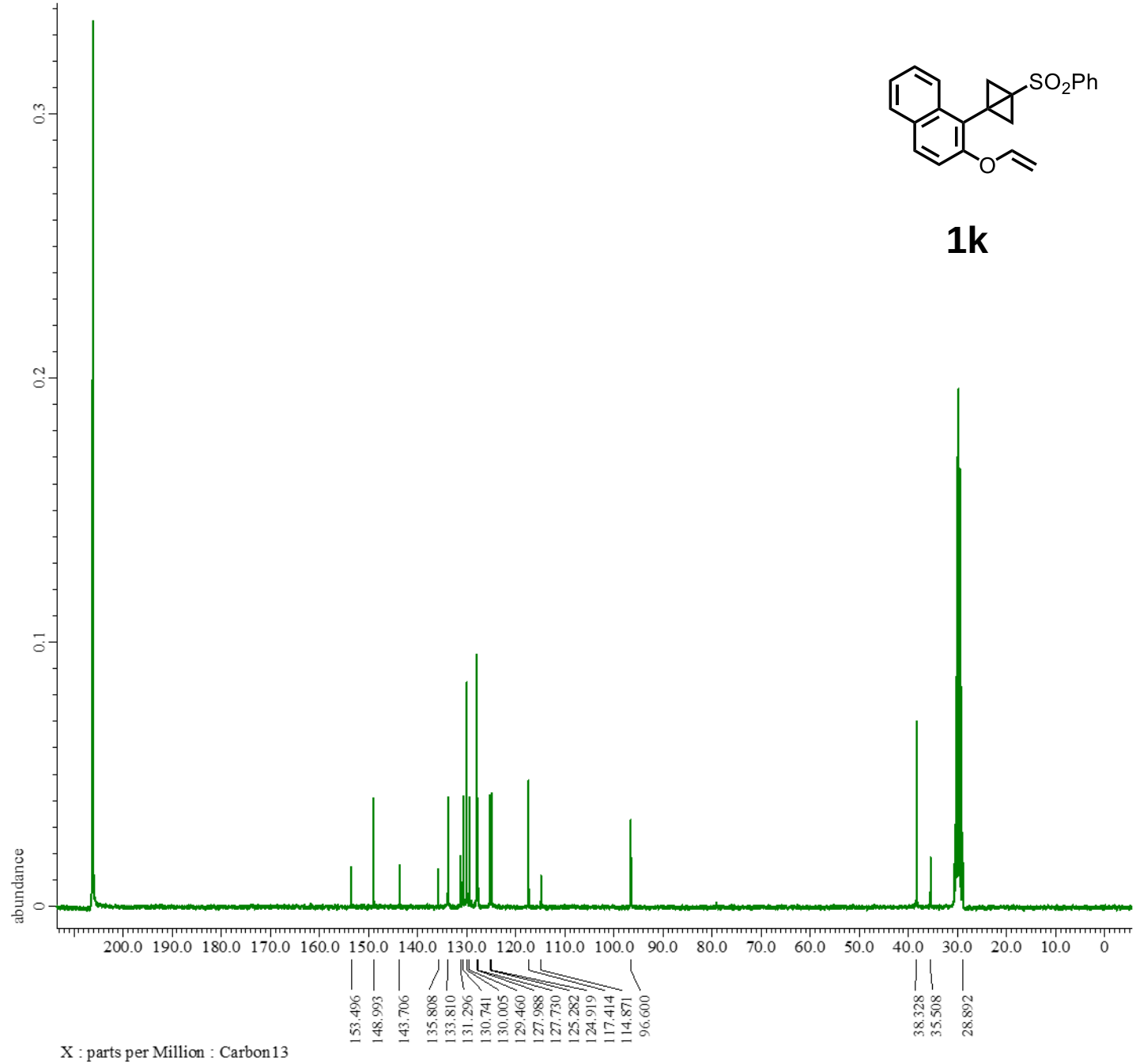


1k

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 34 1,2-naph Acetone-d6_Pr
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210684 check Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 16-MAR-2023 15:47:21
Revision_Time       = 17-MAR-2023 10:16:21

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 44
Temp_Get            = 20[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

[1] H NMR spectrum of **1k** (301 MHz, Acetone-d_6)

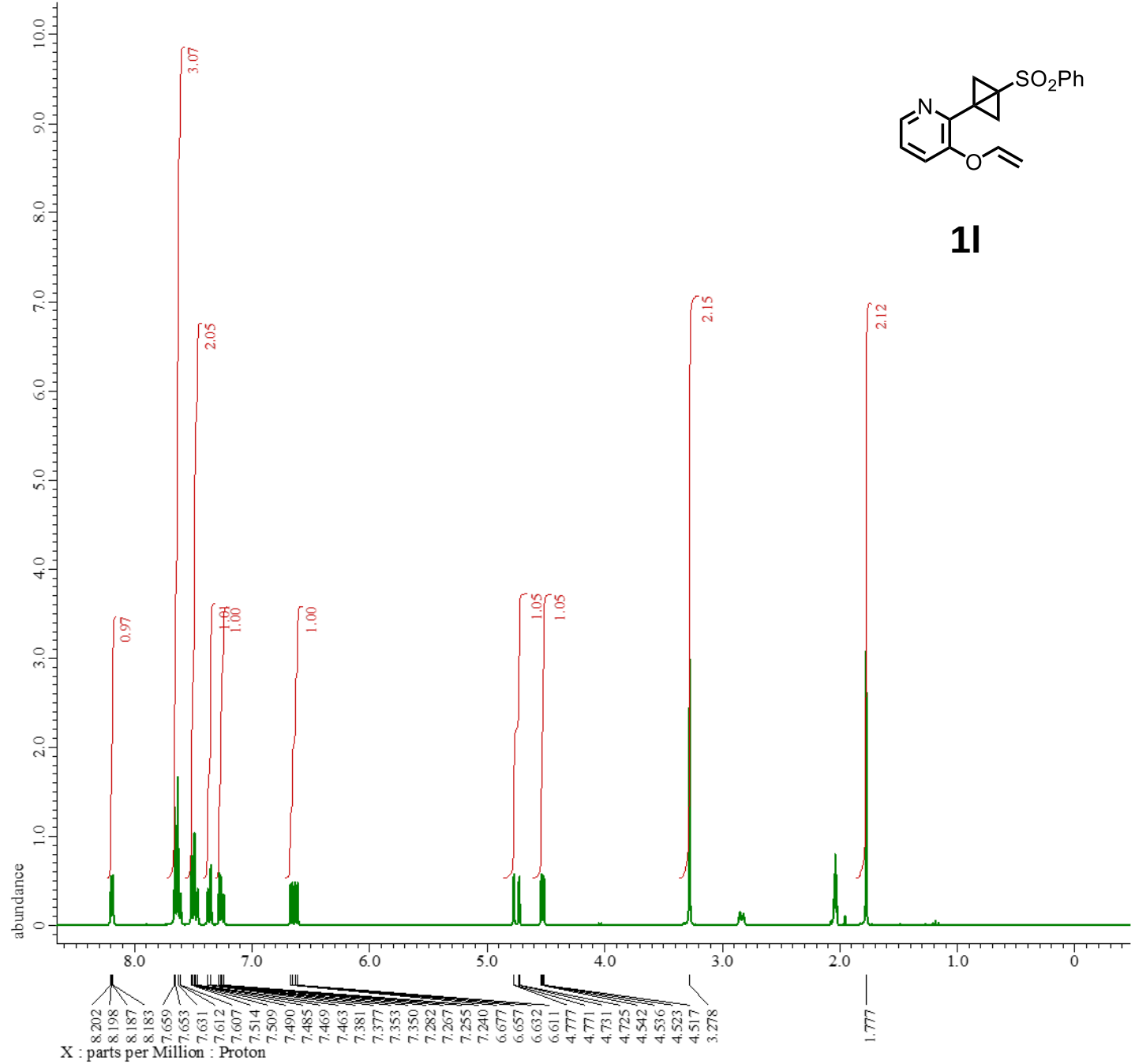


1k

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

```
Filename            = 210684 check2 Acetone-d6_
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210684 check2 Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 17-MAR-2023 10:01:08
Revision_Time       = 17-MAR-2023 11:47:17

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 1.38412032[s]
X_Domain            = 13C
X_Freq              = 75.56823426[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.72248054[Hz]
X_Sweep             = 23.67424242[kHz]
X_Sweep_Clipped     = 18.93939394[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 2501
Total_Scans         = 2501

Relaxation_Delay    = 1[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 11.4[us]
X_Acq_Time          = 1.38412032[s]
X_Angle             = 30[deg]
X_Atn               = 5.4[dB]
X_Pulse             = 3.8[us]
Irr_Atn_Dec         = 21.6[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.118[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = FALSE
Repetition_Time     = 2.38412032[s]
```

^{13}C NMR spectrum of **1k** (76 MHz, Acetone-d_6)

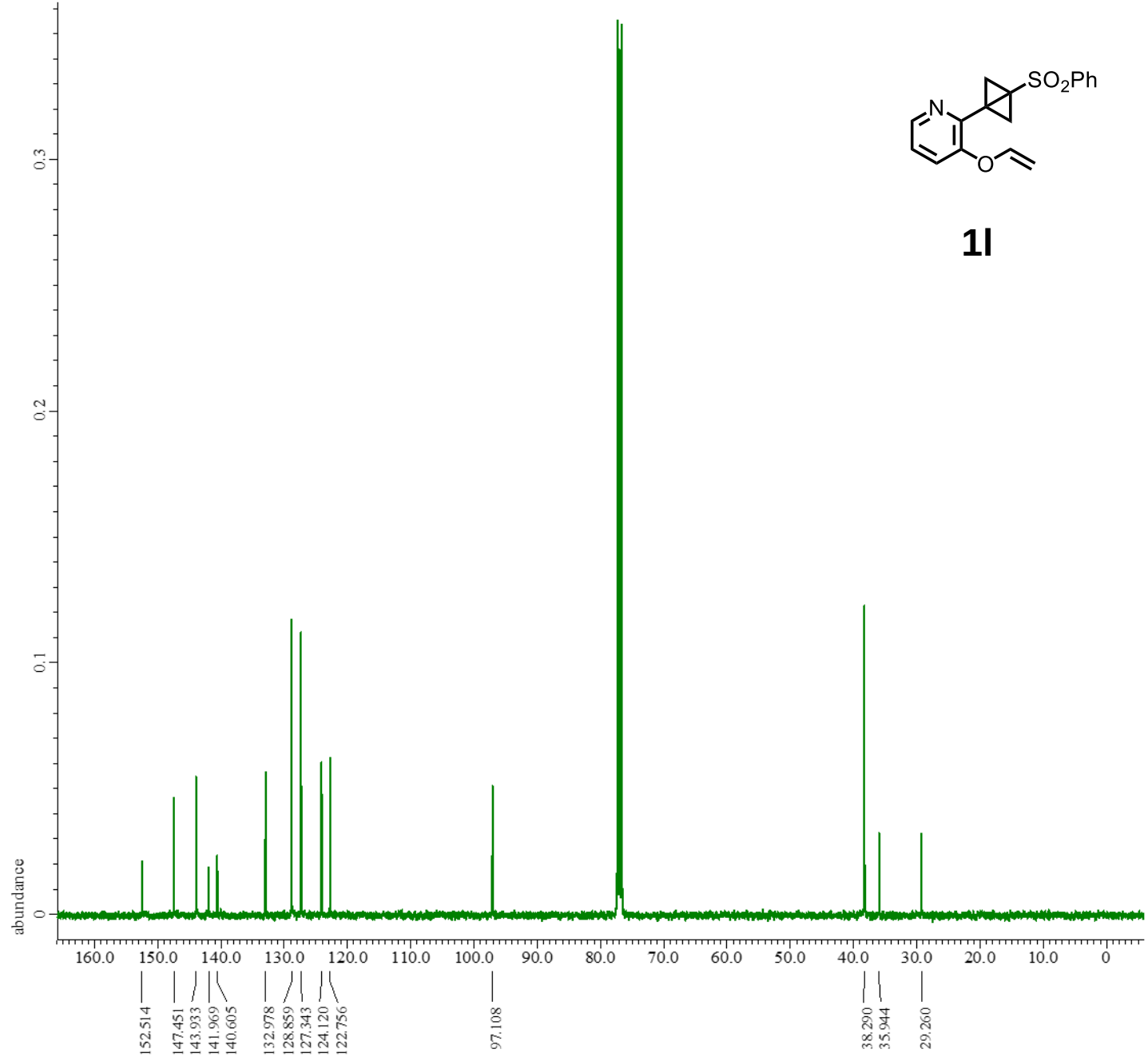


1l

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 33 pyridine Acetone-d6_Pr
Author              = delta
Experiment          = proton.jxp
Sample_Id           = pyridine sub check Aceton
Solvent             = ACETONE-D6
Actual_Start_Time   = 19-JAN-2023 18:23:39
Revision_Time       = 15-MAR-2023 22:34:02

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 38
Temp_Get            = 19.7[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

[1] H NMR spectrum of **1l** (301 MHz, Acetone-d_6)

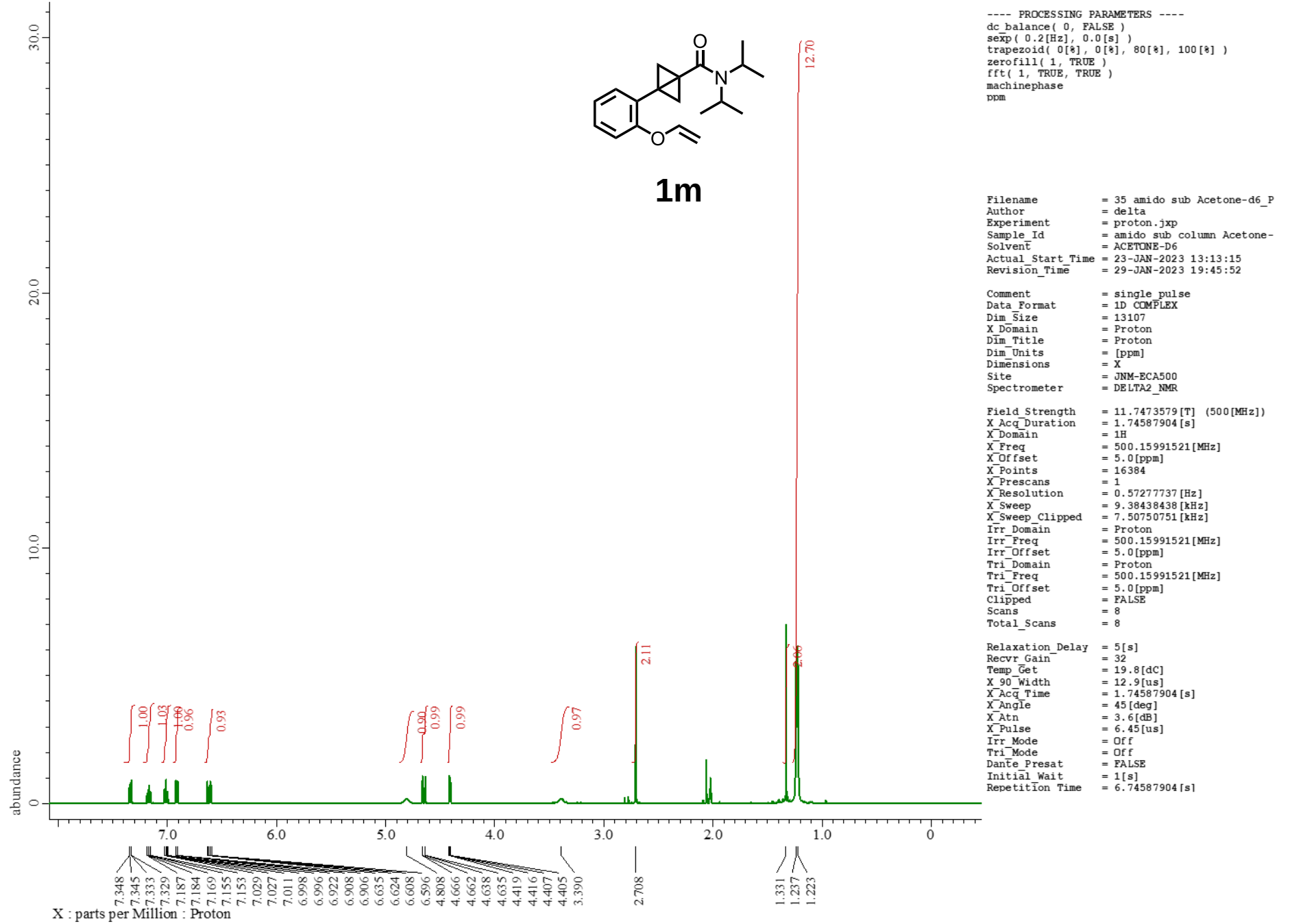


1l

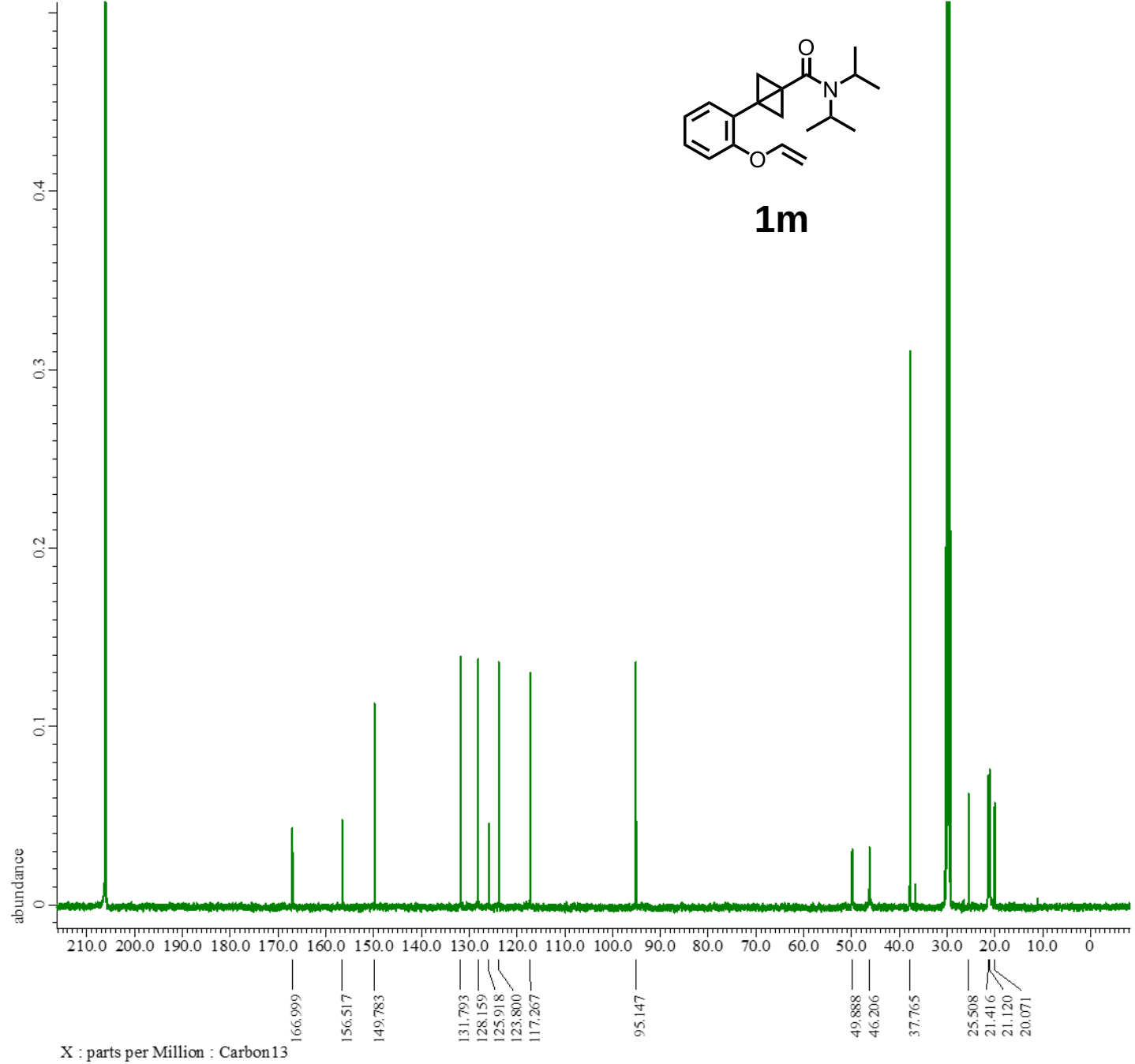
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 33 pyridine CDCl3_Carbon-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = pyridine sub check CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 25-JAN-2023 11:59:51
Revision_Time       = 29-JAN-2023 18:55:44

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 19.9[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **1l** (101 MHz, $CDCl_3$)



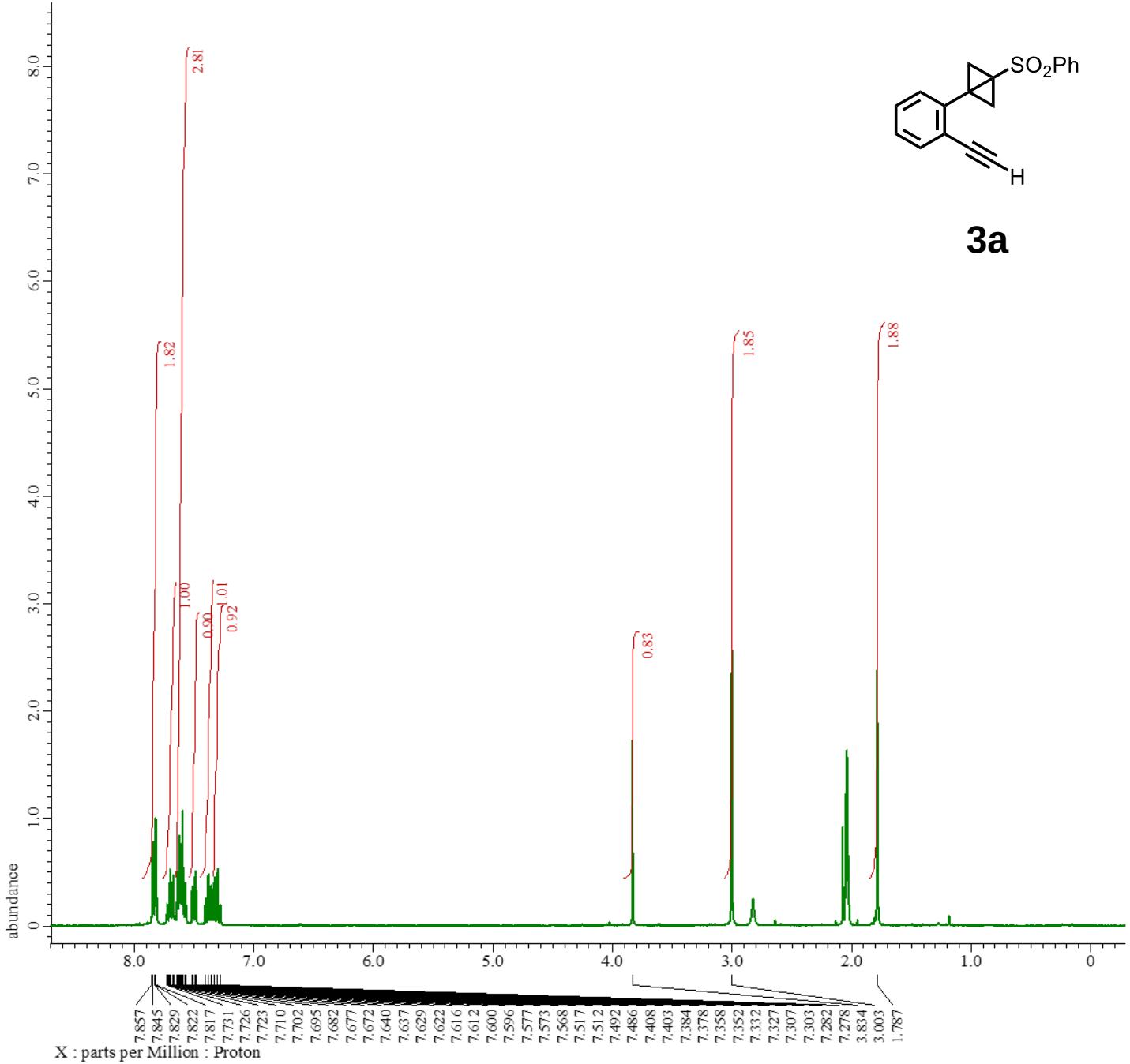
[1] H NMR spectrum of **1m** (500 MHz, Acetone-d_6)




