Supporting Information

Brønsted acid-catalyzed synthesis of spirocyclobutanes *via* heteroannulation of vinyloxyphenylbicyclobutanes with water

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Contents

1. General Information

- ^{1}H-, ^{13}C- and ^{19}F-NMR spectra were recorded 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).

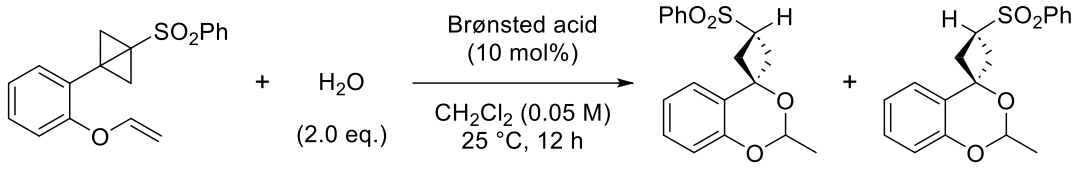
  ^{13}C NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of triplet for $CDCl_3$ at 77 ppm, septet for acetone-d_6 at 29.8 ppm, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (Hz).

  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

2-1. Screening of Brønsted acid catalysts

Table S1



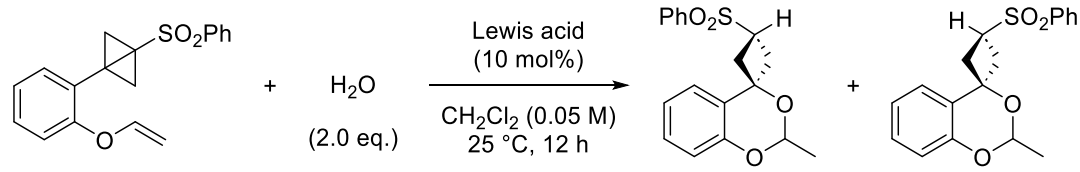
1a (0.10 mmol) **2a** **2a'**

entry	Brønsted acid	**2a**/**2a'** (%, *dr*)[a]
1	TfOH	70% (0.31 : 1)
2	conc. HCl	<5%
3	conc. H_2SO_4	10% (0.16 : 1)
4	60% HNO_3 aq.	73% (0.86 : 1)
5	HCOOH	24% (0.30 : 1)
6	TFA	<5%
7	*p*-TSA•H_2O	76% (0.61 : 1)
8	60% $HClO_4$ aq.	94% (0.94 : 1)
9	60% $HClO_4$ aq. (20 mol%)	95% (1.03 : 1)
10	60% $HClO_4$ aq. (30 mol%)	98% (1.10 : 1)

[a] NMR yield using 1,3,5-trimethoxybenzene as an internal standard.

2-2. Screening of Lewis acid catalysts

Table S2



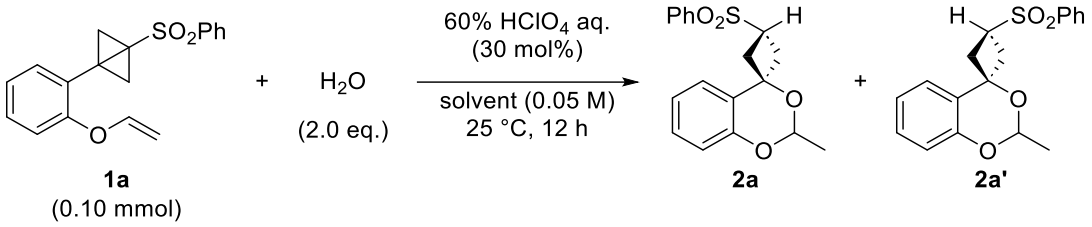
1a (0.10 mmol) **2a** **2a'**

entry	Lewis acid	**2a**/**2a'** (%, *dr*)[a]
1	BF_3•OEt_2	70% (0.78 : 1)
2	$CeCl_3$•$7H_2O$	not detected
3	$TiCl_4$	<5%
4	$AlCl_3$•$6H_2O$	<5%
5	$Bi(OTf)_3$	94% (0.94 : 1)
6	$Yb(OTf)_3$	81% (0.62 : 1)

[a] NMR yield using 1,3,5-trimethoxybenzene as an internal standard.

2-3. Screening of solvents

Table S3



1a
(0.10 mmol)

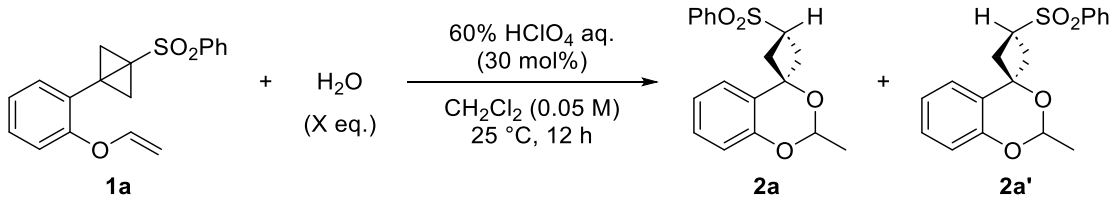
2a **2a'**

entry	solvent	**2a**/**2a'** (%, *dr*)[a]
0	CH_2Cl_2	98% (1.10 : 1)
1	$CHCl_3$	38% (0.83 : 1)
2	PhMe	84% (0.87 : 1)
3	$MeNO_2$	55% (0.56 : 1)
4	EtOAc	91% (0.50 : 1)
5	MeCN	83% (0.88 : 1)
6	1,4-dioxane	90% (0.49 : 1)
7	DMSO	not detected
8	H_2O	no reaction

[a] NMR yield using 1,3,5-trimethoxybenzene as an internal standard.

2-4. Screening of H_2O loading

Table S4



1a
(0.10 mmol)

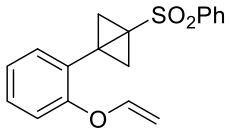
2a **2a'**

entry	X	**2a**/**2a'** (%, *dr*)[a]
0	2	98% (1.10 : 1)
1	0	45% (0.32 : 1)
2	1	89% (0.39 : 1)
3	3	90% (1.18 : 1)
4	5	81% (0.96 : 1)
5	10	48% (0.34 : 1)

[a] NMR yield using 1,3,5-trimethoxybenzene as an internal standard.

2-5. Screening of nucleophiles

Table S5



1a
(0.10 mmol)

+ nucleophile (2.0 eq.)

60% $HClO_4$ aq. (30 mol%)
CH_2Cl_2 (0.05 M)
25 °C, 12 h

→ product

entry	nucleophile	results
1	H_2N-Boc	complex mixture
2	H_2N-Ts	not detected
3	H_2N-Bn	no reaction
4	H_2N-Ph	no reaction
5	$AcONH_4$	no reaction

3. Mechanistic Studies

3-1. NMR time course experiments

We conducted a time-course experiment using NMR to collect any information to shed light on the reaction mechanism. A solution of **1a** (0.10 mmol), H_2O (0.20 mmol) and 30 mol% of 60% $HClO_4$ in CD_2Cl_2 was filled in an NMR tube. We recorded the ^{1}H-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'**.

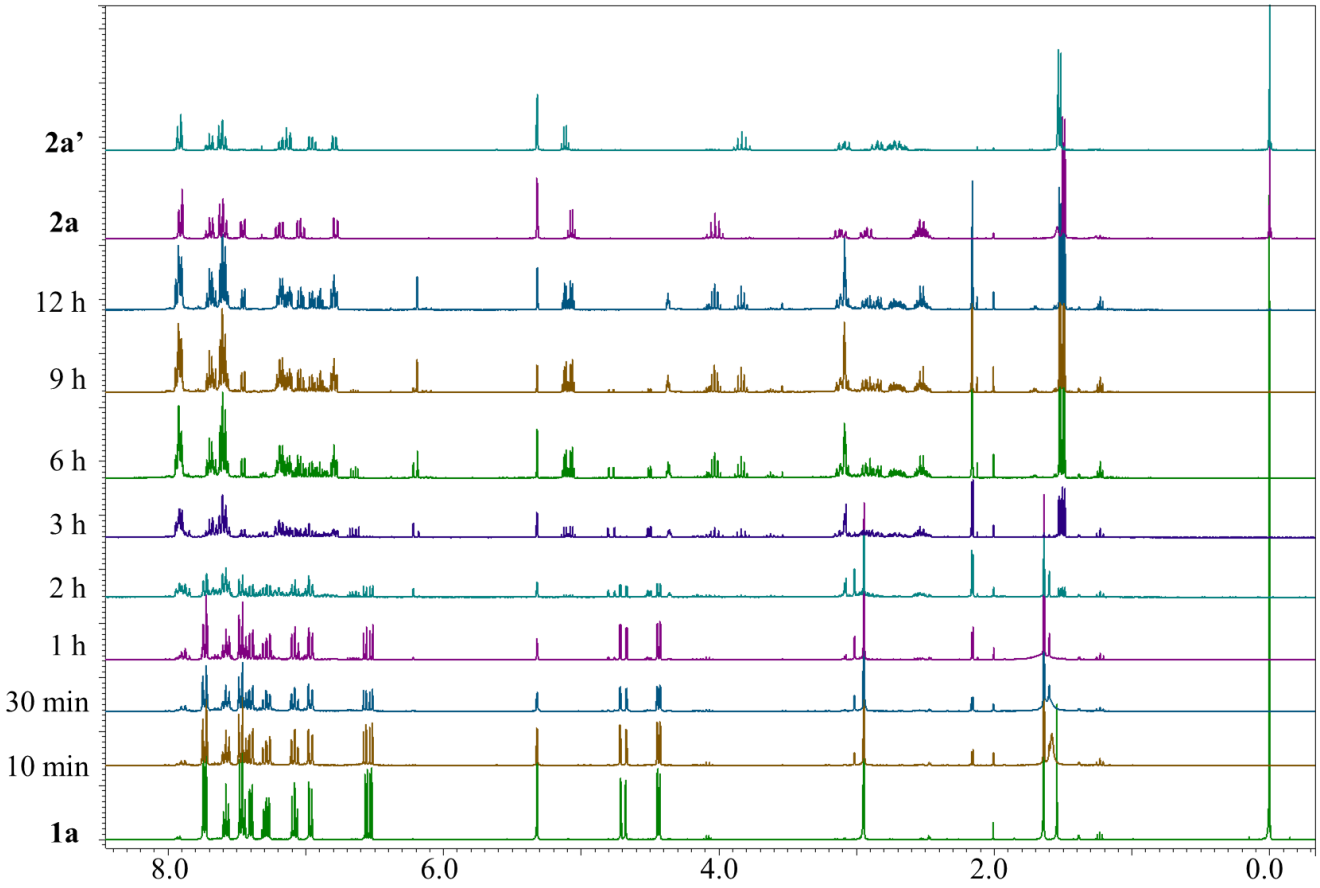
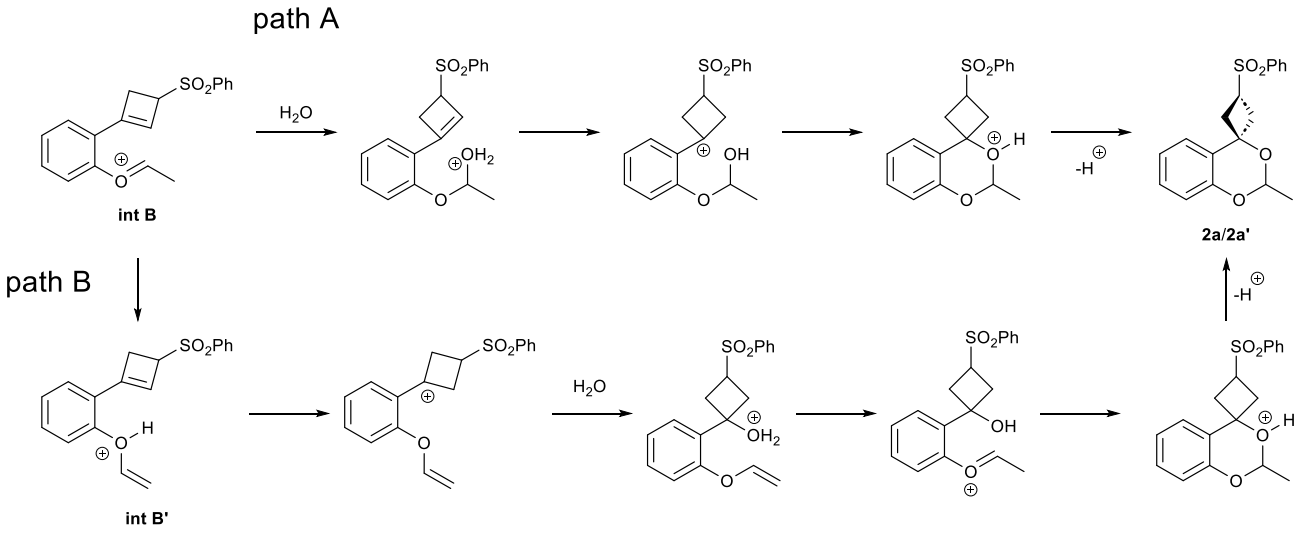


Figure S1

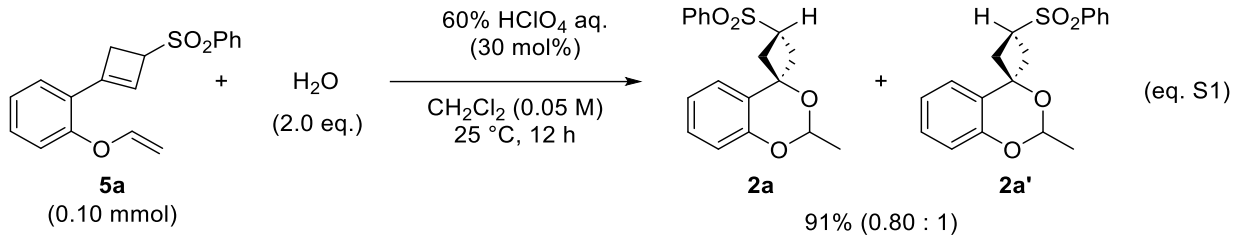
Several pathways from Int B to the products can be considered, and we have described the hypothesized reaction mechanisms (Scheme S1). Based on the NMR experiments above (Figure S1), we did not observe clear peaks attributable to a hemiacetal, benzylic cation, or benzylic alcohol. Therefore, we have chosen not to specify the structures of intermediates between **Int B** and the products in the reaction mechanism described in the main text (Scheme 3).



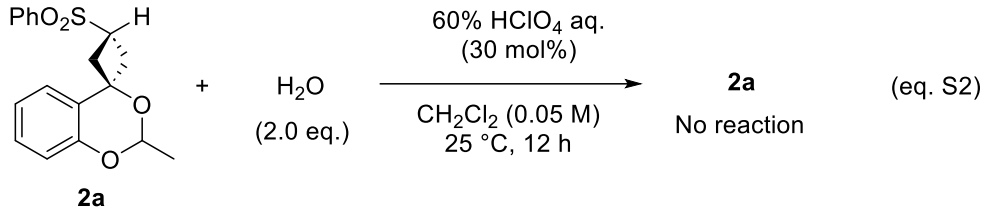
Scheme S1

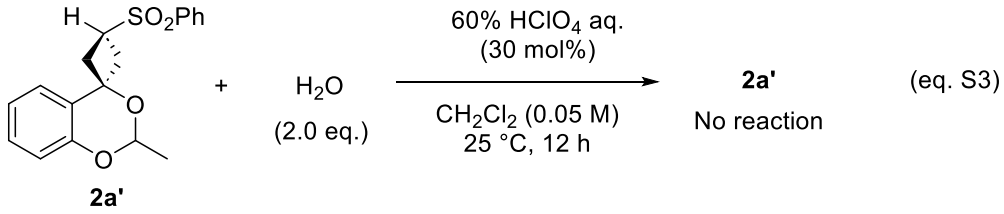
3-2. Control experiments

The reaction using cyclobutene **5a** afforded the corresponding products **2a**/**2a'** somoothly (eq. S1).

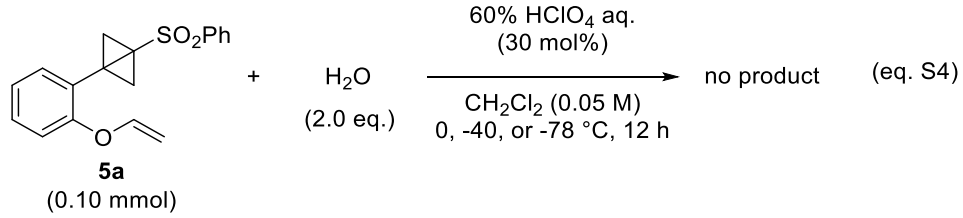


When the product **2a** or **2a'** was subjected to the heteroannulation reaction conditions, no epimerization and no decomposition were observed (eq. S2 and eq. S3).





At lower reaction temperatures (0 °C, -40 °C, -78°C), no products were obtained (eq. S4).

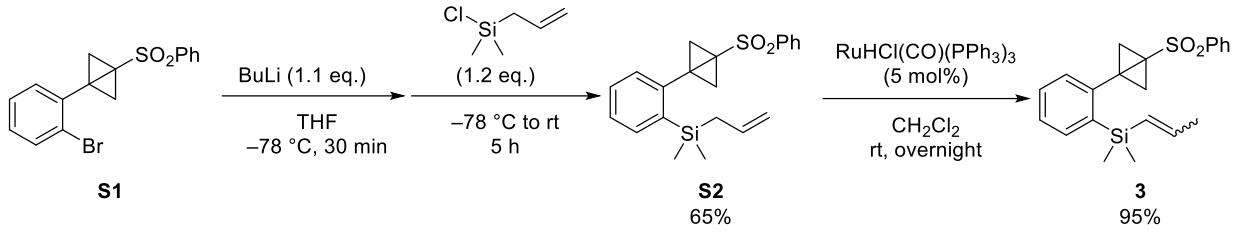


4. Experimental procedure

4-1. Preparation of starting materials

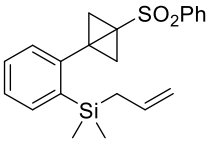
Starting materials **1a**-**1m** and **S1** were prepared through our reported method.[1]

Starting material **3** was prepared according to Scheme S2.



Scheme S2

allyldimethyl(2-(3-(phenylsulfonyl)bicyclo[1.1.0]butan-1-yl)phenyl)silane (**S2**)



A flame dried round-bottom flask equipped with a magnetic stirring bar was charged with the compound **S1**[1] (1.05 g, 3.0 mmol) and dry THF (0.2 M) under N_2. The resulting solution was cooled to -78 °C and added *n*-BuLi (2.08 mL, 1.6 M in hexane, 1.1 eq.) was added dropwise to the solution. The mixture was stirred for 30 min, then a solution of allyl(chloro)dimethylsilane (0.54 mL, 3.6 mmol, 1.2 eq.) was added, and the reaction was stirred at -78 °C before warming to rt. After 5 h, it was diluted with water and EtOAc. The combined organic layer was dried over anhydrous Na_2SO_4 and filtered. The filtrate was evaporated in vacuo and the crude BCB derivatives purified by column chromatography on silica gel (hexane/EtOAc = 20:1) to get compound **S2** as white solid (1.11 g, 65%).

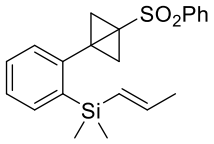
m.p. 75.3 - 76.2 °C

^{1}H NMR (300 MHz, Acetone-d_6) δ 7.99–7.96 (m, 2H), 7.89-7.86 (m, 1H), 7.75-7.63 (m, 3H), 7.57 (d, J = 7.2, 1.7 Hz, 1H), 7.41-7.30 (m, 2H), 5.80-5.66 (m, 1H), 4.88-4.75 (m, 2H), 2.56 (s, 2H), 1.81 (d, J = 8.3 Hz, 2H), 1.68 (s, 2H), 0.29 (s, 6H).

^{13}C{^{1}H} NMR (101 MHz, Acetone-d_6) δ 143.5, 141.5, 138.4, 136.0, 135.5, 134.2, 130.3, 130.2, 129.7, 128.0, 127.9, 113.9, 41.4, 35.1, 34.4, 24.5, -1.7.

HRMS (MALDI) m/z calcd for $C_{21}H_{24}O_2NaSiS$ ([M+Na]$^+$): 391.1158, found 391.1160.

(*E*)-dimethyl(2-(3-(phenylsulfonyl)bicyclo[1.1.0]butan-1-yl)phenyl)(prop-1-en-1-yl)silane (**3**)



A solution of **S2** (84.4 mg, 0.26 mmol, 1.0 eq.) in CH_2Cl_2 (6.5 mL, 0.20 M) was added $RuHCl(CO)(PPh_3)_3$ (12.3 mg, 0.0129 mmol, 5.0 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 = 15:1) to get compound **3** as white solid (80 mg, 95%).

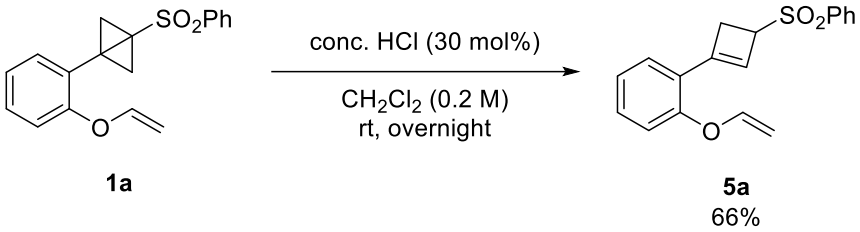
m.p. 95.7 – 96.5 °C

^{1}H NMR (300 MHz, Acetone-d_6) δ 7.99-7.95 (m, 2H), 7.87 (dd, J = 7.4, 1.5 Hz, 1H), 7.77-7.63 (m, 3H), 7.60 (d, J = 6.9 Hz, 1H), 7.42-7.31 (m, 2H), 6.18-6.05 (m, 1H), 5.81 (m, 1H), 2.53 (s, 2H), 1.80 (dd, J = 4.8, 1.4 Hz, 3H), 1.58 (s, 2H), 0.33 (s, 7H).

$^{13}C\{^1H\}$ NMR (76 MHz, Acetone-d_6) δ 144.1, 143.5, 142.1, 138.4, 136.1, 134.1, 131.0, 130.2, 130.1, 129.5, 127.9, 127.9, 41.5, 35.1, 34.7, 22.7, -1.0

HRMS (MALDI) *m/z* calcd for $C_{21}H_{24}O_2NaSiS$ ([M+Na]$^+$): 391.1158, found 391.1154.

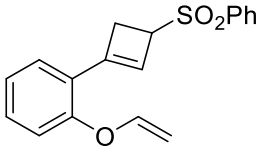
Cyclobutene derivative **5a** was prepared according to Scheme S3.



1a **5a** 66%

Scheme S3

1-(3-(phenylsulfonyl)cyclobut-1-en-1-yl)-2-(vinyloxy)benzene (**5a**)



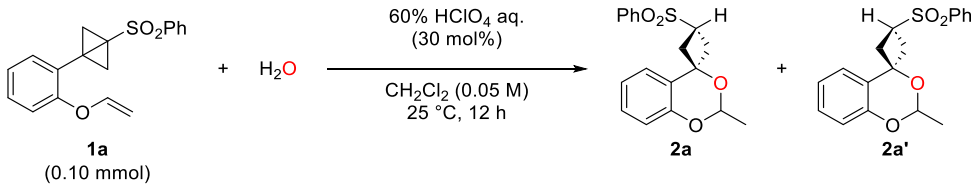
A solution of **1a** (31.2 mg, 0.10 mmol) in CH_2Cl_2 (2.0 mL, 0.20 M) was added a conc. HCl (3.3 μL, 0.030 mmol, 30 mol%). The resulting mixture was stirred at room temperature for overnight. It was diluted with water and EtOAc. The combined organic layer was dried over anhydrous Na_2SO_4 and filtered. The filtrate was evaporated in vacuo and the crude mixture was purified by column chromatography on silica gel (hexane/EtOAc = 20:1) to get compound **5a** as colorless oil (21 mg, 66%).

