

Supporting Information

**Benzannulation of Isobenzopyryliums with Electron-Rich Alkynes:
A Modular Access to β -Functionalized Naphthalenes**

An Wu, Hui Qian, Wanxiang Zhao, and Jianwei Sun*

*Department of Chemistry, the Hong Kong University of Science and Technology,
Clear Water Bay, Kowloon, Hong Kong SAR, China;
HKUST-Shenzhen Research Institute, No. 9 Yuexing 1st Rd, South Area,
Hi-tech Park, Nanshan, Shenzhen 518057, China*

Table of Contents

I.	General Information	S-2
II.	Substrate Preparation	S-3
III.	Synthesis of β -Naphthols from Siloxy Alkynes	S-12
IV.	Synthesis of β -Naphthyl Thioethers from Thioalkynes.....	S-29
V.	Synthesis of β -Naphthylamines from Ynamides	S-34
VI.	Mechanistic Study	S-37
VII.	Product Structure Determination	S-38

^1H NMR and ^{13}C NMR Spectra

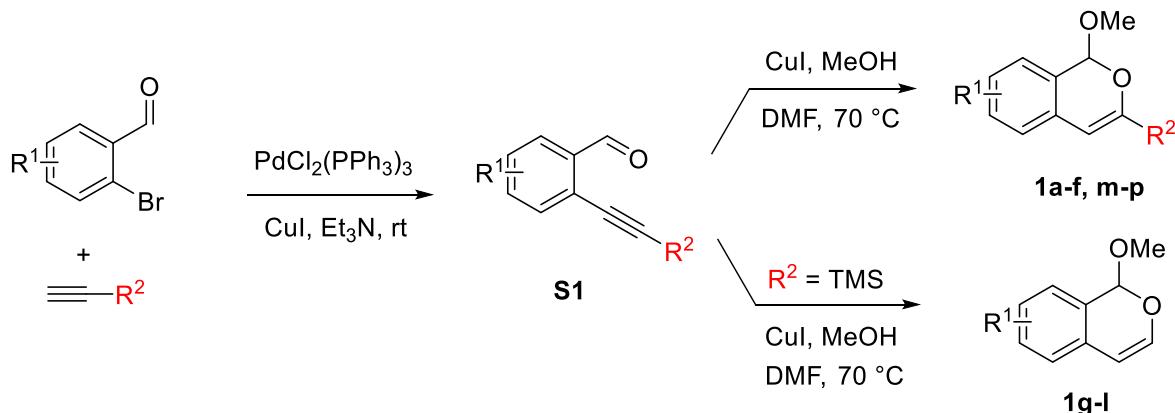
I. General Information

All air or moisture sensitive reactions were conducted in oven-dried glassware under nitrogen atmosphere using dry solvents. Flash column chromatography was performed over silica gel (230-400 mesh) purchased from Qindao Puke Co., China. Anhydrous *N,N*-dimethylformamide and anhydrous acetonitrile were purified by the Innovative® solvent purification system. Anhydrous dichloromethane was distilled with calcium hydride. Toluene and 1, 2-dichloroethane were purchased from Sigma-Aldrich and used as received. Triethylamine was purchased from Acros and used as received. ¹H and ¹³C NMR spectra were collected on a Bruker AV 400 MHz NMR spectrometer using residue solvent peaks as an internal standard (¹H NMR: CDCl₃ at 7.26 ppm, C₆D₆ at 7.16 ppm, CD₂Cl₂ at 5.32 ppm, ¹³C NMR: CDCl₃ at 77.0 ppm, C₆D₆ at 128.06 ppm, CD₂Cl₂ at 53.84 ppm). Mass spectra were collected on an Agilent GC/MS 5975C system, or a MALDI Micro MX mass spectrometer, or an API QSTAR XL System.

II. Substrate Preparation

Alkynes used this work are all known compounds. They were prepared according to the procedures in the literature and the characterization data match the reported data.¹

General Procedure A.



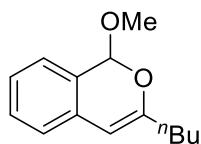
This procedure was modified from the literature.^{2,3} Under N₂, to a flame-dried flask charged with the 2-bromobenzaldehyde (10 mmol, 1.0 equiv), CuI (38.1 mg, 0.2 mmol, 0.02 equiv) and PdCl₂(PPh₃)₃ (140.4 mg, 0.2 mmol, 0.02 equiv) were sequentially added Et₃N (17 mL) and the terminal alkyne (20 mmol, 2.0 equiv). The reaction was stirred at room temperature and the progress was monitored by ¹H NMR of the reaction mixture. Upon completion, DCM (50 mL) was added. The reaction mixture was filtered through a short pad of celite, and the filtrate was concentrated. The residue was purified by silica gel column chromatography to afford 2-alkynylbenzaldehyde **S1**.