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

```
Filename            = 35 amido sub Acetone-d6_C
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = amido sub column Acetone-
Solvent             = ACETONE-D6
Actual_Start_Time   = 23-JAN-2023 13:18:39
Revision_Time       = 17-MAR-2023 16:00:05

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T]  (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = TRUE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 4.1[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 21.078[dB]
Irr_Atn_Noe         = 20.664[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

^{13}C NMR spectrum of **1m** (126 MHz, Acetone-d_6)

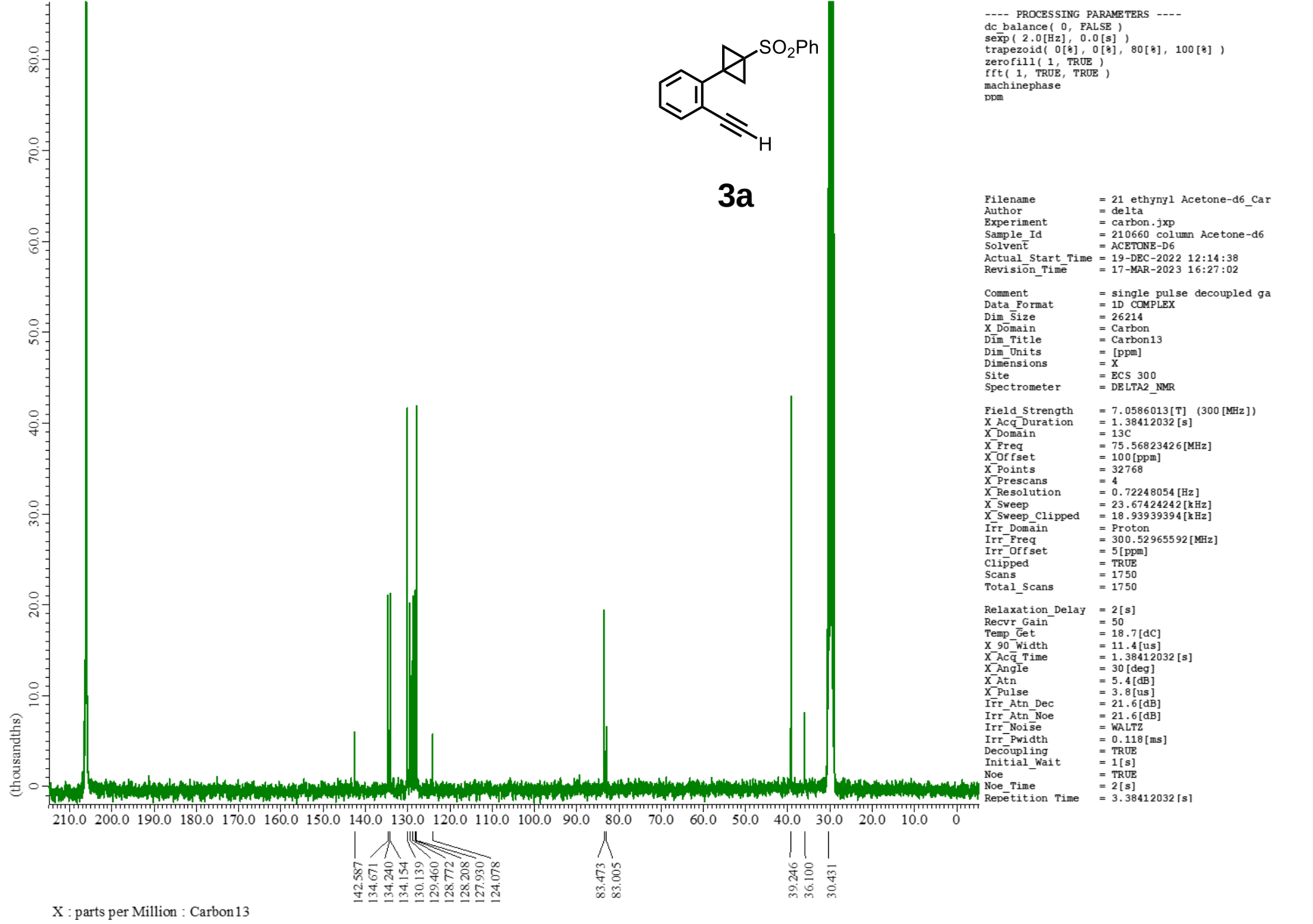


3a

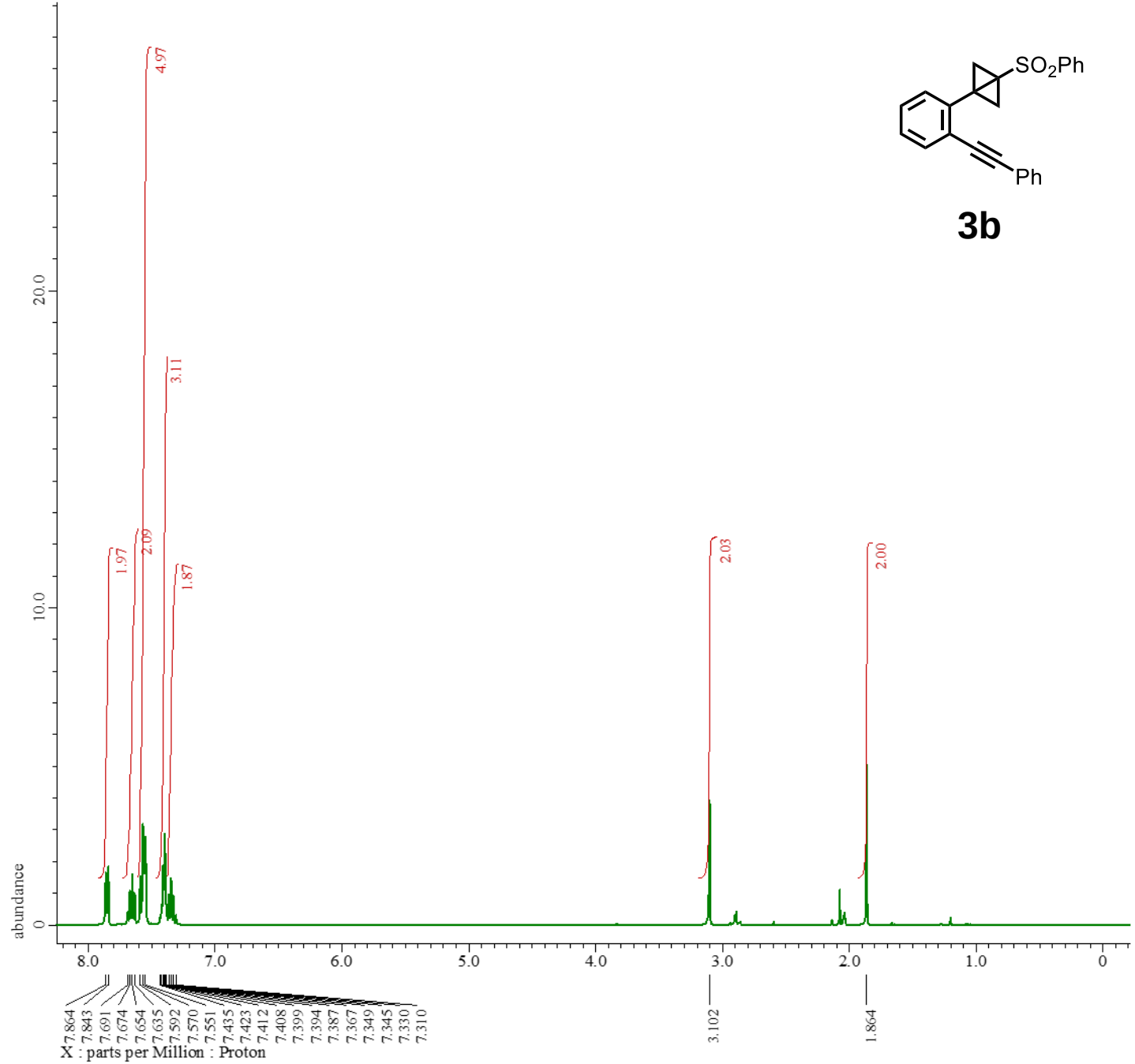
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 21 ethynyl Acetone-d6_Pro
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210660 column Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 19-DEC-2022 11:59:44
Revision_Time       = 15-MAR-2023 22:46:41

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 42
Temp_Get            = 18.6[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

[1] H NMR spectrum of **3a** (301 MHz, Acetone-d_6)



^{13}C NMR spectrum of **3a** (76 MHz, Acetone-d_6)

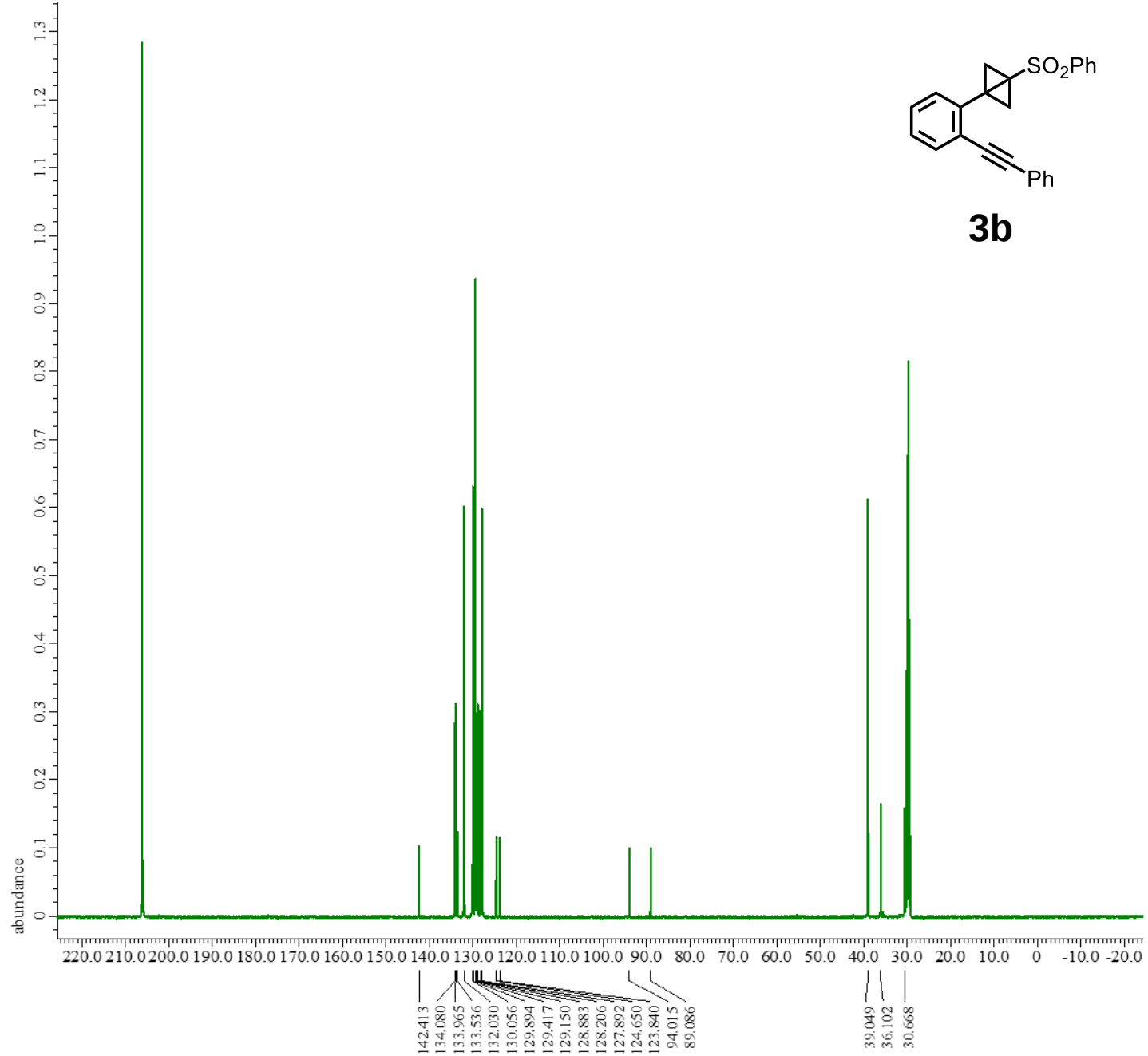


3b

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 22 phenylethynyl Acetone-
Author            = delta
Experiment        = proton.jxp
Sample_Id         = phenylethynyl Acetone-d6
Solvent           = ACETONE-D6
Actual_Start_Time = 25-NOV-2022 22:37:16
Revision_Time     = 16-JAN-2023 17:39:24

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proton
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 19.8[dC]
X_90_Width        = 12.4[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

[1] H NMR spectrum of **3b** (400 MHz, Acetone-d_6)

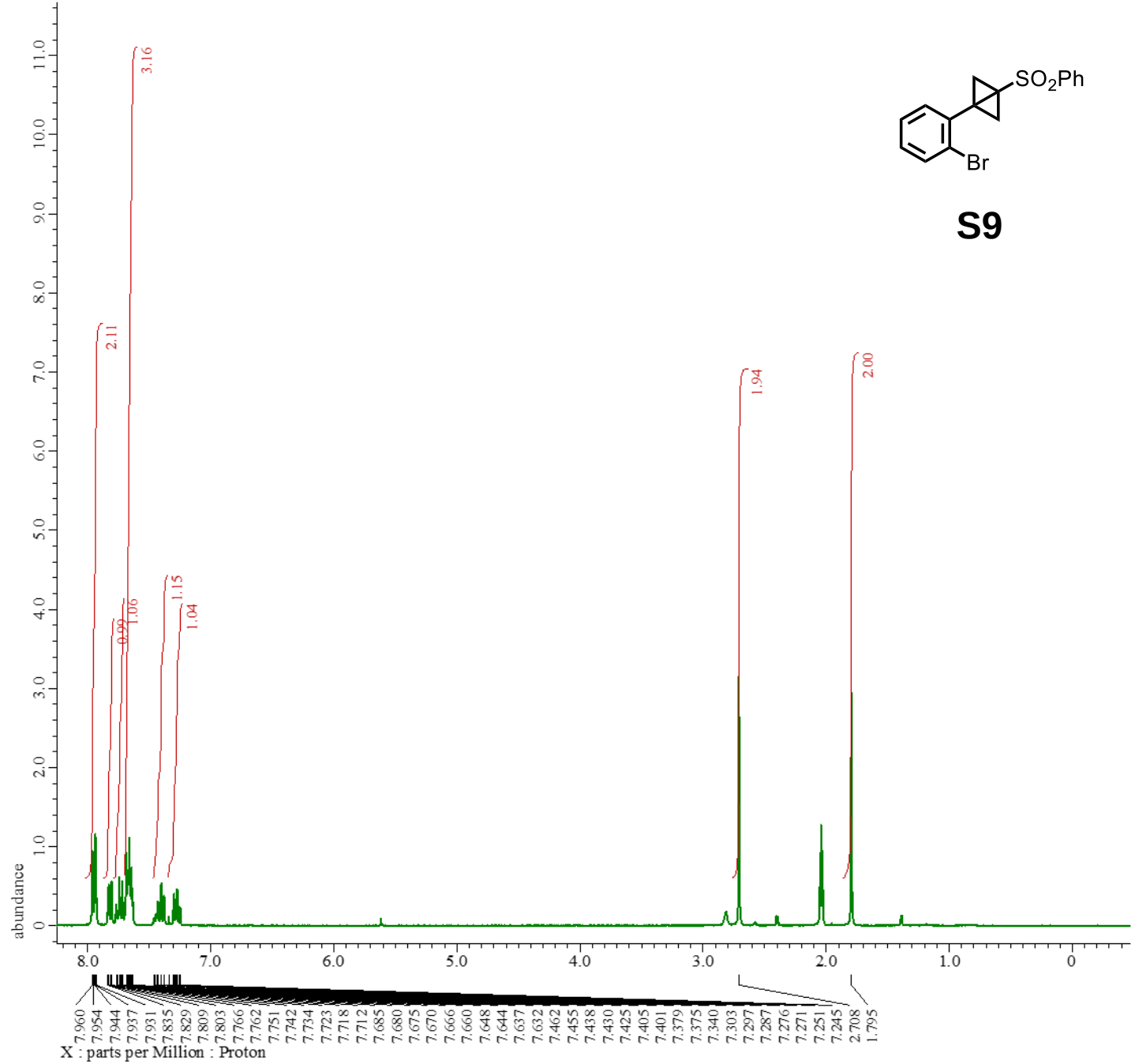


3b

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

```
Filename            = 22 phenylethynyl Acetone-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = phenylethynyl Acetone-d6
Solvent             = ACETONE-D6
Actual_Start_Time   = 29-NOV-2022 00:08:34
Revision_Time       = 17-MAR-2023 16:47:17

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 19.9[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **3b** (101 MHz, Acetone-d_6)

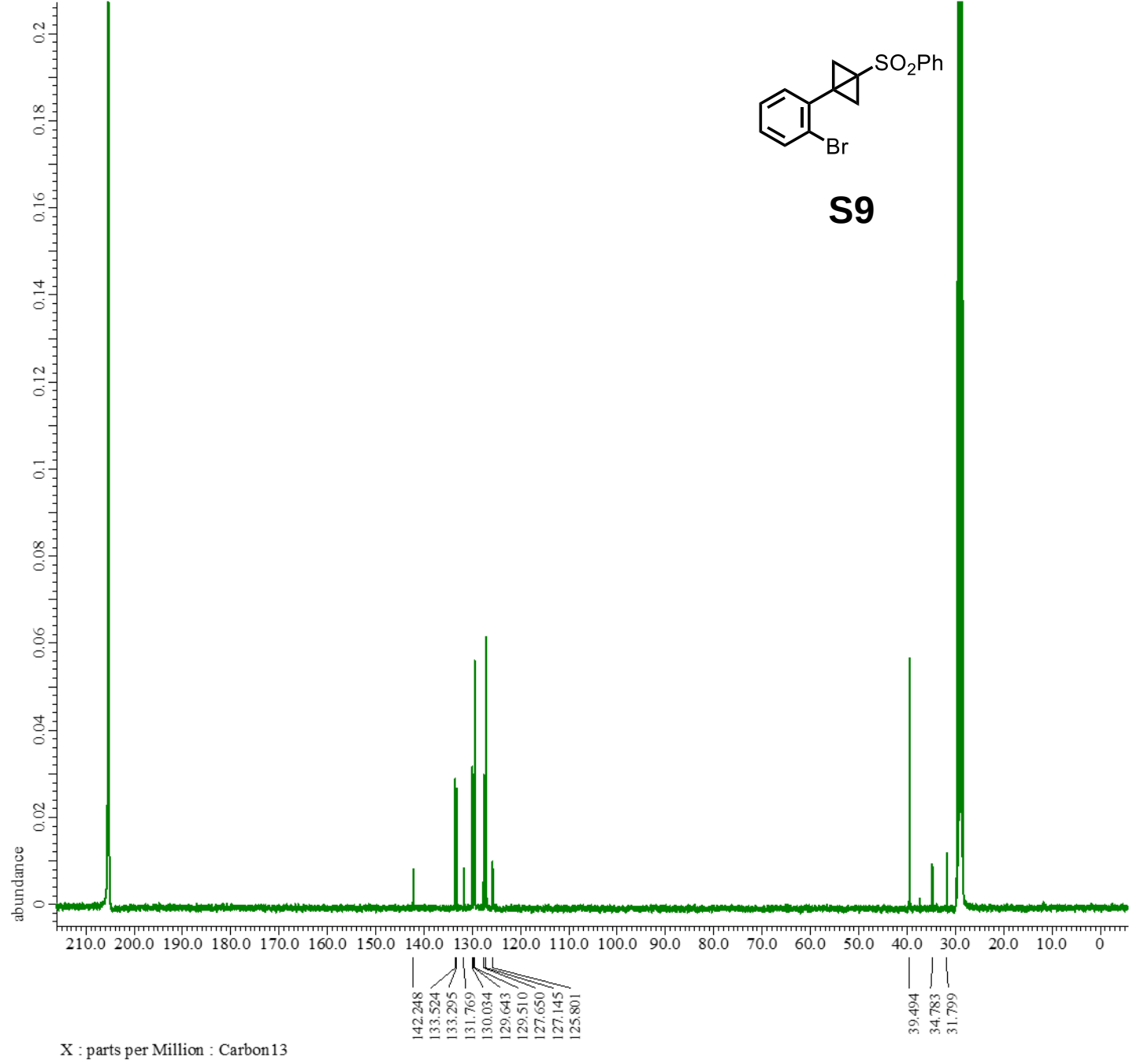


S9

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 25 Bromo sub Acetone-d6_P
Author            = delta
Experiment        = proton.jxp
Sample_Id         = Bromo sub Acetone-d6
Solvent           = ACETONE-D6
Actual_Start_Time =  5-NOV-2022 20:48:03
Revision_Time     = 18-JAN-2023 09:41:05

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proton
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 300
Spectrometer      = DELTA2_NMR