^1H NMR (400 MHz, Acetone-d_6) δ 7.98-7.91 (m, 2H), 7.78-7.63 (m, 3H), 7.42-7.30 (m, 2H), 7.17-7.07 (m,2H), 6.84-6.77 (m, 1H), 6.24 (d, J = 0.9 Hz), 4.77 (dd, J = 1.7, 13 Hz), 4.52-4.49 (dd, J = 1.4, 6.2 Hz), 3.15-3.02 (m, 2H),

$^{13}C\{^1H\}$ NMR (101 MHz, Acetone-d_6) δ 156.1, 148.6, 147.8, 139.6, 134.5, 131.4, 130.0, 129.3, 128.5, 125.9, 124.0, 116.9, 95.9, 61.8, 40.8, 32.5.

HRMS (MALDI) *m/z* calcd for $C_{18}H_{16}NaO_3S$ ([M+Na]$^+$): 335.0712, found 335.0709.

4-2. Brønsted acid-catalyzed heteroannulation reaction

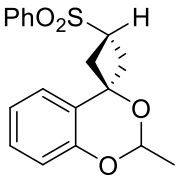


1a (0.10 mmol) **2a** **2a'**

General procedure

A flame dried test tube equipped with magnetic stirring bar charged with a 60% $HClO_4$ aq. (30 mol%) under N_2 and added dry CH_2Cl_2 (2 mL, 0.05 M). Subsequently, H_2O (3.6 μL, 0.20 mmol, 2.0 eq.) was added to resulting solution at ambient temperature. The mixture was stirred at 25 °C for 5 min, and then the compound **1** (0.10 mmol) was added. After 12 h, the mixture was filtered through a short pad of silica gel. The obtained residue was purified by flash column chromatography on silica gel (hexane/EtOAc) to give compounds **2** and **2'**.

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



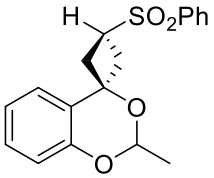
49% yield, pale yellow solid, m.p. 149.5-150.3 ℃.

^{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 $C_{18}H_{28}O_4NaS$ ([M+Na]$^+$): 353.0818, found 353.0820.

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



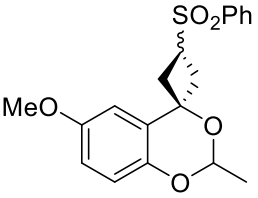
49% yield, pale yellow solid, m.p. 139.1-141.1 ℃.

^{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'**) (diastereo mixture)



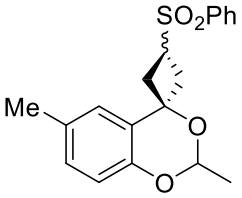
quant. (**2b**/**2b'** = 0.68 : 1), colorless oil.

^{1}H NMR (300 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, 0.43H), 6.76-6.68 (m, 2H), 6.60 (m, 0.56H), 5.02 (q, *J* = 4.7 Hz, 0.58H), 4.97 (q, *J* = 5.0 Hz, 0.46H), 4.00 (quin., *J* = 8.6 Hz, 0.66H), 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, 0.54H), 2.85 (dd, *J* = 12.0, 8.6 Hz, 0.52H), 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'**) (diastereo mixture)



quant. (**2c**/**2c'** = 1.03 : 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, 0.51H), 6.99-6.94 (m, 1H), 6.85 (d, *J* = 1.8 Hz, 0.50H), 6.69 (dd, *J* = 8.2, 1.8 Hz, 1H), 5.06 (q, *J* = 5.0 Hz, 0.50H), 5.01 (q, *J* = 5.0 Hz, 0.49H), 4.06-3.97 (m, 0.51H), 3.85-3.76 (m, 0.52H), 3.17-3.10 (m, 1H), 2.97-2.85 (m, 1H), 2.75-2.64 (m, 1H),

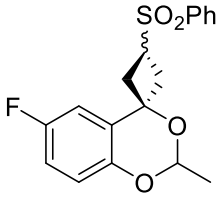
2.58-2.44 (m, 1H), 2.29-2.38 (s, 1.5H), 2.20-2.29 (s, 1.5H), 1.50 (m, 3H).

$^{13}C\{^{1}H\}$ 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, 20.7, 20.7.

HRMS (MALDI) *m/z* calcd for $C_{19}H_{20}O_4NaS$ ($[M+Na]^+$): 367.0975, found 367.0971.

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

95% yield (**2d**/**2d'** = 0.62 : 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, 0.65H), 6.92-6.85 (m, 1H), 6.81-6.78 (m, 0.34H), 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, 0.66H), 3.82-3.73 (m, 0.38H), 3.18 (dd, *J* = 13.3, 9.2 Hz, 0.40H), 3.09 (dd, *J* = 13.3, 8.7 Hz, 0.66H), 2.98-2.88 (m, 1H), 2.76-2.48 (m, 2H), 1.53 (d, *J* = 5.0 Hz, 1.2H), 1.51 (d, *J* = 5.5 Hz, 1.8H).

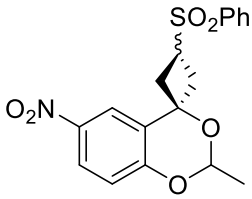
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 157.5 (d, $J_{C\text{-}F}$ = 241 Hz), 157.3 (d, $J_{C\text{-}F}$ = 241 Hz), 148.4, 148.1, 137.9, 137.9, 134.1, 129.6, 128.5, 128.4, 127.3 (d, $J_{C\text{-}F}$ = 6.7 Hz), 126.7 (d, $J_{C\text{-}F}$ = 6.7 Hz), 118.1 (d, $J_{C\text{-}F}$ = 7.7 Hz), 117.6 (d, $J_{C\text{-}F}$ = 7.7 Hz), 116.2 (d, $J_{C\text{-}F}$ = 23 Hz), 115.9 (d, $J_{C\text{-}F}$ = 23 Hz), 112.6 (d, $J_{C\text{-}F}$ = 24 Hz), 109.8 (d, $J_{C\text{-}F}$ = 24 Hz), 93.4, 93.0, 75.1, 72.1, 50.8, 49.5, 40.4, 39.5, 37.3, 37.2, 20.7, 20.7.

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

HRMS (MALDI) *m/z* calcd for $C_{18}H_{17}O_4NaS$ ($[M+Na]^+$): 371.0724, found 371.0725.

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

quant. (**2e**/**2e'** = 0.32 : 1), colorless oil.



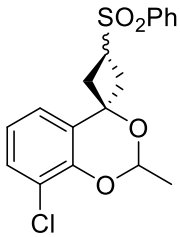
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 2.7 Hz, 0.74H), 8.10-8.06 (m, 1H), 8.02 (d, *J* = 2.7 Hz, 0.34H), 7.96-7.93 (m, 2H), 7.73-7.68 (m, 1H), 7.63-7.59 (m, 2H), 6.92-6.88 (m, 1H), 5.21 (q, *J* = 5.0 Hz, 0.26H), 5.13 (q, *J* = 5.2 Hz, 0.71H), 4.10-4.01 (m, 0.73H), 3.94-3.85 (m, 0.22H), 3.27-3.21 (m, 0.23H), 3.16 (dd, *J* = 13.3, 8.7 Hz, 0.76H), 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, 2.1H).

$^{13}C\{^{1}H\}$ 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'**) (diastereo mixture)

86% yield (**2f**/**2f'** = 0.66 : 1), colorless oil.



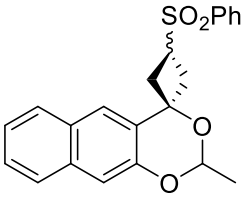
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.89 (m, 2H), 7.69-7.65 (m, 1H), 7.60-7.55 (m, 2H), 7.43 (dd, *J* = 7.8, 1.4 Hz, 0.58H), 7.27-7.22 (m, 1H), 7.00-6.94 (m, 1H), 6.86 (t, *J* = 8.0 Hz, 0.36H), 5.15 (q, *J* = 5.2 Hz, 0.40H), 5.08 (q, *J* = 5.2 Hz, 0.58H), 4.06-3.97 (m, 0.59H), 3.83-3.74 (m, 0.37H), 3.20-3.12 (m, 1H), 3.01-2.95 (m, 0.58H), 2.92-2.87 (m, 0.36H), 2.75-2.48 (m, 2H), 1.59 (m, 3H).

$^{13}C\{^1H\}$ 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'**) (diastereo mixture)

95% yield (**2g**/**2g'** = 0.62 : 1), colorless oil.



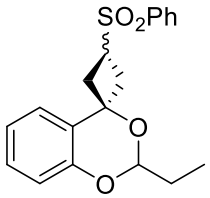
^1H NMR (300 MHz, $CDCl_3$) δ 7.99-7.96 (m, 2.7H), 7.93 (s, 0.70H), 7.73-7.57 (m, 4.7H), 7.45-7.29 (m, 2.1H), 7.20 (s, 1H), 5.23 (q, J = 5.2 Hz, 0.37H), 5.16 (q, J = 5.0 Hz, 0.59H), 4.14-4.05 (m, 0.62H), 4.02-3.94 (m, 0.40H), 3.35-3.23 (m, 1H), 3.10-2.95 (m, 1H), 2.90-2.60 (m, 2H), 1.61-1.57 (m, 3H).

$^{13}C\{^1H\}$ 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'**) (diastereo mixture)

88% (**2h**/**2h'** = 0.82 : 1), colorless oil.



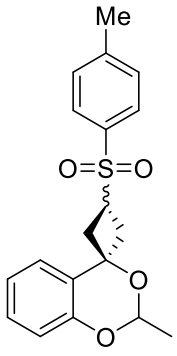
^1H NMR (400 MHz, $CDCl_3$) δ 7.95-7.92 (m, 2H), 7.70-7.65 (m, 1H), 7.61-7.56 (m, 2H), 7.49-7.47 (m, 0.57H), 7.27-7.23 (m, 2H), 7.09 (dd, J = 7.8, 1.8 Hz, 0.45H), 7.0-7.01 (m, 1H), 6.95-6.92 (m, 0.45H), 6.83-6.80 (m, 1H), 4.88 (t, J = 5.0 Hz, 0.41H), 4.83 (t, J = 5.0 Hz, 0.50H), 4.06-3.98 (m, 0.51H), 3.87-3.78 (m, 0.47H), 3.16 (dd, J = 13.3, 9.2 Hz, 1H), 3.00-2.97 (m, 0.52H), 2.88-2.83 (m, 0.43H), 2.78-2.66 (m, 1H), 2.62-2.47 (m, 1H), 1.89-1.77 (m, 2H), 1.07-1.01 (m, 3H).

$^{13}C\{^1H\}$ 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, 27.5, 27.5, 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.

2-methyl-3'-tosylspiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2i, 2i'**) (diastereo mixture)

85% yield (**2i**/**2i'** = 0.58 : 1), colorless oil.

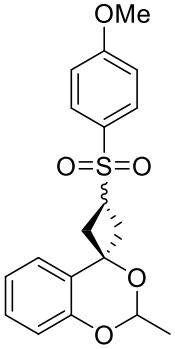


^1H NMR (400 MHz, $CDCl_3$) δ 7.82-7.79 (m, 2H), 7.49 (dd, J = 7.8, 1.4 Hz, 0.51H), 7.37 (dd, J = 8.2, 2.7 Hz, 2H), 7.21-7.14 (m, 1H), 7.09 (dd, J = 8.0, 1.6 Hz, 0.29H), 7.06-7.02 (m, 0.57H), 6.96-6.92 (m, 0.33H), 6.82-6.79 (m, 1H), 5.11 (q, J = 5.0 Hz, 0.34H), 5.06 (q, J = 5.0 Hz, 0.58H), 4.05-3.96 (m, 0.57H), 3.83-3.74 (m, 0.34H), 3.18-3.12 (m,1H), 3.00-2.95 (m,0.59H), 2.91-2.86 (m, 0.33H), 2.76-2.65 (m, 1H), 2.61-2.48 (m, 1H), 2.46 (s, 3H), 1.54 (d, J = 5.0 Hz, 1.1H), 1.52 (d, J = 5.0 Hz, 1.9H).

$^{13}C\{^1H\}$ 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, 20.7, 20.7.

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

3'-((4-methoxyphenyl)sulfonyl)-2-methylspiro[benzo[d][1,3]dioxine-4,1'-cyclobutane] (**2j, 2j'**) (diastereo mixture)



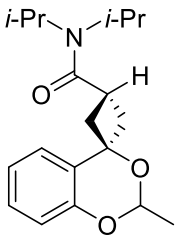
quant. (**2j**/**2j'** = 0.62 : 1), colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.82 (m, 2H), 7.48 (dd, J = 7.8, 1.4 Hz, 0.62H), 7.21-7.14 (m, 1H), 7.09 (dd, J = 7.8, 1.4 Hz, 0.39H), 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, 0.39H), 5.06 (q, J = 5.2 Hz, 0.61H), 4.03-3.95 (m, 0.54H), 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, 0.63H), 2.86 (dd, J = 12.4, 8.7 Hz, 0.39H), 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, 20.7, 20.7.

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

(3'r,4r)-*N*,*N*-diisopropyl-2-methylspiro[benzo[d][1,3]dioxine-4,1'-cyclobutane]-3'-carboxamide (**2k**)



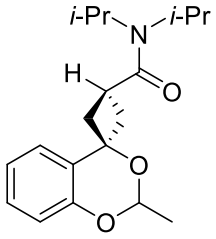
38% yield, colorless oil. (minor product)

^{1}H NMR (400 MHz, Acetone-d_6) δ 7.50-7.48 (m, 1H), 7.17-7.13 (m, 1H), 6.99-6.94 (m, 1H), 6.75 (d, J = 8.2 Hz, 1H), 5.19 (q, J = 5.0 Hz, 1H), 4.11-4.01 (m, 1H), 3.50-3.32 (m, 2H), 2.89-2.81 (m, 2H), 2.66-2.57 (m, 2H), 1.44 (d, J = 5.0 Hz, 3H), 1.39-1.37 (m, 6H), 1.20-1.17 (m, 6H).

^{13}C{^{1}H} NMR (126 MHz, Acetone-d_6) δ 172.0, 152.7, 129.4, 128.8, 125.1, 122.1, 116.8, 93.2, 74.6, 60.5, 48.7, 45.9, 42.8, 39.0, 21.1, 21.1, 20.8.

HRMS (MALDI) *m*/*z* calcd for $C_{19}H_{28}NO_3$ ([M+H]$^+$): 318.2064, found 318.2059.

(3's,4s)-*N*,*N*-diisopropyl-2-methylspiro[benzo[d][1,3]dioxine-4,1'-cyclobutane]-3'-carboxamide (**2k'**)



62% yield, colorless oil. (major product)

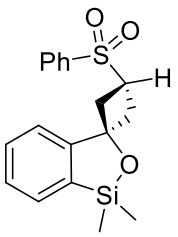
^{1}H NMR (400 MHz, Acetone-d_6) δ 7.36 (dd, J = 7.8, 1.8 Hz, 1H), 7.16-7.12 (m, 1H), 6.99-6.96 (m, 1H), 6.74 (dd, J = 8.2, 0.9 Hz, 1H), 5.18 (q, J = 5.1 Hz, 1H), 4.03-3.96 (m, 1H), 3.51-3.41 (m, 2H), 2.98-2.92 (m, 1H), 2.57 (dd, J = 8.9, 2.5 Hz, 2H), 2.46-2.40 (m, 1H), 1.49 (d, J = 5.1 Hz, 3H), 1.41-1.38 (m, 6H), 1.20-1.17 (m, 6H).

^{13}C{^{1}H} NMR (126 MHz, Acetone-d_6) δ 172.6, 153.1, 128.9, 128.6, 127.1, 122.0, 116.6, 93.8, 77.3, 48.7, 45.9, 42.4, 38.5, 31.4, 21.1, 20.8, 20.8.

HRMS (MALDI) *m*/*z* calcd for $C_{19}H_{28}NO_3$ ([M+H]$^+$): 318.2064, found 318.2059.

(3r,3'r)-1,1-dimethyl-3'-(phenylsulfonyl)-1H-spiro[benzo[c][1,2]oxasilole-3,1'-cyclobutane] (**4**)

87% yield, white solid



m.p. 154.4 – 155.4 ℃.

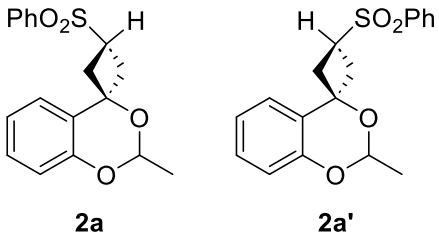
^{1}H NMR (400 MHz, Acetone-d_6) δ 7.99-7.97 (m, 2H), 7.79-7.75 (m, 1H), 7.70-7.66 (m, 2H), 7.61 (d, J = 7.8 Hz, 2H), 7.55-7.51 (m, 1H), 7.36-7.33 (m, 1H), 4.30-4.21 (m, 1H), 3.03-2.97 (m, 2H), 2.47-2.41 (m, 2H), 0.31 (s, 6H).

^{13}C NMR (101 MHz, Acetone-d_6) δ 154.8, 139.5, 136.2, 134.6, 131.3, 131.3, 130.3, 129.0, 128.4, 123.8, 82.8, 51.5, 40.5, 0.7.

HRMS (MALDI) m/z calcd for $C_{19}H_{20}O_3NaSiS$ ($[M+Na]^+$): 367.0795, found 367.0795.

5. Computational Studies

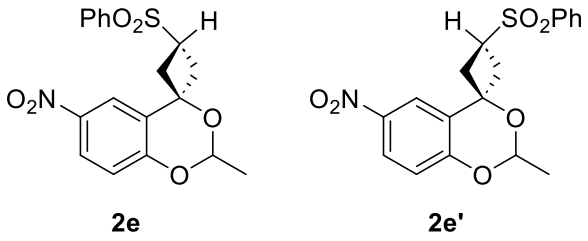
All calculations were carried with the Gaussian 16 program package.[2] Geometry optimizations were performed using density functional theory (DFT) with the B3LYP functional, and the basis sets 6-311G(d,p) with the PCM solvation model in CH_2Cl_2. Harmonic vibrational analyses were performed at the same level of theory with the geometry optimization to confirm no imaginary vibration was observed for the optimized structure, and only a single imaginary vibration was observed for the transition state.



2a **2a'**

(**2a**:**2a'** = 1.01:1)

$\Delta G_{\mathbf{2a'\text{-}2a}}$ = 4.08 kcal/mol



2e **2e'**

(**2e**:**2e'** = 0.32:1)

$\Delta G_{\mathbf{2e'\text{-}2e}}$ = 4.14 kcal/mol

2a

EE + Thermal Free Energy Correction = -1395.7669 Hartree

charge = 0, spin = 1

C	-0.60044	-1.68515	-0.15933
C	0.132397	-0.84271	-1.22753
C	1.246453	-0.57941	-0.17539
C	0.234651	-1.00056	0.945158
C	1.836777	3.202875	-0.15126
C	3.231035	3.252877	-0.08802
C	3.968231	2.078395	-0.01628
C	3.311903	0.845491	-0.01507
C	1.913555	0.77213	-0.10517
C	1.192678	1.970737	-0.16146
O	4.085357	-0.27969	0.073882
C	3.358253	-1.45628	0.447466
C	4.266885	-2.64567	0.267821
O	2.230366	-1.60586	-0.37835
S	-2.41208	-1.66967	-0.0729
O	-2.79235	-2.34863	1.178818
O	-2.91261	-2.18991	-1.35781
C	-2.92369	0.052003	0.05813
C	-3.06298	0.625393	1.322103
C	-3.47475	1.95245	1.416772
C	-3.74281	2.68597	0.26113
C	-3.60919	2.096492	-0.99588
C	-3.19803	0.77029	-1.10569
H	-0.38503	-2.75077	-0.24802
H	0.458537	-1.3555	-2.13025
H	-0.401	0.070971	-1.48797
H	-0.26312	-0.13078	1.375635
H	0.620748	-1.63495	1.741719
H	1.257489	4.117005	-0.19967
H	3.743433	4.208007	-0.0862
H	5.049792	2.090267	0.045967
H	0.110622	1.942312	-0.22235
H	3.042617	-1.33	1.492377
H	5.173965	-2.51393	0.859096

H	4.535898	-2.74433	-0.78537
H	3.757481	-3.55311	0.594513
H	-2.8756	0.036867	2.210869
H	-3.59311	2.408992	2.392014
H	-4.06604	3.717231	0.340715
H	-3.83213	2.664627	-1.8909
H	-3.11307	0.292678	-2.07314

2a'

EE + Thermal Free Energy Correction = -1395.7604 Hartree

charge = 0, spin = 1

C	-0.434599	-1.45326	0.160805
C	0.205973	-0.37548	1.061055
C	1.074538	0.08455	-0.16804
C	0.162369	-0.77678	-1.09267
C	4.318352	-1.95662	-0.04314
C	5.271933	-0.95104	-0.2138
C	4.872083	0.372862	-0.333
C	3.51381	0.697398	-0.28969
C	2.538429	-0.29892	-0.14134
C	2.968511	-1.62518	-0.01096
O	3.184999	2.019808	-0.41308
C	1.860539	2.354523	0.022622
C	1.807839	2.485453	1.539511
O	0.921306	1.458634	-0.52687
S	-2.229344	-1.73299	0.247879
O	-2.583839	-2.62167	-0.87323
O	-2.534101	-2.15467	1.626896
C	-3.029201	-0.14496	-0.03248
C	-3.333492	0.664676	1.061873
C	-3.968708	1.883996	0.83988
C	-4.291708	2.278584	-0.45813
C	-3.990316	1.454318	-1.54193
C	-3.356023	0.231953	-1.33517
H	-0.051841	-2.45908	0.341173
H	0.730397	-0.73566	1.944574
H	-0.488994	0.416918	1.338481
H	0.653136	-1.42796	-1.8132
H	-0.542142	-0.12145	-1.60504
H	4.623774	-2.9911	0.056934
H	6.326799	-1.19792	-0.24646
H	5.591033	1.174025	-0.45582
H	2.239418	-2.41881	0.108256
H	1.646582	3.310596	-0.45227
H	2.504956	3.264118	1.854085
H	2.082855	1.556611	2.042096
H	0.801597	2.772537	1.850217
H	-3.099174	0.334185	2.065463
H	-4.216843	2.520277	1.680794
H	-4.787658	3.227552	-0.62488
H	-4.254362	1.757789	-2.54776
H	-3.13892	-0.42813	-2.16489

2e

EE + Thermal Free Energy Correction = -1600.3304 Hartree

charge = 0, spin = 1

C	-1.182348	-1.99944	-0.20871
C	-0.273074	-1.30601	-1.24862
C	0.862532	-1.33235	-0.18915
C	-0.223102	-1.55449	0.917207
C	2.328353	2.184593	-0.07672
C	3.696591	1.932714	0.036423
C	4.12675	0.622325	0.110398
C	3.197712	-0.42652	0.07172
C	1.819084	-0.17068	-0.07229
C	1.39785	1.153016	-0.13656
O	3.683243	-1.68319	0.175869
C	2.690239	-2.70125	0.436834
C	3.332469	-4.04244	0.197673
O	1.603586	-2.53891	-0.43066
S	-2.946823	-1.58533	-0.11778
O	-3.472645	-2.21549	1.105902

O	-3.537915	-1.93119	-1.42223
C	-3.061697	0.198943	0.085601
C	-3.07475	0.737483	1.372451
C	-3.170269	2.118797	1.521575
C	-3.251948	2.940207	0.397407
C	-3.250767	2.386804	-0.88272
C	-3.156288	1.007143	-1.04763
N	1.857797	3.568025	-0.14505
O	0.649114	3.766821	-0.24288
O	2.694468	4.465617	-0.10058
H	-1.207521	-3.08266	-0.33364
H	-0.059845	-1.84899	-2.1671
H	-0.59108	-0.29016	-1.48141
H	0.00603	-2.2832	1.693225
H	-0.517303	-0.60988	1.376433
H	4.395073	2.756165	0.073133
H	5.177316	0.381393	0.209428
H	0.352413	1.406298	-0.24167
H	2.371856	-2.57046	1.478826
H	3.618023	-4.13025	-0.85191
H	2.626973	-4.83615	0.446013
H	4.220022	-4.14676	0.822738
H	-3.033948	0.086611	2.23619
H	-3.186292	2.551093	2.514691
H	-3.323737	4.014398	0.519332
H	-3.328007	3.026868	-1.75314
H	-3.178228	0.562414	-2.03413

2e’

EE + Thermal Free Energy Correction = -1600.3238 Hartree

charge = 0, spin = 1

C	-0.698339	-1.23283	0.162167
C	-0.340898	-0.02486	1.054361
C	0.39365	0.620154	-0.17768
C	-0.285187	-0.43817	-1.09589
C	4.02443	-0.5566	-0.09891
C	4.729135	0.639632	-0.24022
C	4.018788	1.820497	-0.3242
C	2.617755	1.8075	-0.26784
C	1.907009	0.59678	-0.15134
C	2.634779	-0.58547	-0.06076
O	1.994664	3.003725	-0.34347
C	0.597844	3.010825	0.028852
C	0.46427	3.130597	1.539568
O	-0.067332	1.919082	-0.55123
S	-2.370551	-1.94392	0.251856
O	-2.492951	-2.89446	-0.86696
O	-2.560509	-2.4212	1.632556
C	-3.533652	-0.60082	-0.03393
C	-4.023414	0.117281	1.05714
C	-4.937279	1.143399	0.830509
C	-5.350387	1.438165	-0.46852
C	-4.860382	0.704708	-1.54871
C	-3.946344	-0.3245	-1.33752
N	4.76211	-1.81559	0.00018
O	4.121591	-2.85607	0.129773
O	5.988191	-1.77188	-0.05048
H	-0.080024	-2.11239	0.348801
H	0.250925	-0.23887	1.942656
H	-1.207854	0.578436	1.322399
H	0.343815	-0.95307	-1.81925
H	-1.128155	0.028415	-1.60582
H	5.808908	0.629101	-0.27403
H	4.523174	2.772831	-0.4252
H	2.145825	-1.54561	0.029258
H	0.190608	3.888788	-0.46824
H	0.947693	4.052203	1.867525
H	0.930951	2.29435	2.062935
H	-0.591784	3.175603	1.811158
H	-3.713412	-0.13967	2.061742
H	-5.331212	1.705449	1.668623
H	-6.063509	2.23614	-0.63878
H	-5.194126	0.927132	-2.55499

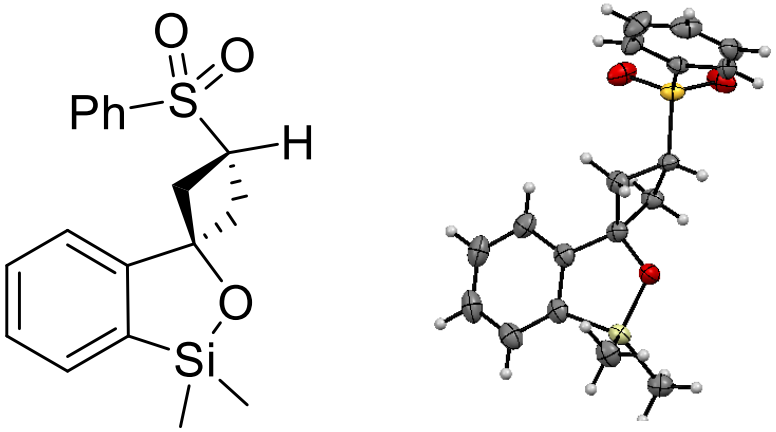
H	-3.577684	-0.91845	-2.1637

6. References

1. M. Takatsuki, H. Aoyama, K. Murai, M. Arisawa and M. Sako, *Chem. Commun*., **2023**, *59*, 7467.
2. Gaussian 16, Revision C.02, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2019.