1 (a) M. P. Schramm, V. Shubinets, S. A. Kozmin, *Org. Synth.*, 2010, **87**, 253-262; (b) W. Zhao, Z. Wang, J. Sun, *Angew. Chem., Int. Ed.*, 2012, **51**, 6209-6213; (c) Y. Wang, L.-J. Song, X. Zhang, J. Sun, *Angew. Chem., Int. Ed.*, 2016, **55**, 9704-9708; (d) T. Hamada, X. Ye, S. S. Stahl, *J. Am. Chem. Soc.*, 2008, **130**, 833-835; (e) S. Ding, G. Jia, J. Sun, *Angew. Chem., Int. Ed.*, 2014, **53**, 1877-1880.

2 Y. Luan, K. S. Barbato, P. N. Moquist, T. Kodama, S. E. Schaus, *J. Am. Chem. Soc.*, 2015, **137**, 3233-3236.

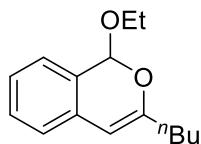
3 N.-T. Patil, Y. Yamamoto, *J. Org. Chem.*, 2004, **69**, 5139-5142.

Under N₂, to a flame-dried flask charged with **S1** (5 mmol, 1.0 equiv) and CuI (95.2 mg, 0.5 mmol, 0.1 equiv) were added DMF (2 mL) and MeOH (0.4 mL, 10 mmol, 2.0 equiv) *via* syringe. The reaction was heated to 70 °C, and the progress was monitored by ¹H NMR of the reaction mixture. Upon completion, water (10 mL) and EtOAc (10 mL) were added. The organic layer was separated, and the aqueous layer was extracted with EtOAc (10 mL×3). The combined organic layers were washed with water (10 mL×3), and dried over Na₂SO₄, filtered, and concentrated. The crude product was purified by silica gel (washed with Et₃N) column chromatography to afford the substrate **1**.



1a

3-Butyl-1-methoxy-1*H*-isochromene (1a) was prepared as an oil according to the General Procedure A. It is a known compound. The spectra data are consistent with the literature.³



1a'

3-Butyl-1-ethoxy-1*H*-isochromene (1a') was prepared as a yellow oil according to the General Procedure A, in which EtOH was used in place of MeOH.

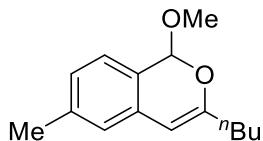
¹H NMR (400 MHz, C₆D₆) δ 7.15 - 7.08 (m, 1H), 7.08 - 6.97 (m, 2H), 6.96 - 6.89 (m, 1H), 5.97 (s, 1H), 5.67 (s, 1H), 3.92 - 3.80 (m, 1H), 3.54 - 3.41 (m, 1H), 2.33 - 2.13 (m, 2H), 1.65 - 1.50 (m, 2H), 1.39 - 1.23 (m, 2H), 1.06 (t, *J* = 7.1 Hz, 3H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 154.5, 129.2, 127.9, 127.5, 126.4, 125.9, 123.6, 100.7, 99.0,

63.1, 34.1, 29.5, 22.4, 15.4, 14.1.

IR (thin film) 3070, 3028, 2957, 2930, 2870, 1657, 1077 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₂₀O₂ (M⁺): 232.1463, Found: 232.1469.



1b

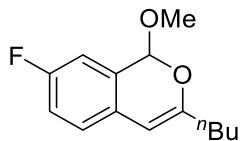
3-Butyl-1-methoxy-6-methyl-1H-isochromene (1b) was prepared as a yellow oil according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 6.99 (d, *J* = 7.7 Hz, 1H), 6.88 - 6.83 (m, 1H), 6.74 (s, 1H), 5.89 (s, 1H), 5.68 (s, 1H), 3.31 (s, 3H), 2.32 - 2.17 (m, 2H), 2.11 (s, 3H), 1.66 - 1.53 (m, 2H), 1.37 - 1.25 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 154.5, 138.8, 131.3, 126.7, 126.4, 124.7, 124.2, 100.7, 100.3, 54.5, 34.1, 29.6, 22.5, 21.4, 14.1.

IR (thin film) 3013, 2956, 2828, 1657, 1078 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₂₀O₂ (M⁺): 232.1463, Found: 232.2467.



1c

3-Butyl-7-fluoro-1-methoxy-1H-isochromene (1c) was prepared as a yellow oil according to the General Procedure A.

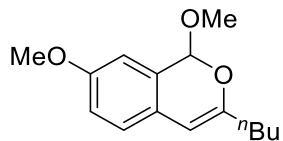
¹H NMR (400 MHz, C₆D₆) δ 6.82 - 6.74 (m, 1H), 6.74 - 6.68 (m, 1H), 6.68 - 6.60 (m, 1H), 5.62 (s, 1H), 5.54 (s, 1H), 3.22 (s, 3H), 2.26 - 2.08 (m, 2H), 1.61 - 1.47 (m, 2H), 1.36 - 1.21 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 161.4 (d, *J* = 243.9 Hz), 153.7 (d, *J* = 2.5 Hz), 128.7 (d, *J* = 7.1 Hz), 127.5 (d, *J* = 2.9 Hz), 125.1 (d, *J* = 7.6 Hz), 116.3 (d, *J* = 21.7 Hz), 113.3 (d, *J* = 22.5

Hz), 99.8, 99.5 (d, J = 2.4 Hz), 54.7, 33.9, 29.5, 22.4, 14.1.