Field_Strength    = 7.0586013[T] (300[MHz])
X_Acq_Duration    = 2.90717696[s]
X_Domain          = 1H
X_Freq            = 300.52965592[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.34397631[Hz]
X_Sweep           = 5.63570784[kHz]
X_Sweep_Clipped   = 4.50856628[kHz]
Irr_Domain        = Proton
Irr_Freq          = 300.52965592[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 300.52965592[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 20.4[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.90717696[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.90717696[s]
```

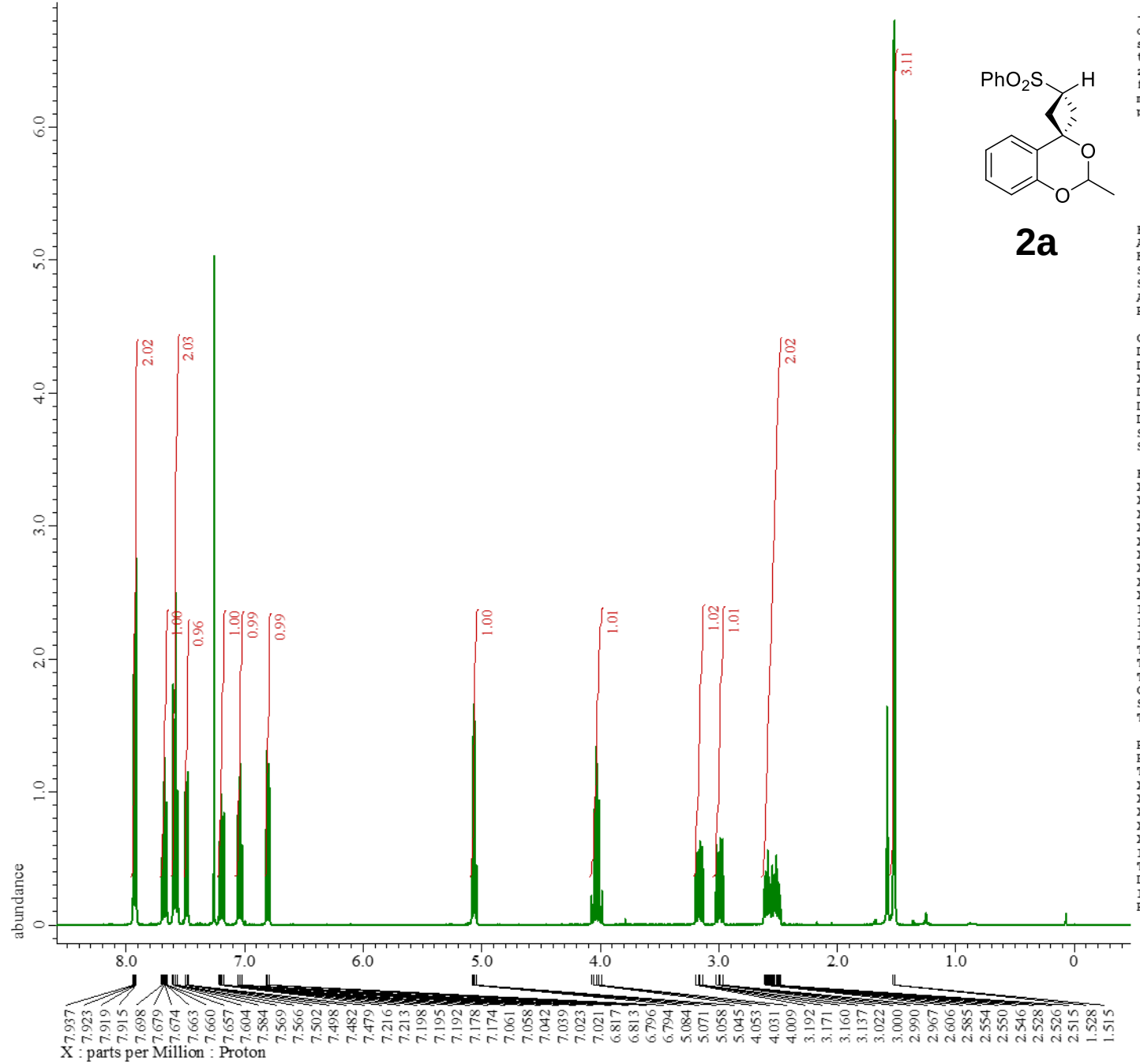
[1] H NMR spectrum of **S9** (301 MHz, Acetone-d_6)




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

```
Filename            = 25 Bromo sub_Carbon-1-3.j
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = Bromo sub
Solvent             = ACETONE-D6
Actual_Start_Time   = 13-NOV-2022 00:05:43
Revision_Time       = 17-MAR-2023 16:48:26

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 4650
Total_Scans         = 4650

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **S9** (101 MHz, Acetone-d_6)

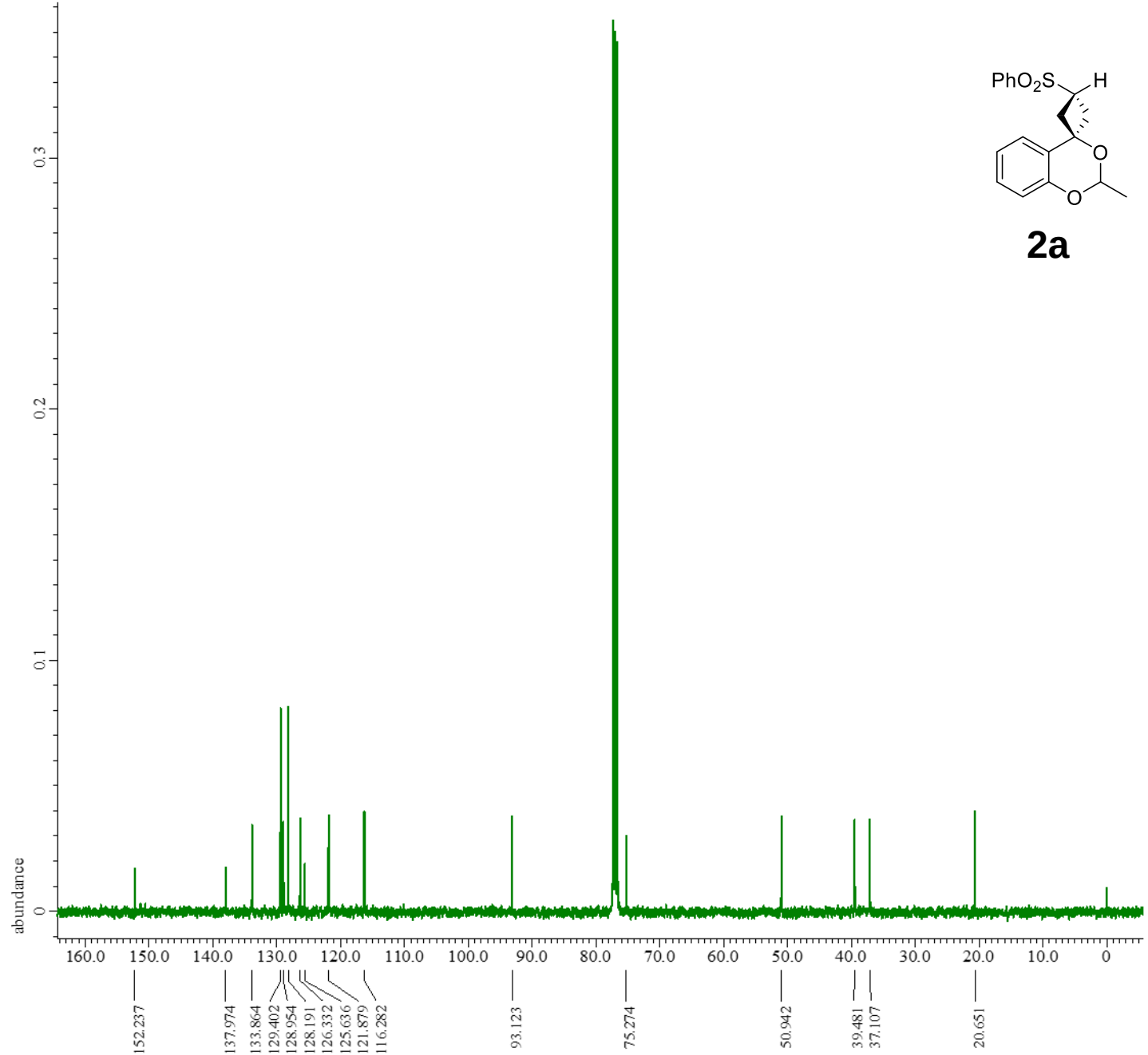


2a

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 11 ue CDCl3_Proton-1-3.jd
Author              = delta
Experiment          = proton.jxp
Sample_Id           = ue CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-NOV-2022 13:41:35
Revision_Time       = 16-MAR-2023 10:48:11

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 44
Temp_Get            = 20.1[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2a** (400 MHz, $CDCl_3$)

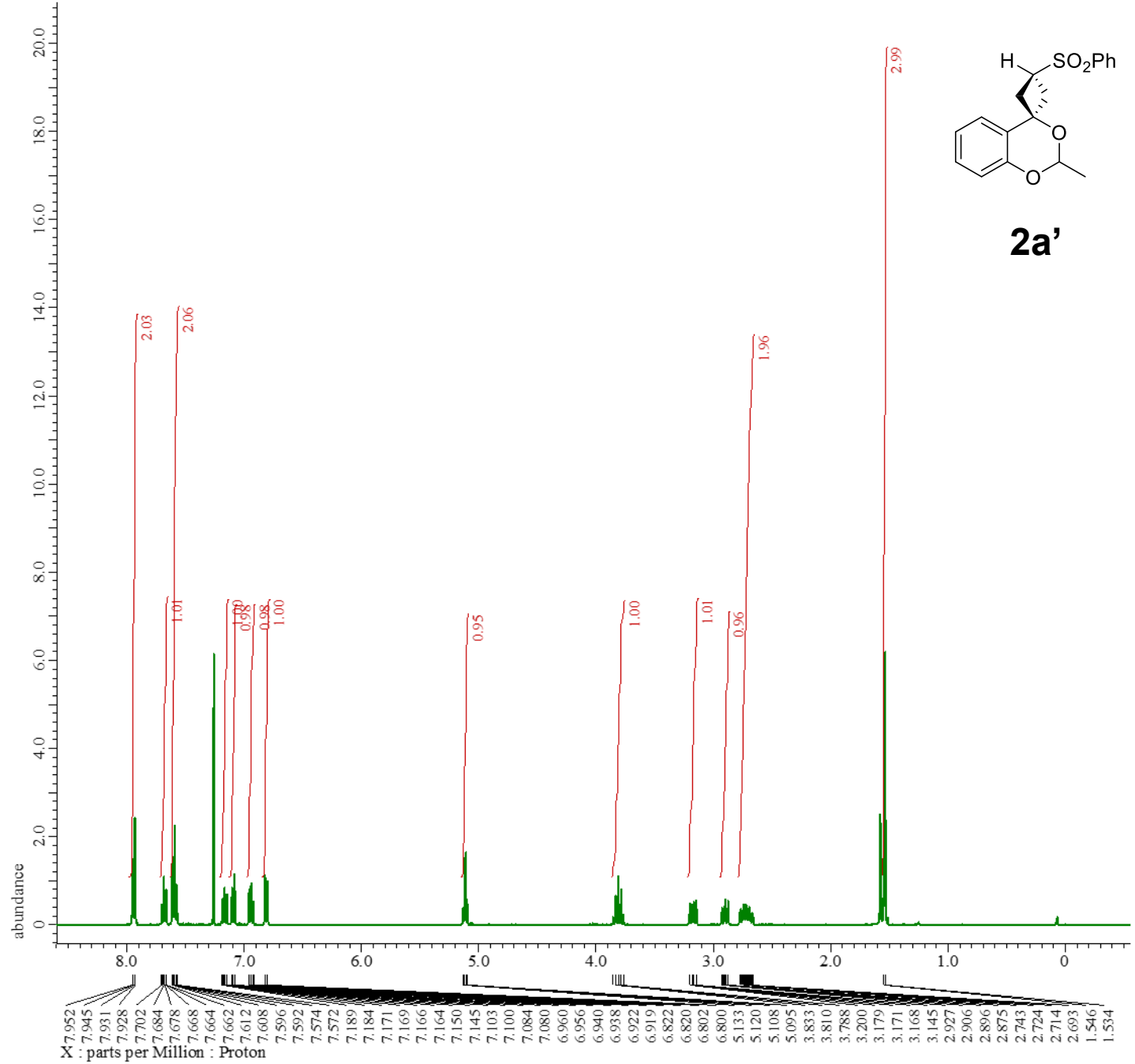


2a

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

```
Filename            = 11 ue CDCl3_Carbon-1-2.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = ue CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 31-OCT-2022 09:34:22
Revision_Time       =  5-JAN-2023 16:44:24

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 512
Total_Scans         = 512

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.2[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2a** (101 MHz, $CDCl_3$)

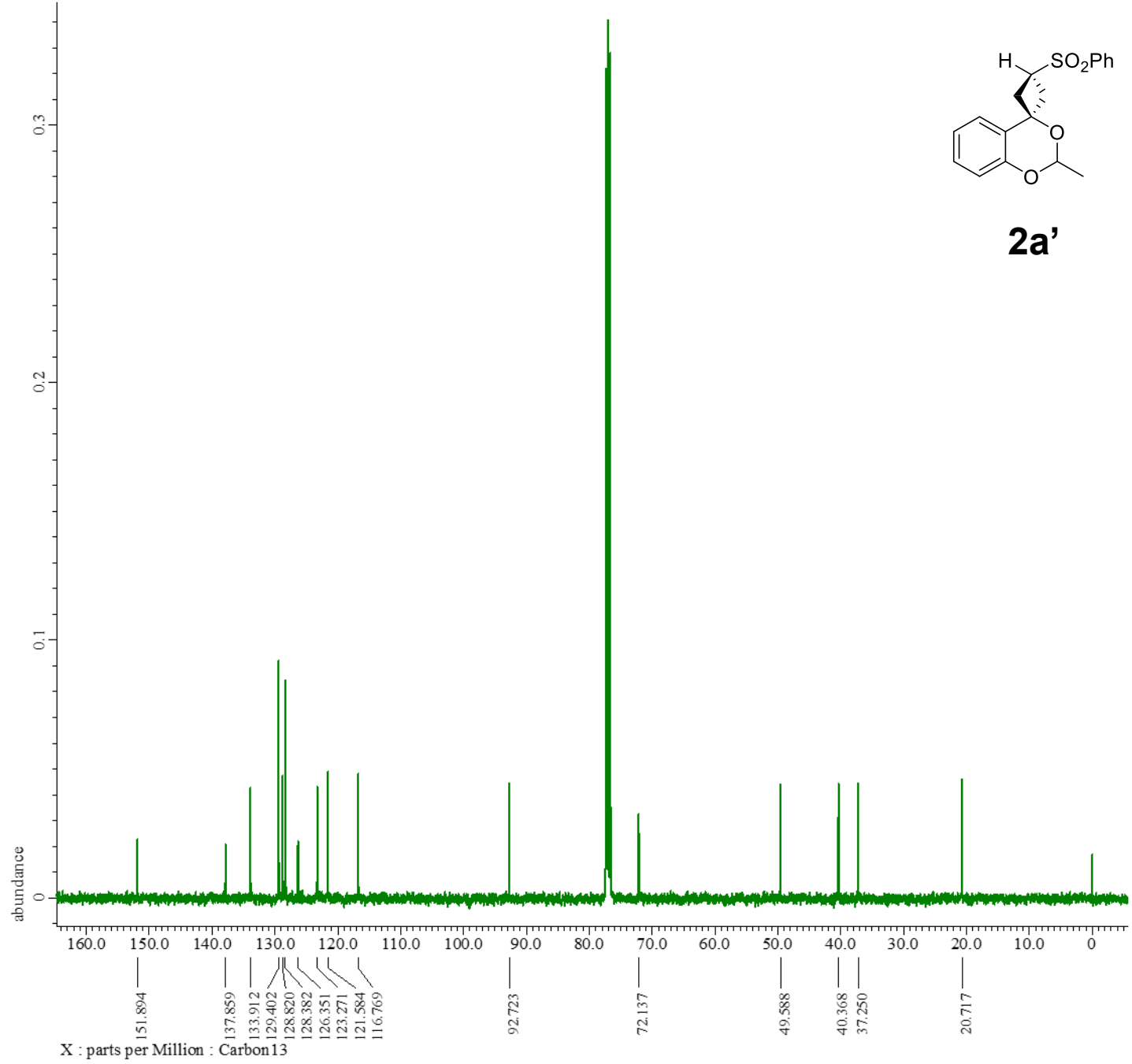


2a'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 11 shita CDCl3_Proton-1-3
Author              = delta
Experiment          = proton.jxp
Sample_Id           = shita CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-NOV-2022 13:47:56
Revision_Time       = 16-MAR-2023 10:48:41

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 46
Temp_Get            = 20.1[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2a'** (400 MHz, $CDCl_3$)

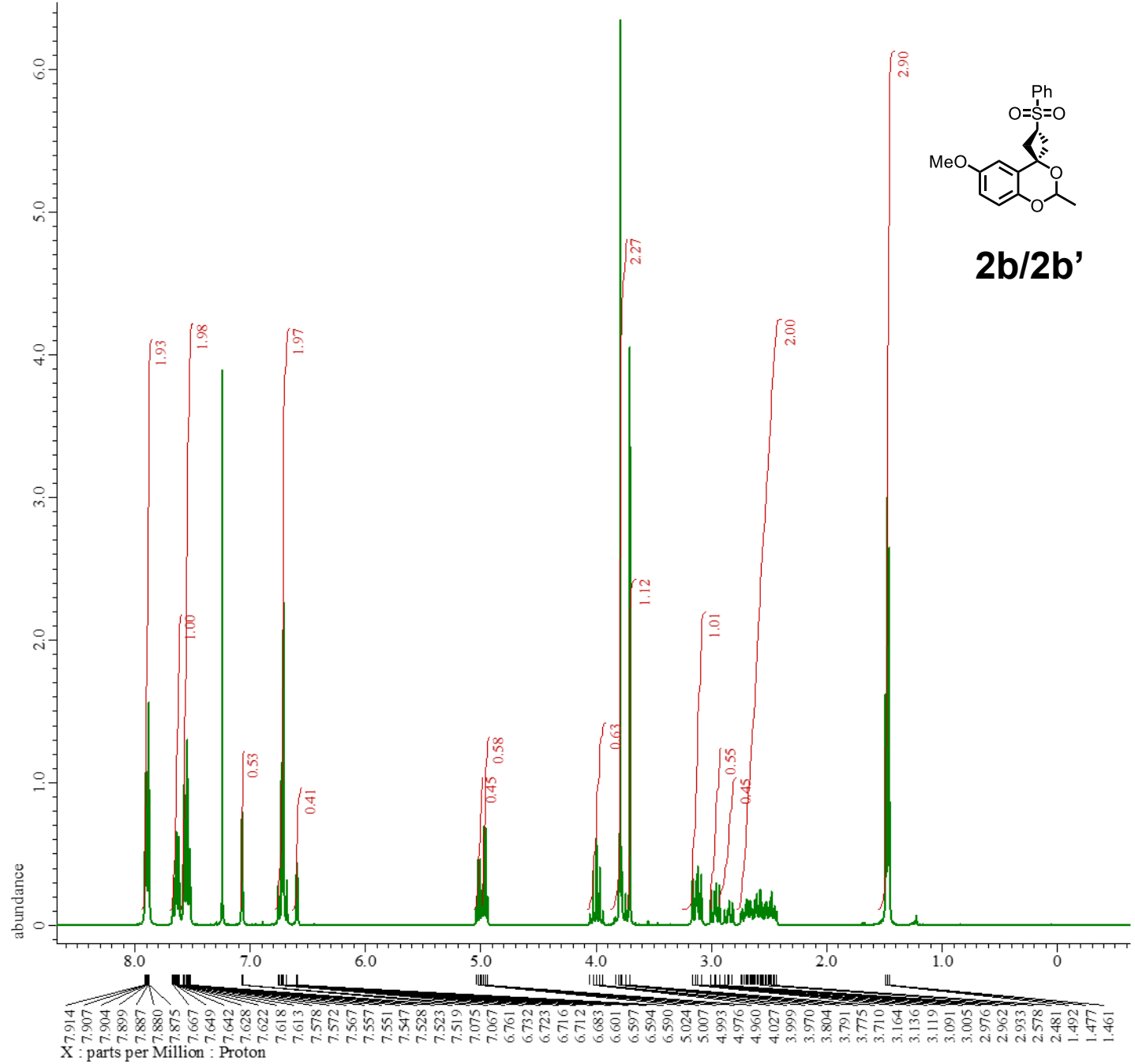


2a'

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

```
Filename            = 11 shita CDCl3_Carbon-1-2
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = shita CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 31-OCT-2022 10:07:39
Revision_Time       =  7-JAN-2023 09:41:49

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 512
Total_Scans         = 512

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2a'** (101 MHz, $CDCl_3$)

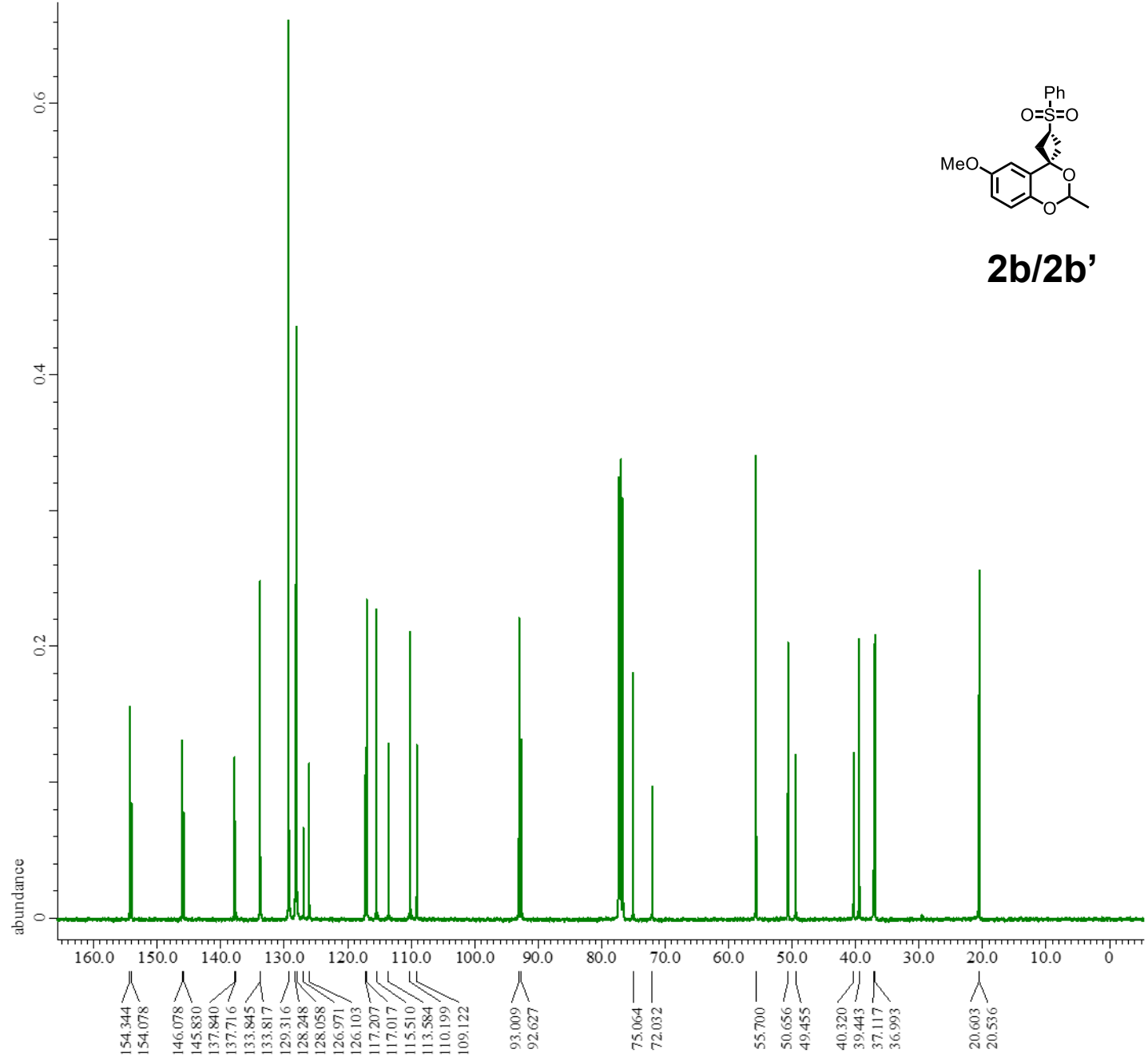


2b/2b'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 12 Ar 4-OMe CDCl3_Proton-
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 4-OMe PLC CDC13
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-DEC-2022 11:42:52
Revision_Time       = 16-MAR-2023 11:20:25