7. X-ray Crystallographic Analysis

(3r,3'r)-1,1-dimethyl-3'-(phenylsulfonyl)-1H-spiro[benzo[c][1,2]oxasilole-3,1'-cyclobutane] (**4**)



CCDC number 2324350

Bond precision:	C-C = 0.0035 A	Wavelength=1.54187	
Cell:	a=13.1061(7)	b=8.2101(4)	c=16.7736(9)
	alpha=90	beta=90.719(6)	gamma=90
Temperature:	143 K		

	Calculated	Reported
Volume	1804.74(16)	1804.73(16)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C18 H20 O3 S Si	C18 H20 O3 S Si
Sum formula	C18 H20 O3 S Si	C18 H20 O3 S Si
Mr	344.49	344.50
Dx,g cm-3	1.268	1.268
Z	4	4
Mu (mm-1)	2.324	2.324
F000	728.0	728.0
F000'	732.18	
h,k,lmax	15,9,20	15,9,20
Nref	3309	3259
Tmin,Tmax	0.455,0.793	0.492,0.793
Tmin'	0.236	

Correction method= # Reported T Limits: Tmin=0.492 Tmax=0.793
AbsCorr = MULTI-SCAN

Data completeness= 0.985 Theta(max)= 68.234

R(reflections)= 0.0485(2831) wR2(reflections)= 0.1295(3259)

S = 1.044 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

```
PLAT230_ALERT_2_C Hirshfeld Test Diff for    S1       --O3       .        6.2 s.u.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.596 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         41 Report
PLAT934_ALERT_3_C Number of (Iobs-Icalc)/Sigma(W) > 10 Outliers ..          1 Check
```

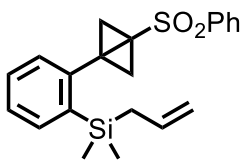
Alert level G

```
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 H20 O3 S1 Si1
PLAT882_ALERT_1_G No Datum for _diffrn_reflns_av_unetI/netI ......     Please Do !
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600          9 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..........        3.7 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          2 Info
```

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

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
2 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 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

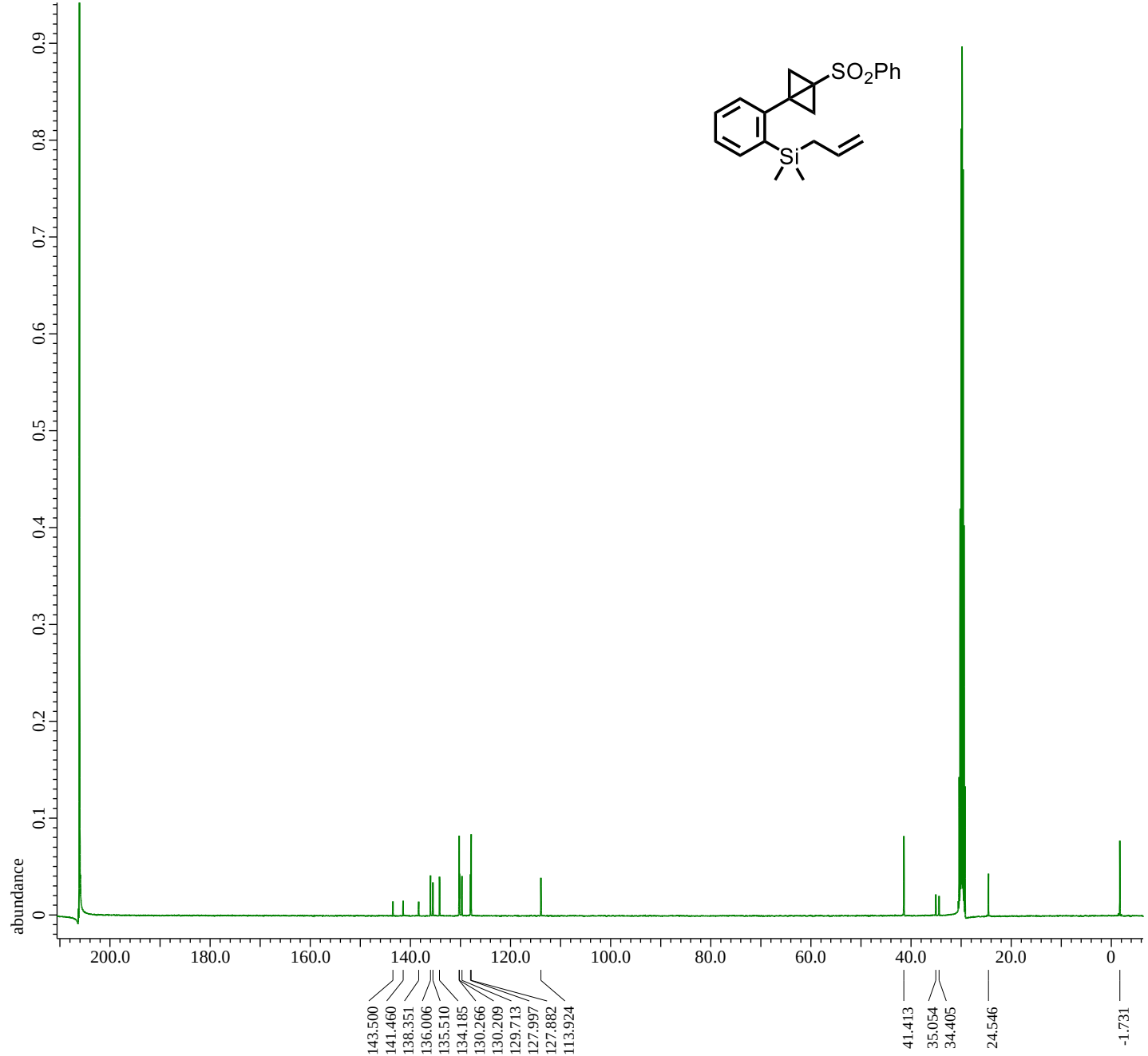
7. NMR Spectra




```
---- 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          = 36 silicon propenyl Aceto
Author            = delta
Experiment        = proton.jxp
Sample_Id         = silicon propenyl recrysta
Solvent           = ACETONE-D6
Actual_Start_Time = 29-JAN-2023 12:51:10
Revision_Time     = 15-DEC-2023 17:29:05

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          = 18.7[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]
```

7.988 7.983 7.966 7.960 7.885 7.882 7.860 7.857 7.752 7.738 7.726 7.718 7.708 7.683 7.658 7.641 7.629 7.586 7.581 7.580 7.579 7.562 7.556 7.410 7.405 7.386 7.361 7.354 7.345 7.326 7.302 7.297 5.802 5.769 5.746 5.720 5.692 5.659 4.879 4.874 4.871 4.862 4.821 4.817 4.810 4.788 4.785 4.781 4.755 4.752 4.747 2.560 1.828 1.801 1.675 0.286

[1] H NMR spectrum of **S2** (300 MHz, Acetone-d_6)



X : parts per Million : Carbon13

```
---- 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          = 36 silicon propenyl Aceto
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = silicon propenyl recrysta
Solvent           = ACETONE-D6
Actual_Start_Time = 30-JAN-2023 04:41:21
Revision_Time     = 30-JAN-2023 09:04:57

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             = 5017
Total_Scans       = 5017

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]
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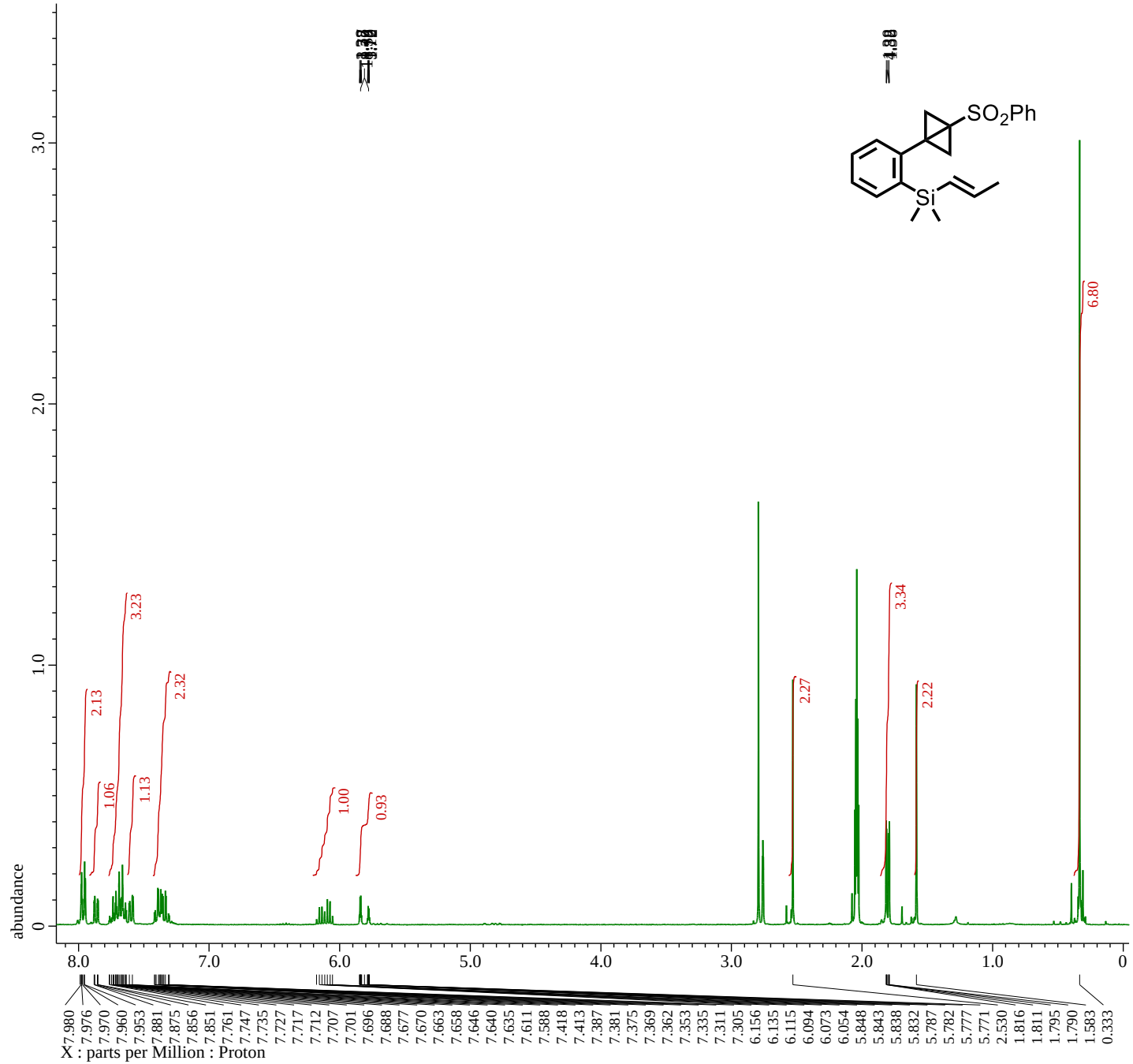
^{13}C NMR spectrum of **S2** (101 MHz, Acetone-d_6)

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---- 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

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Author              = delta
Experiment          = proton.jxp
Sample_Id           = 211196 column 4-5 Acetone
Solvent             = ACETONE-D6
Actual_Start_Time   = 20-FEB-2024 14:51:10
Revision_Time       = 21-FEB-2024 23:07:00

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    = 0.5[s]
Recvr_Gain          = 44
Temp_Get            = 25.1[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     = 3.40717696[s]
```
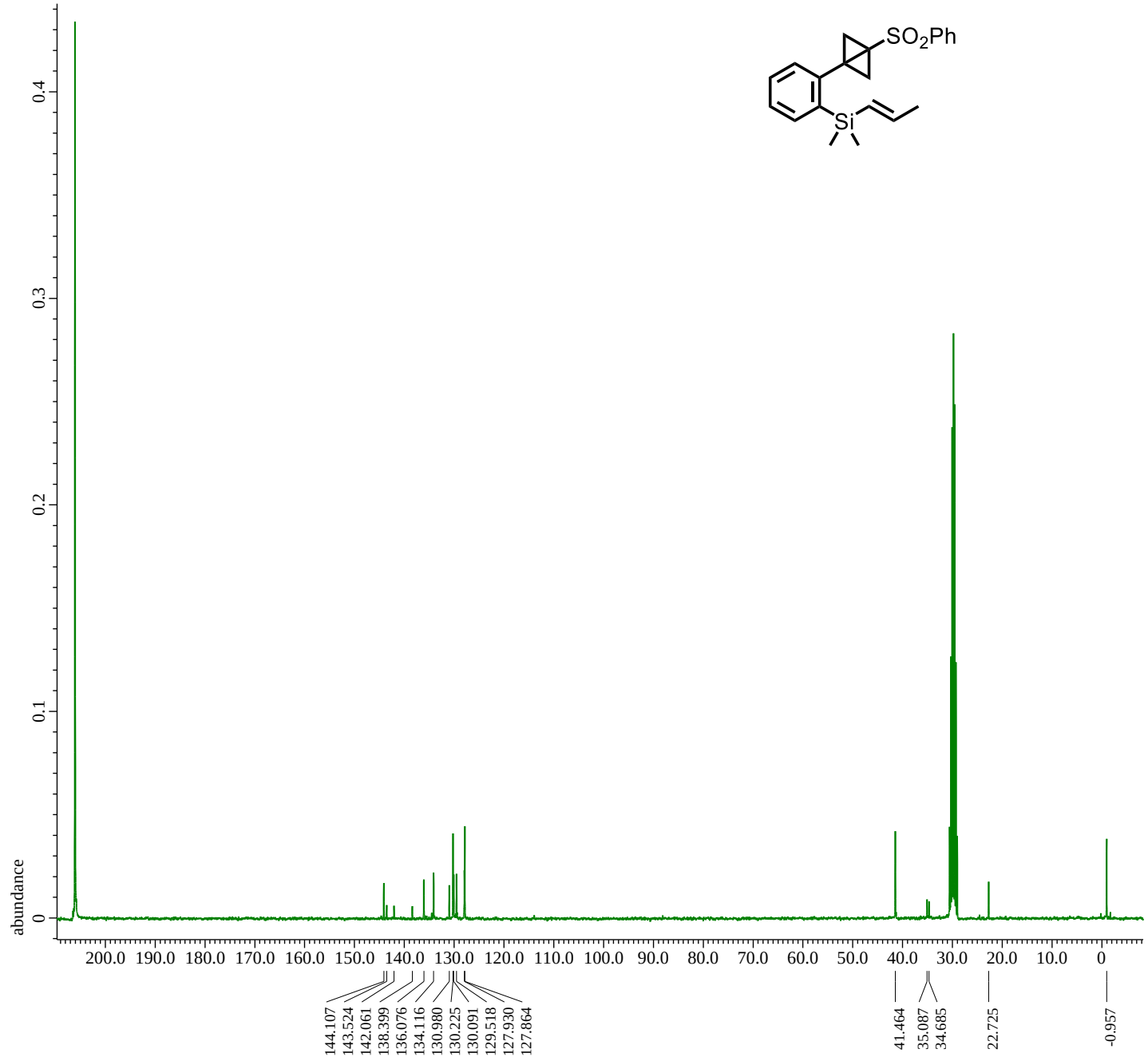
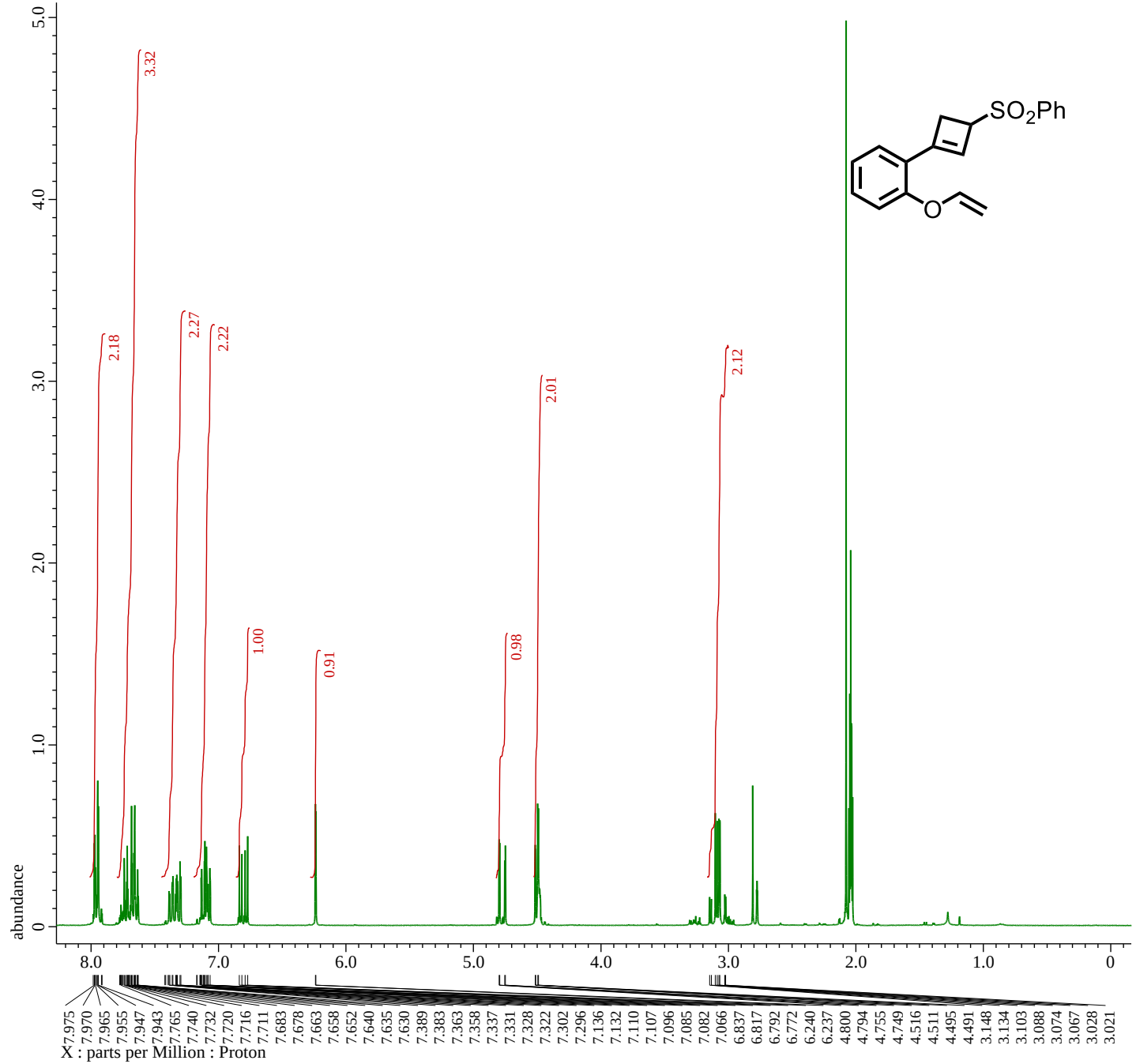

7.980 7.976 7.970 7.960 7.953 7.881 7.875 7.856 7.851 7.761 7.747 7.735 7.727 7.717 7.712 7.707 7.701 7.696 7.688 7.677 7.670 7.663 7.658 7.646 7.640 7.635 7.611 7.588 7.418 7.413 7.387 7.381 7.375 7.369 7.362 7.353 7.335 7.311 7.305 6.156 6.135 6.115 6.094 6.073 6.054 5.848 5.843 5.838 5.832 5.787 5.782 5.777 5.771 2.530 1.816 1.811 1.795 1.790 1.583 0.333

[1] H NMR spectrum of **3** (300 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          = 211196 column 4-5 CDCl3 2
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 211196 column 4-5 CDCl3 2
Solvent           = ACETONE-D6
Actual_Start_Time = 22-FEB-2024 21:56:58
Revision_Time     = 28-FEB-2024 15:03:23

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             = 2302
Total_Scans       = 2302