IR (thin film) 3069, 2958, 2932, 2870, 2831, 1660, 1500 cm⁻¹.

HRMS (CI) Calcd for C₁₄H₁₇FO₂ (M⁺): 236.1213, Found: 236.1203.



1d

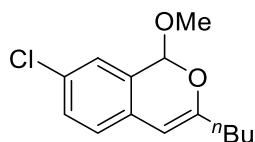
3-Butyl-1,7-dimethoxy-1H-isochromene (1d) was prepared as a yellow oil according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 6.87 - 6.85 (m, 1H), 6.83 - 6.76 (m, 1H), 6.76 - 6.71 (m, 1H), 5.84 (s, 1H), 5.67 (s, 1H), 3.32 (s, 3H), 3.29 (s, 3H), 2.33 - 2.16 (m, 2H), 1.67 - 1.53 (m, 2H), 1.40 - 1.25 (m, 2H), 0.88 (t, J = 7.4 Hz, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 158.6, 152.3, 128.5, 124.8, 124.4, 115.7, 111.7, 100.3, 100.2, 54.9, 54.6, 33.9, 29.6, 22.5, 14.1.

IR (thin film) 3067, 2996, 2956, 2870, 2832, 1661, 1504 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₂₀O₃ (M⁺): 248.1412, Found: 248.1408.



1e

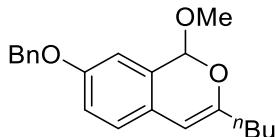
3-Butyl-7-chloro-1-methoxy-1H-isochromene (1e) was prepared as a yellow oil according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 7.09 - 7.03 (m, 1H), 7.01 (s, 1H), 6.58 (d, J = 8.2 Hz, 1H), 5.58 (s, 1H), 5.50 (s, 1H), 3.21 (s, 3H), 2.25 - 2.06 (m, 2H), 1.60 - 1.45 (m, 2H), 1.33 - 1.21 (m, 2H), 0.86 (t, J = 7.3 Hz, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 154.9, 131.0, 129.7, 129.4, 128.5, 126.6, 124.8, 99.8, 99.4, 54.8, 33.9, 29.4, 22.4, 14.1.

IR (thin film) 3072, 3031, 2957, 2868, 2831, 1656, 1487 cm⁻¹.

HRMS (CI) Calcd for C₁₄H₁₇ClO₂ (M⁺): 252.0917, Found: 252.0970.



1f

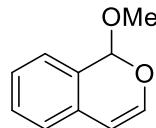
7-(Benzylxy)-3-butyl-1-methoxy-1H-isochromene (1f) was prepared as a colorless oil according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 7.27 - 7.22 (m, 2H), 7.19 - 7.13 (m, 2H), 7.11 - 7.07 (m, 1H), 6.88 - 6.84 (m, 2H), 6.83 - 6.80 (m, 1H), 5.85 (s, 1H), 5.67 (s, 1H), 4.66 (s, 2H), 3.32 (s, 3H), 2.35 - 2.13 (m, 2H), 1.67 - 1.52 (m, 2H), 1.39 - 1.24 (m, 2H), 0.89 (t, J = 7.4 Hz, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 157.8, 152.5, 137.7, 128.7, 128.5, 127.9, 127.7, 124.8, 124.7, 116.5, 112.8, 100.3, 100.2, 70.2, 54.6, 33.9, 29.6, 22.5, 14.1.

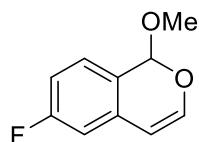
IR (thin film) 3064, 3033, 2956, 2868, 2829, 1660, 1502 cm⁻¹.

HRMS (CI) Calcd for C₂₁H₂₄O₃ (M⁺): 324.1725, Found: 324.1720.



1g

1-Methoxy-1H-isochromene (1g) was prepared as an oil according to the General Procedure A. It is a known compound. The spectra data are consistent with the literature.^[3]



1h

6-Fluoro-1-methoxy-1H-isochromene (1h) was prepared as a yellow solid according

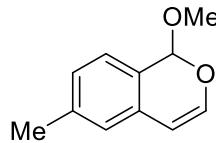
to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 6.76 - 6.68 (m, 1H), 6.68 - 6.59 (m, 1H), 6.53 - 6.45 (m, 1H), 6.36 (d, *J* = 5.7 Hz, 1H), 5.65 (s, 1H), 5.48 (d, *J* = 5.7 Hz, 1H), 3.19 (s, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 163.6 (d, *J* = 245.8 Hz), 143.3, 132.0 (d, *J* = 9.2 Hz), 128.5 (d, *J* = 8.9 Hz), 123.9 (d, *J* = 2.9 Hz), 113.5 (d, *J* = 22.3 Hz), 110.4 (d, *J* = 22.4 Hz), 104.1 (d, *J* = 2.4 Hz), 99.0, 54.9.

IR (thin film) 3076, 2997, 2957, 2932, 2908, 2831, 1638, 1026 cm⁻¹.

HRMS (CI) Calcd for C₁₀H₉FO₂ (M⁺): 180.0587, Found: 180.0585.