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 28
Temp_Get            = 19.8[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

[1] H NMR spectrum of **2b**/**2b'** (301 MHz, $CDCl_3$)

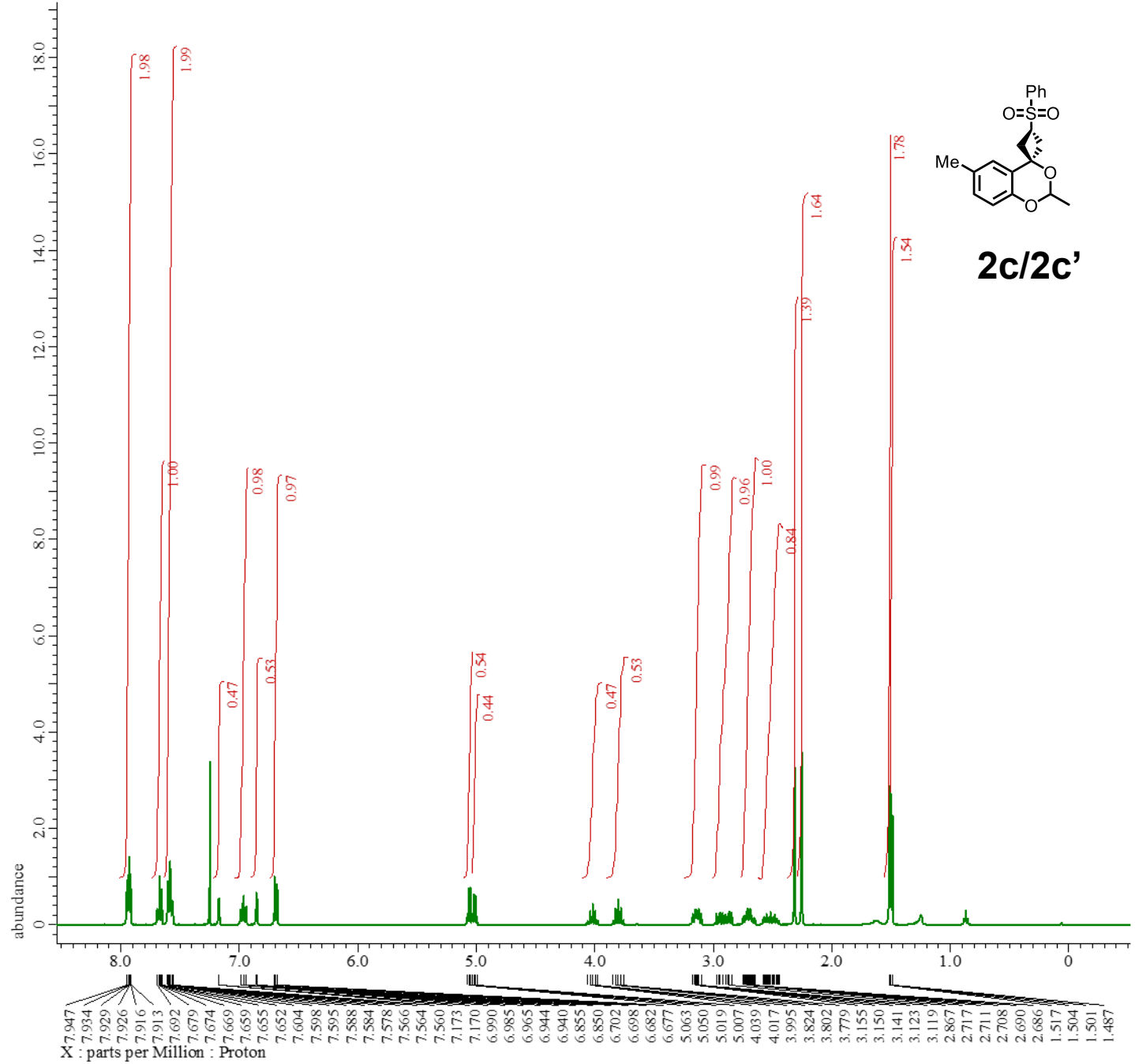


2b/2b'

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

```
Filename            = 12 Ar 4-OMe PLC CDCl3_Car
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 4-OMe PLC CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-DEC-2022 15:42:44
Revision_Time       = 11-JAN-2023 10:01:41

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1650
Total_Scans         = 1650

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 21.1[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

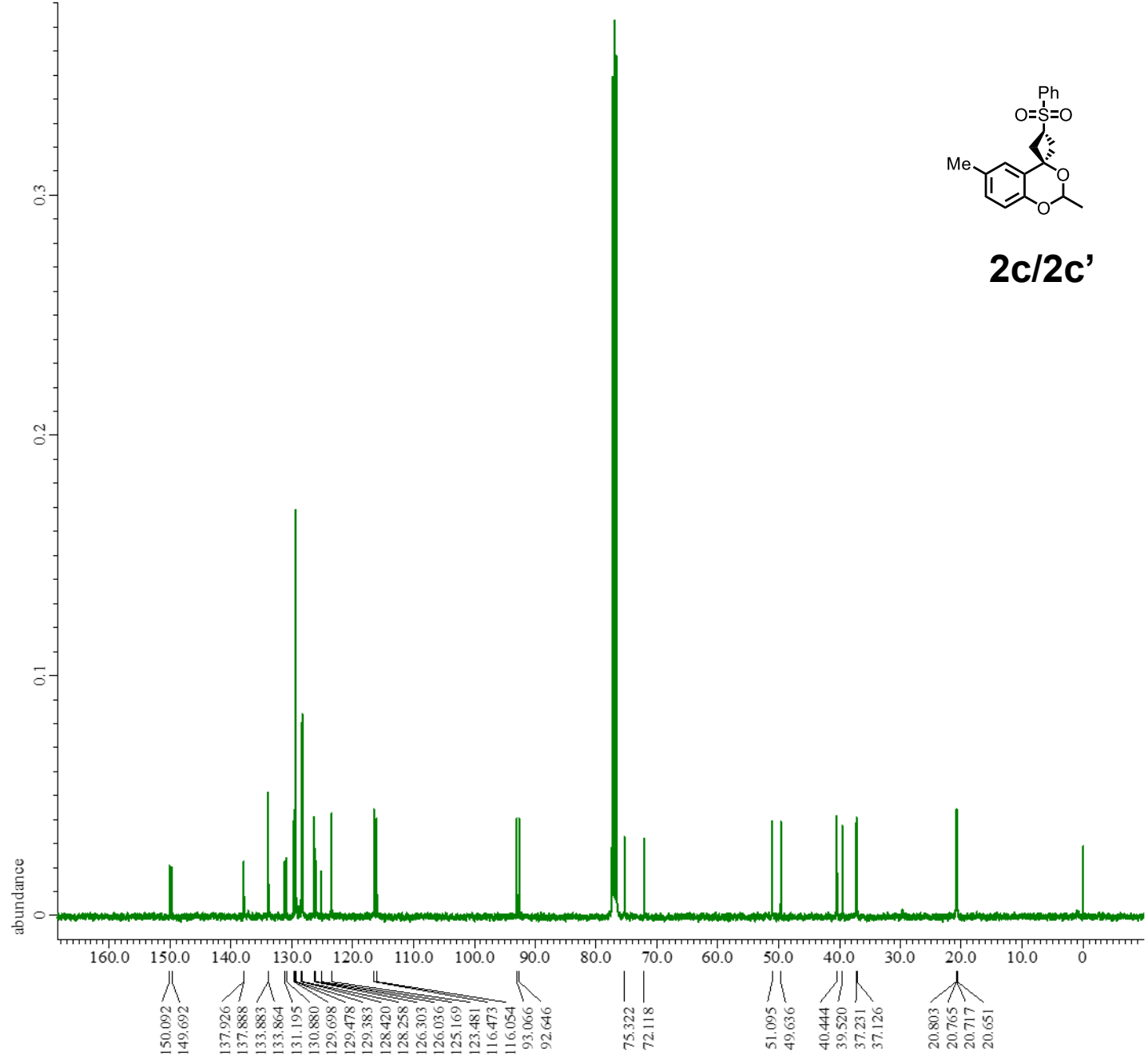
^{13}C NMR spectrum of **2b**/**2b'** (101 MHz, $CDCl_3$)



2c/2c'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 15 Ar 4-Me CDCl3_Proton-1
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210556 CDC13
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 25-NOV-2022 22:43:24
Revision_Time       = 11-JAN-2023 10:08:11

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 38
Temp_Get            = 19.7[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2c**/**2c'** (400 MHz, $CDCl_3$)

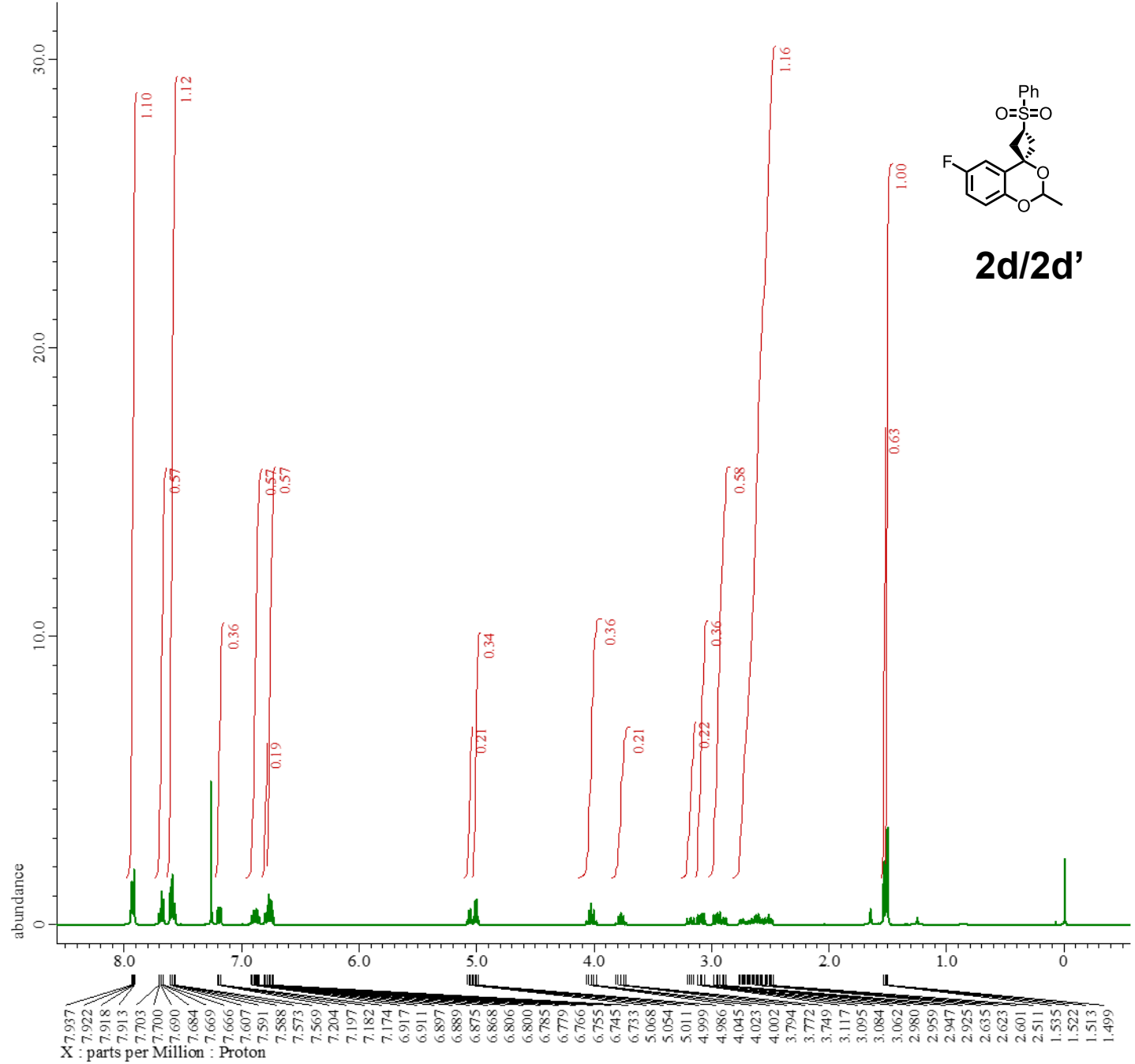


2c/2c'

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

```
Filename            = 15 Ar 4-Me CDCl3_Carbon-1
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210556 3 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-NOV-2022 03:05:51
Revision_Time       = 11-JAN-2023 11:03:31

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1150
Total_Scans         = 1150

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2c/2c'** (101 MHz, $CDCl_3$)

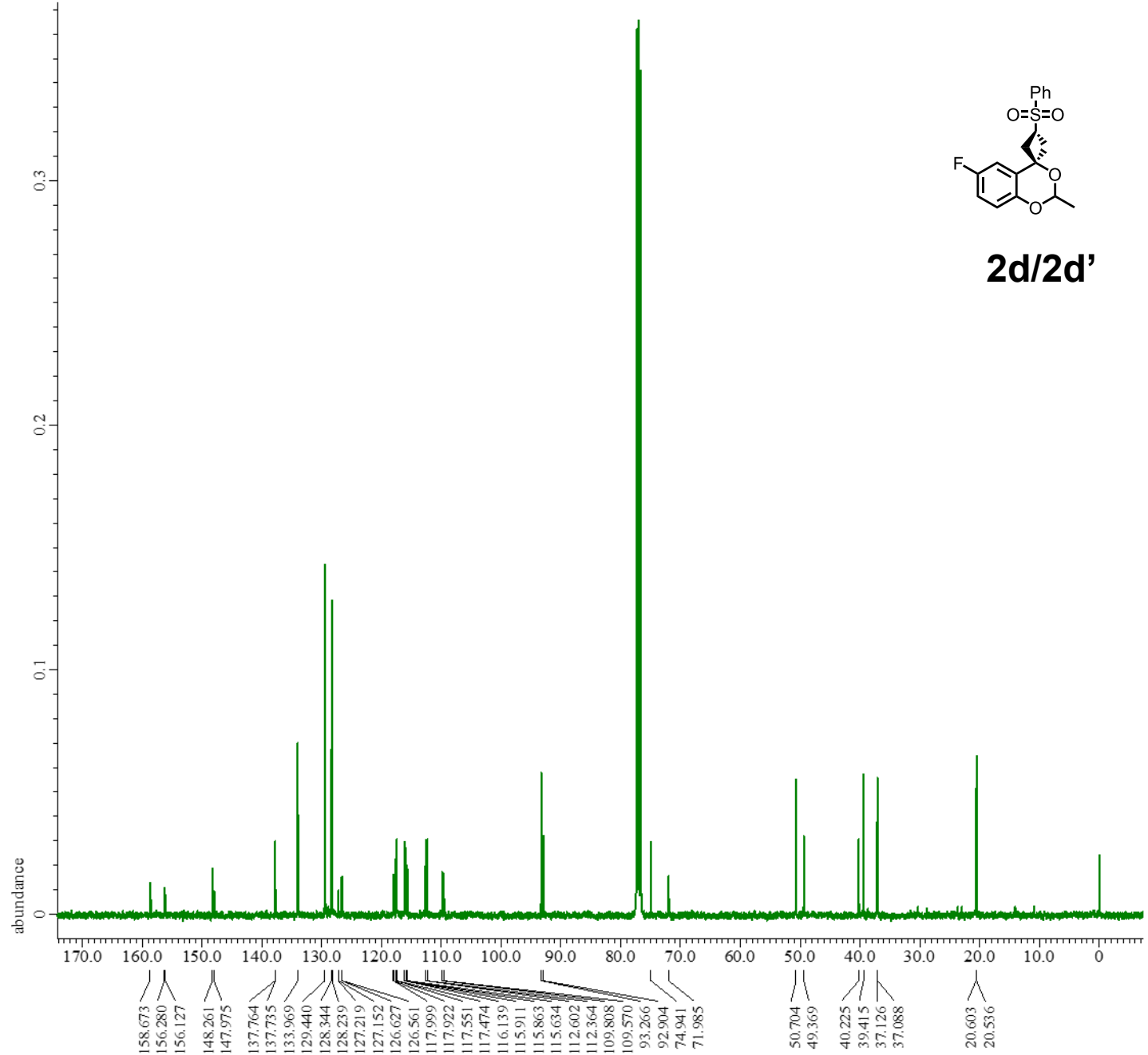


2d/2d'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 14 Ar 4-F CDCl3_Proton-1-
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210594 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 14-NOV-2022 15:31:42
Revision_Time       = 11-JAN-2023 11:52:53

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 36
Temp_Get            = 20[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2d/2d'** (400 MHz, $CDCl_3$)

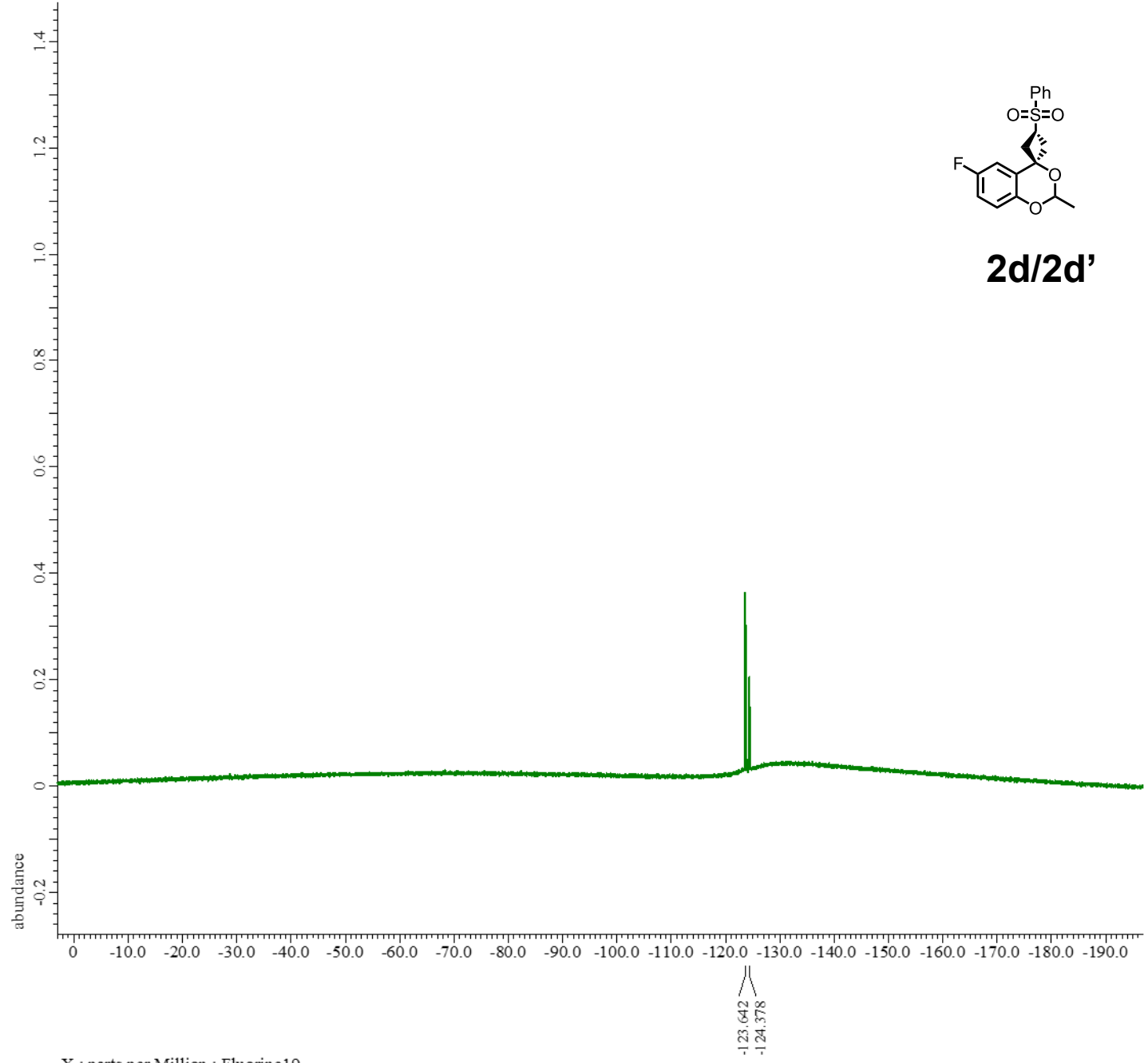


2d/2d'