Relaxation_Delay  = 0.5[s]
Recvr_Gain        = 50
Temp_Get          = 23[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          = 0.5[s]
Repetition_Time   = 1.88412032[s]
```

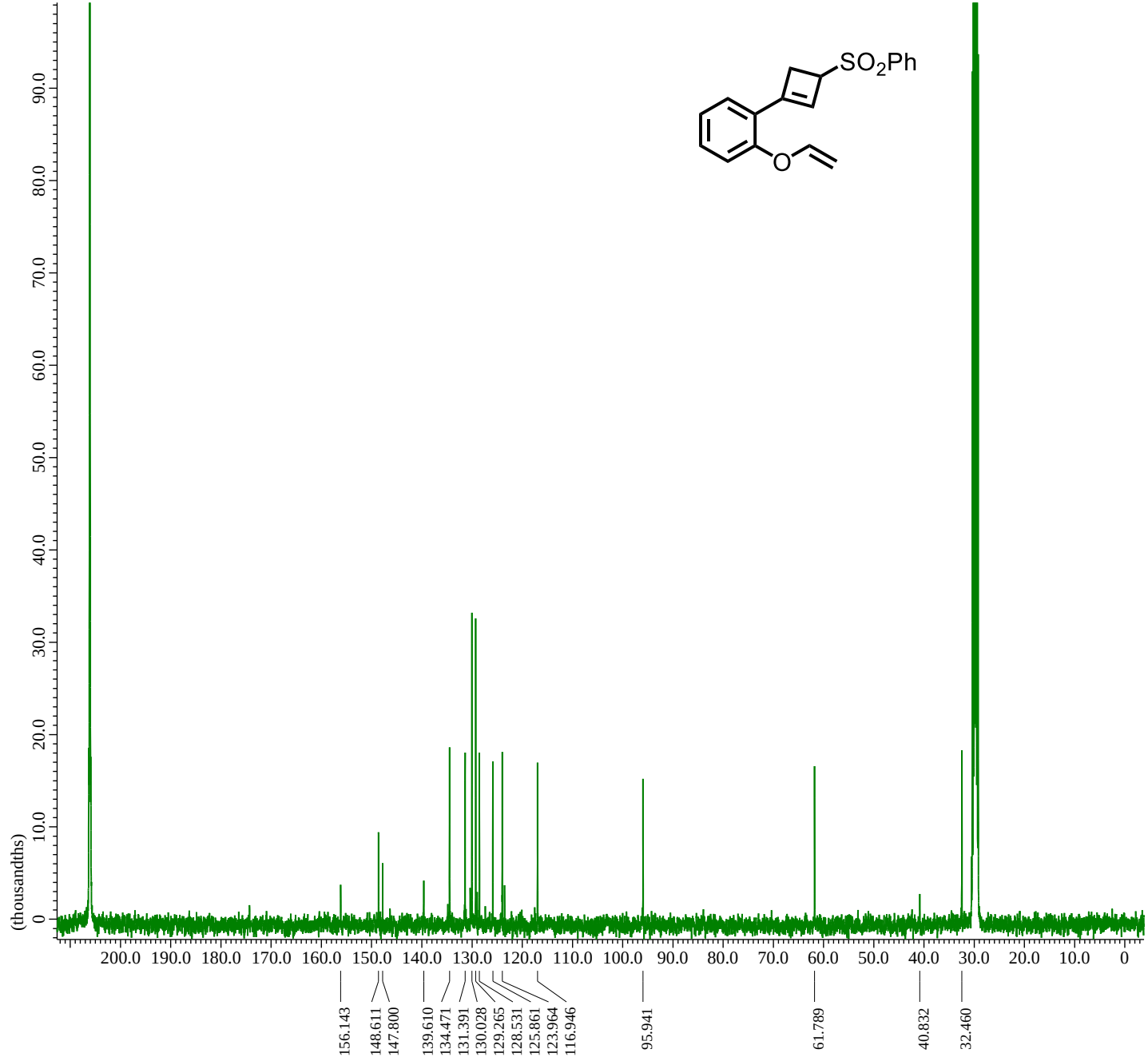
^{13}C NMR spectrum of **3** (76 MHz, Acetone-d_6)




```
---- 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	= 211118,20,21 column Aceto
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 211118,20,21 column Aceto
Solvent	= ACETONE-D6
Actual_Start_Time	= 30-DEC-2023 20:42:50
Revision_Time	= 12-JAN-2024 22:25:15
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 7368
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	= 32
Total_Scans	= 32
Relaxation_Delay	= 1[s]
Recvr_Gain	= 46
Temp_Get	= 22.6[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	= 3.90717696[s]

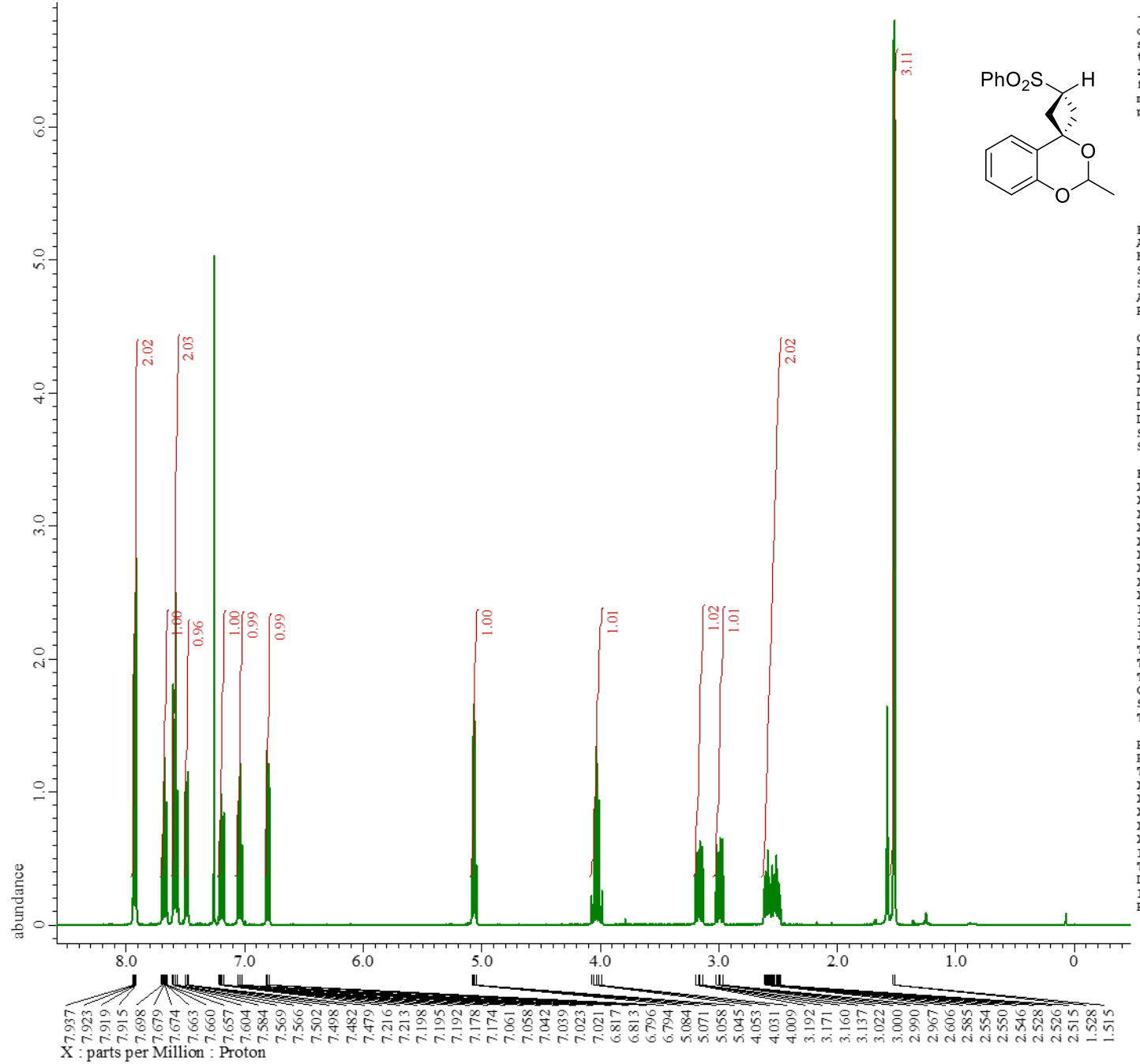
[1] H NMR spectrum of **5a** (400 MHz, Acetone-d_6)




```
---- 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
```

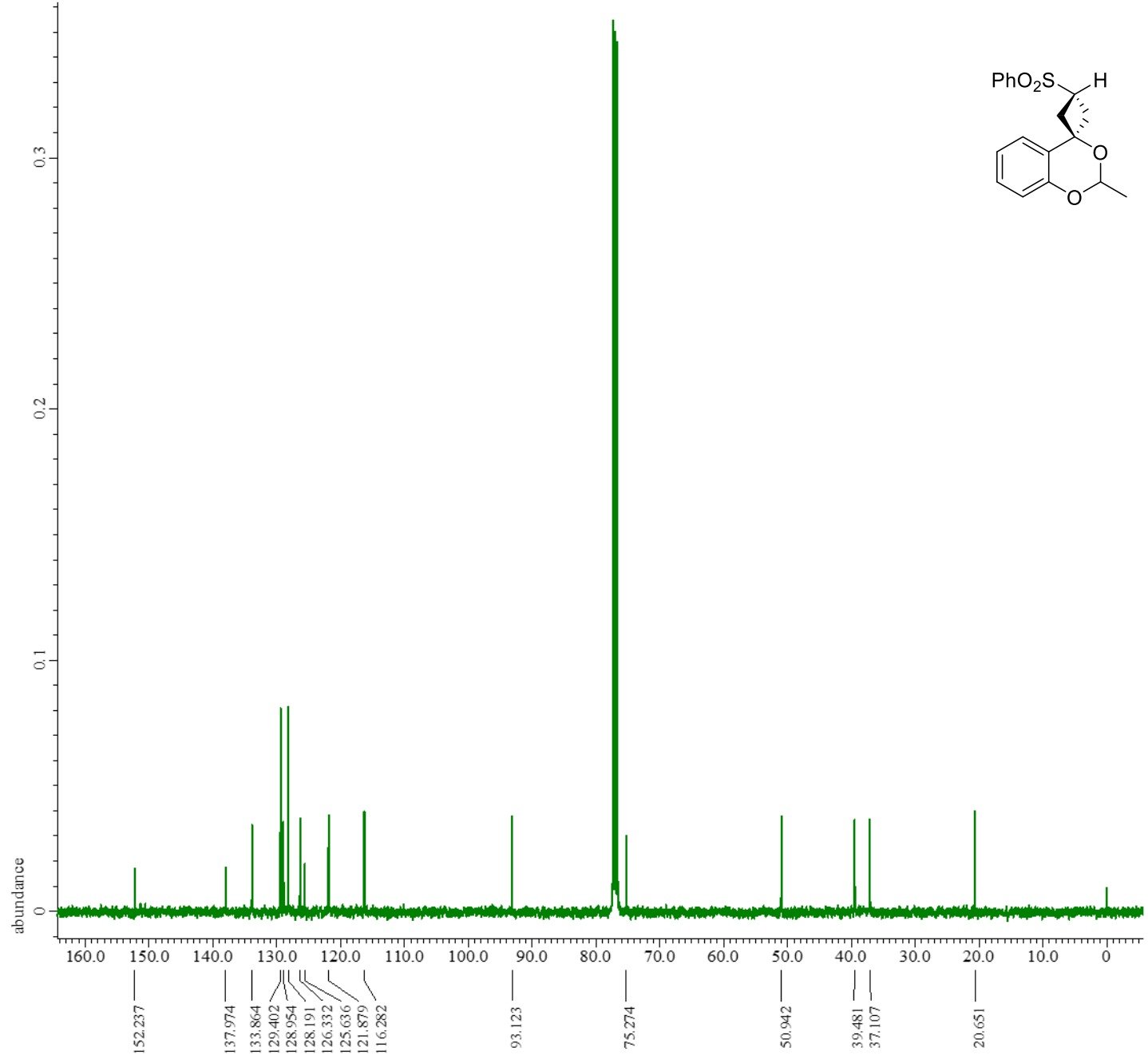
```
Filename           = 211118,20,21 column Aceto
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = 211118,20,21 column Aceto
Solvent            = ACETONE-D6
Actual_Start_Time  = 31-DEC-2023 00:06:52
Revision_Time      = 12-JAN-2024 22:37:37

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              = 1750
Total_Scans        = 1750

Relaxation_Delay   = 1[s]
Recvr_Gain         = 50
Temp_Get           = 20.4[dC]
X_90_Width         = 9.2[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 4.7[dB]
X_Pulse            = 3.06666667[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           = 1[s]
Repetition_Time    = 2.04333312[s]
```

^{13}C NMR spectrum of **5a** (101 MHz, Acetone-d_6)



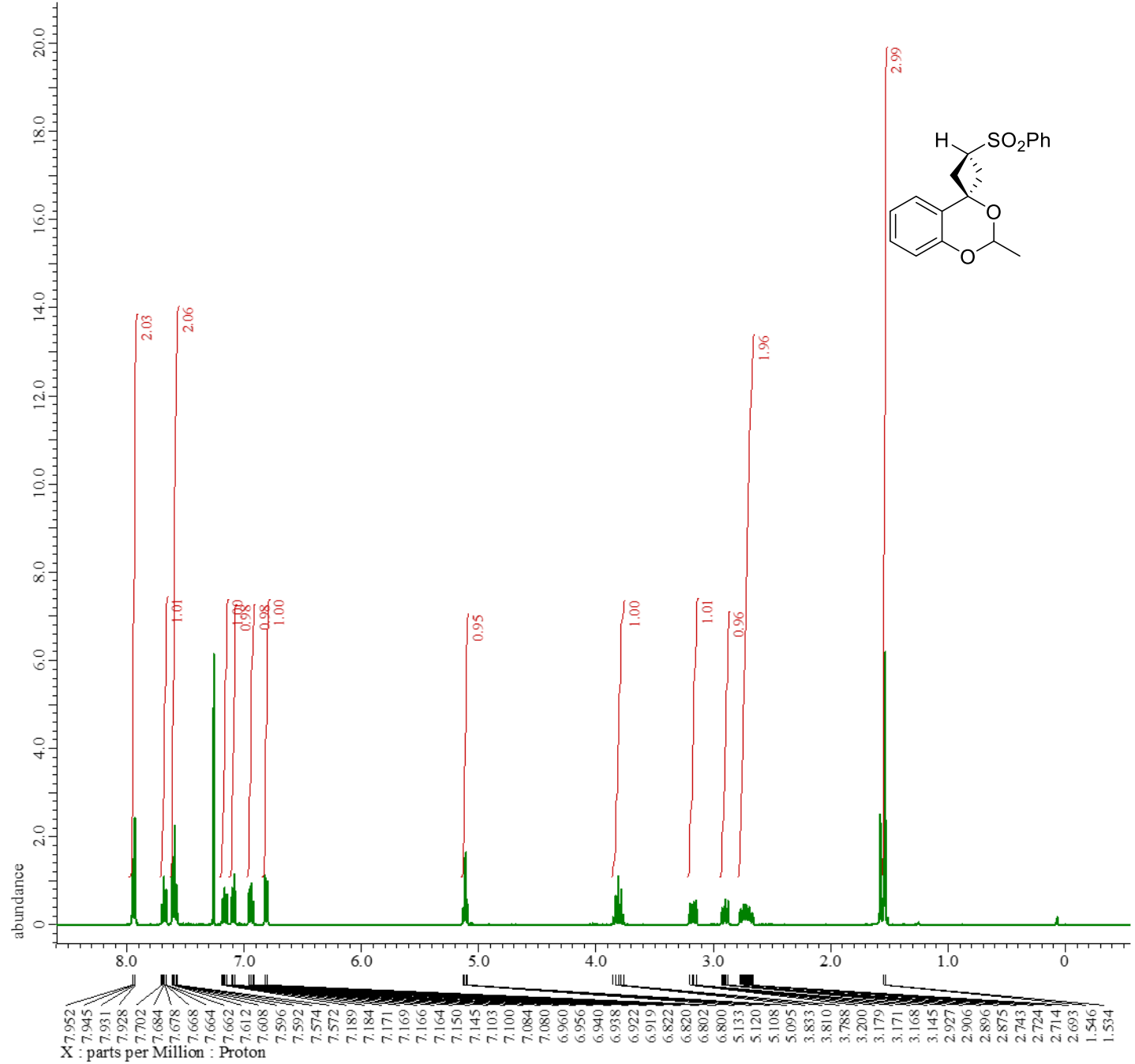

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 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, $CDCl_3$)




```
---- 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]
```

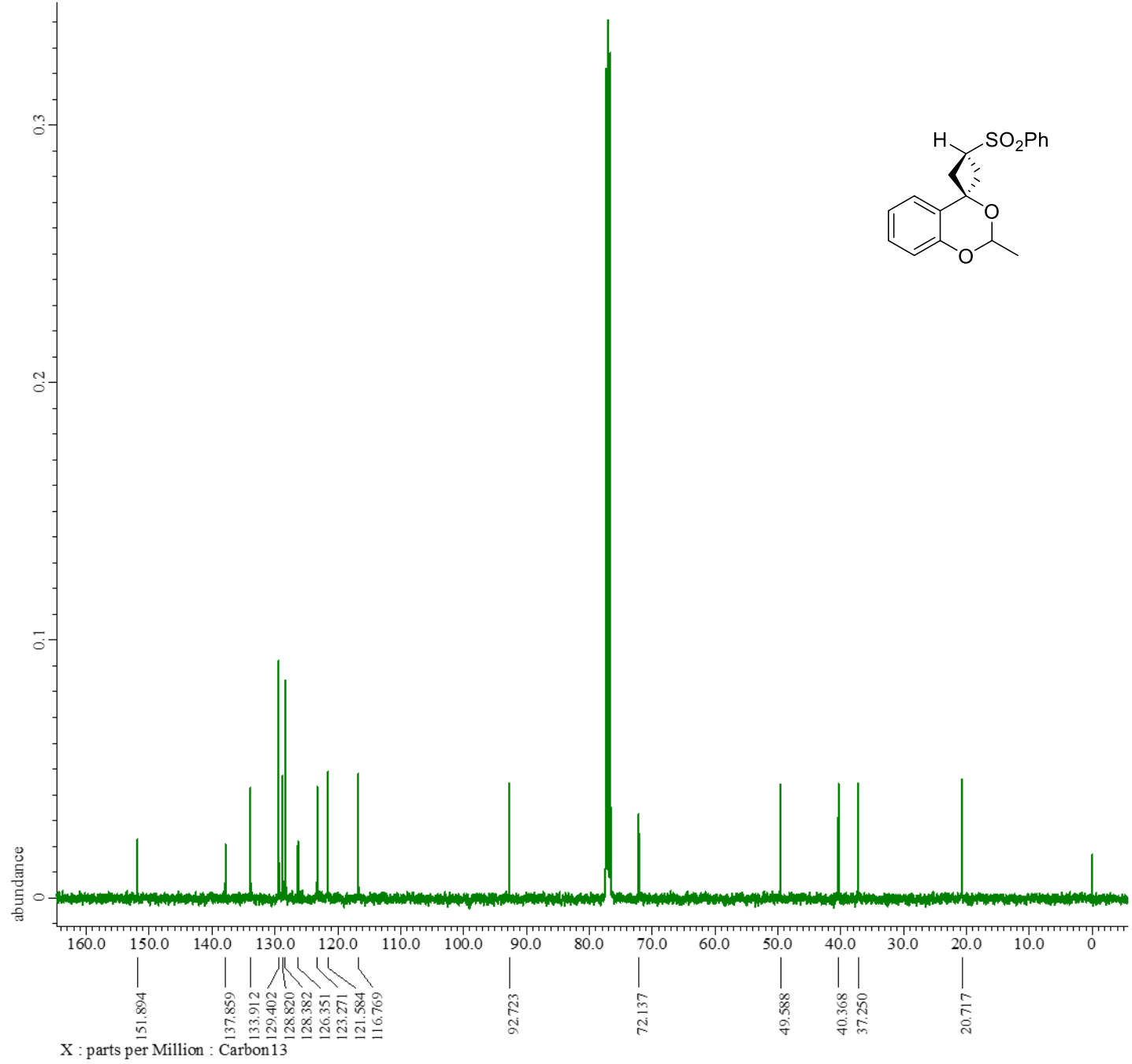
^{13}C NMR spectrum of **2a** (101 MHz, $CDCl_3$)




```
---- 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]

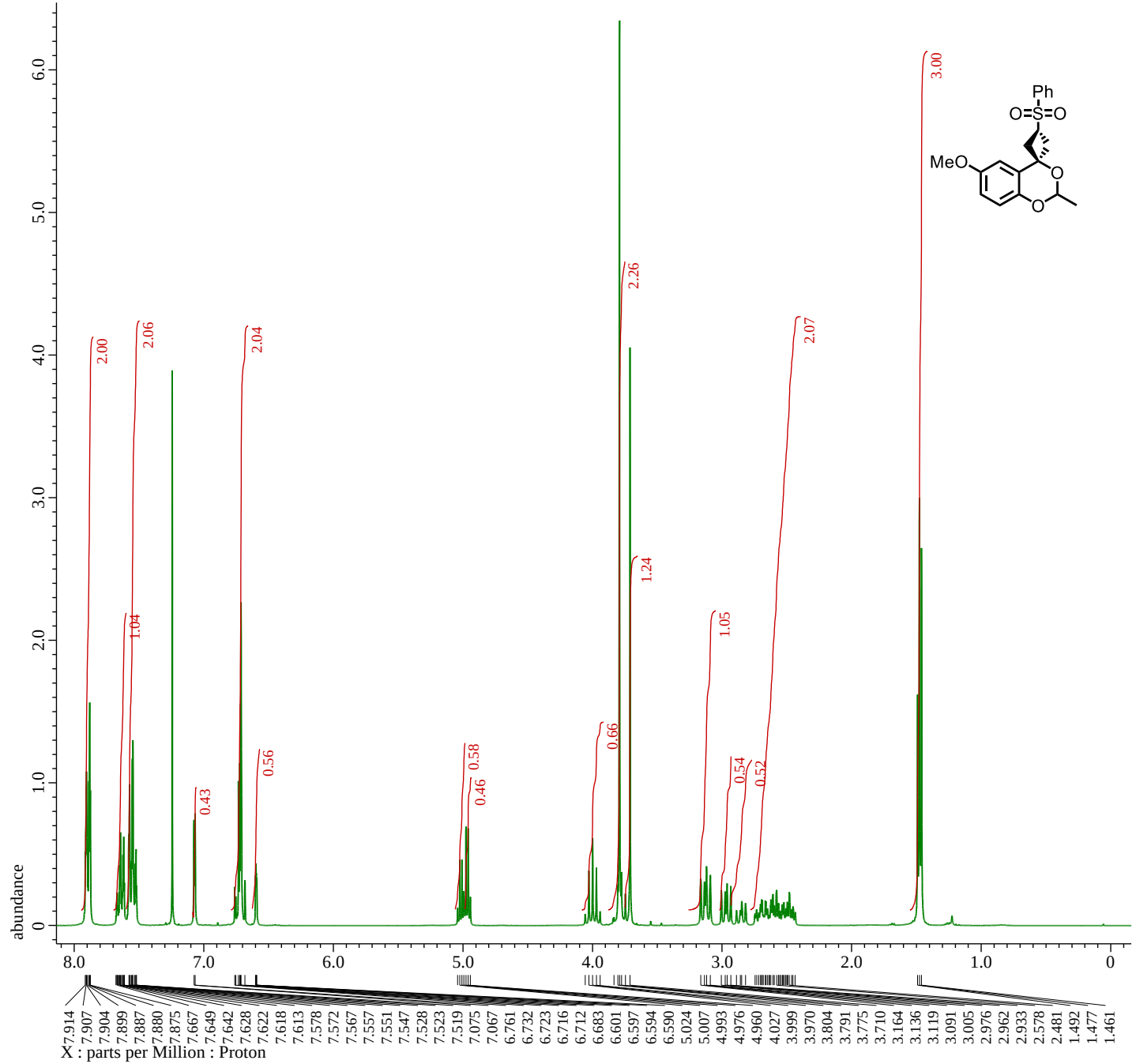
[1] H NMR spectrum of **2a'** (400 MHz, $CDCl_3$)