1i

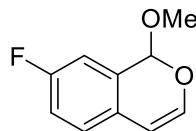
1-Methoxy-6-methyl-1*H*-isochromene (1i) was prepared as a yellow oil according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 6.94 (d, *J* = 7.7 Hz, 1H), 6.84 (d, *J* = 7.7 Hz, 1H), 6.65 (s, 1H), 6.48 (d, *J* = 5.7 Hz, 1H), 5.83 (s, 1H), 5.71 (d, *J* = 5.7 Hz, 1H), 3.27 (s, 3H), 2.06 (s, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 142.5, 138.9, 127.9, 127.6, 126.6, 125.6, 124.6, 104.8, 99.6, 54.9, 21.3.

IR (thin film) 3071, 3022, 2994, 2905, 2828, 1636, 1025 cm⁻¹.

HRMS (CI) Calcd for C₁₁H₁₂O₂ (M⁺): 176.0837, Found: 176.0839.



1j

7-Fluoro-1-methoxy-1*H*-isochromene (1j) was prepared as a yellow solid according to the General Procedure A.

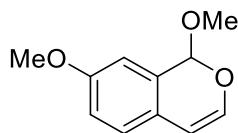
¹H NMR (400 MHz, C₆D₆) δ 6.78 - 6.60 (m, 2H), 6.60 - 6.49 (m, 1H), 6.35 (d, *J* = 5.7 Hz,

1H), 5.57 (d, J = 5.7 Hz, 1H), 5.55 (s, 1H), 3.18 (s, 3H).

^{13}C NMR (101 MHz, C_6D_6) δ 161.8 (d, J = 245.0 Hz), 141.6 (d, J = 2.5 Hz), 129.7 (d, J = 7.2 Hz), 126.0 (d, J = 3.0 Hz), 125.6 (d, J = 7.7 Hz), 116.3 (d, J = 21.8 Hz), 113.5 (d, J = 22.7 Hz), 104.0, 98.8 (d, J = 2.3 Hz), 55.1.

IR (thin film) 3085, 3054, 2981, 2932, 2832, 1640, 1500, 1025 cm^{-1} .

HRMS (CI) Calcd for $\text{C}_{10}\text{H}_9\text{FO}_2$ (M^+): 180.0587, Found: 180.0583.



1k

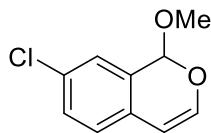
1,7-Dimethoxy-1*H*-isochromene (1k) was prepared as a yellow oil according to the General Procedure A.

^1H NMR (400 MHz, C_6D_6) δ 6.80 - 6.72 (m, 2H), 6.72 - 6.66 (m, 1H), 6.43 (d, J = 5.7 Hz, 1H), 5.78 (s, 1H), 5.73 (d, J = 5.7 Hz, 1H), 3.28 (s, 3H), 3.25 (s, 3H).

^{13}C NMR (101 MHz, C_6D_6) δ 159.1, 140.5, 129.5, 125.3, 122.9, 115.6, 111.8, 104.6, 99.5, 54.9, 54.9.

IR (thin film) 2999, 2942, 2832, 1618, 1502, 1029 cm^{-1} .

HRMS (CI) Calcd for $\text{C}_{11}\text{H}_{12}\text{O}_3$ (M^+): 192.0786, Found: 192.0788.



1l

7-Chloro-1-methoxy-1*H*-isochromene (1l) was prepared as a yellow solid according to the General Procedure A.

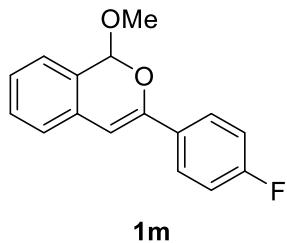
^1H NMR (400 MHz, C_6D_6) δ 7.03 - 6.97 (m, 1H), 6.97 - 6.93 (m, 1H), 6.52 - 6.45 (m, 1H), 6.34 (d, J = 5.7 Hz, 1H), 5.53 (d, J = 5.7 Hz, 1H), 5.51 (s, 1H), 3.16 (s, 3H).

^{13}C NMR (101 MHz, C_6D_6) δ 142.6, 131.9, 129.5, 129.4, 128.2, 126.8, 125.3, 103.9, 98.7,

55.1.

IR (thin film) 3075, 2998, 2958, 2931, 2830, 1634, 1488, 1027 cm⁻¹.

HRMS (CI) Calcd for C₁₀H₉ClO₂ (M⁺): 196.0291, Found: 196.0291.



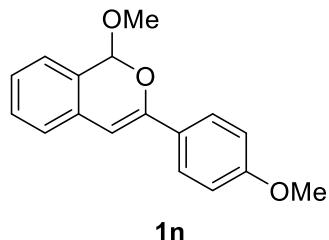
3-(4-Fluorophenyl)-1-methoxy-1H-isochromene (1m) was prepared as a colorless solid according to the General Procedure A.

¹H NMR (400 MHz, CD₂Cl₂) δ 7.86 - 7.77 (m, 2H), 7.42 - 7.33 (m, 1H), 7.31 - 7.19 (m, 3H), 7.17 - 7.08 (m, 2H), 6.56 (s, 1H), 6.13 (s, 1H), 3.58 (s, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 163.5 (d, *J* = 248.2 Hz), 149.1, 131.4 (d, *J* = 3.3 Hz), 131.0, 129.5, 127.1 (d, *J* = 8.1 Hz), 126.8, 126.4, 124.6, 115.7, 115.5, 100.7, 100.4, 54.8.