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

```
Filename            = 14 Ar 4-F CDCl3_Carbon-1-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210594 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-NOV-2022 08:30:16
Revision_Time       = 16-MAR-2023 11:58:52

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1150
Total_Scans         = 1150

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2d/2d'** (101 MHz, $CDCl_3$)

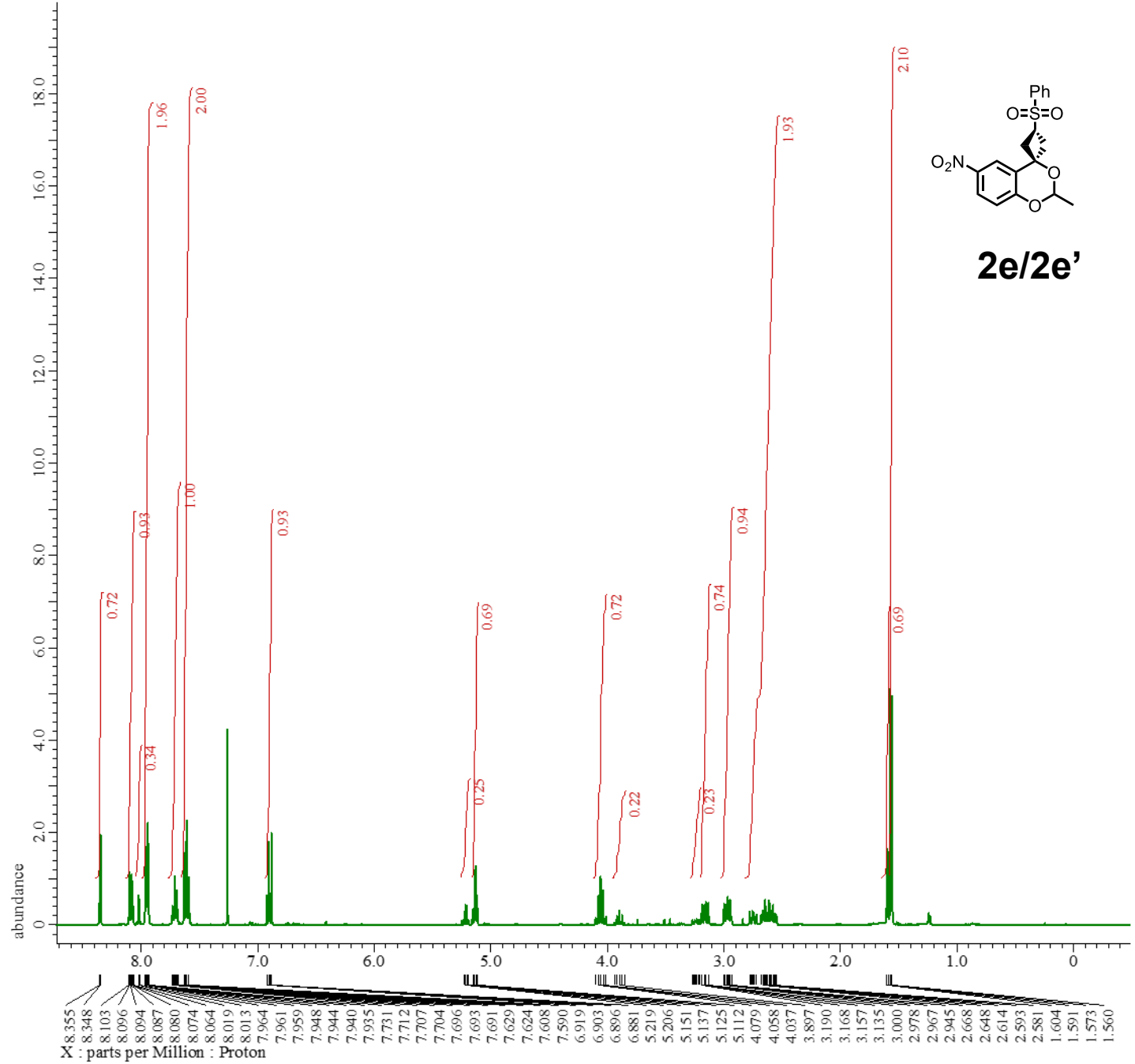


2d/2d'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 14 Ar 4-F CDCl3-4.jdf
Author              = delta
Experiment          = single_pulse.jxp
Sample_Id           = 210664-665 column CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 22-DEC-2022 10:23:58
Revision_Time       = 17-MAR-2023 16:50:43

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Fluori
Dim_Title           = Fluorine19
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 0.15368192[s]
X_Domain            = 19F
X_Freq              = 282.78036857[MHz]
X_Offset            = -80[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 6.5069463[Hz]
X_Sweep             = 106.6098081[kHz]
X_Sweep_Clipped     = 85.28784648[kHz]
Irr_Domain          = Fluorine19
Irr_Freq            = 282.78036857[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Fluorine19
Tri_Freq            = 282.78036857[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 32
Temp_Get            = 18.9[dC]
X_90_Width          = 12[us]
X_Acq_Time          = 0.15368192[s]
X_Angle             = 45[deg]
X_Atn               = 2.8[dB]
X_Pulse             = 6[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 5.15368192[s]
```

^{19}F NMR spectrum of **2d/2d'** (283 MHz, $CDCl_3$)

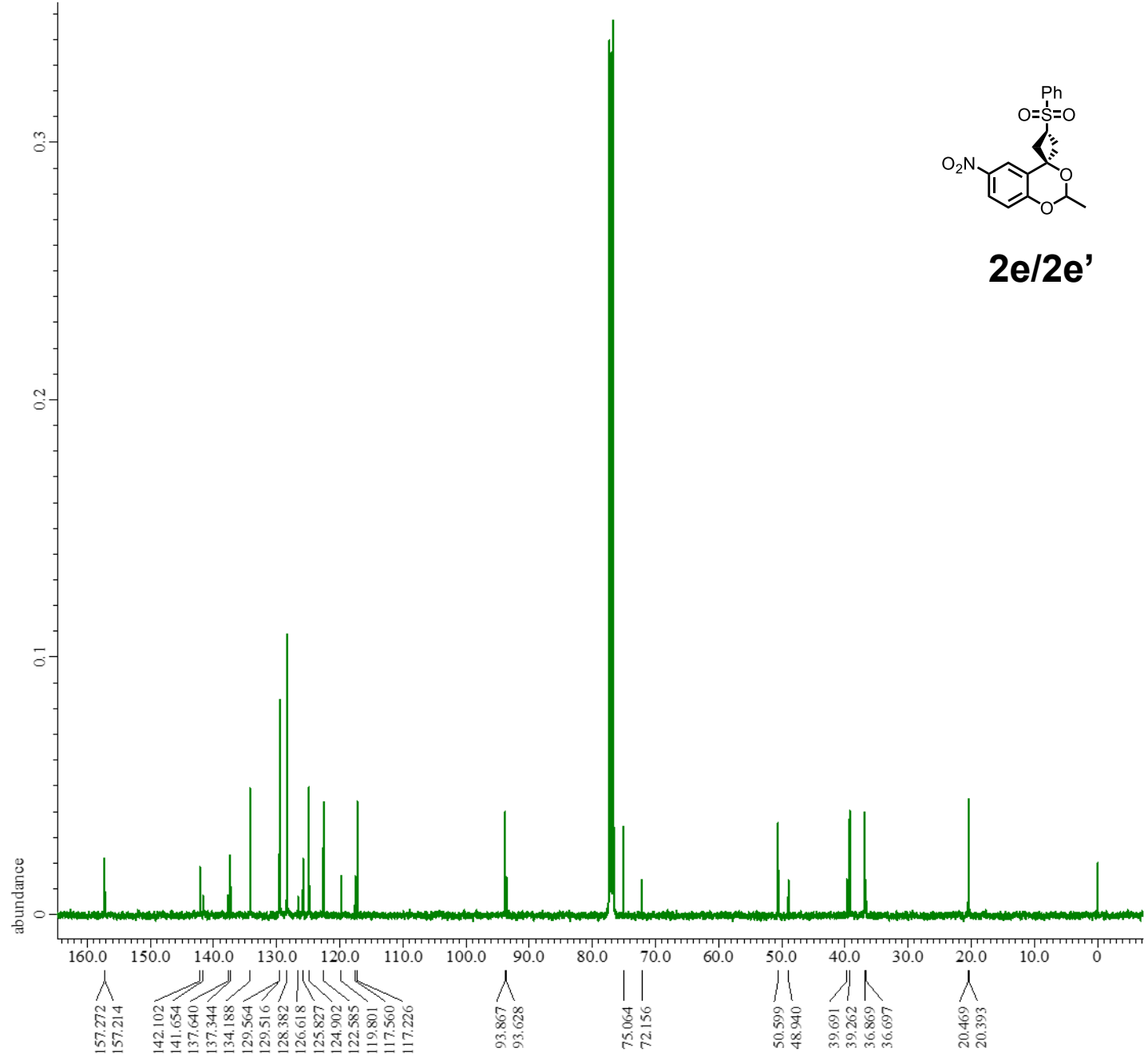


2e/2e'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 13 Ar 4-NO2 CDCL3_Proton-
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 4-NO2 PLC CDCL3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 19-DEC-2022 22:24:43
Revision_Time       = 11-JAN-2023 16:31:15

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 38
Temp_Get            = 20.6[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

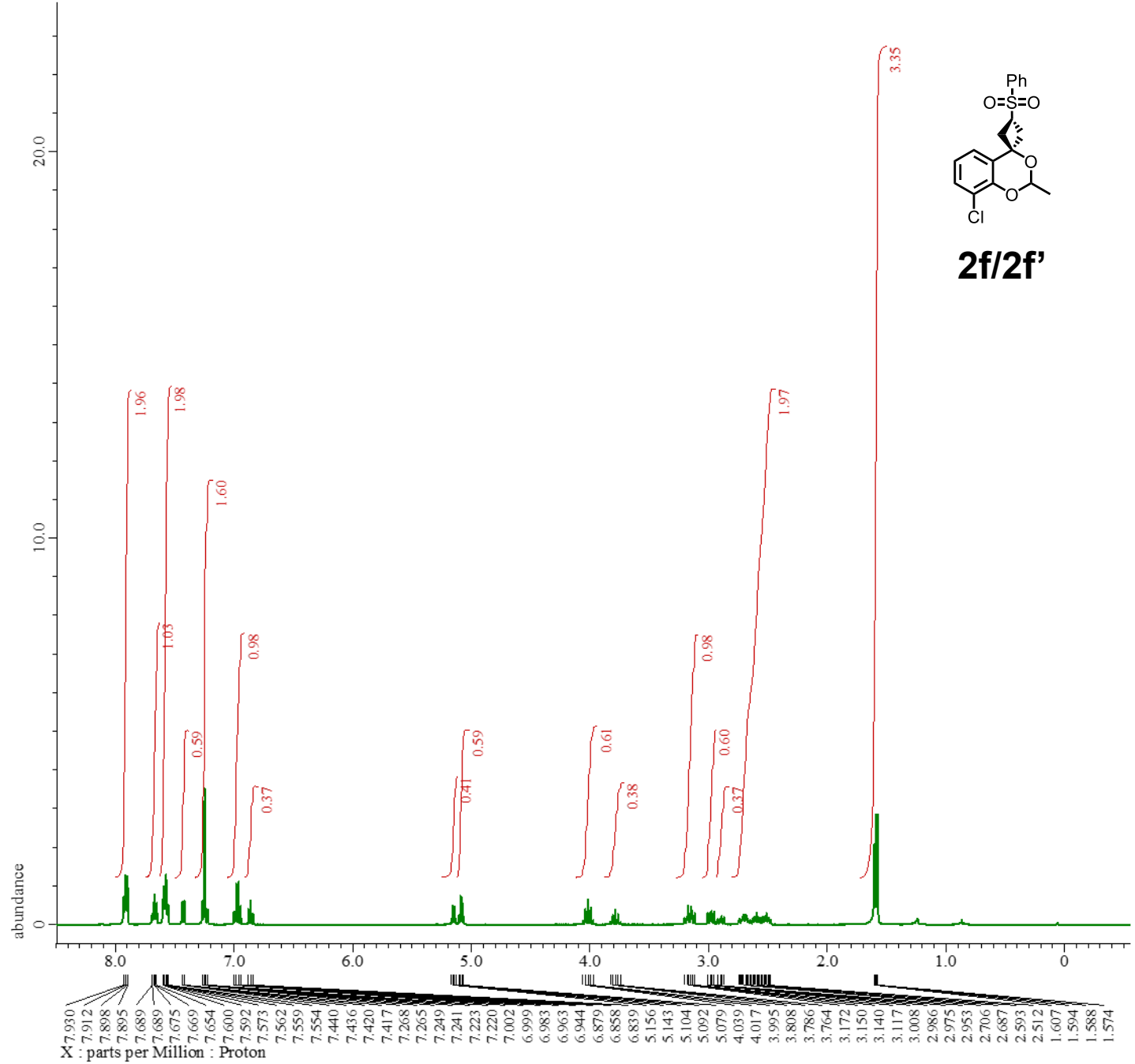
[1] H NMR spectrum of **2e**/**2e'** (400 MHz, $CDCl_3$)



2e/2e'

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

Filename            = 13 Ar 4-NO2 CDCl3_Carbon-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210591 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-NOV-2022 06:20:18
Revision_Time       = 11-JAN-2023 16:52:32

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1150
Total_Scans         = 1150

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2e**/**2e'** (101 MHz, $CDCl_3$)

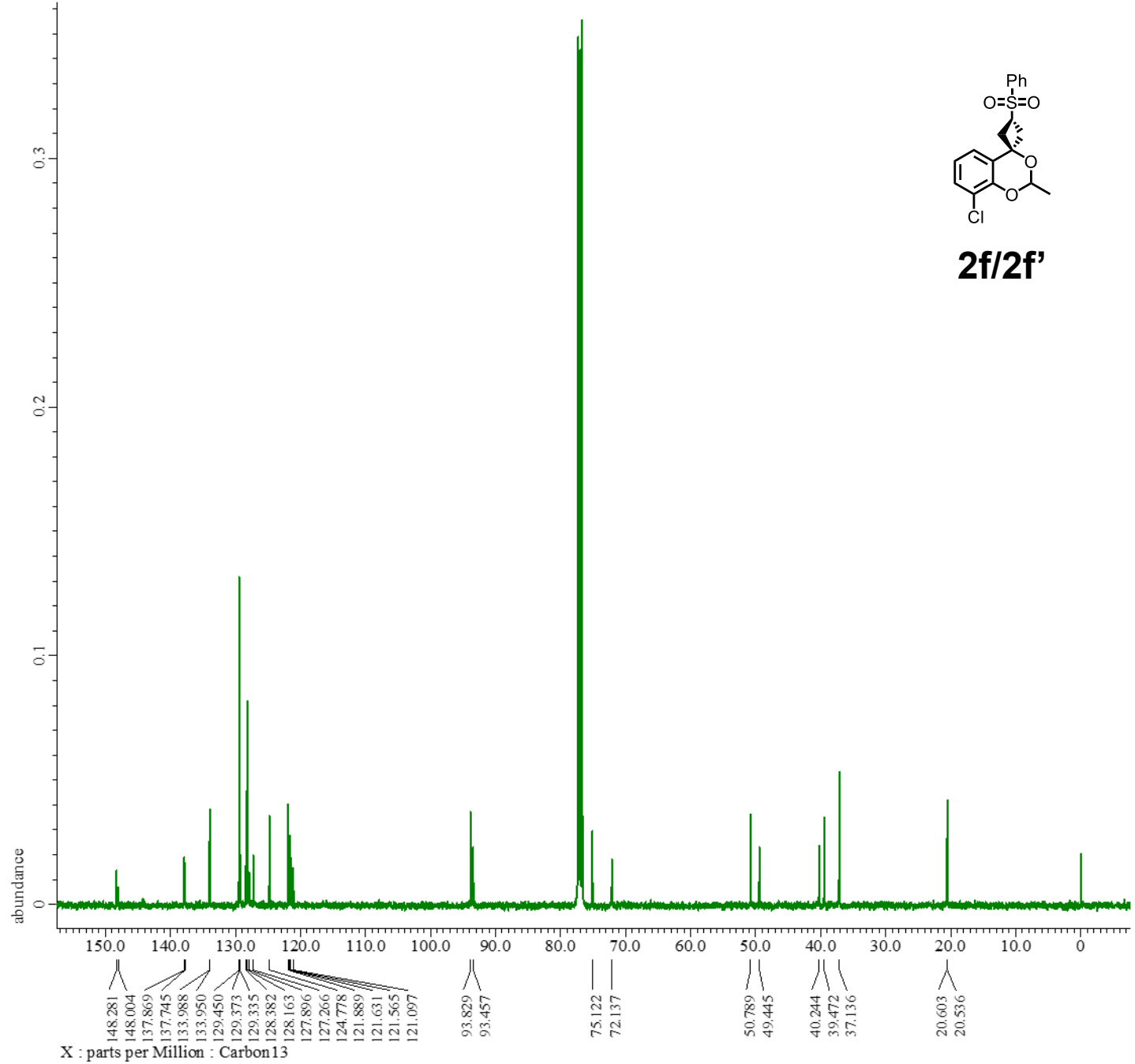


2f/2f'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 17 Ar 6-Cl acid CDCl3_Pro
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210592 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 25-NOV-2022 22:51:11
Revision_Time       = 16-MAR-2023 12:08:56

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 40
Temp_Get            = 19.6[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2f/2f'** (400 MHz, $CDCl_3$)

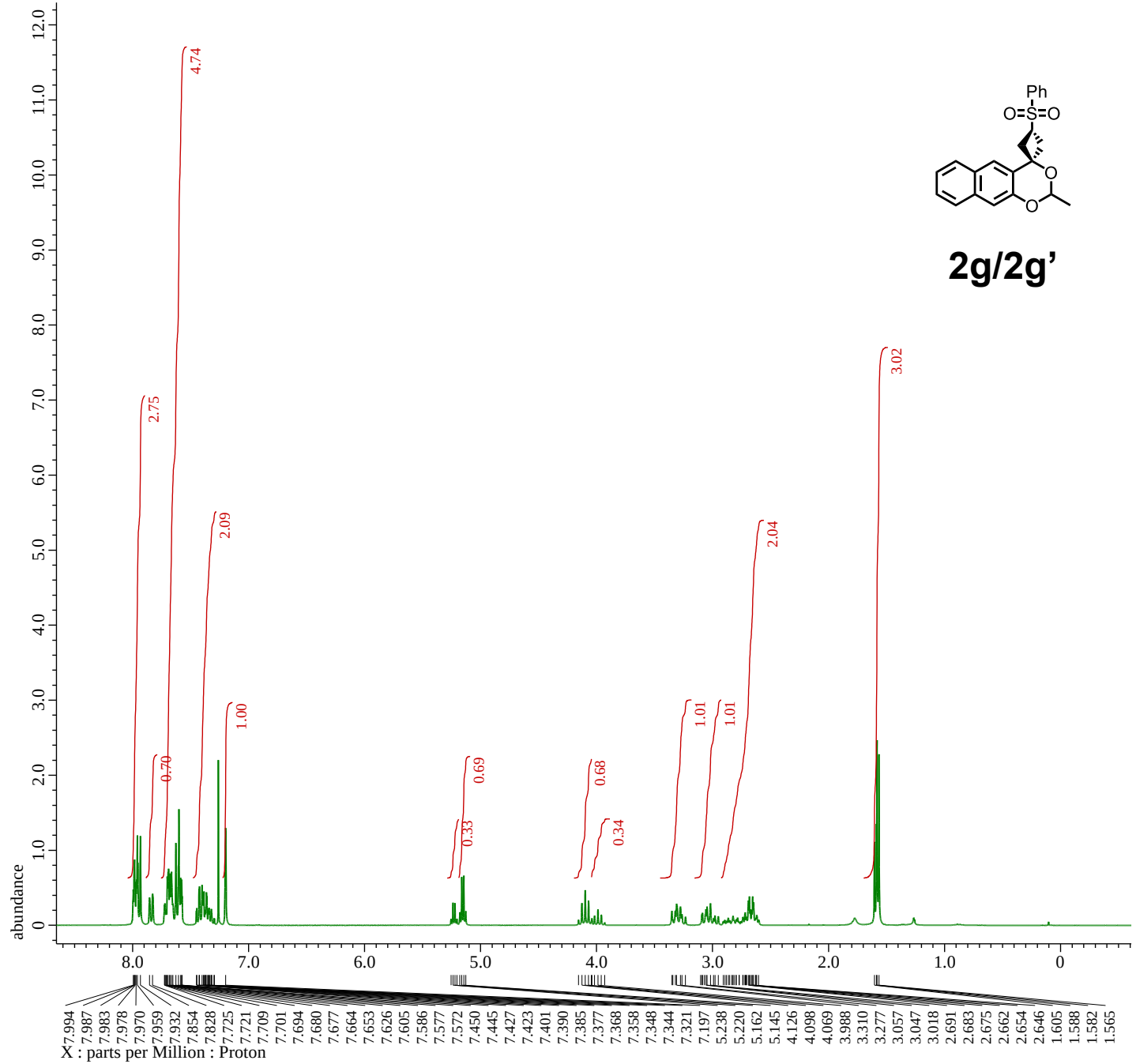


2f/2f'

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

```
Filename            = 17 Ar 6-Cl CDCl3_Carbon-1
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210592 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-NOV-2022 07:25:59
Revision_Time       = 11-JAN-2023 15:54:48

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1150
Total_Scans         = 1150

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2f/2f'** (101 MHz, $CDCl_3$)



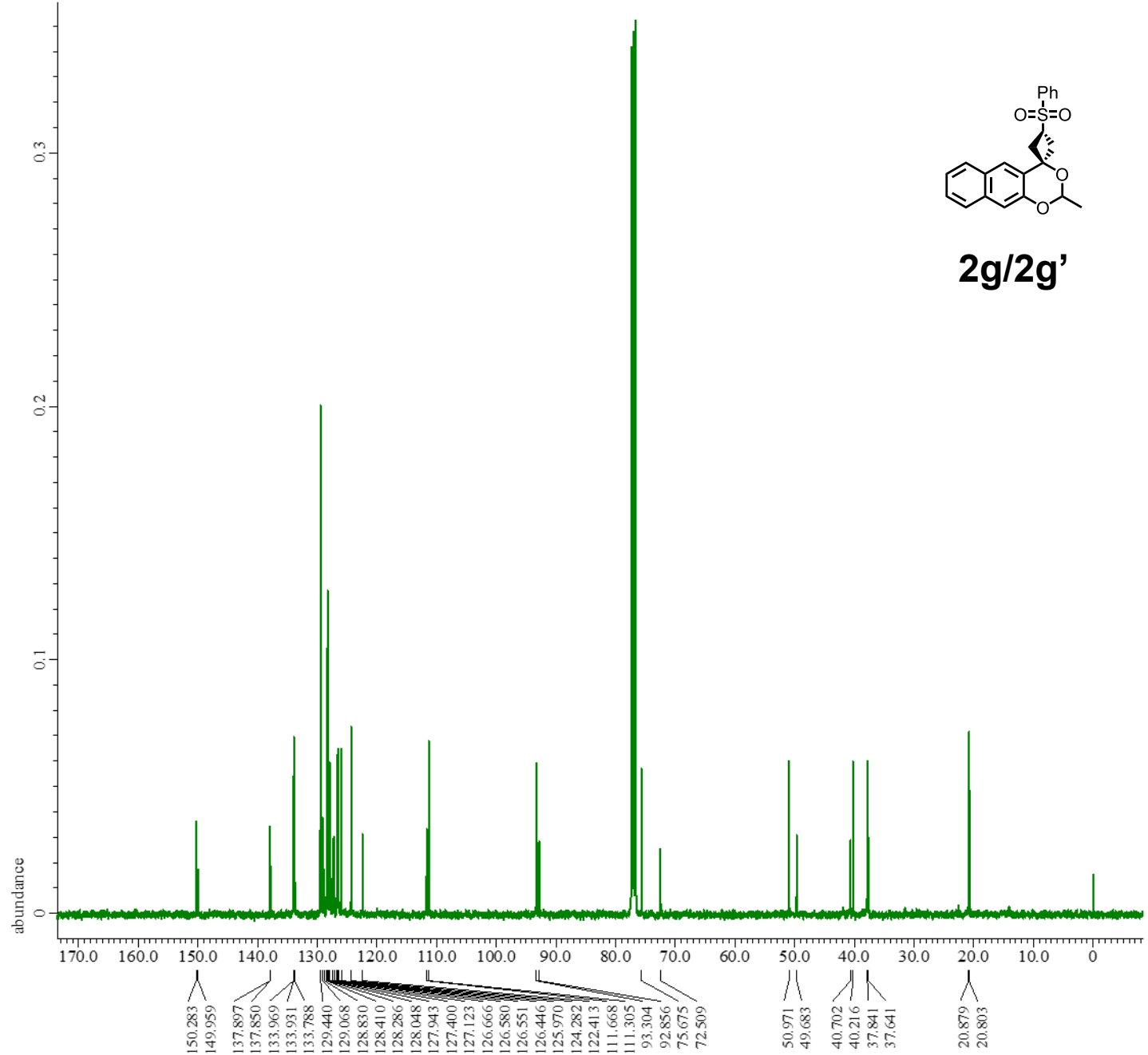
2g/2g'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 18 2,3-naphthyl CDCl3_Pro
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210695-696 column2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-MAR-2023 09:57:19
Revision_Time       = 16-MAR-2023 11:38:08

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 300
Spectrometer        = DELTA2_NMR

Field_Strength      = 7.0586013[T] (300[MHz])
X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 19.6[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 1[dB]
X_Pulse             = 5.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.90717696[s]
```