```
---- 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
```

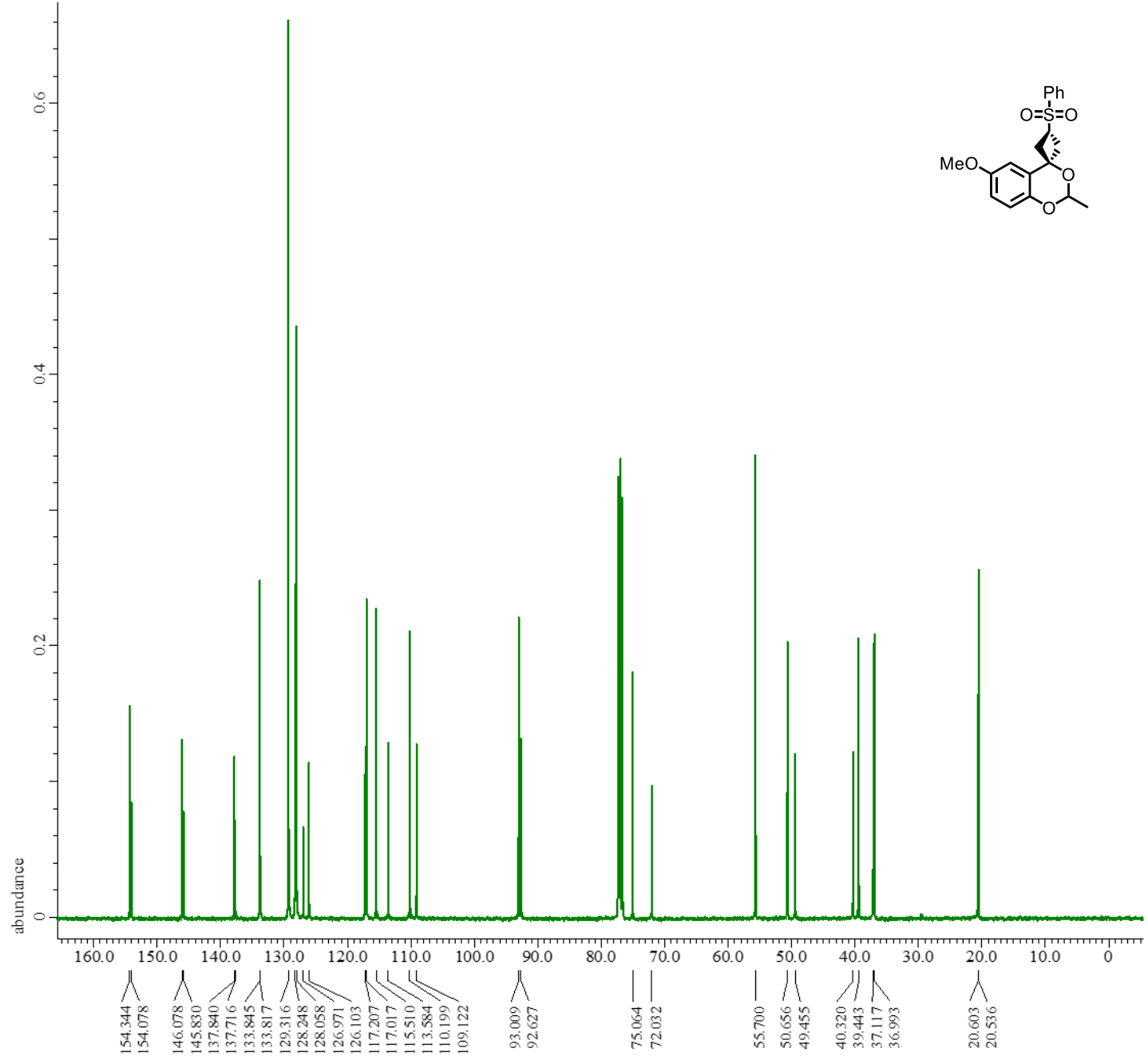
```
Filename            = 11 shita CDCl3_Carbon-1-2
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = shita CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 31-OCT-2022 10:07:39
Revision_Time       =  7-JAN-2023 09:41:49

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, $CDCl_3$)




```
---- 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 CDCl3
Solvent           = CHLOROFORM-D
Actual_Start_Time = 16-DEC-2022 11:42:52
Revision_Time     = 14-DEC-2023 21:11:56

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]
```

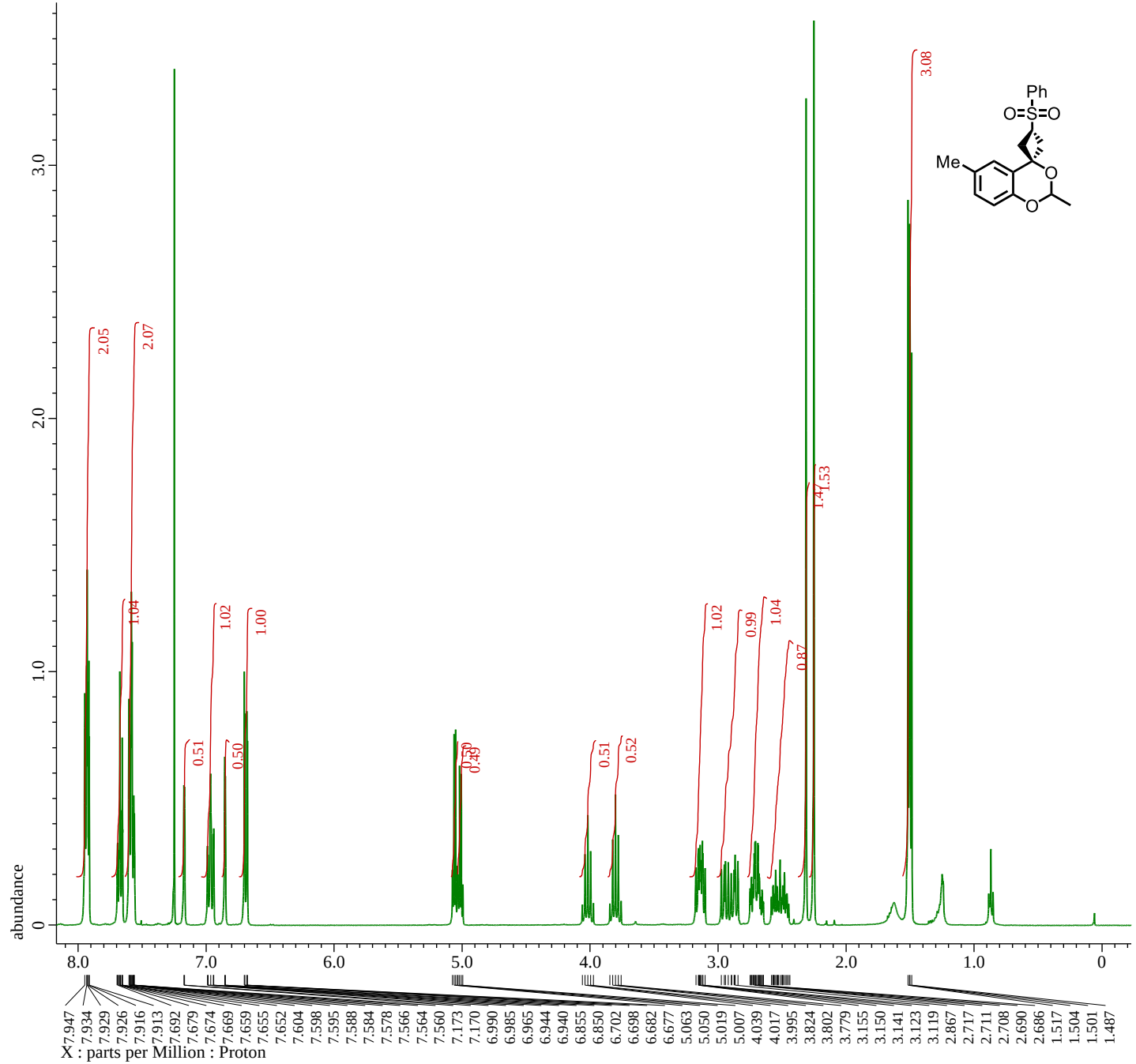
^{1}H NMR spectrum of **2b**/**2b'** (300 MHz, $CDCl_3$)




```
---- 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
```

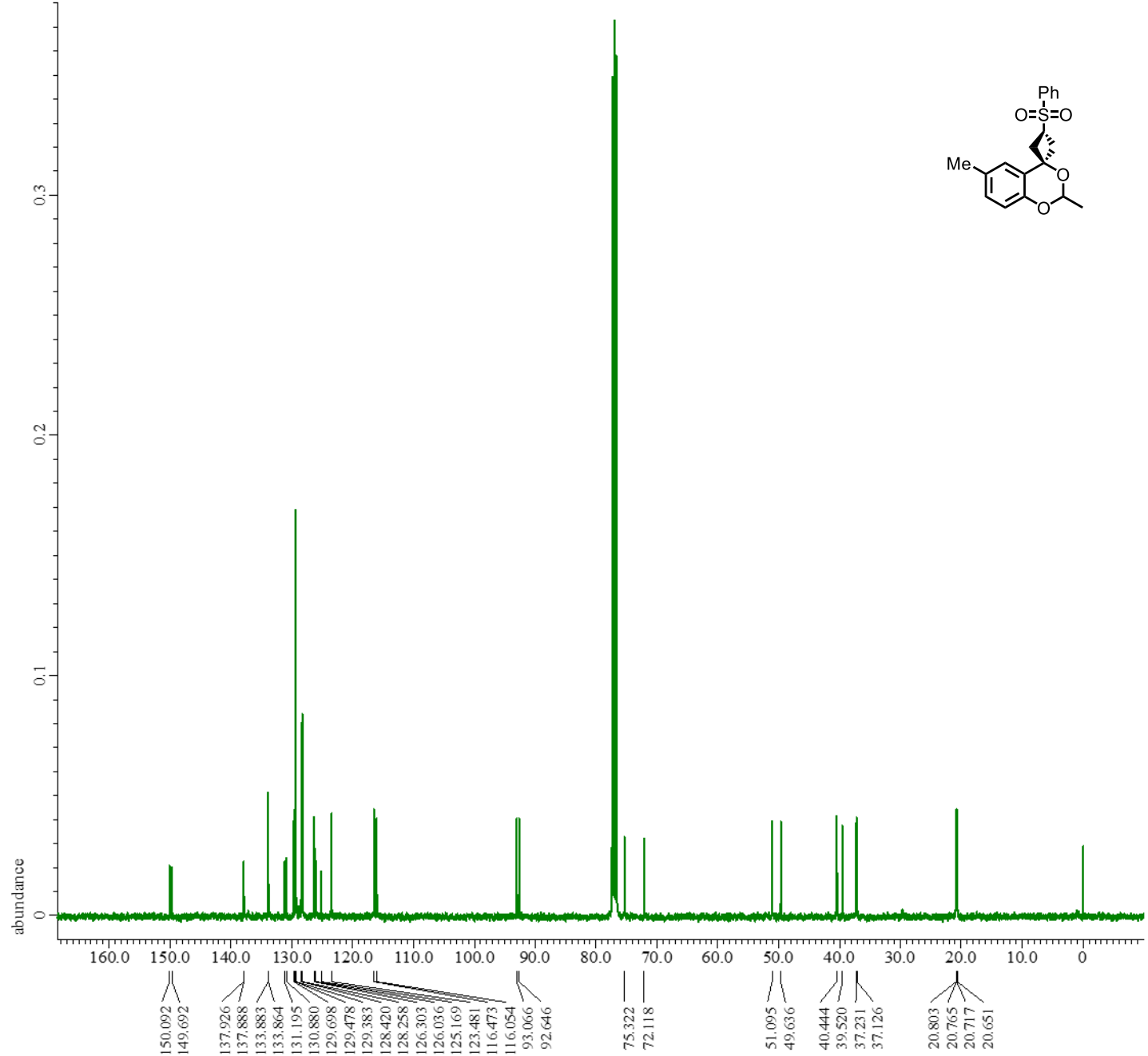
```
Filename            = 12 Ar 4-OMe PLC CDCl3_Car
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 4-OMe PLC CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-DEC-2022 15:42:44
Revision_Time       = 11-JAN-2023 10:01:41

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, $CDCl_3$)




```
---- 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 CDCl3
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 25-NOV-2022 22:43:24
Revision_Time      = 15-DEC-2023 10:08:14

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]
```

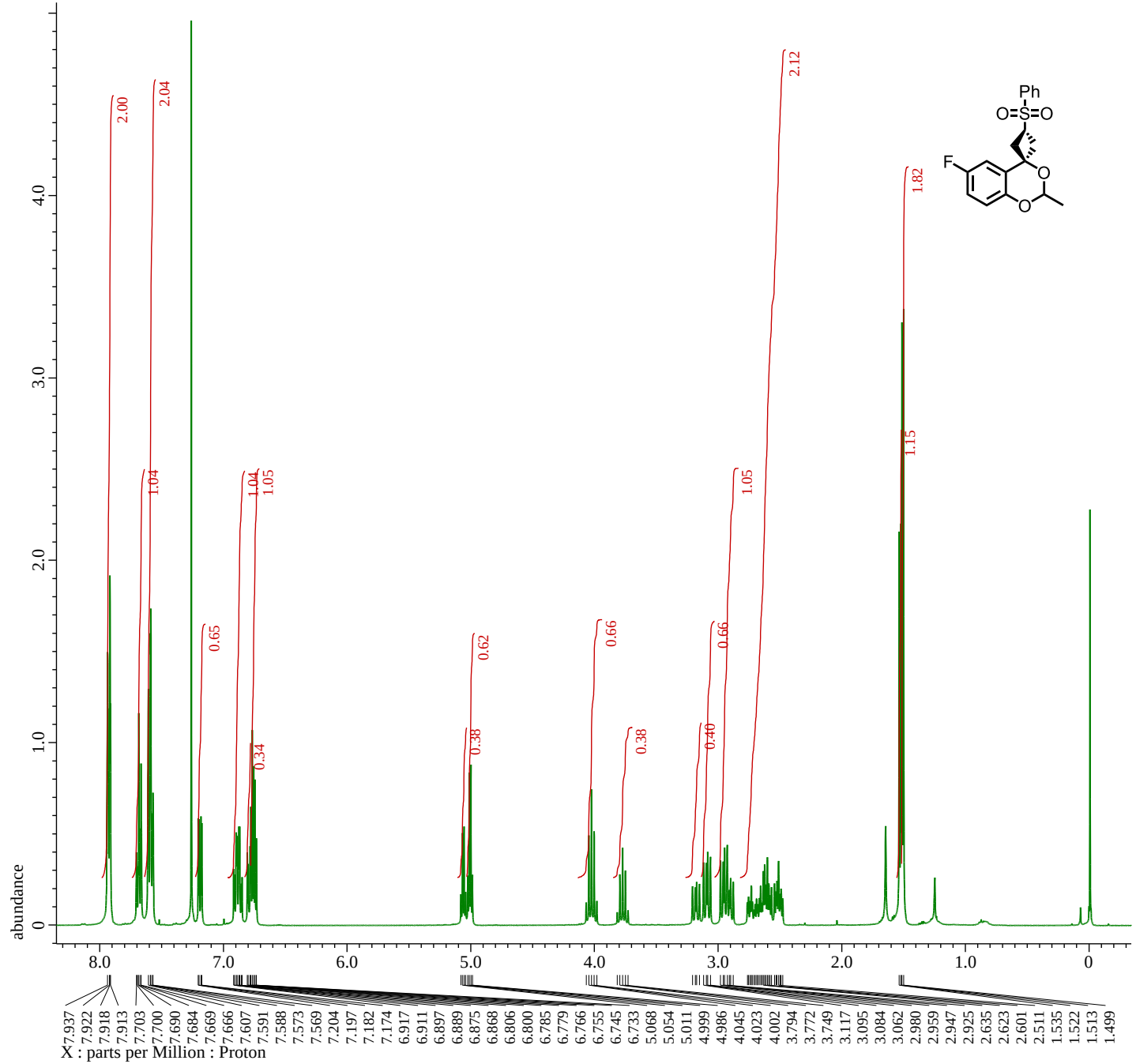
^{1}H NMR spectrum of **2c**/**2c'** (400 MHz, $CDCl_3$)




```
---- 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
```

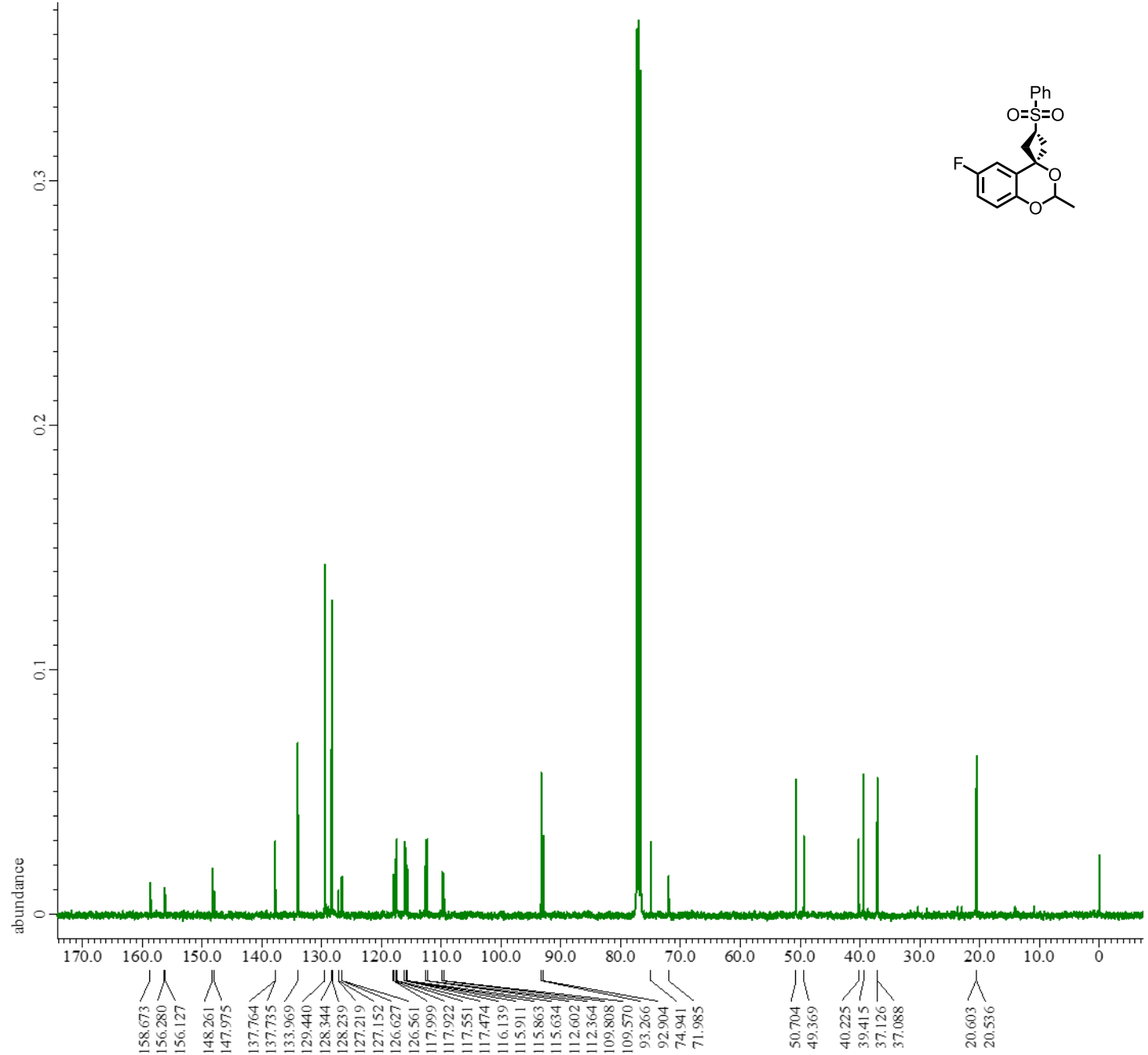
```
Filename            = 15 Ar 4-Me CDCl3_Carbon-1
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210556 3 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-NOV-2022 03:05:51
Revision_Time       = 11-JAN-2023 11:03:31

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, $CDCl_3$)




```
---- 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       = 15-DEC-2023 10:16:09

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]
```

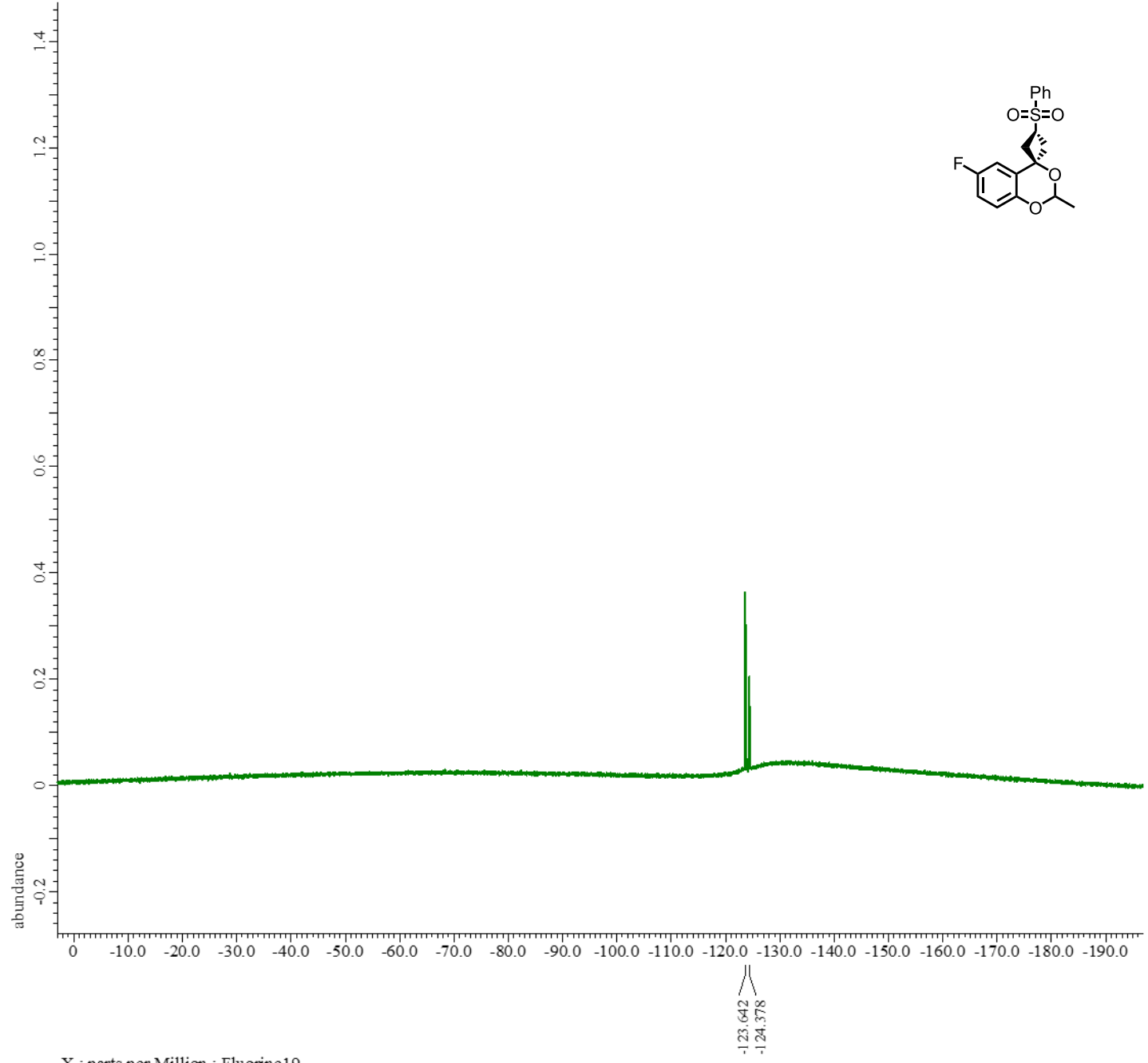
^{1}H NMR spectrum of **2d**/**2d'** (400 MHz, $CDCl_3$)




```
---- 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]
```