IR (thin film) 3070, 2999, 2917, 2829, 1634, 1604, 1503, 1029 cm⁻¹.

HRMS (CI) Calcd for C₁₆H₁₃FO₂ (M⁺): 256.0900, Found: 256.0899.



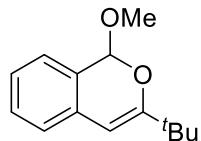
1-Methoxy-3-(4-methoxyphenyl)-1H-isochromene (1n) was prepared as a colorless solid according to the General Procedure A.

¹H NMR (400 MHz, CD₂Cl₂) δ 7.79 - 7.72 (m, 2H), 7.38 - 7.33 (m, 1H), 7.28 - 7.16 (m, 3H), 6.99 - 6.91 (m, 2H), 6.49 (s, 1H), 6.11 (s, 1H), 3.84 (s, 3H), 3.58 (s, 3H).

¹³C NMR (101 MHz, C₆D₆) δ 160.8, 150.2, 131.5, 129.5, 128.2, 127.9, 126.8, 126.4, 126.3, 124.4, 114.3, 100.4, 99.4, 54.9, 54.7.

IR (thin film) 3068, 3000, 2924, 2833, 1606, 1508, 1032 cm⁻¹.

HRMS (CI) Calcd for C₁₇H₁₆O₃ (M⁺): 268.1099, Found: 268.1093.



1o

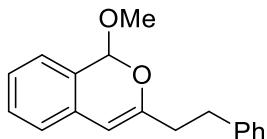
3-(Tert-butyl)-1-methoxy-1H-isochromene (1o) was prepared as a yellow solid according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 7.14 - 7.08 (m, 1H), 7.05 - 6.99 (m, 2H), 6.98 - 6.90 (m, 1H), 5.84 (s, 1H), 5.81 (s, 1H), 3.29 (s, 3H), 1.22 (s, 9H).

¹³C NMR (101 MHz, C₆D₆) δ 161.3, 131.4, 129.3, 127.2, 126.3, 126.0, 124.1, 100.3, 97.4, 55.0, 35.6, 28.3.

IR (thin film) 3073, 3030, 2965, 2911, 2828, 1645, 1064 cm⁻¹.

HRMS (CI) Calcd for C₁₄H₁₈O₂ (M⁺): 218.1307, Found: 218.1305.



1p

1-Methoxy-3-phenethyl-1H-isochromene (1p) was prepared as a pale yellow oil according to the General Procedure A.

¹H NMR (400 MHz, C₆D₆) δ 7.19 - 6.96 (m, 8H), 6.86 (d, J = 7.5 Hz, 1H), 5.86 (s, 1H), 5.58 (s, 1H), 3.29 (s, 3H), 2.86 (t, J = 7.9 Hz, 2H), 2.60 - 2.40 (m, 2H).

¹³C NMR (101 MHz, C₆D₆) δ 153.6, 141.7, 131.1, 129.3, 128.8, 128.7, 127.2, 126.44, 126.36, 126.1, 123.7, 101.1, 100.3, 54.7, 36.4, 33.9.

IR (thin film) 3067, 3028, 2915, 2829, 1656, 1610, 1078 cm⁻¹.

HRMS (CI) Calcd for C₁₈H₁₈O₂ (M⁺): 266.1307, Found: 266.1306.

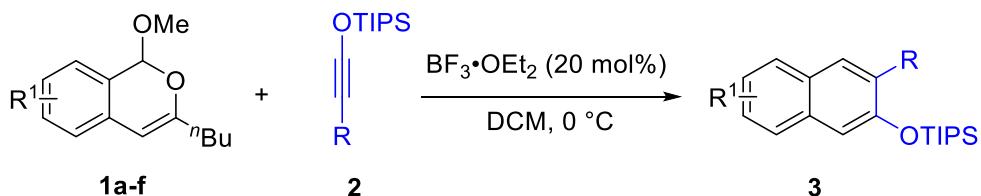
III. Synthesis of β -Naphthols from Siloxy Alkynes

Table S1. Condition Optimization for the Synthesis of **4a.**

entry	catalyst	additive	solvent	yield (%) ^[a]
1	HNTf ₂	-	DCM	<5
2	CH ₃ SO ₃ H	-	DCM	<5
3	AgOTf	-	DCM	<5
4	AgNTf ₂	-	DCM	<5
5	TMSOTf	-	DCM	53
6	TiCl ₄	-	DCM	<5
7	BF ₃ •OEt ₂	-	DCM	64
8	BF ₃ •OEt ₂ (1.0 equiv)	-	DCM	68
9	BF ₃ •OEt ₂ (2.0 equiv)	-	DCM	72
10	BF ₃ •OEt ₂ (2.0 equiv)	2,4-di- <i>tert</i> -butylpyridine	DCM	72
11	BF ₃ •OEt ₂ (2.0 equiv)	2,6-lutidine	DCM	81
12	BF ₃ •OEt ₂ (2.0 equiv)	2,4,6-collidine	DCM	85 (78) ^[b]
13	BF ₃ •OEt ₂ (2.0 equiv)	2,4,6-collidine	MeCN	6
14	BF ₃ •OEt ₂ (2.0 equiv)	2,4,6-collidine	toluene	35
15	BF ₃ •OEt ₂ (2.0 equiv)	2,4,6-collidine	DCE	82