7.994 7.987 7.983 7.978 7.970 7.959 7.932 7.854 7.828 7.725 7.721 7.709 7.701 7.694 7.680 7.677 7.664 7.653 7.626 7.605 7.586 7.577 7.572 7.450 7.445 7.427 7.423 7.401 7.390 7.385 7.377 7.368 7.358 7.348 7.344 7.321 7.197 5.238 5.220 5.162 5.145 4.126 4.098 4.069 3.988 3.310 3.277 3.057 3.047 3.018 2.691 2.683 2.675 2.662 2.654 2.646 1.605 1.588 1.582 1.565

[1] H NMR spectrum of **2g/2g'** (301 MHz, $CDCl_3$)

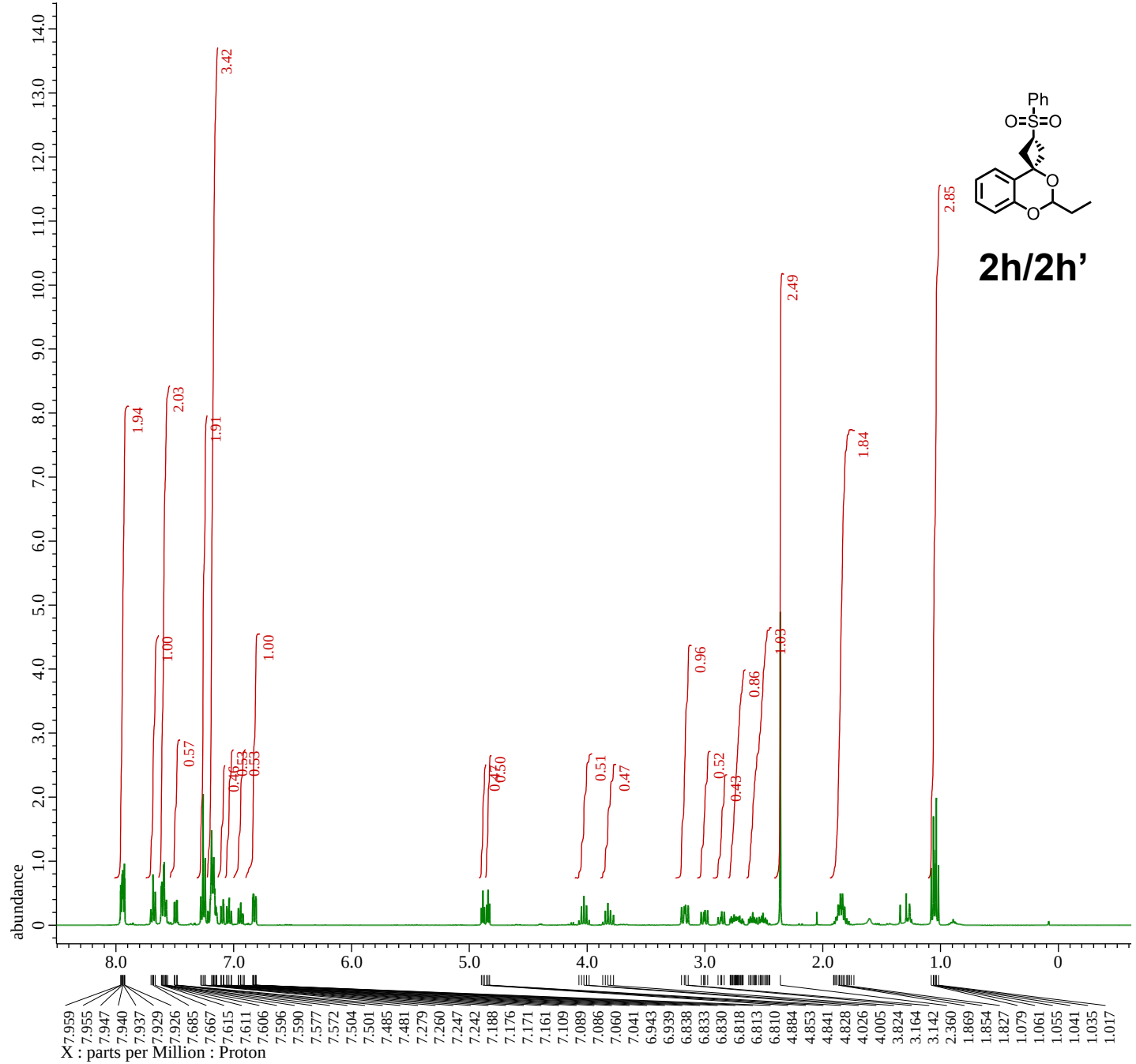


2g/2g'

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

```
Filename            = 16 Ar 2,3-naphthyl CDCl3_
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210552 3 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-NOV-2022 20:52:23
Revision_Time       = 16-JAN-2023 20:33:14

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2g/2g'** (101 MHz, $CDCl_3$)

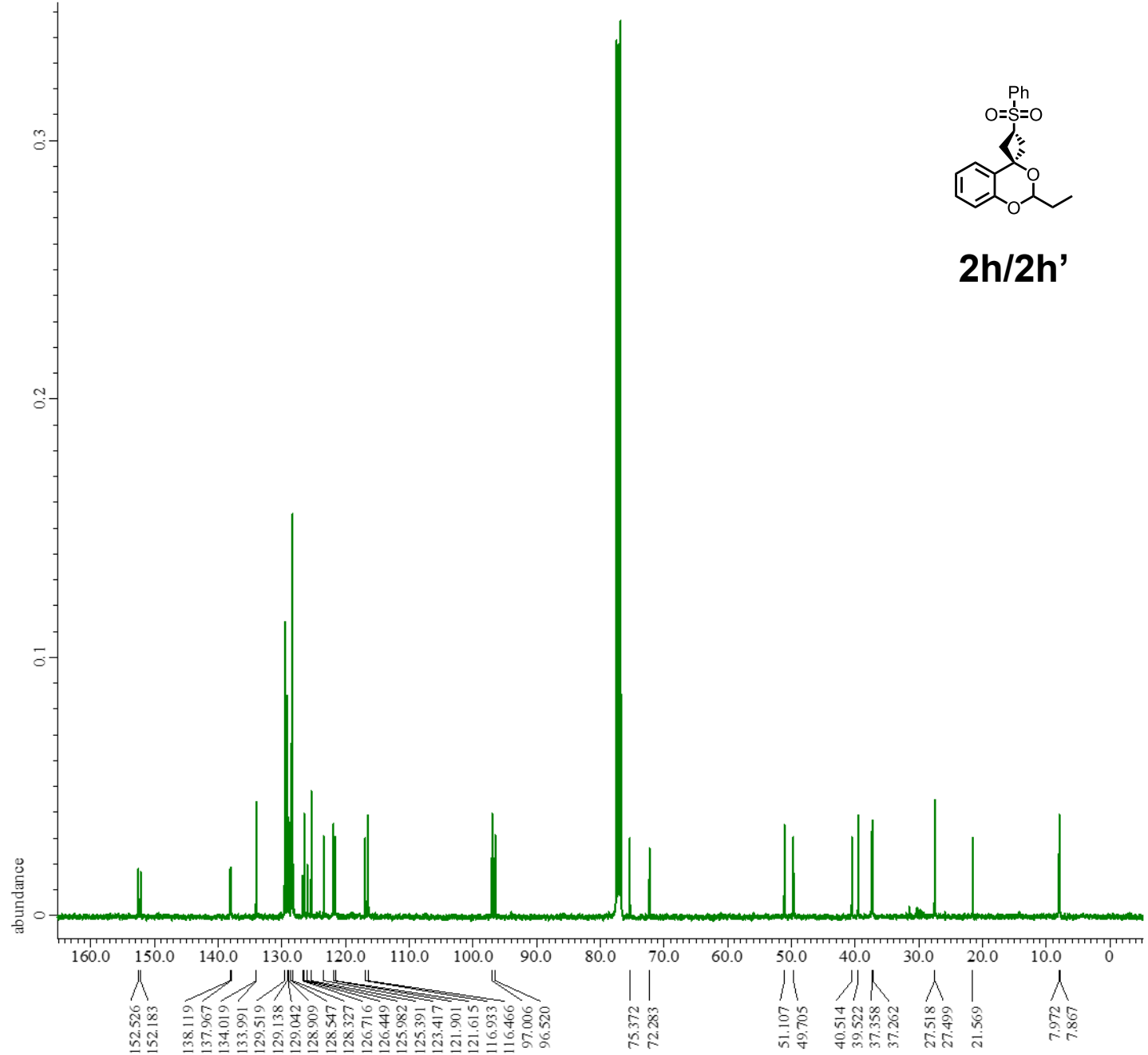


2h/2h’

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 18 O-propenyl_Proton-1-2.
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210680 check CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 11-FEB-2023 00:06:06
Revision_Time       = 16-MAR-2023 12:33:27

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 36
Temp_Get            = 20.8[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2h**/**2h’** (400 MHz, $CDCl_3$)

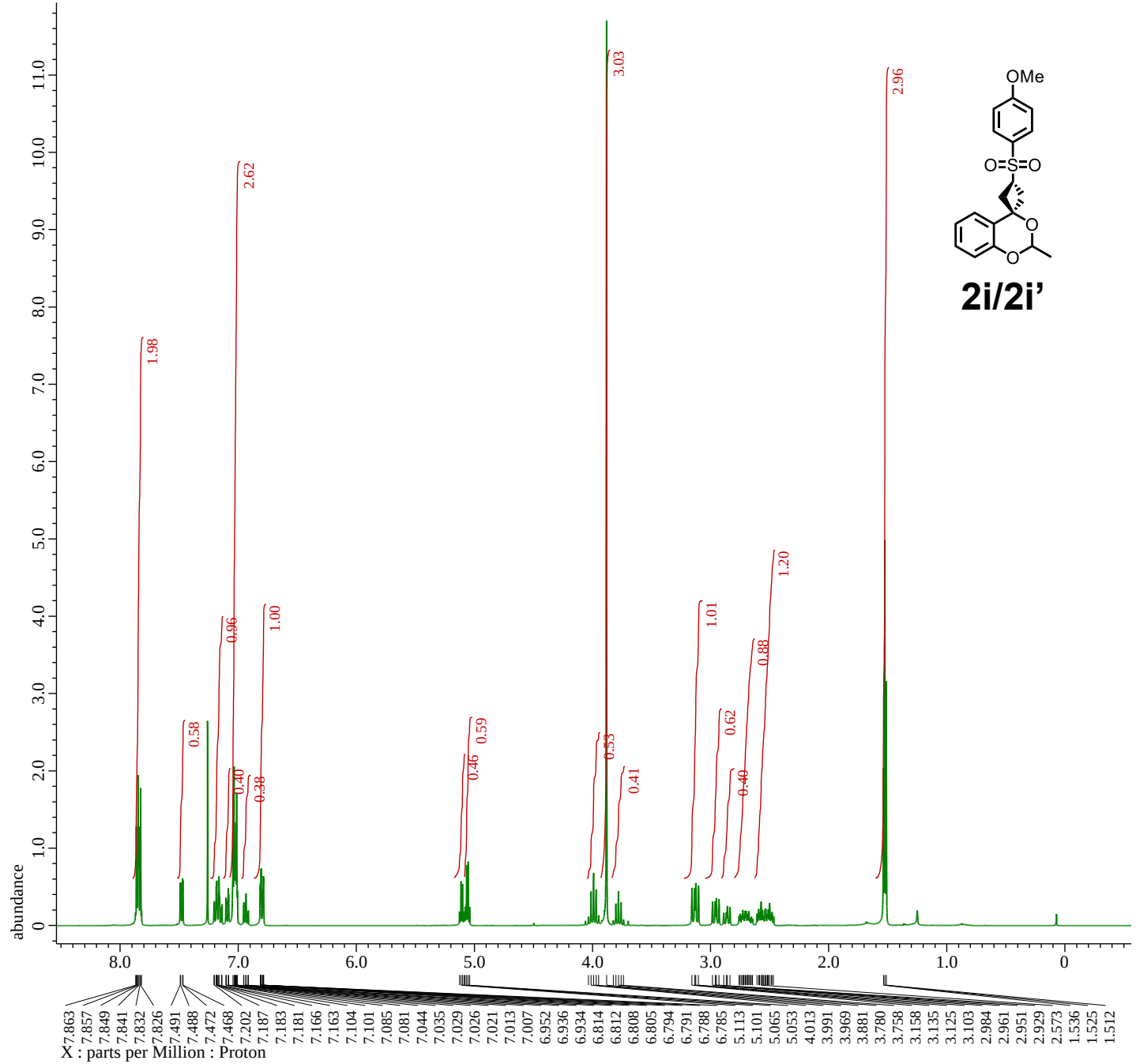


2h/2h'

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

```
Filename            = 18 O-propenyl CDCl3_Carbo
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210680 check CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 11-FEB-2023 00:09:37
Revision_Time       = 16-MAR-2023 12:20:36

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 2500
Total_Scans         = 2500

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.9[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **2h/2h'** (101 MHz, $CDCl_3$)

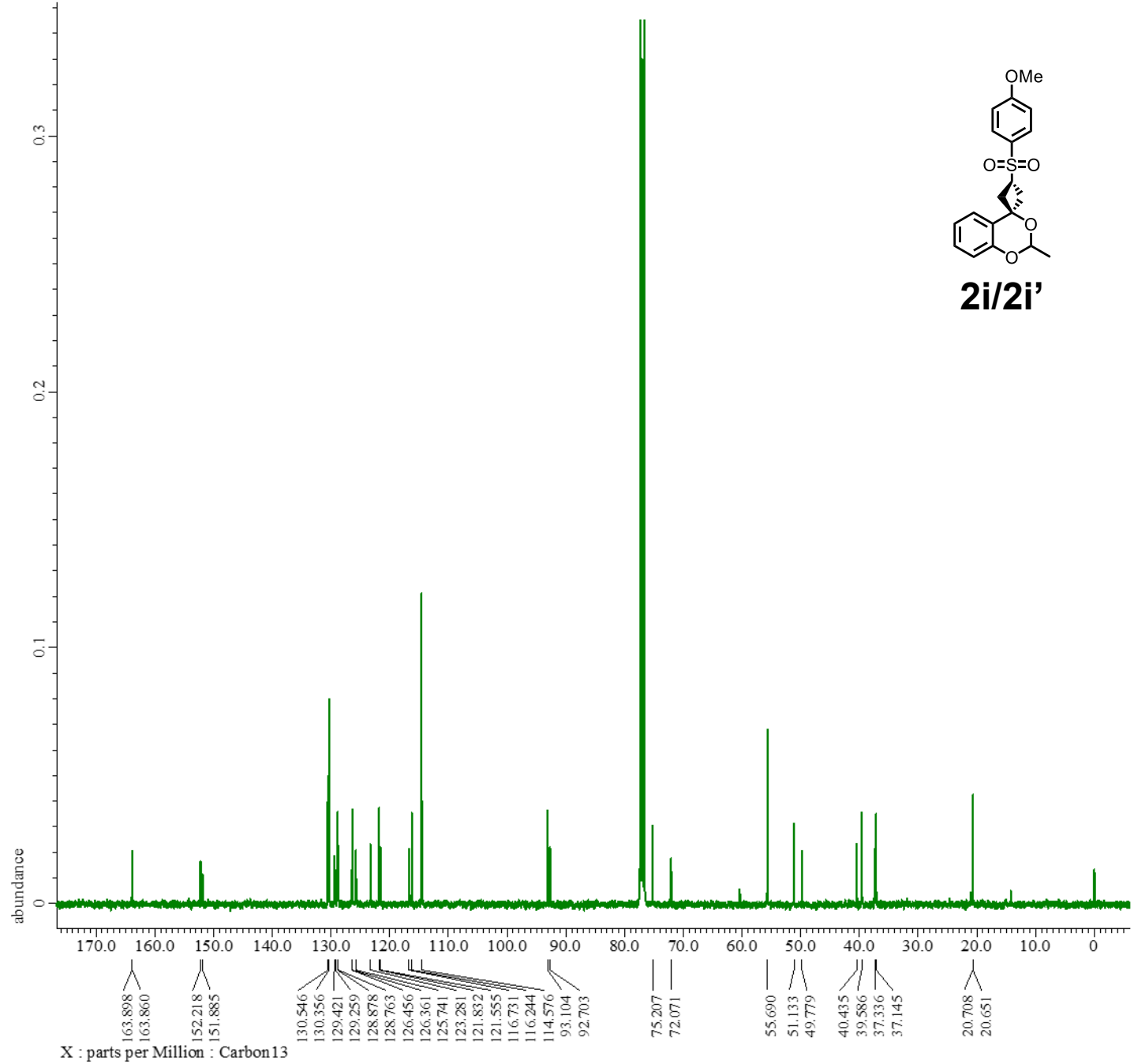


2i/2i'

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 20 EWG methoxyphenyl CDCl
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210669-670 column2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 30-DEC-2022 21:51:28
Revision_Time       = 18-JAN-2023 10:49:18

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 20.7[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2i/2i'** (400 MHz, $CDCl_3$)

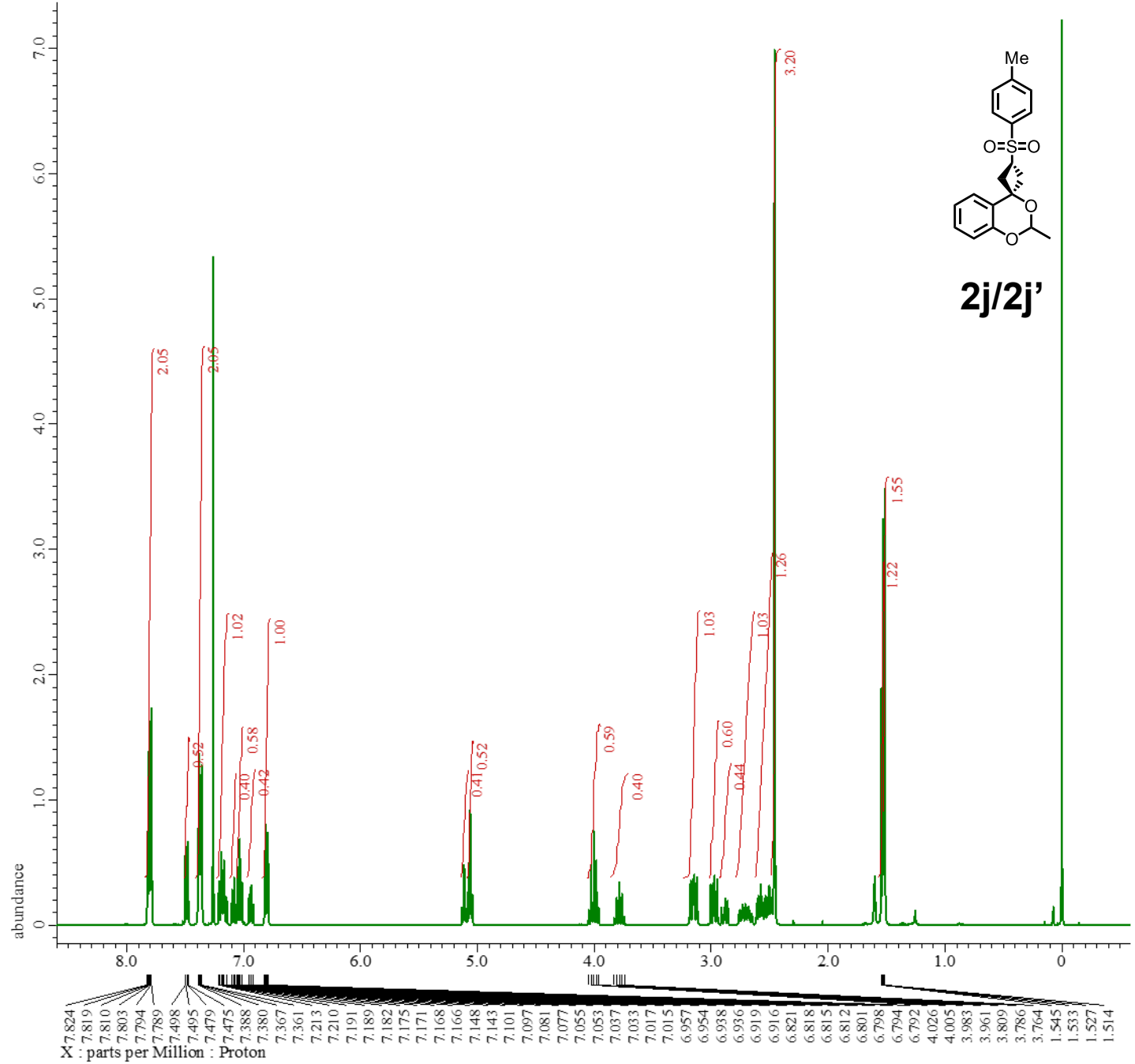


2i/2i'