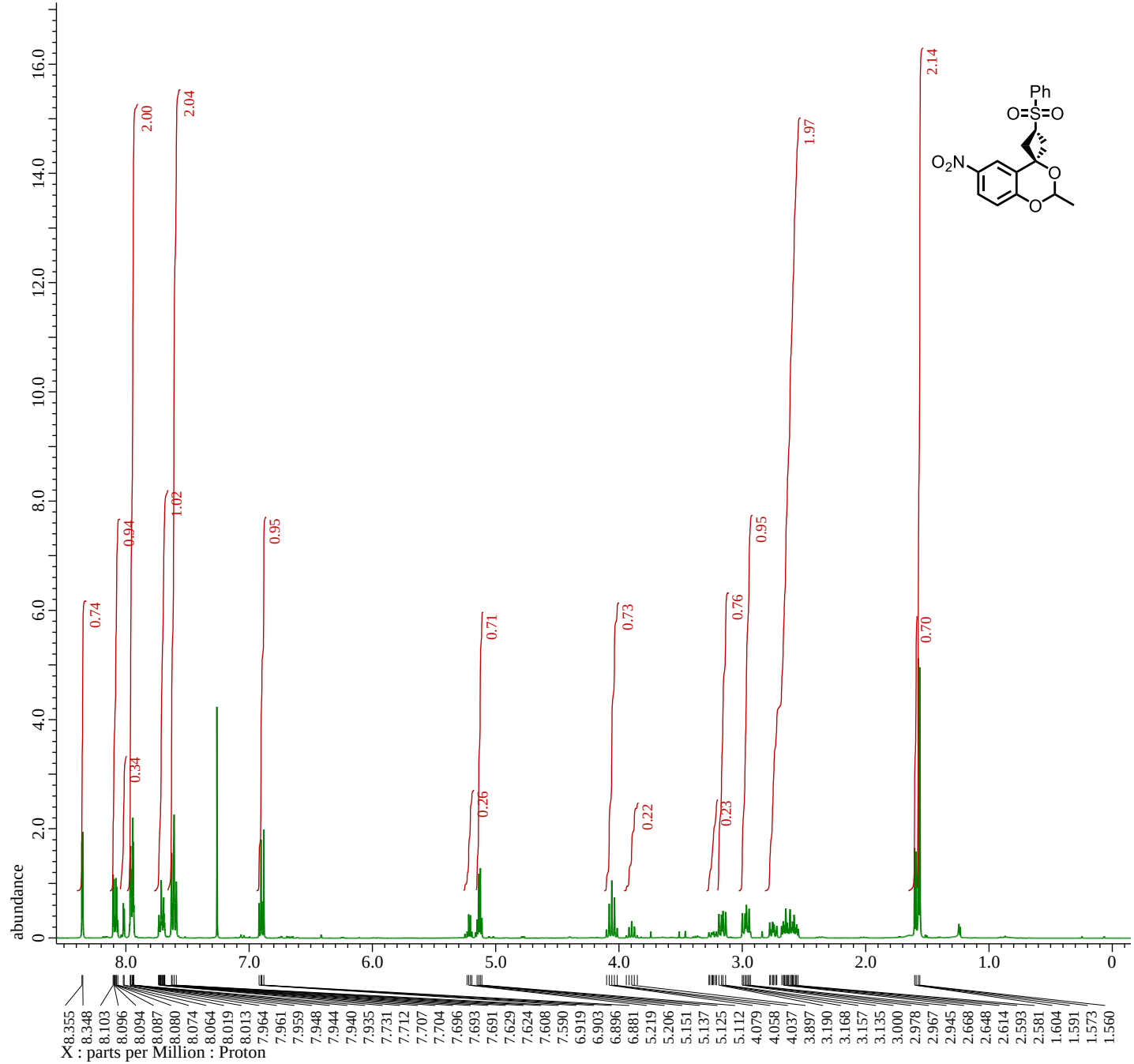
^{13}C NMR spectrum of **2d**/**2d'** (101 MHz, $CDCl_3$)




```
---- 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]
```

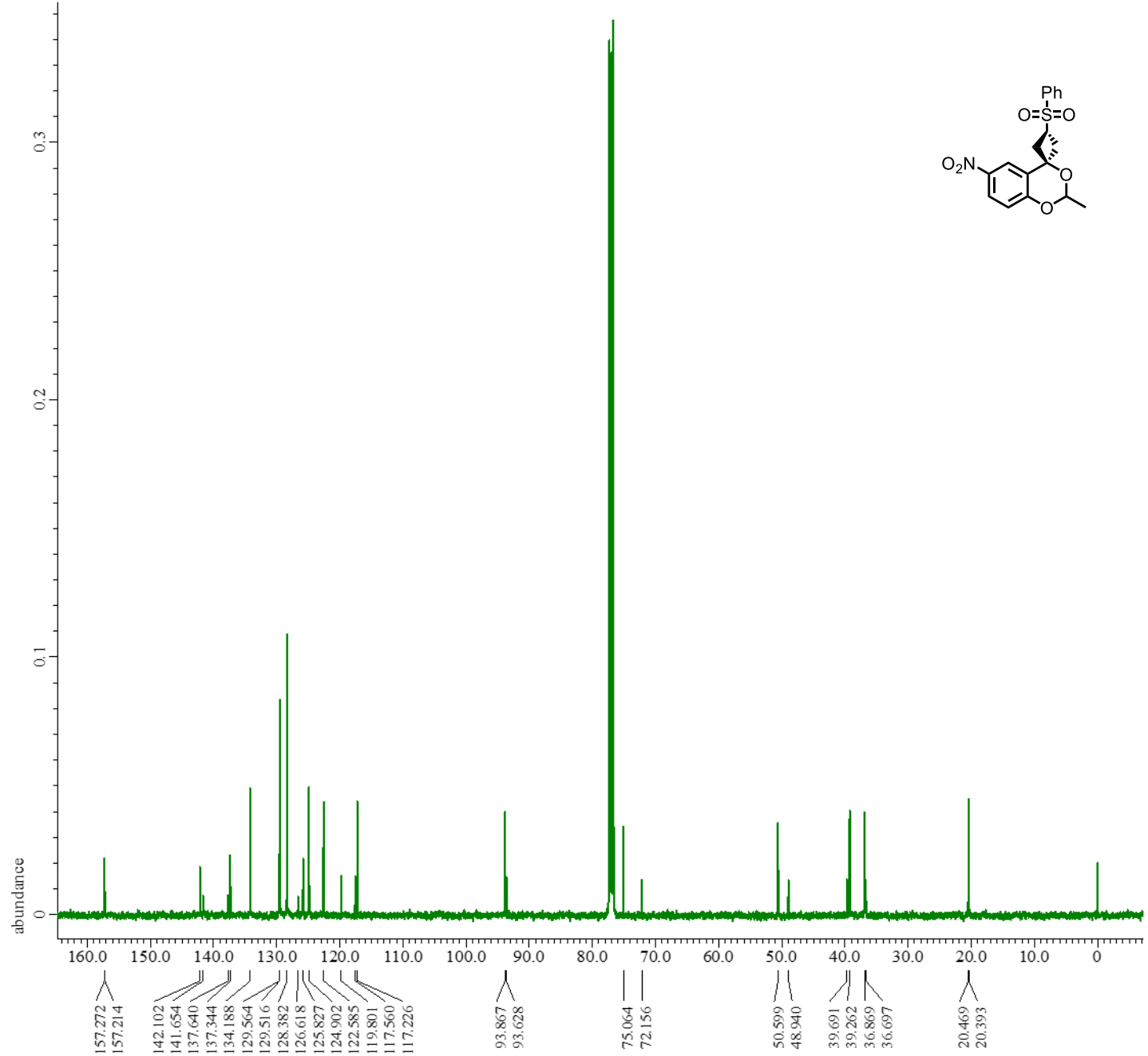
^{19}F NMR spectrum of **2d**/**2d'** (283 MHz, $CDCl_3$)




```
---- 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	= 15-DEC-2023 10:25:04
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]

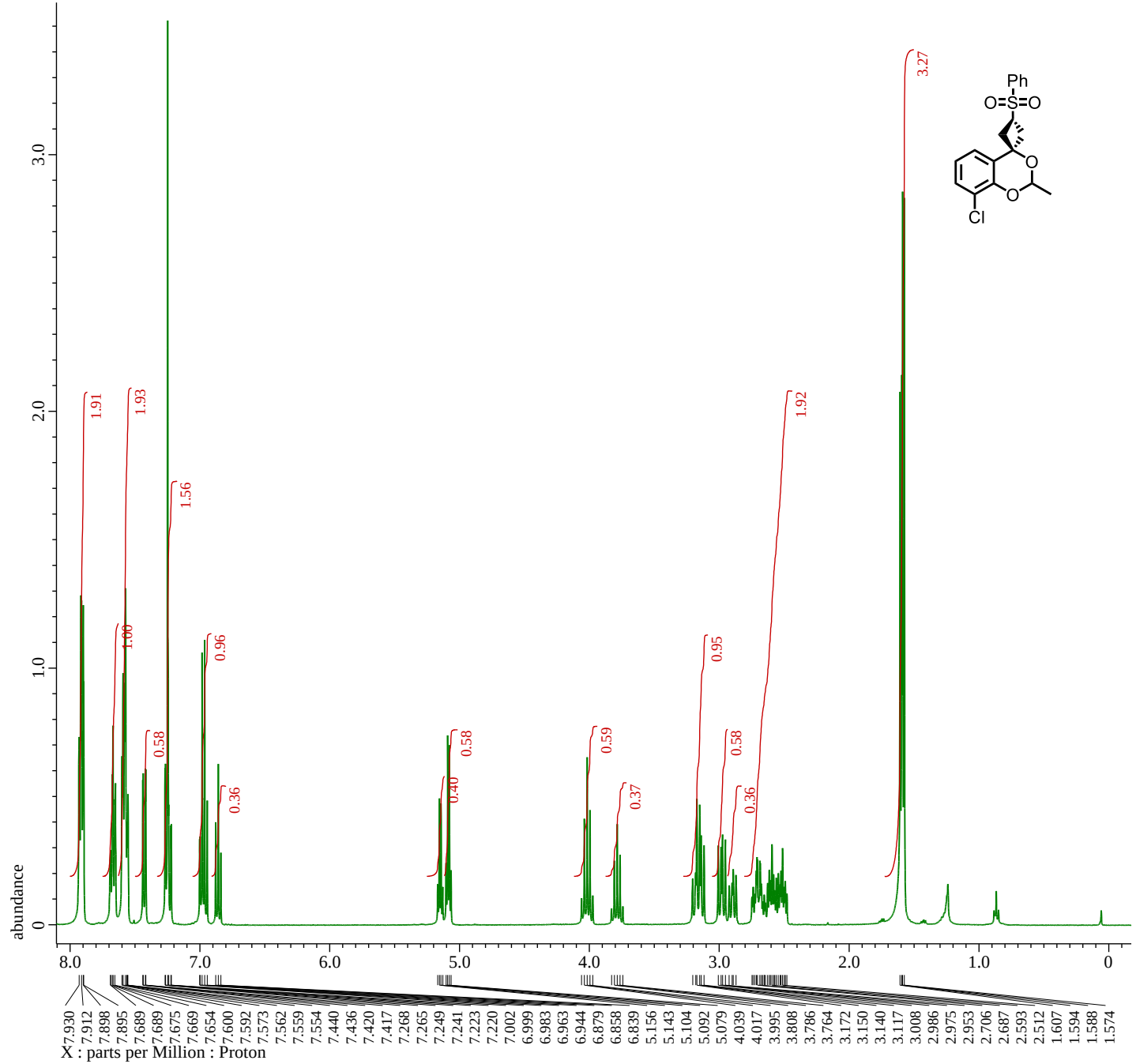
^{1}H NMR spectrum of **2e**/**2e'** (400 MHz, $CDCl_3$)




```
---- 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]
```

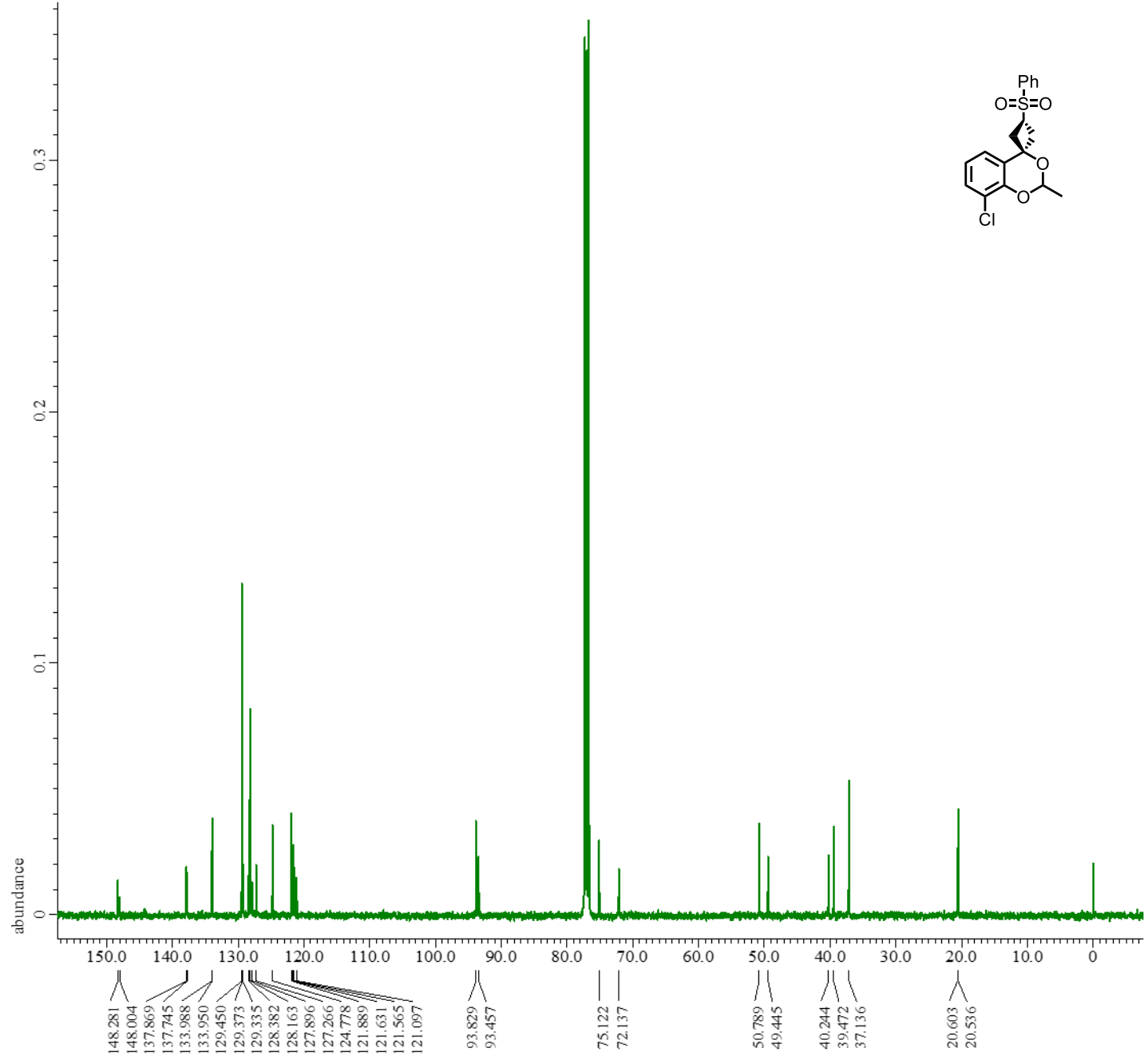
^{13}C NMR spectrum of **2e**/**2e'** (101 MHz, $CDCl_3$)




```
---- 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	= 15-DEC-2023 10:47:15
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	= 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]

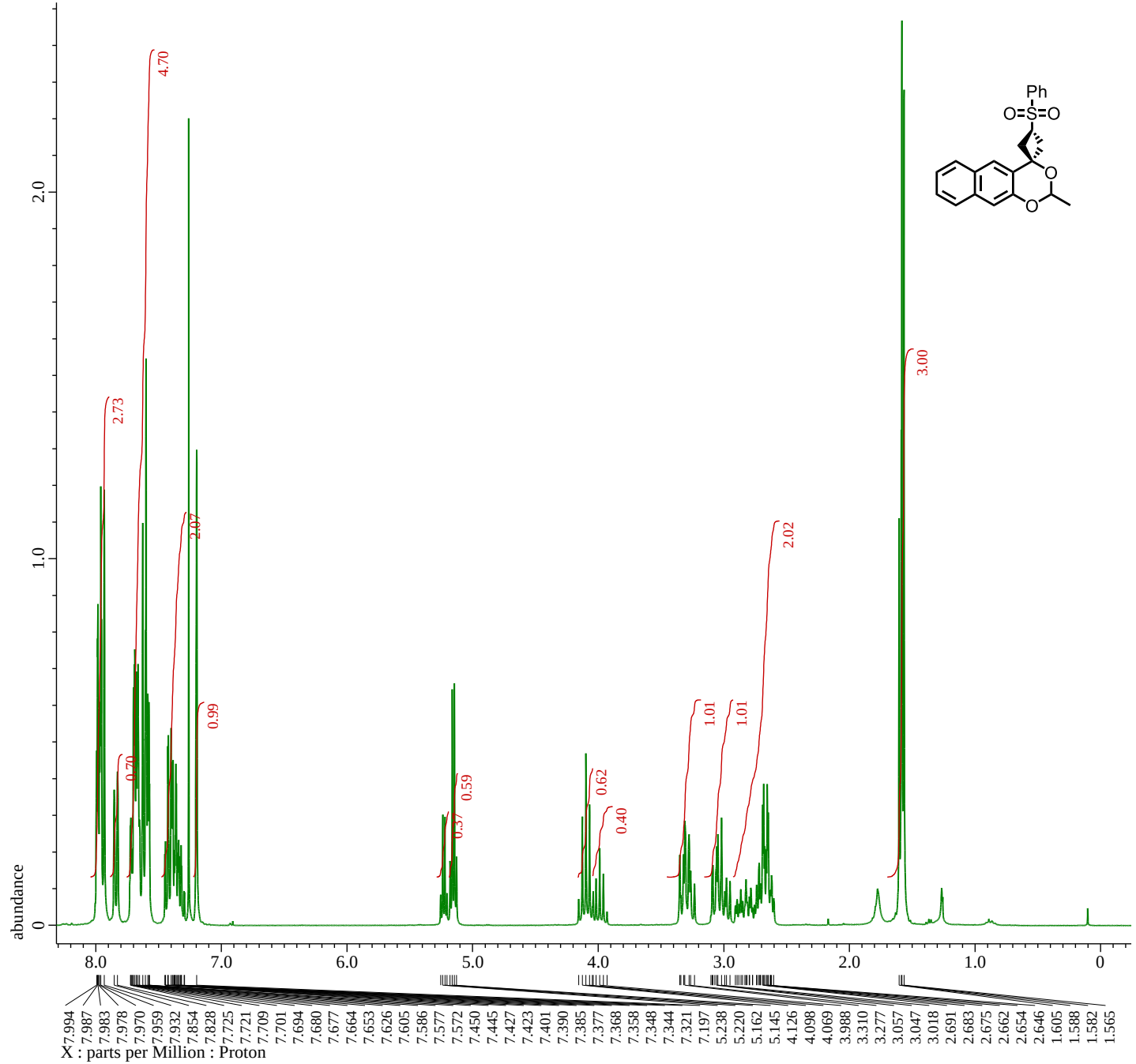
^{1}H NMR spectrum of **2f/2f'** (400 MHz, $CDCl_3$)




```
---- 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, $CDCl_3$)

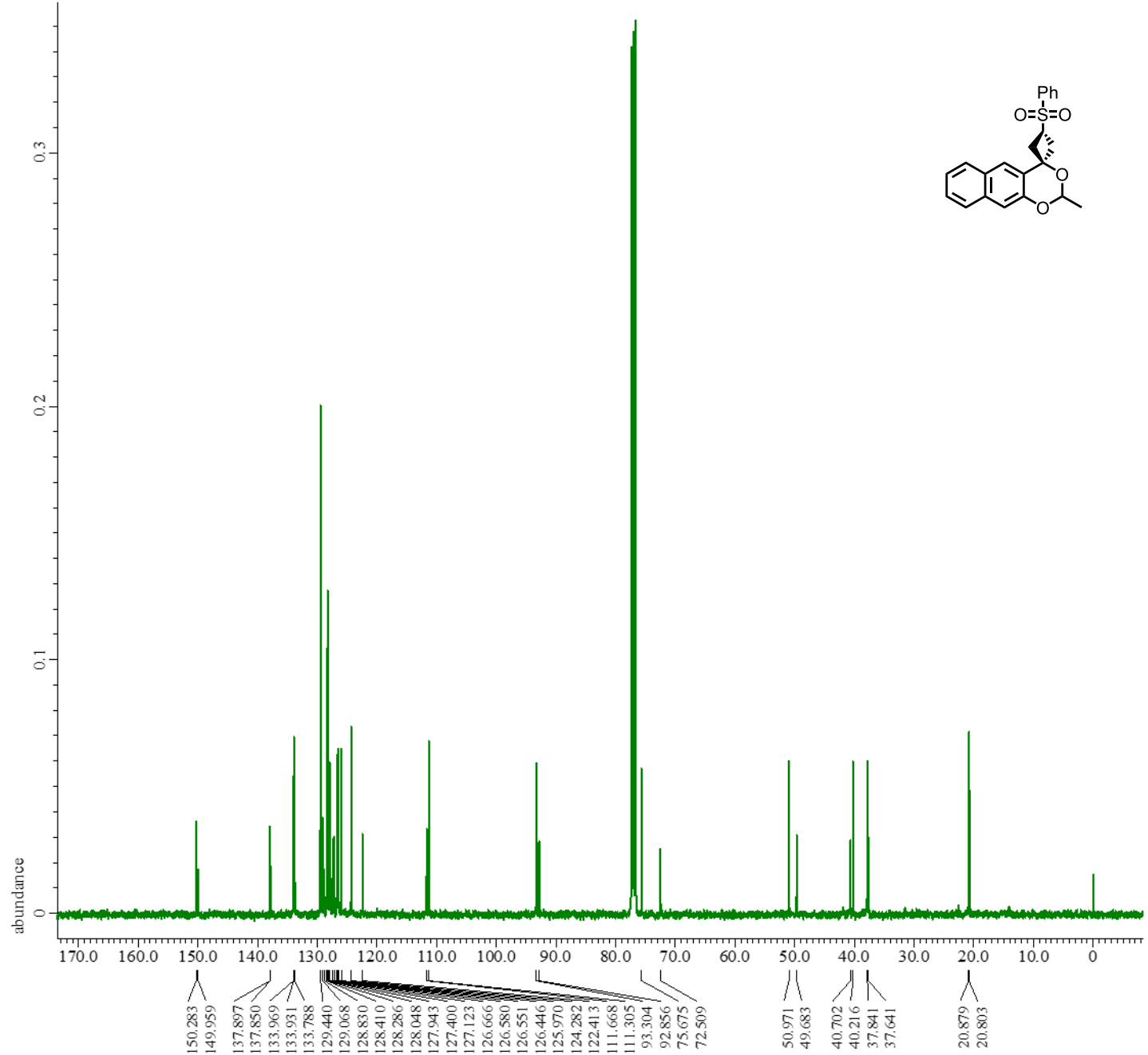



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= 16 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	= 15-DEC-2023 10:57:03
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

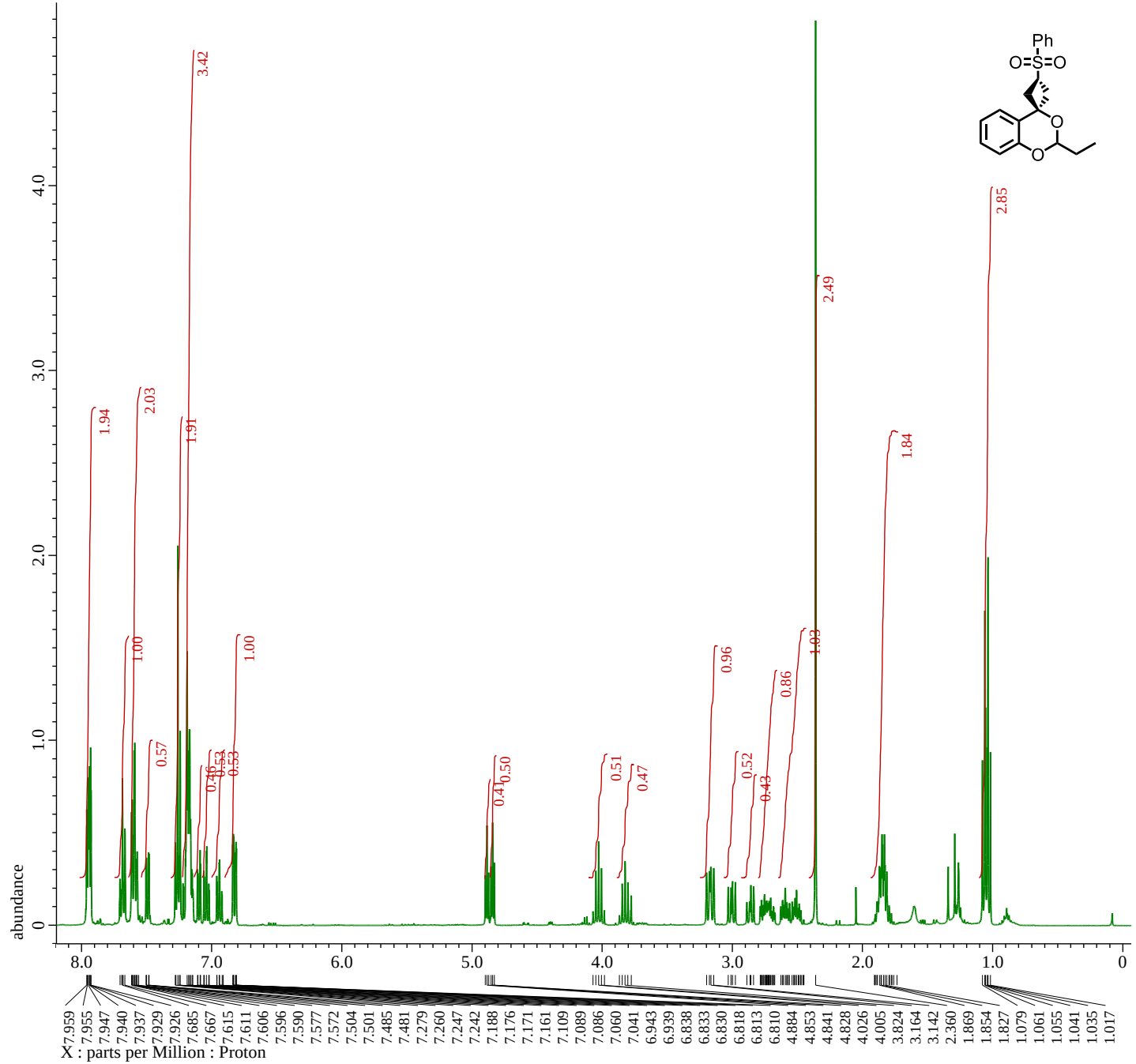
^{1}H NMR spectrum of **2g/2g'** (300 MHz, $CDCl_3$)