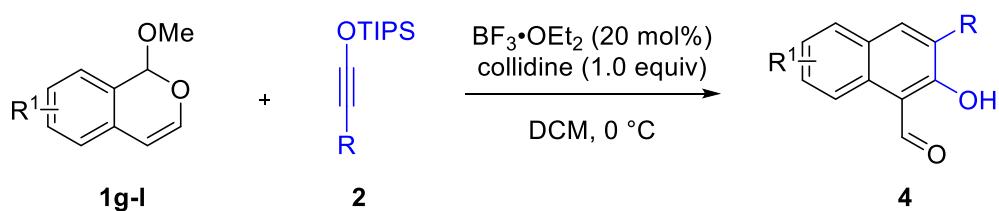
[a] Reaction scale: **1g** (0.05 mmol), **2a** (0.06 mmol), catalyst (10 mol%), additive (0.05 mmol), solvent (0.5 mL). Yield is based on analysis of the ¹H NMR spectrum of the crude product using CH₂Br₂ as the internal standard. [b] Yield in parentheses is isolated yield.

General Procedure B.



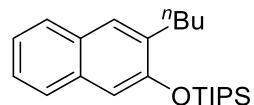
Under N_2 at room temperature, an oven-dried 25-mL Schlenk tube was charged with acetal **1** (0.5 mmol, 1.0 equiv), alkyne **2** (0.6 mmol, 1.2 equiv) and CH_2Cl_2 (5.0 mL). The mixture was cooled to 0 °C and then $\text{BF}_3\cdot\text{OEt}_2$ (12.7 μL , 0.10 mmol, 0.2 equiv) was added. The reaction mixture was then slowly warmed to room temperature and stirred overnight. Upon completion (reaction time is specified in each case shown below), the reaction was quenched by addition of a saturated aqueous NaHCO_3 solution (8 mL). The aqueous layer was separated and extracted by DCM (10 mL $\times 3$). The combined organic layers were washed with water (10 mL $\times 3$), dried with Na_2SO_4 , filtered, and concentrated. The crude product was purified by silica gel to afford the pure naphthalene product **3**.

General Procedure C.



Under N_2 at room temperature, an oven-dried 25-mL Schlenk tube was charged with starting materials **1g-l** (0.5 mmol, 1.0 equiv), **2** (0.6 mmol, 1.2 equiv), 2,4,6-collidine (66.1 μL , 0.5 mmol, 1.0 equiv) and CH_2Cl_2 (5.0 mL). The mixture was cooled to 0 °C and then $\text{BF}_3\cdot\text{OEt}_2$ (126.7 μL , 1.0 mmol, 2.0 equiv) was added. The reaction mixture was then slowly warmed to room temperature and stirred overnight. Upon completion (reaction time is specified in each case shown below), the reaction was quenched by

addition of a saturated aqueous NaHCO_3 solution (8 mL). The aqueous layer was separated and extracted by DCM (10 mL $\times 3$). The combined organic layers were washed with water (10 mL $\times 3$), dried with Na_2SO_4 , filtered, and concentrated. The crude product was purified by silica gel to afford the pure naphthalene product **3**.



3a

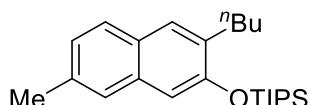
((3-Butylnaphthalen-2-yl)oxy)triisopropylsilane (3a) was prepared as a colorless oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (109.2 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure B (12 h, eluent: hexanes) in 81% yield (144.3 mg).

¹H NMR (400 MHz, CDCl_3) δ 7.70 (d, $J = 8.0$ Hz, 1H), 7.63 (d, $J = 8.1$ Hz, 1H), 7.57 (s, 1H), 7.39 - 7.32 (m, 1H), 7.32 - 7.27 (m, 1H), 7.10 (s, 1H), 2.79 (t, $J = 7.6$ Hz, 2H), 1.67 (m, 2H), 1.49 - 1.35 (m, 5H), 1.16 (d, $J = 7.5$ Hz, 18H), 0.96 (t, $J = 7.3$ Hz, 3H).

¹³C NMR (101 MHz, CDCl_3) δ 153.0, 135.0, 133.2, 129.0, 128.2, 127.0, 126.0, 125.1, 123.3, 112.4, 32.2, 31.1, 22.8, 18.1, 14.1, 13.1.

IR (thin film) 3056, 2949, 2867, 2724, 1466, 1257 cm^{-1} .

HRMS (CI) Calcd for $\text{C}_{23}\text{H}_{36}\text{OSi} (\text{M}^+)$: 356.2535, Found: 356.2531.