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

```
Filename            = 20 EWG 4-MeOPh CDCl3_Carb
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210571 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-NOV-2022 04:11:38
Revision_Time       = 18-JAN-2023 10:50:41

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1150
Total_Scans         = 1150

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.1[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

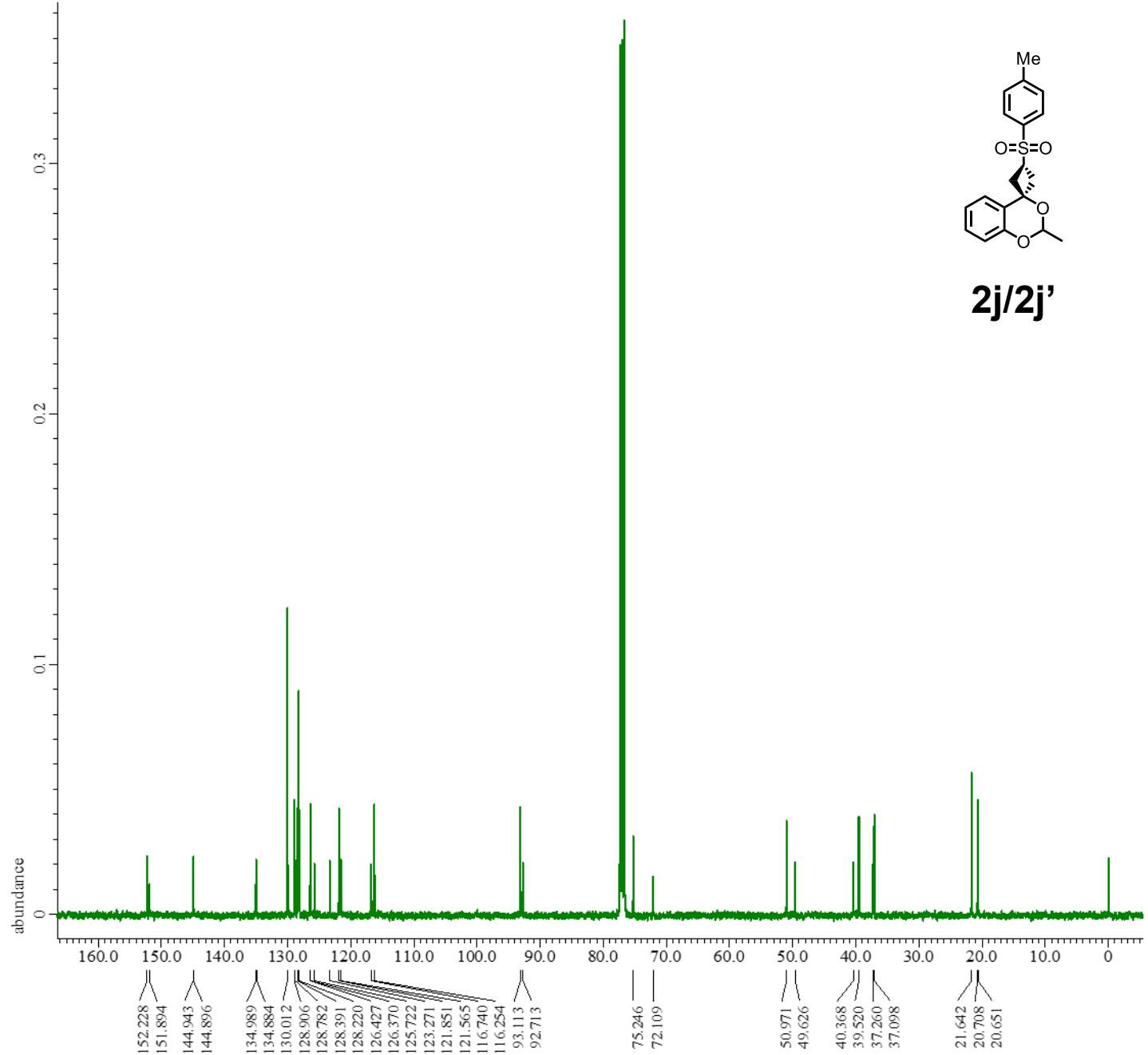
^{13}C NMR spectrum of **2i/2i'** (101 MHz, $CDCl_3$)




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 19 EWG methylphenyl CDCl3
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210588 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-NOV-2022 20:22:51
Revision_Time       = 16-JAN-2023 21:10:08

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 40
Temp_Get            = 19.9[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **2j/2j'** (400 MHz, $CDCl_3$)

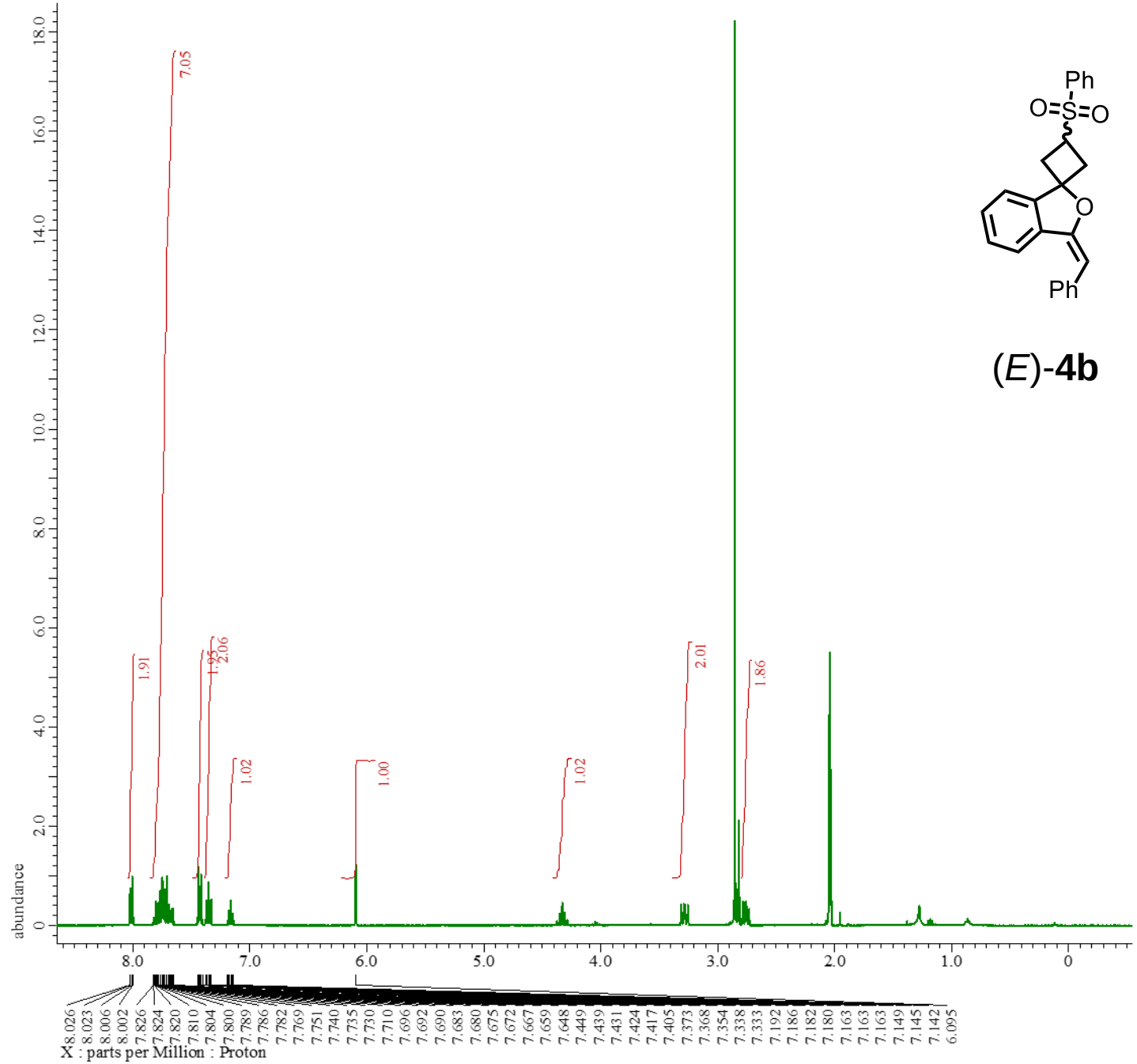


2j/2j'

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

Filename	= 19 EWG 4-MePh CDCl3_Carbo
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 210588 2 CDCl3
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 16-NOV-2022 05:16:14
Revision_Time	= 16-JAN-2023 21:46:08
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1150
Total_Scans	= 1150
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 20.1[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 4.7[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 22.346[dB]
Irr_Atn_Noe	= 22.346[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]

^{13}C NMR spectrum of **2j/2j'** (101 MHz, $CDCl_3$)

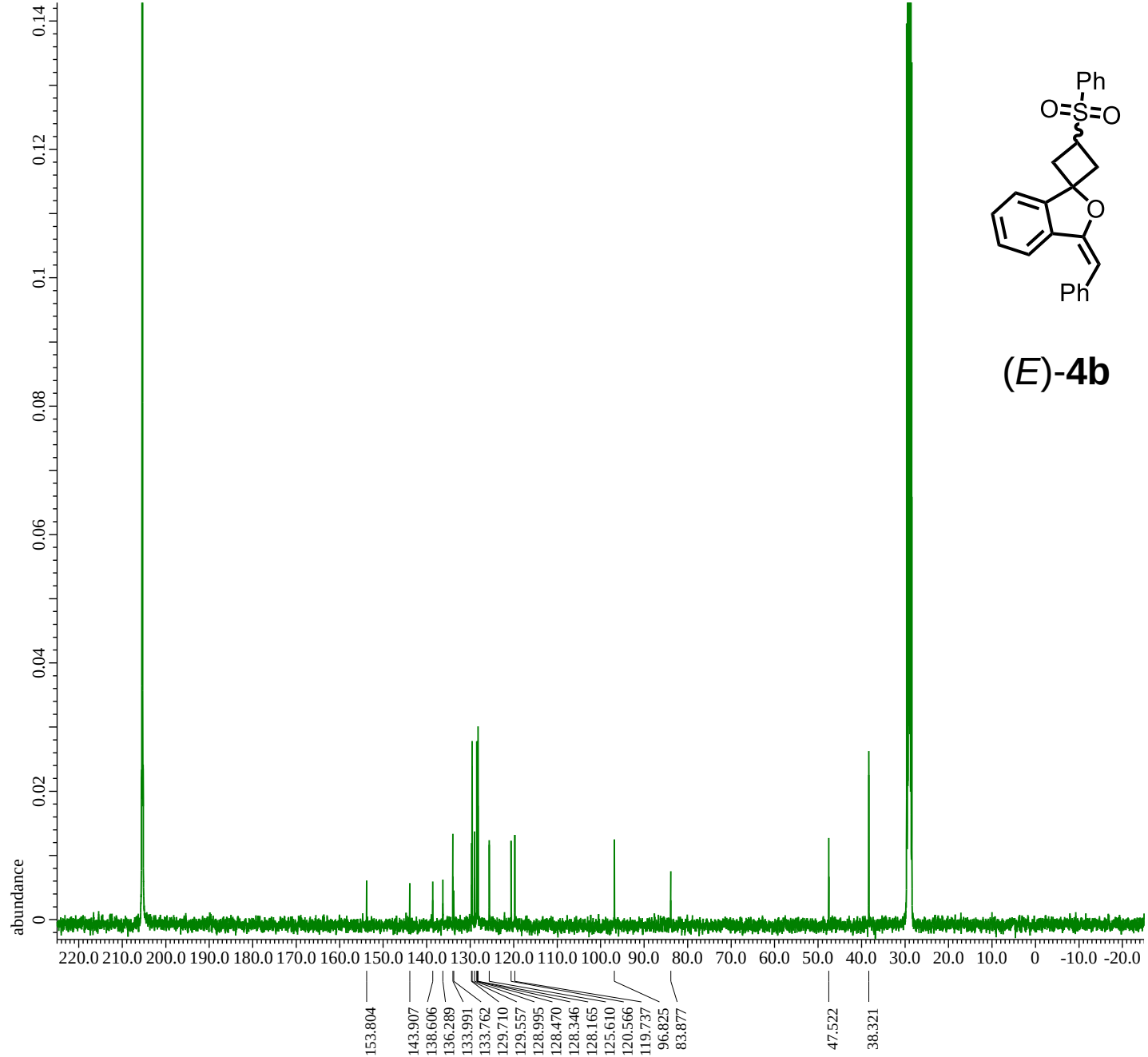


(*E*)-**4b**

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 210697-698 column down Ac
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210697-698 column down Ac
Solvent             = ACETONE-D6
Actual_Start_Time   = 12-FEB-2023 17:04:47
Revision_Time       = 17-MAR-2023 11:56:05

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 44
Temp_Get            = 20.1[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

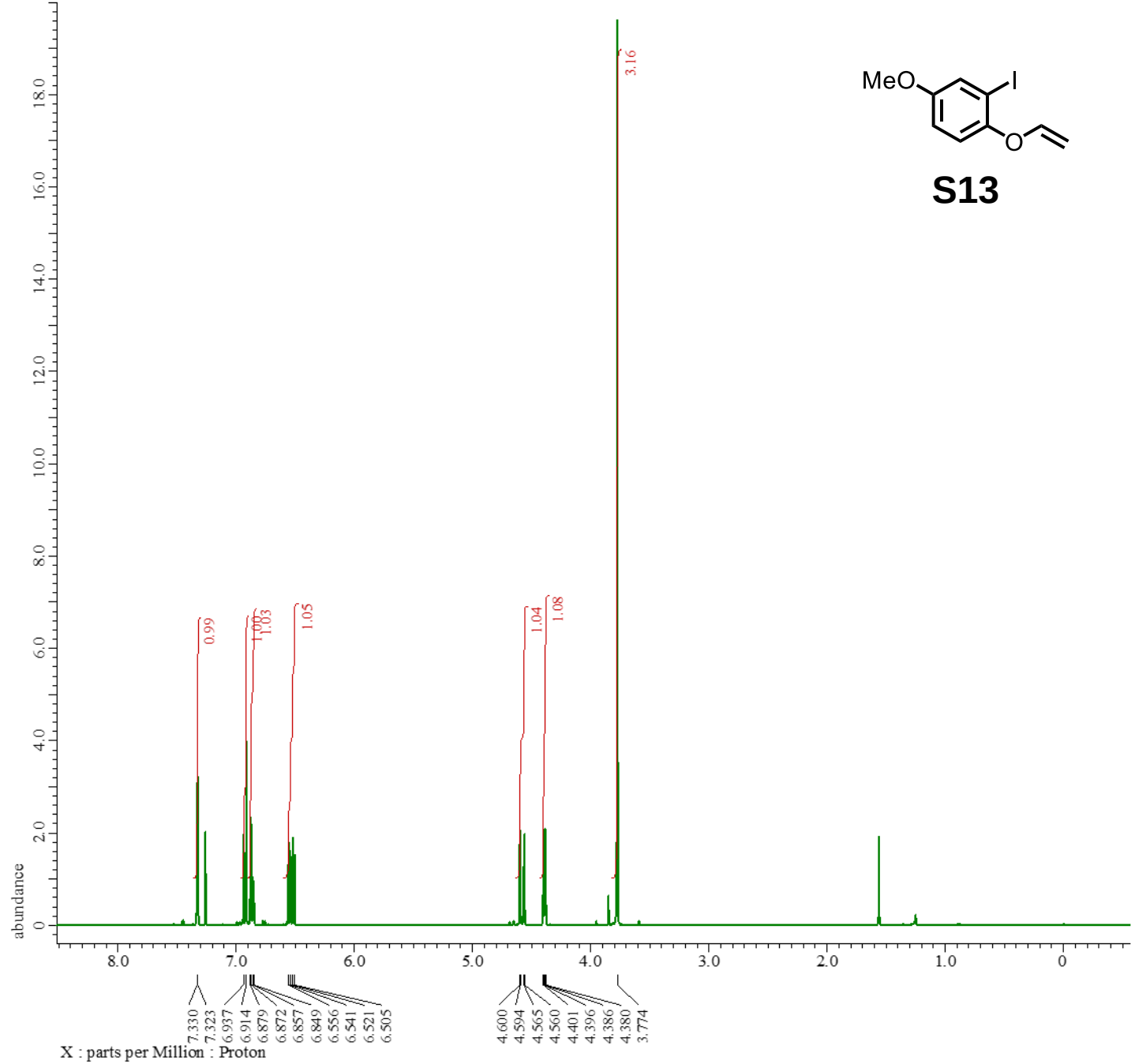
[1] H NMR spectrum of (*E*)-**4b** (301 MHz, Acetone-d_6)



(*E*)-**4b**

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

Filename           = 210697-698 column down Ac
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = 210697-698 column down Ac
Solvent            = ACETONE-D6
Actual_Start_Time  = 12-FEB-2023 17:08:59
Revision_Time      = 17-MAR-2023 16:53:20

Comment            = single pulse decoupled ga
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = Carbon
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = ECS 400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 1512
Total_Scans        = 1512

Relaxation_Delay   = 2[s]
Recvr_Gain         = 50
Temp_Get           = 20.1[dC]
X_90_Width         = 9[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.7[dB]
X_Pulse            = 3[us]
Irr_Atn_Dec        = 22.346[dB]
Irr_Atn_Noe        = 22.346[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
```

^{13}C NMR spectrum of (*E*)-**4b** (101 MHz, Acetone-d_6)

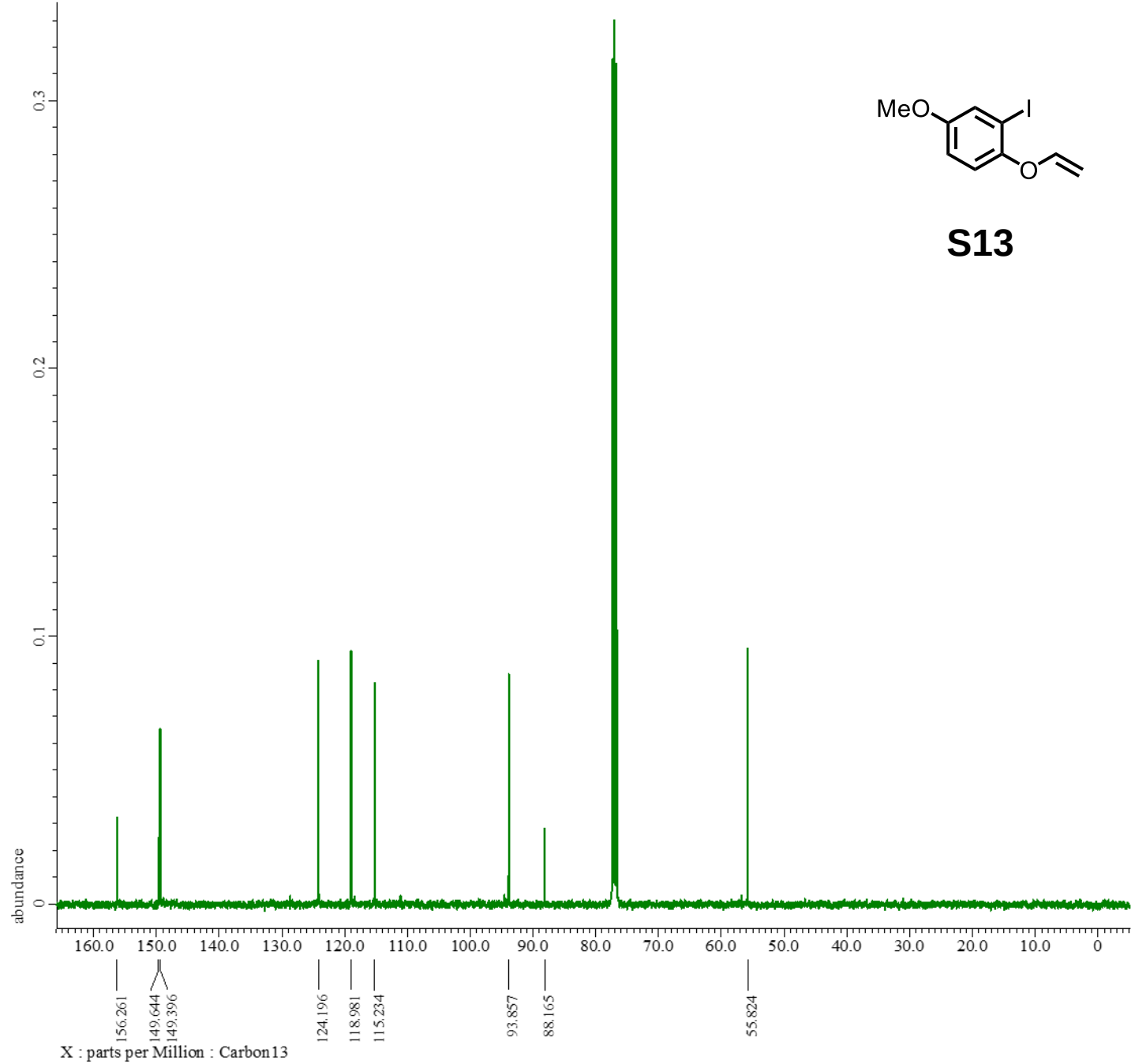


S13

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 32 Ar 4-OMe CDCl3_Proton-
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 210679 column CDCl3
Solvent           = CHLOROFORM-D
Actual_Start_Time = 25-JAN-2023 11:00:18
Revision_Time     = 30-JAN-2023 17:42:18

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proton
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 20.3[dC]
X_90_Width        = 12.4[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

^{1}H NMR spectrum of **S13** (400 MHz, $CDCl_3$)