```
---- 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
```

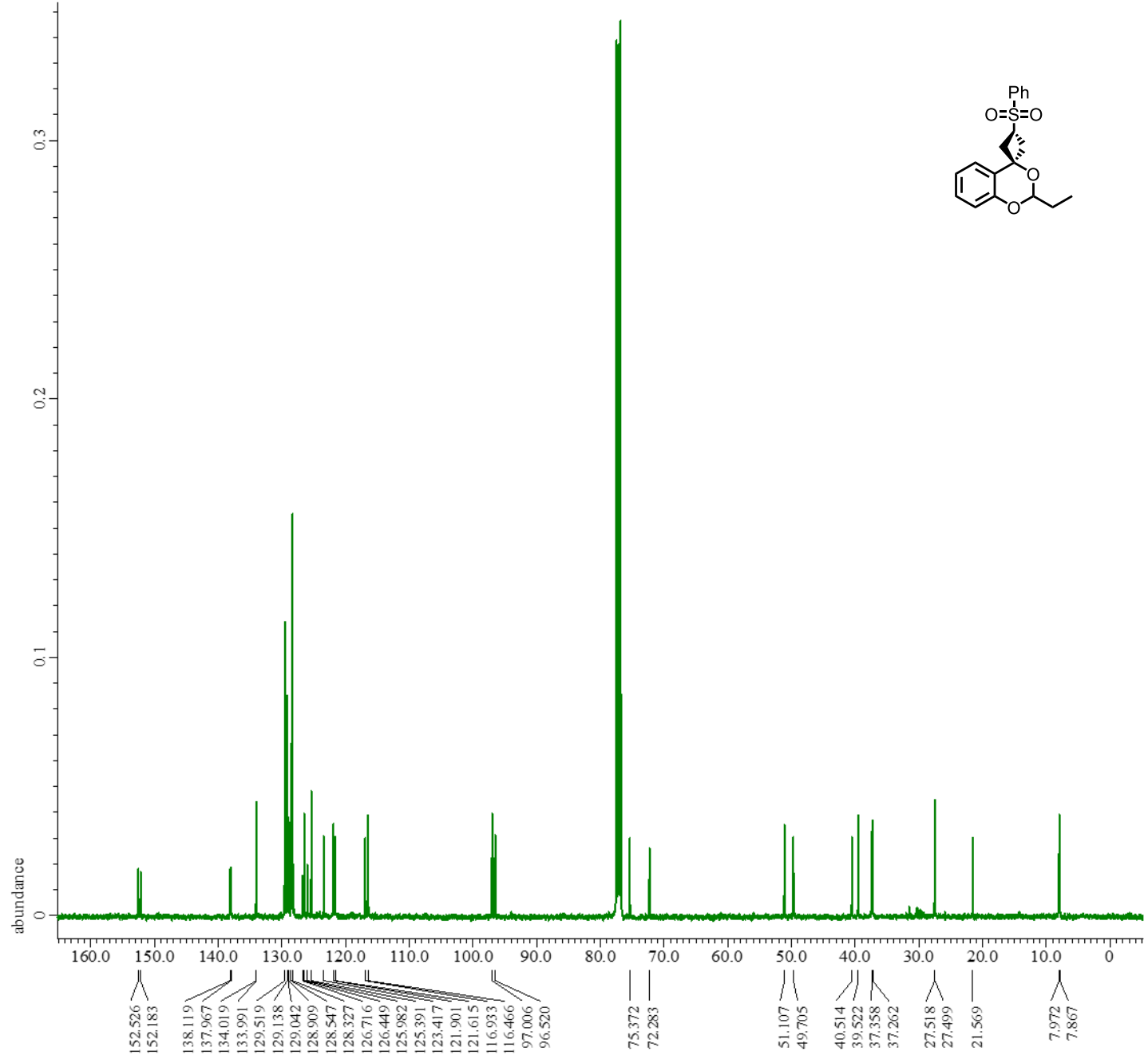
```
Filename            = 16 Ar 2,3-naphthyl CDCl3_
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = 210552 3 CDCl3
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-NOV-2022 20:52:23
Revision_Time       = 16-JAN-2023 20:33:14

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, $CDCl_3$)




```
---- 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-1-3.
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 210680 check CDCl3
Solvent           = CHLOROFORM-D
Actual_Start_Time = 11-FEB-2023 00:06:06
Revision_Time     = 15-DEC-2023 11:11:20

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]
```

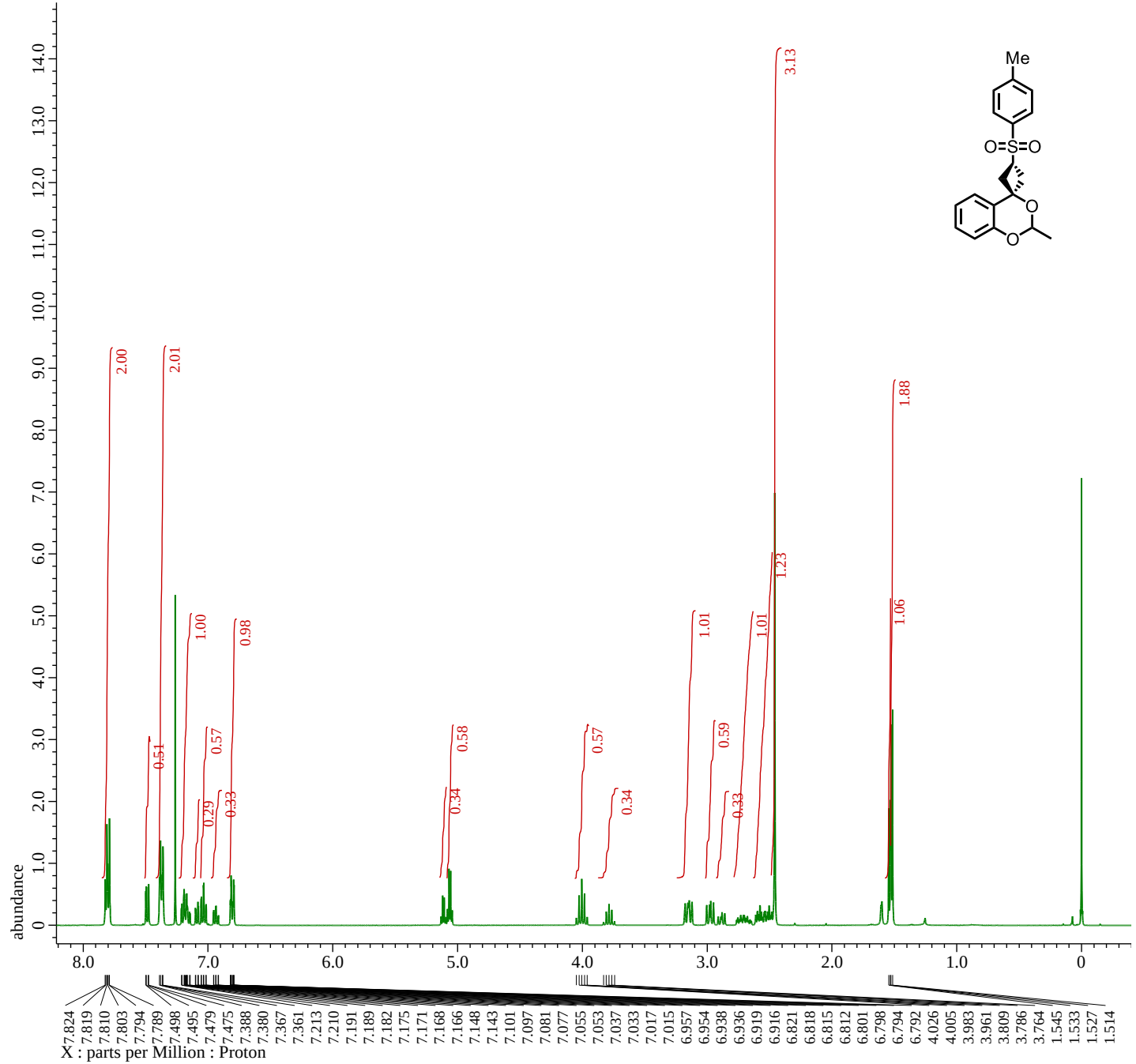
^{1}H NMR spectrum of **2h**/**2h'** (400 MHz, $CDCl_3$)




```
---- 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, $CDCl_3$)

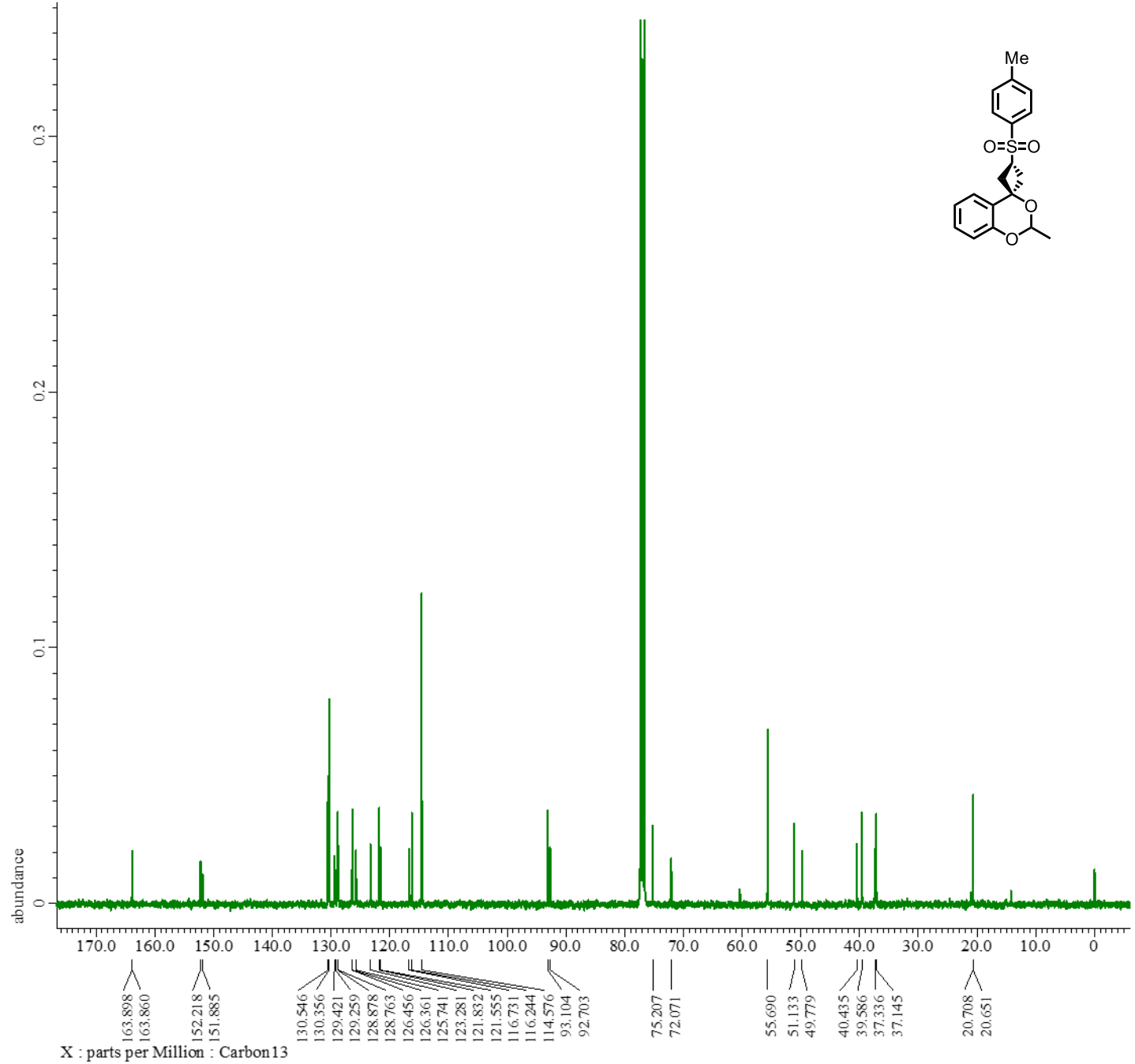



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
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     = 15-DEC-2023 12:23:35

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          = 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]
```

7.824 7.819 7.810 7.803 7.794 7.789 7.498 7.495 7.479 7.475 7.388 7.380 7.367 7.361 7.213 7.210 7.191 7.189 7.182 7.175 7.171 7.168 7.166 7.148 7.143 7.101 7.097 7.081 7.077 7.055 7.053 7.037 7.033 7.017 7.015 6.957 6.954 6.938 6.936 6.919 6.916 6.821 6.818 6.815 6.812 6.801 6.798 6.794 6.792 4.026 4.005 3.983 3.961 3.809 3.786 3.764 1.545 1.533 1.527 1.514

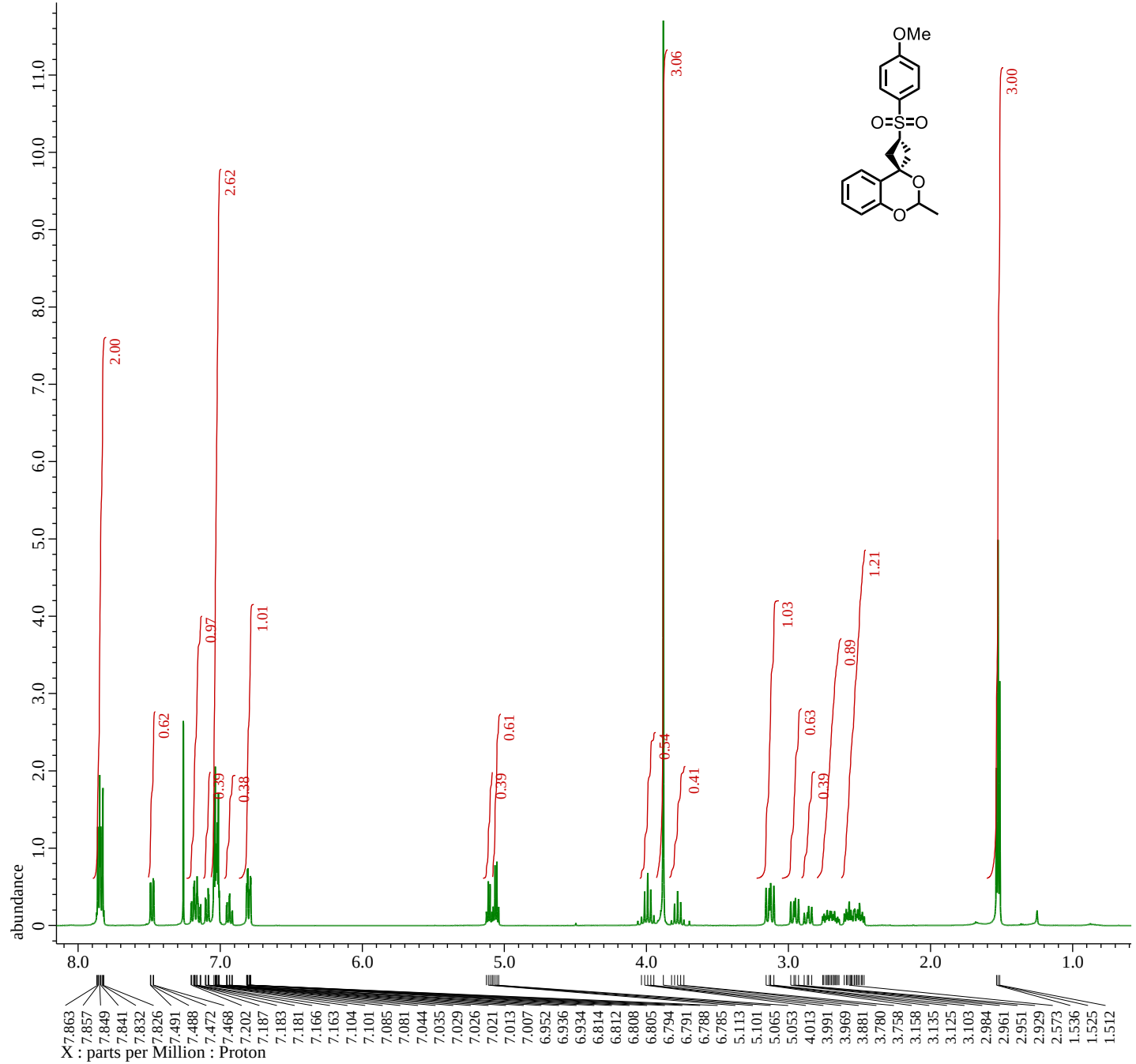
[1] H NMR spectrum of **2i/2i'** (400 MHz, $CDCl_3$)




```
---- 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, $CDCl_3$)

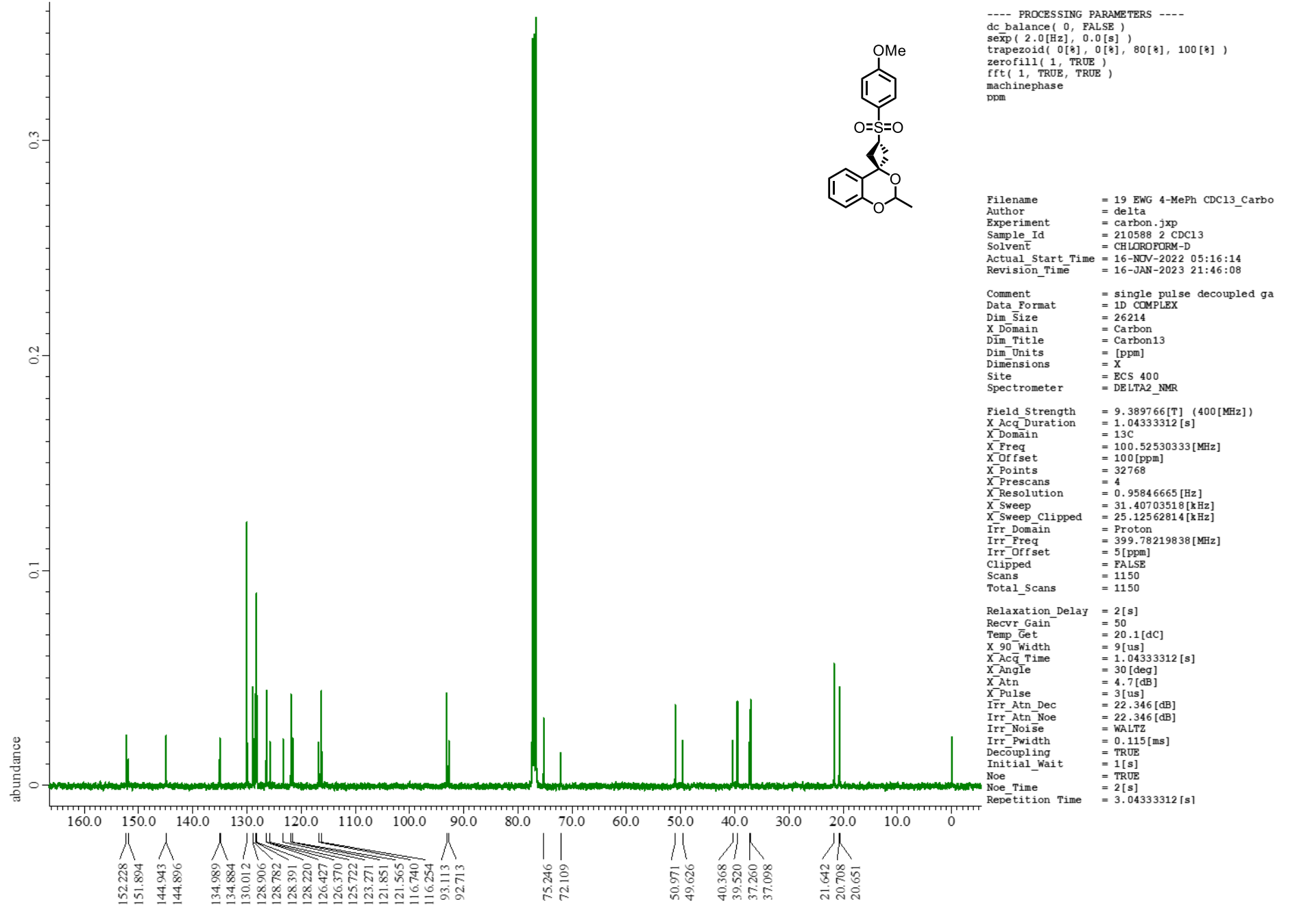



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 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       = 15-DEC-2023 14:47:08

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          = 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]
```

7.863 7.857 7.849 7.841 7.832 7.826 7.491 7.488 7.472 7.468 7.202 7.187 7.183 7.181 7.166 7.163 7.104 7.101 7.085 7.081 7.044 7.035 7.029 7.026 7.021 7.013 7.007 6.952 6.936 6.934 6.814 6.812 6.808 6.805 6.794 6.791 6.788 6.785 5.113 5.101 5.065 5.053 4.013 3.991 3.969 3.881 3.780 3.758 3.158 3.135 3.125 3.103 2.984 2.961 2.951 2.929 2.573 1.536 1.525 1.512