3b

((3-Butyl-7-methylnaphthalen-2-yl)oxy)triisopropylsilane (3b) was prepared as a colorless oil from 3-butyl-1-methoxy-6-methyl-1*H*-isochromene **1b** (116.2 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure B (12 h, eluent: hexanes) in 84% yield (155.8 mg).

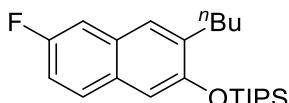
¹H NMR (400 MHz, CDCl_3) δ 7.59 (d, $J = 8.3$ Hz, 1H), 7.51 (s, 1H), 7.41 (s, 1H), 7.12 (d, $J = 8.3$ Hz, 1H), 7.02 (s, 1H), 2.75 (t, $J = 7.6$ Hz, 2H), 2.46 (s, 3H), 1.72 - 1.58 (m, 2H), 1.48

- 1.32 (m, 5H), 1.15 (d, J = 7.4 Hz, 18H), 0.94 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 153.1, 134.6, 133.9, 133.4, 127.9, 127.2, 126.8, 125.6, 125.1, 111.9, 32.3, 31.1, 22.8, 21.6, 18.1, 14.1, 13.1.

IR (thin film) 3048, 2948, 2867, 2727, 1470, 1252 cm^{-1} .

HRMS (CI) Calcd for $\text{C}_{24}\text{H}_{38}\text{OSi}$ (M^+): 370.2692, Found: 370.2699.



3c

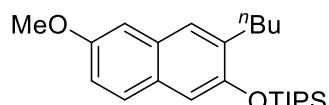
((3-Butyl-6-fluoronaphthalen-2-yl)oxy)triisopropylsilane (3c) was prepared as a colorless oil from 3-butyl-7-fluoro-1-methoxy-1*H*-isochromene **1c** (118.1 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure B (24 h, eluent: hexanes) in 68% yield (127.3 mg).

^1H NMR (400 MHz, CDCl_3) δ 7.62 - 7.55 (m, 1H), 7.50 (s, 1H), 7.34 - 7.28 (m, 1H), 7.17 - 7.09 (m, 1H), 7.09 (s, 1H), 2.77 (t, J = 7.6 Hz, 2H), 1.71 - 1.57 (m, 2H), 1.48 - 1.34 (m, 5H), 1.15 (d, J = 7.5 Hz, 18H), 0.95 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 159.4 (d, J = 242.1 Hz), 152.3, 136.2, 130.1, 129.4 (d, J = 8.9 Hz), 128.1 (d, J = 8.6 Hz), 127.4 (d, J = 5.1 Hz), 115.3 (d, J = 25.2 Hz), 112.4, 110.0 (d, J = 20.4 Hz), 32.1, 31.1, 22.8, 18.1, 14.0, 13.1.

IR (thin film) 2949, 2868, 1608, 1503, 1468, 1377, 1253 cm^{-1} .

HRMS (CI) Calcd for $\text{C}_{23}\text{H}_{35}\text{FOSi}$ (M^+): 374.2441, Found: 374.2437.



3d

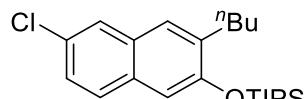
((3-Butyl-6-methoxynaphthalen-2-yl)oxy)triisopropylsilane (3d) was prepared as a colorless oil from 3-butyl-1,7-dimethoxy-1*H*-isochromene **1d** (124.2 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure B (24 h, eluent: hexanes) in 77% yield (149.6 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, *J* = 9.6 Hz, 1H), 7.47 (s, 1H), 7.06 - 7.00 (m, 3H), 3.89 (s, 3H), 2.76 (t, *J* = 7.6 Hz, 2H), 1.71 - 1.60 (m, 2H), 1.47 - 1.32 (m, 5H), 1.15 (d, *J* = 7.4 Hz, 18H), 0.95 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 156.0, 151.3, 135.3, 129.8, 128.4, 127.5, 127.1, 117.7, 112.5, 105.4, 55.3, 32.2, 31.2, 22.8, 18.1, 14.1, 13.1.

IR (thin film) 3059, 2948, 2867, 2724, 1606, 1503, 1467, 1388, 1250 cm⁻¹.

HRMS (CI) Calcd for C₂₄H₃₈O₂Si (M⁺): 386.2641, Found: 386.2635.



3e

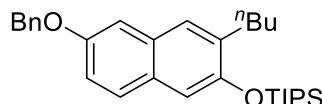
((3-Butyl-6-chloronaphthalen-2-yl)oxy)triisopropylsilane (3e) was prepared as a colorless oil from 3-butyl-7-chloro-1-methoxy-1*H*-isochromene **1e** (101.1 mg, 0.4 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (122.2 mg, 0.48 mmol) according to the General Procedure B (24 h, eluent: hexanes) in 63% yield (98.6 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.67 (s, 1H), 7.55 (d, *J* = 8.7 Hz, 1H), 7.48 (s, 1H), 7.31 - 7.26 (m, 1H), 7.06 (s, 1H), 2.76 (t, *J* = 7.6 Hz, 2H), 1.71 - 1.59 (m, 2H), 1.46 - 1.33 (m, 5H), 1.15 (d, *J* = 7.4 Hz, 18H), 0.95 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 153.2, 136.2, 131.4, 129.6, 128.8, 127.6, 127.3, 125.9, 125.7, 112.3, 32.1, 31.1, 22.7, 18.1, 14.0, 13.1.