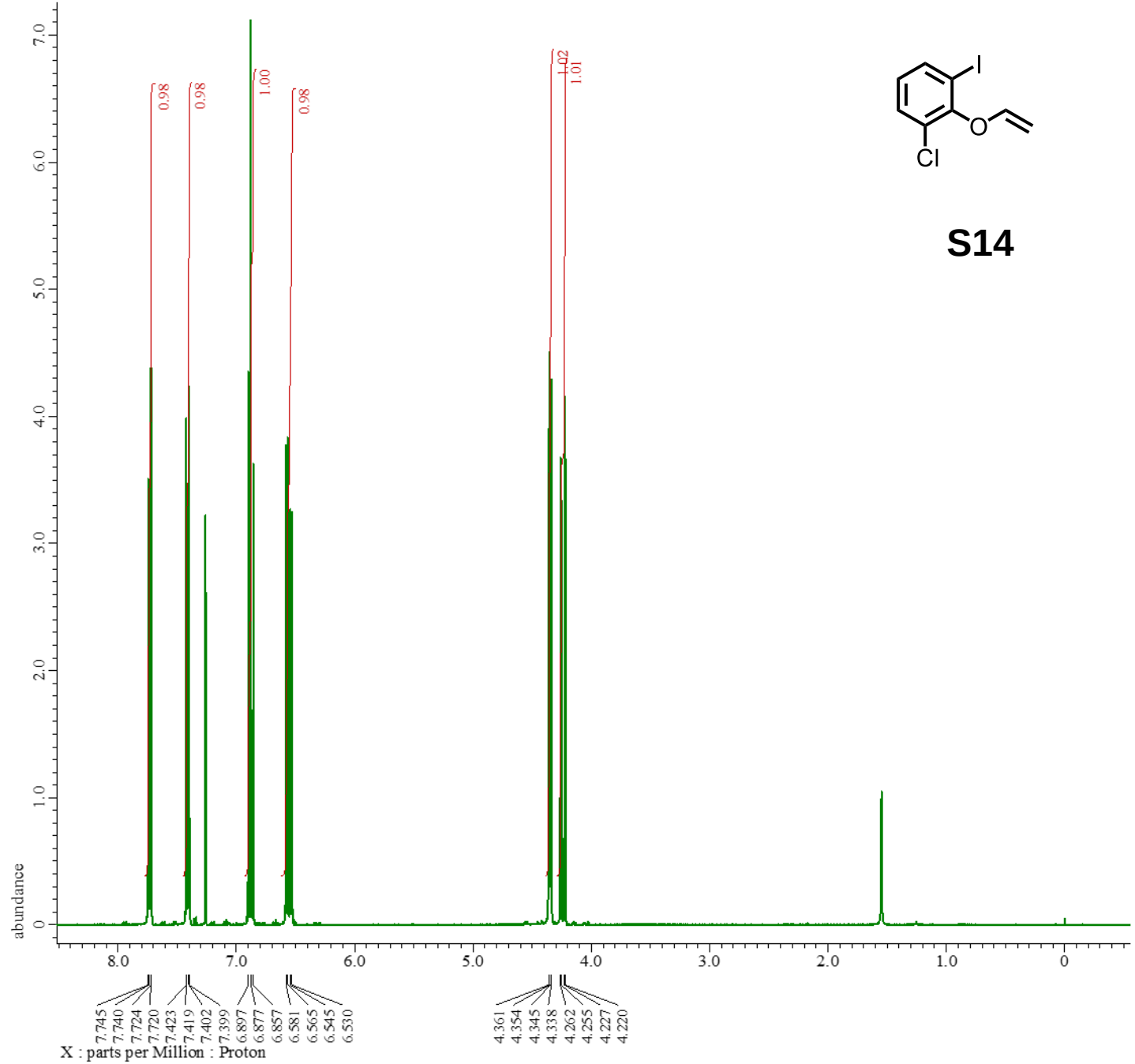


S13

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

```
Filename            = 32 Ar 4-OMe  CDCl3_Carbon
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210679 column CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 25-JAN-2023 10:06:48
Revision_Time       = 17-MAR-2023 10:33:14

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.6[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **S13** (101 MHz, $CDCl_3$)

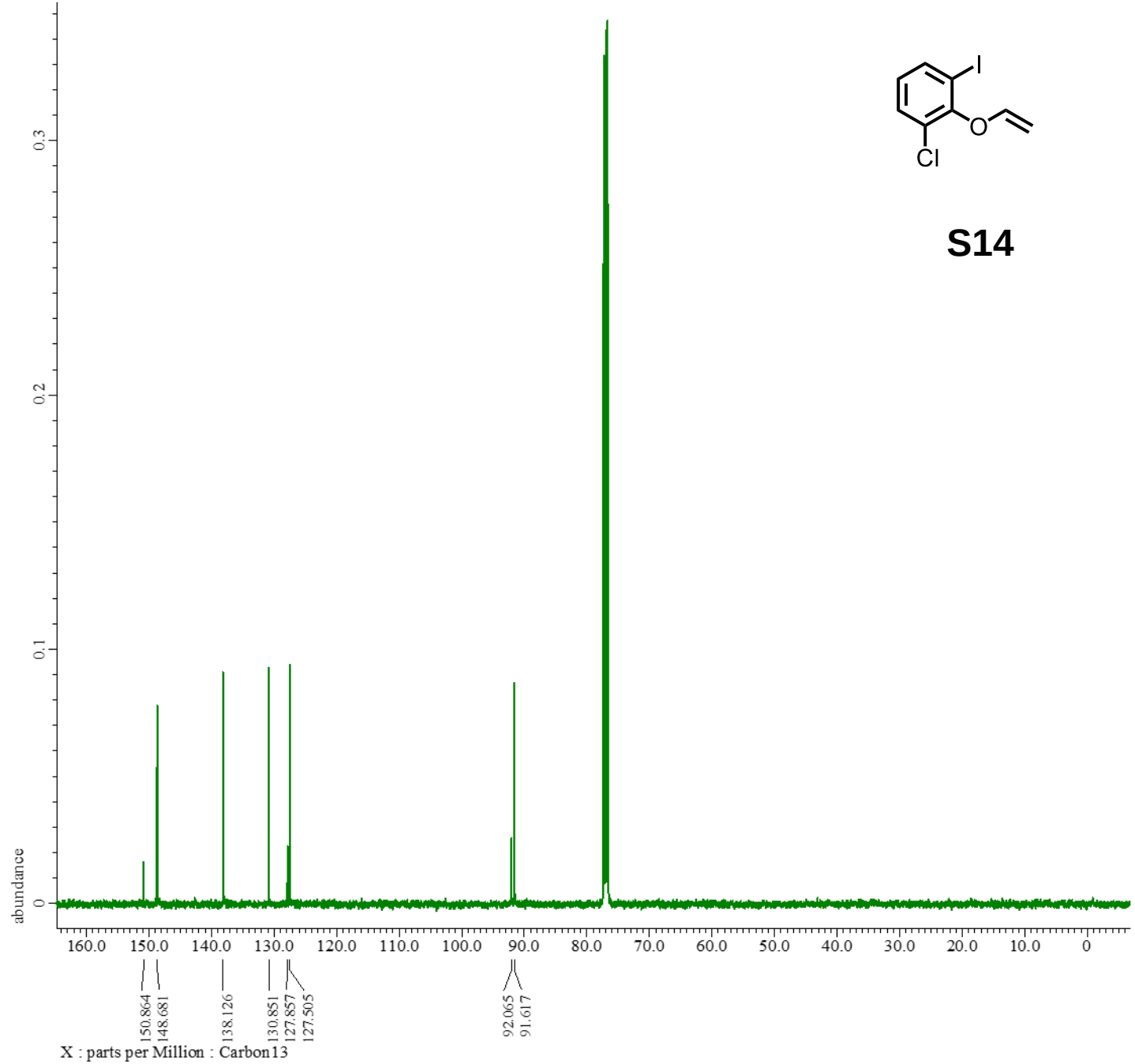


S14

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 30 Ar 6-Cl CDCl3_Proton-1
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210642 column 2 CDC13
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 14-DEC-2022 20:54:08
Revision_Time       = 18-JAN-2023 17:54:17

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 399.78219838[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 46
Temp_Get            = 20.7[dC]
X_90_Width          = 12.4[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3[dB]
X_Pulse             = 6.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```

[1] H NMR spectrum of **S14** (400 MHz, $CDCl_3$)

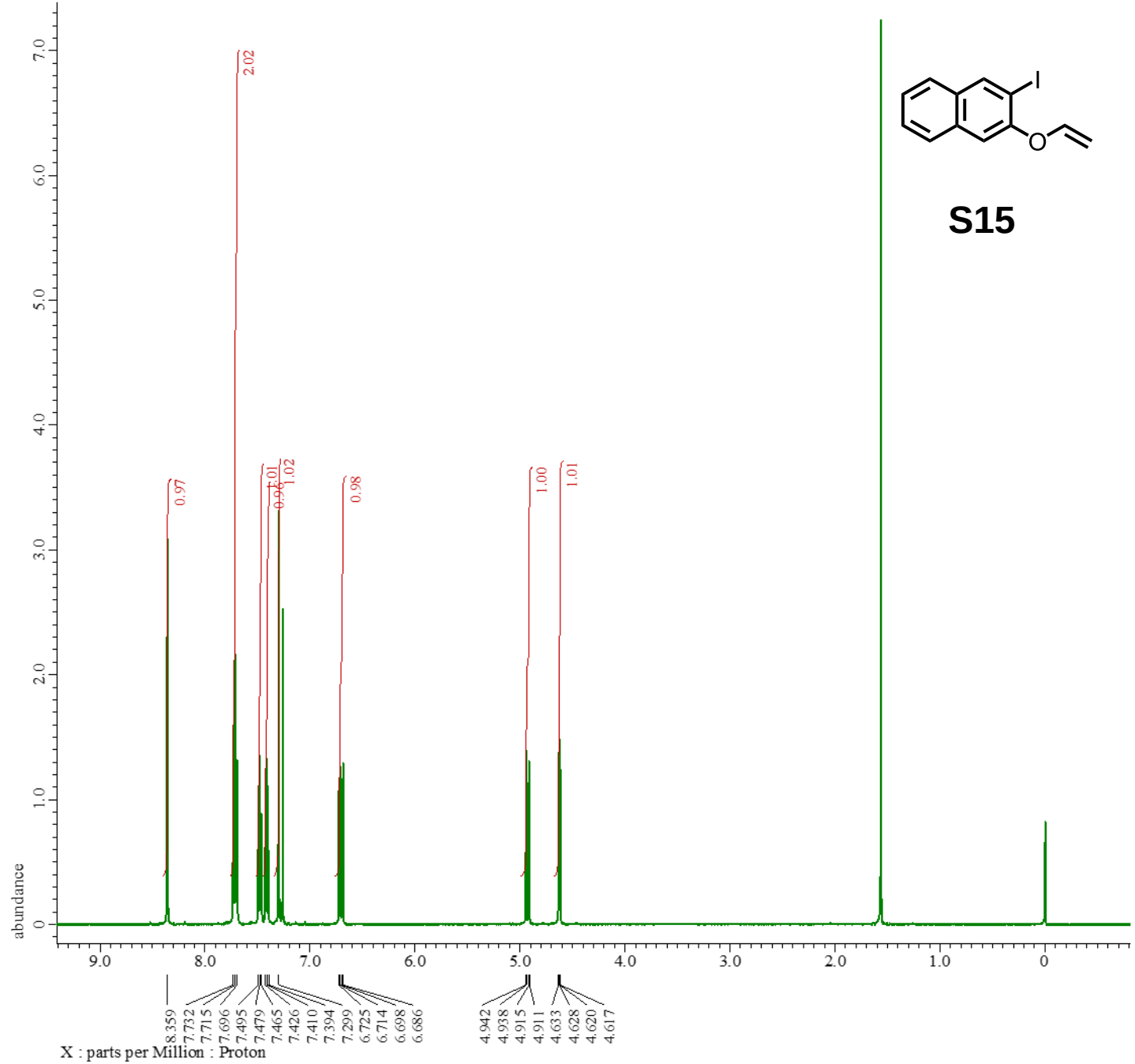


S14

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

```
Filename            = 30 Ar 6-Cl CDCl3_Carbon-1
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210642 column 2 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-DEC-2022 00:07:05
Revision_Time       = 18-JAN-2023 17:57:57

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 21.3[dC]
X_90_Width          = 9[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 4.7[dB]
X_Pulse             = 3[us]
Irr_Atn_Dec         = 22.346[dB]
Irr_Atn_Noe         = 22.346[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

^{13}C NMR spectrum of **S14** (101 MHz, $CDCl_3$)

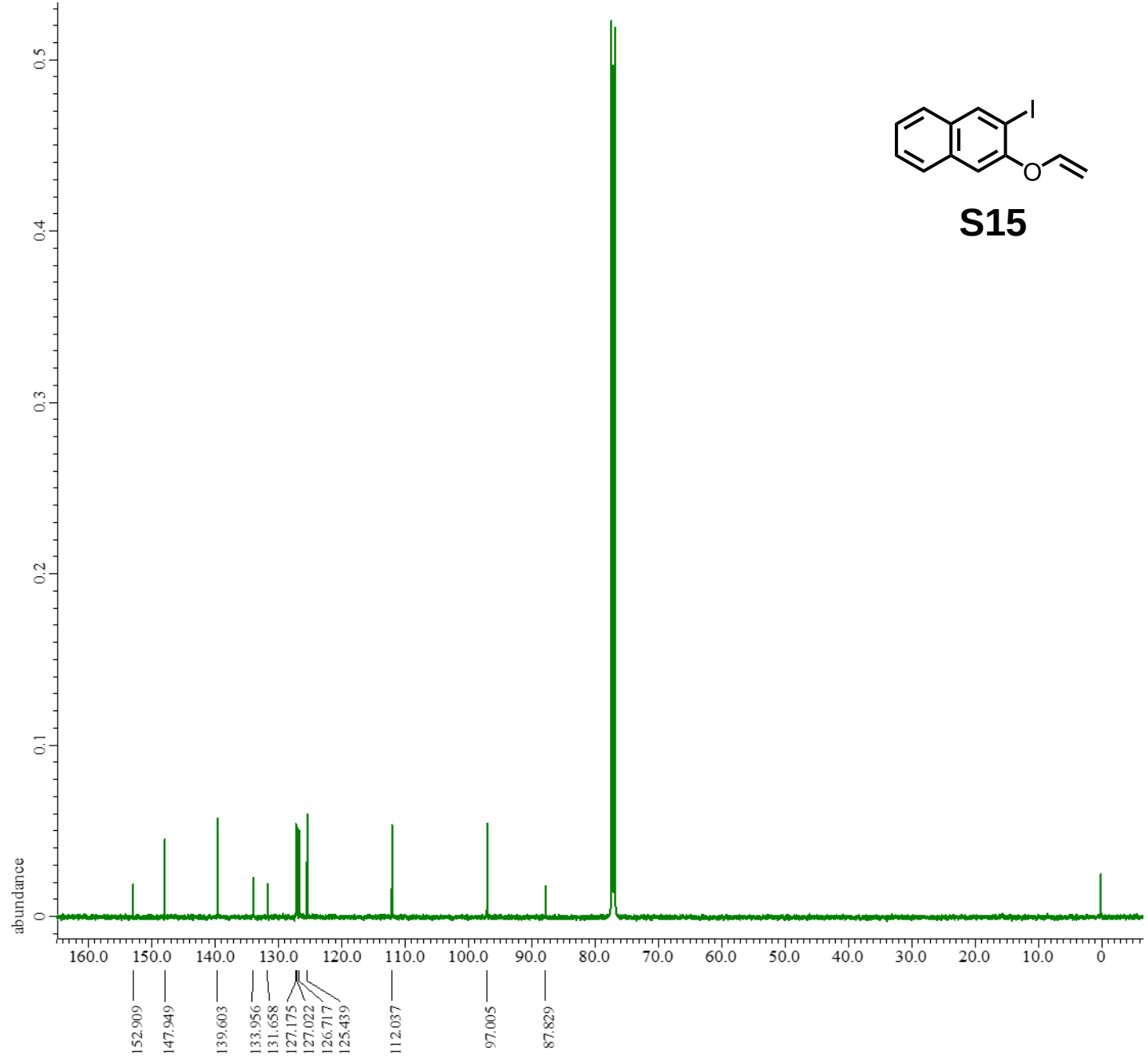


S15

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 29 Ar 2,3-naphthyl CDCl3_
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210433 column CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 30-JUN-2022 15:35:17
Revision_Time       = 18-JAN-2023 20:22:10

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 50
Temp_Get            = 20.5[dC]
X_90_Width          = 13[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.6[dB]
X_Pulse             = 6.5[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

[1] H NMR spectrum of **S15** (500 MHz, $CDCl_3$)

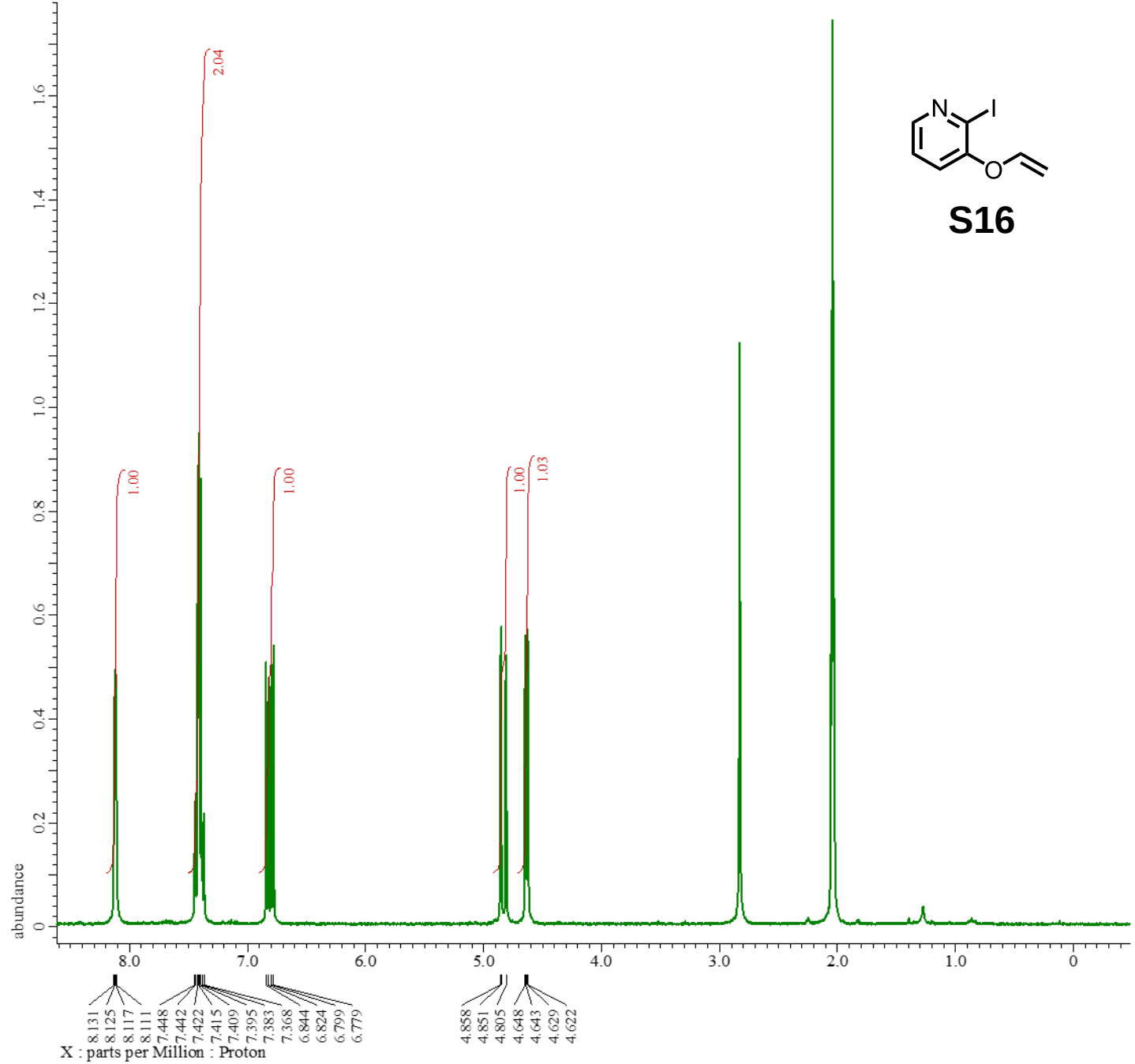


S15

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

```
Filename            = 29 Ar 2,3-naphthyl CDCl3_
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210433 column CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  1-JUL-2022 09:42:34
Revision_Time       = 18-JAN-2023 20:25:33

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T]  (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = TRUE
Scans               = 2350
Total_Scans         = 2350

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 20.7[dC]
X_90_Width          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 4.1[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 21.078[dB]
Irr_Atn_Noe         = 21.078[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

^{13}C NMR spectrum of **S15** (126 MHz, $CDCl_3$)

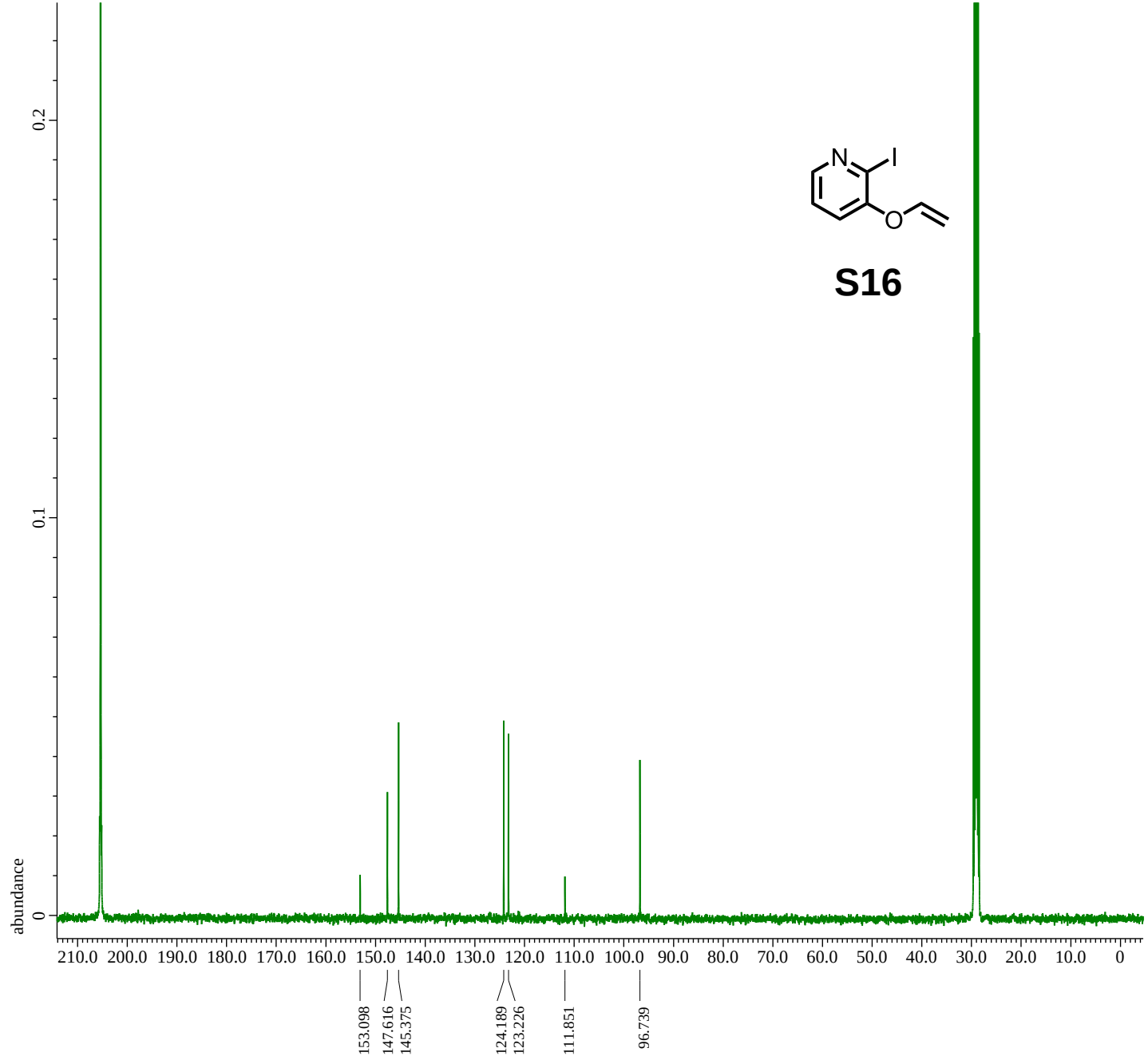


S16

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 31 Ar pyridine Acetone-d6
Author            = delta
Experiment        = proton.jxp
Sample_Id         = pyridine vinyloxy check A
Solvent           = ACETONE-D6
Actual_Start_Time = 19-JAN-2023 11:45:33
Revision_Time     = 19-JAN-2023 16:52:44

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proton
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 300
Spectrometer      = DELTA2_NMR

Field_Strength    = 7.0586013[T] (300[MHz])
X_Acq_Duration    = 2.90717696[s]
X_Domain          = 1H
X_Freq            = 300.52965592[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.34397631[Hz]
X_Sweep           = 5.63570784[kHz]
X_Sweep_Clipped   = 4.50856628[kHz]
Irr_Domain        = Proton
Irr_Freq          = 300.52965592[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 300.52965592[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 18.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.90717696[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.90717696[s]
```

[1] H NMR spectrum of **S16** (301 MHz, Acetone-d_6)



S16

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

Filename	= 31 Ar pyridine Acetone-d6
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= pyridine vinyloxy check A
Solvent	= ACETONE-D6
Actual_Start_Time	= 19-JAN-2023 13:03:39
Revision_Time	= 17-MAR-2023 16:55:06
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1650
Total_Scans	= 1650
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 19.8[dC]
X_90_Width	= 9[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 4.7[dB]
X_Pulse	= 3[us]
Irr_Atn_Dec	= 22.346[dB]
Irr_Atn_Noe	= 22.346[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]

^{13}C NMR spectrum of **S16** (101 MHz, Acetone-d_6)