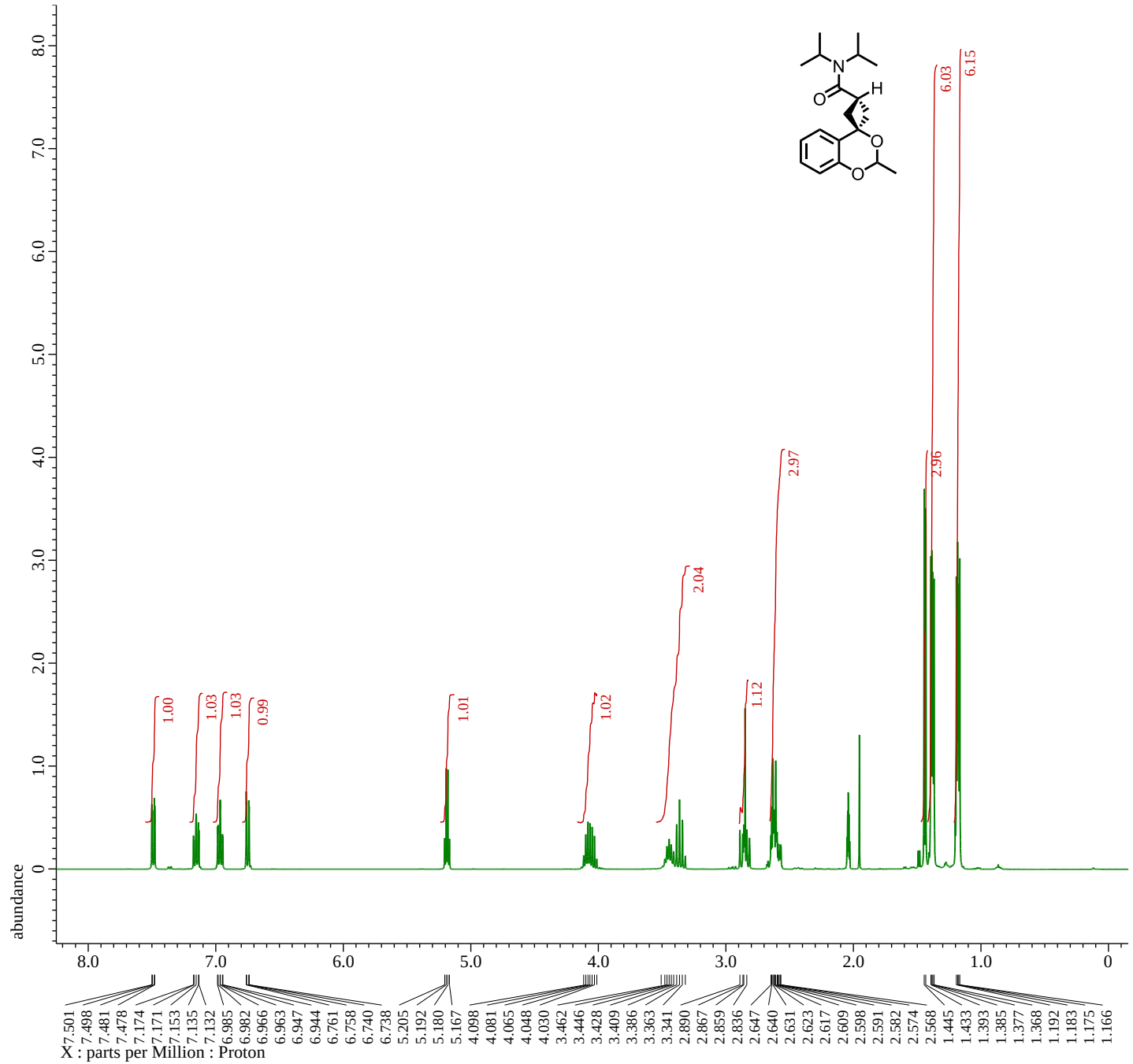
[1] H NMR spectrum of **2j**/**2j'** (400 MHz, $CDCl_3$)




```
---- 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]
```

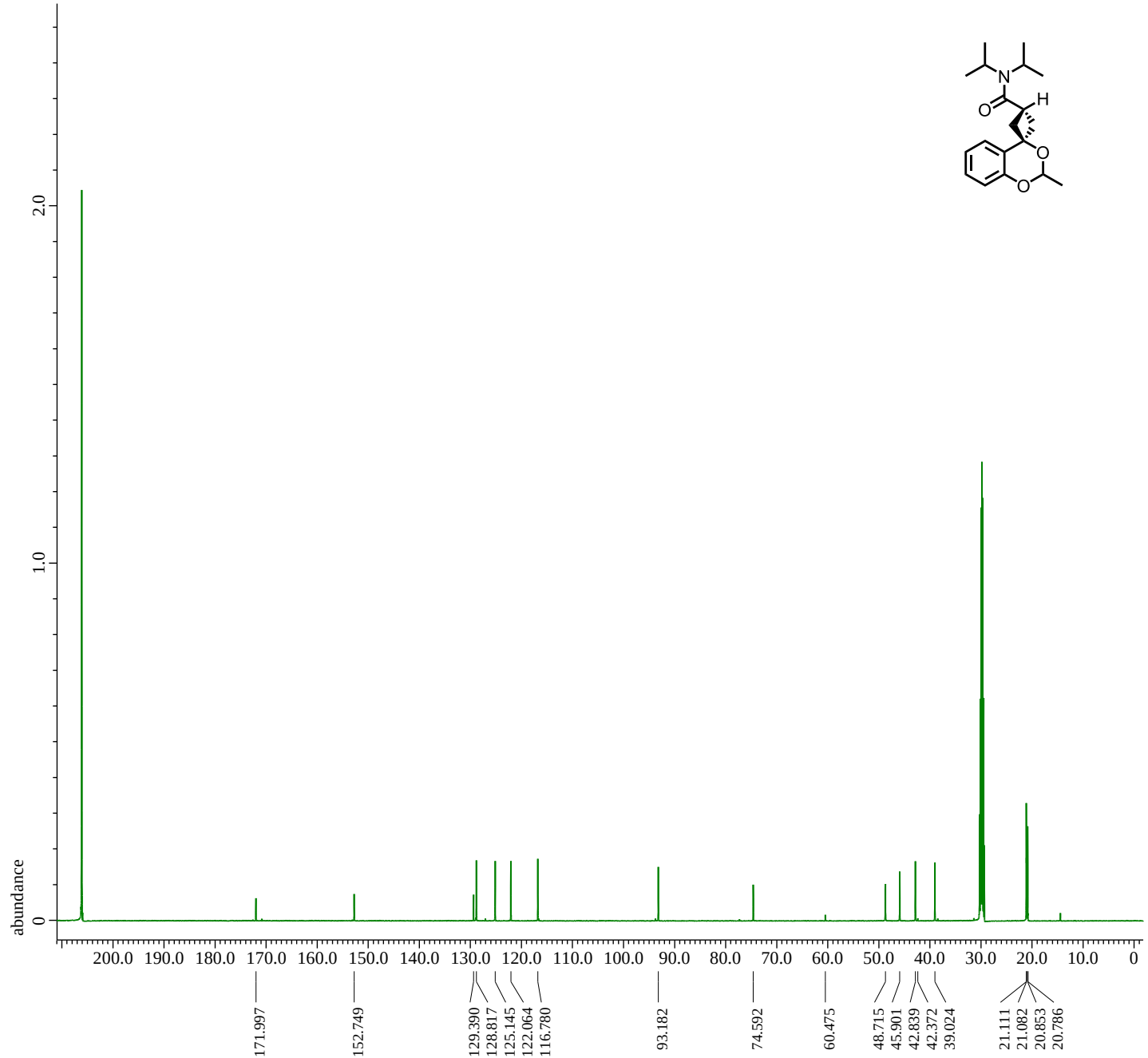
^{13}C NMR spectrum of **2j/2j'** (101 MHz, $CDCl_3$)




```
---- 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            = 210688-690 column shita A
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 210688-690 column shita A
Solvent             = ACETONE-D6
Actual_Start_Time   =  1-FEB-2023 11:13:23
Revision_Time       = 13-DEC-2023 16:43:51

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               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
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]
```

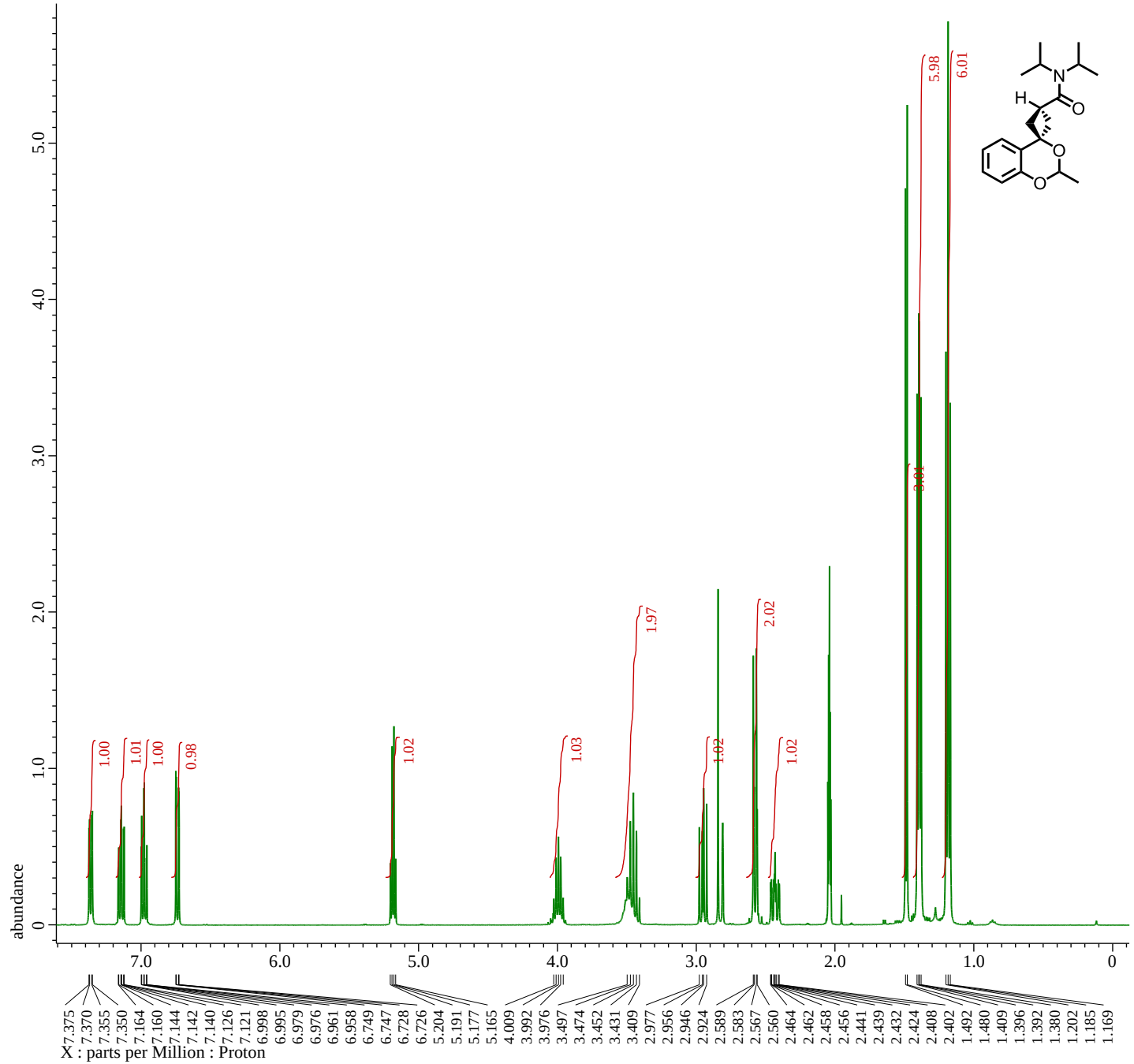
[1] H NMR spectrum of **2k** (400 MHz, Acetone-d_6)




```
---- 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	= 210688-690 column shita A
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 210688-690 column shita A
Solvent	= ACETONE-D6
Actual_Start_Time	= 3-FEB-2023 02:17:34
Revision_Time	= 13-DEC-2023 15:49:44
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= 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	= 3259
Total_Scans	= 3259
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.2[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]

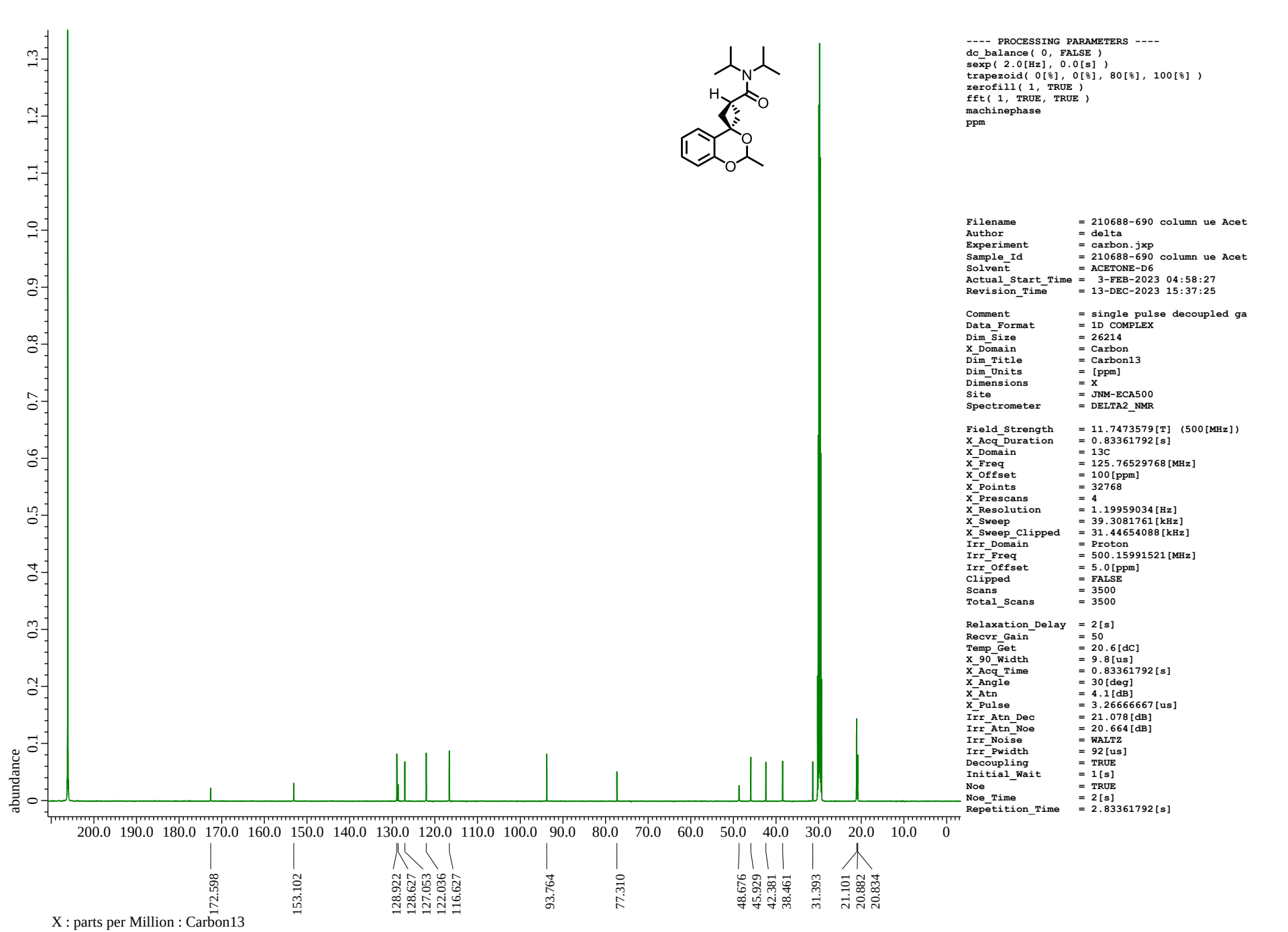
^{13}C NMR spectrum of **2k** (126 MHz, Acetone-d_6)




```
---- 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	= 210688-690 column ue Acet
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 210688-690 column ue Acet
Solvent	= ACETONE-D6
Actual_Start_Time	= 1-FEB-2023 11:07:17
Revision_Time	= 13-DEC-2023 16:32:51
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	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 38
Temp_Get	= 20.2[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]

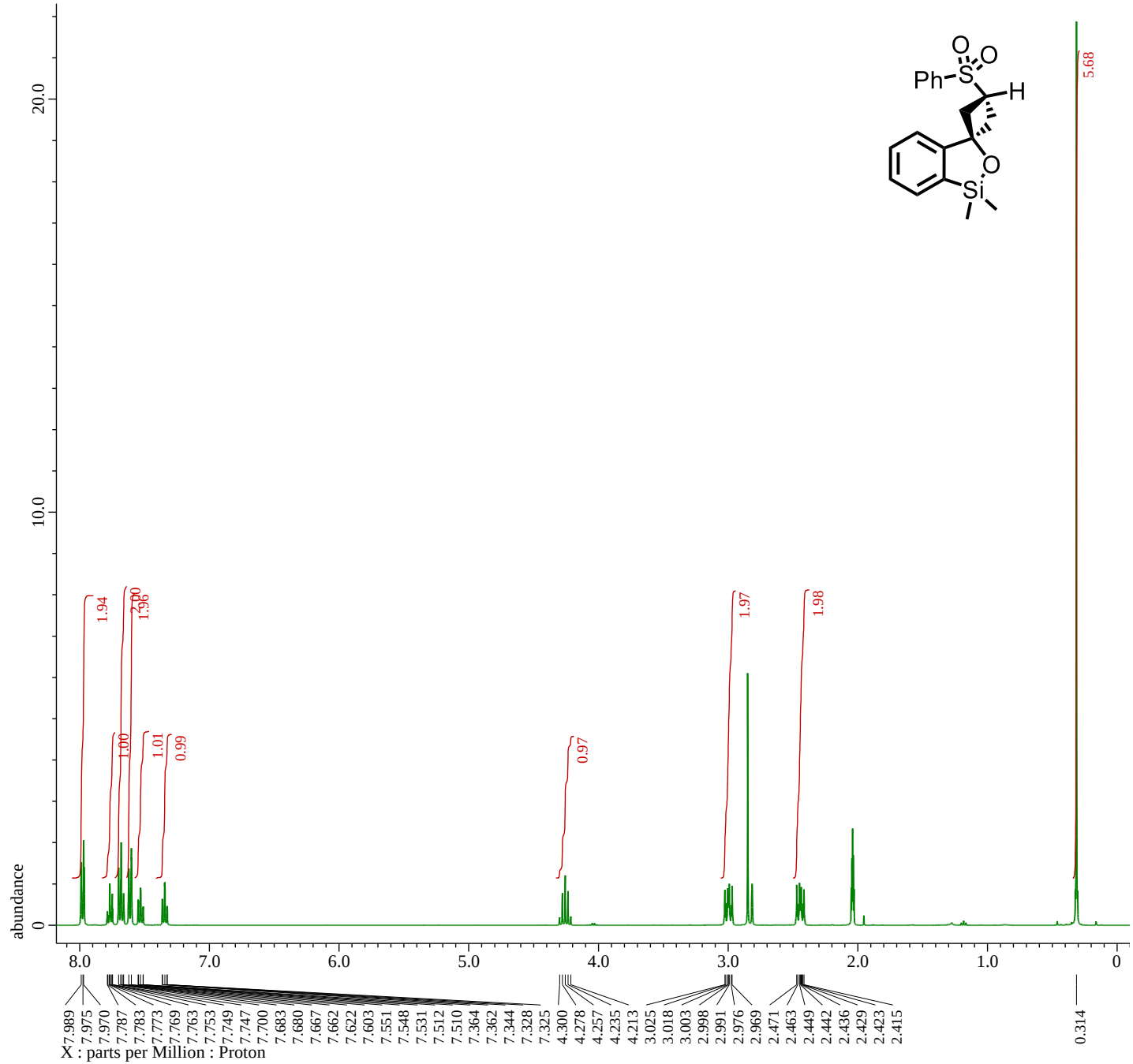
^{1}H NMR spectrum of **2k'** (400 MHz, Acetone-d_6)




```
---- 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           = 210688-690 column ue Acet
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = 210688-690 column ue Acet
Solvent            = ACETONE-D6
Actual_Start_Time  =  3-FEB-2023 04:58:27
Revision_Time      = 13-DEC-2023 15:37:25

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            = FALSE
Scans              = 3500
Total_Scans        = 3500

Relaxation_Delay   = 2[s]
Recvr_Gain         = 50
Temp_Get           = 20.6[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]
```

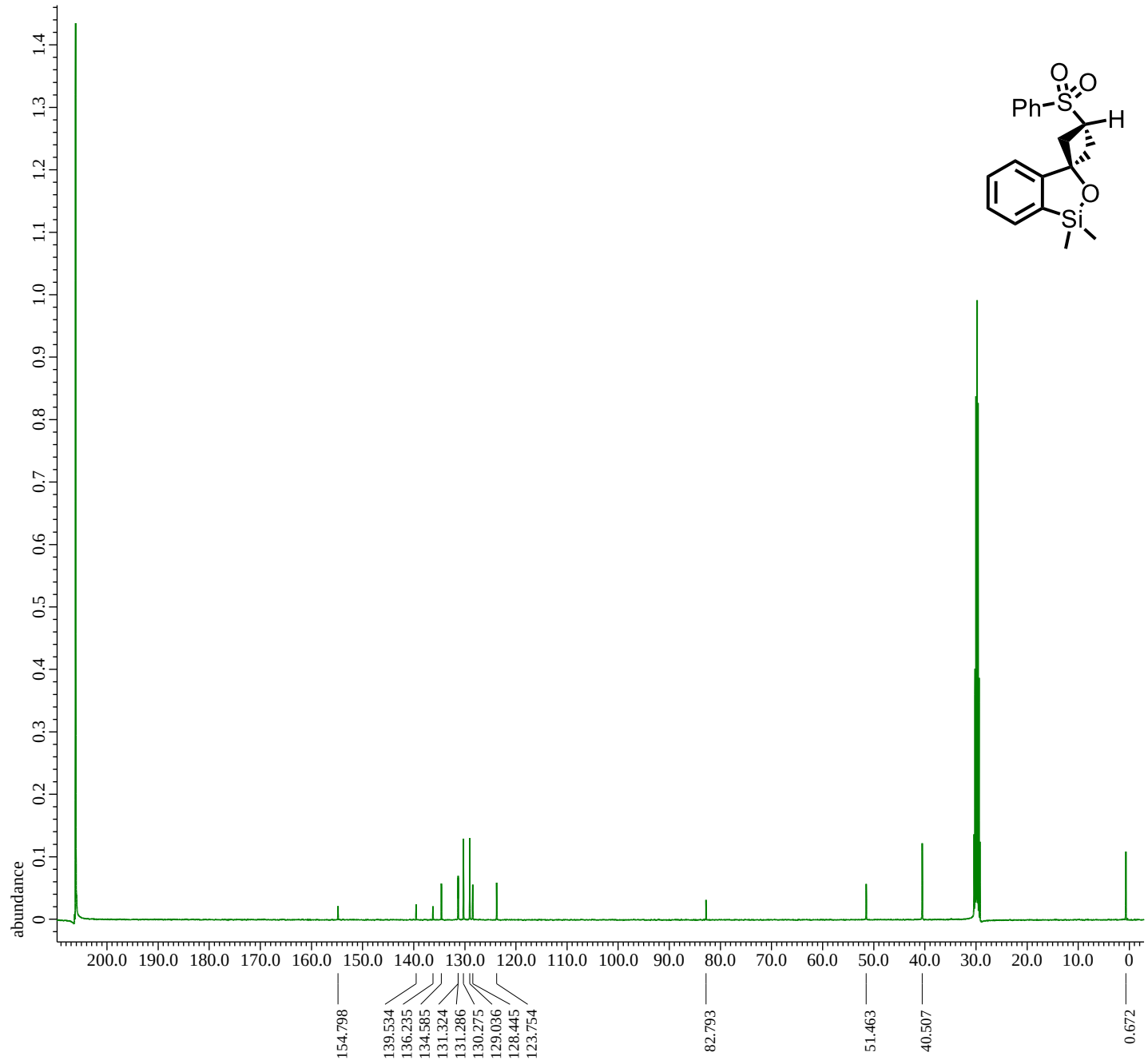
^{13}C NMR spectrum of **2k'** (126 MHz, Acetone-d_6)




```
---- 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           = 210699-700 column Acetone
Author             = delta
Experiment         = proton.jxp
Sample_Id          = 210699-700 column Acetone
Solvent            = ACETONE-D6
Actual_Start_Time  = 11-FEB-2023 06:03:17
Revision_Time      = 12-FEB-2023 11:51:55

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         = 40
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 **4** (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	= 210699-700 column Acetone
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 210699-700 column Acetone
Solvent	= ACETONE-D6
Actual_Start_Time	= 11-FEB-2023 06:06:48
Revision_Time	= 13-DEC-2023 15:09:04
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	= 20.5[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 **4** (101 MHz, Acetone-d_6)