IR (thin film) 2949, 2867, 2278, 1631, 1594, 1494, 1463, 1256 cm⁻¹.

HRMS (CI) Calcd for C₂₃H₃₅ClOSi (M⁺): 390.2146, Found: 390.2140.



3f

((6-(Benzylxy)-3-butylnaphthalen-2-yl)oxy)triisopropylsilane (3f) was prepared as a colorless solid from 7-(benzylxy)-3-butyl-1-methoxy-1*H*-isochromene **1f** (129.8 mg, 0.4 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (122.2 mg, 0.48 mmol) according

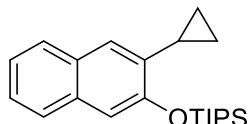
to the General Procedure B (12 h, eluent: hexanes) in 77% yield (142.3 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.59 - 7.52 (m, 1H), 7.51 - 7.44 (m, 3H), 7.44 - 7.37 (m, 2H), 7.37 - 7.30 (m, 1H), 7.15 - 7.09 (m, 2H), 7.05 (s, 1H), 5.15 (s, 2H), 2.75 (t, *J* = 7.6 Hz, 2H), 1.72 - 1.58 (m, 2H), 1.47 - 1.33 (m, 5H), 1.15 (d, *J* = 7.4 Hz, 18H), 0.95 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 155.2, 151.3, 137.2, 135.4, 129.7, 128.6, 128.5, 127.9, 127.6, 127.5, 127.1, 118.1, 112.5, 106.9, 70.1, 32.2, 31.2, 22.8, 18.1, 14.1, 13.1.

IR (thin film) 3063, 3033, 2946, 2867, 1605, 1504, 1250 cm⁻¹.

HRMS (CI) Calcd for C₃₀H₄₂O₂Si (M⁺): 462.2954, Found: 462.2971.



3g

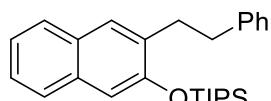
((3-Cyclopropylnaphthalen-2-yl)oxy)triisopropylsilane (3g) was prepared as a colorless oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (65.5 mg, 0.3 mmol) and ((cyclopropylethynyl)oxy)triisopropylsilane (85.8 mg, 0.36 mmol) according to the General Procedure B (12 h, eluent: hexanes) in 81% yield (82.4 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.70 - 7.59 (m, 2H), 7.38 - 7.31 (m, 1H), 7.31 - 7.26 (m, 2H), 7.12 (s, 1H), 2.35 - 2.25 (m, 1H), 1.48 - 1.37 (m, 3H), 1.17 (d, *J* = 7.5 Hz, 18H), 1.03 - 0.95 (m, 2H), 0.79 - 0.72 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 153.8, 135.9, 132.8, 129.0, 127.0, 126.0, 125.1, 123.6, 123.4, 112.4, 18.1, 13.1, 10.9, 8.0.

IR (thin film) 3070, 3012, 2954, 2865, 1632, 1600, 1461, 1259 cm⁻¹.

HRMS (CI) Calcd for C₂₂H₃₂OSi (M⁺): 340.2222, Found: 340.2226.



3h

Triisopropyl((3-phenethylnaphthalen-2-yl)oxy)silane (3h) was prepared as a

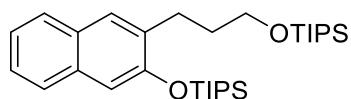
colorless oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (54.6 mg, 0.25 mmol) and triisopropyl((4-phenylbut-1-yn-1-yl)oxy)silane (90.8 mg, 0.3 mmol) according to the General Procedure B (12 h, eluent: hexanes) in 57% yield (57.7 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.70 - 7.62 (m, 2H), 7.54 (s, 1H), 7.39 - 7.33 (m, 1H), 7.31 - 7.26 (m, 3H), 7.24 - 7.17 (m, 3H), 7.14 (s, 1H), 3.14 - 3.06 (m, 2H), 3.03 - 2.95 (m, 2H), 1.49 - 1.38 (m, 3H), 1.18 (d, *J* = 7.5 Hz, 18H).

¹³C NMR (101 MHz, CDCl₃) δ 152.9, 142.2, 133.8, 133.4, 129.0, 128.7, 128.5, 128.3, 127.0, 126.1, 125.8, 125.3, 123.5, 112.5, 36.5, 33.3, 18.2, 13.2.

IR (thin film) 3058, 3027, 2954, 2864, 2722, 1633, 1598, 1461, 1256 cm⁻¹.

HRMS (CI) Calcd for C₂₇H₃₆OSi (M⁺): 404.2535, Found: 404.2539.



3i

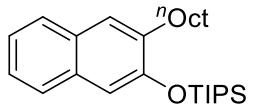
Triisopropyl(3-(3-((triisopropylsilyl)oxy)naphthalen-2-yl)propoxy)silane (3i) was prepared as a colorless solid from 3-butyl-1-methoxy-1*H*-isochromene **1a** (65.5 mg, 0.3 mmol) and 3,3,11,11-tetraisopropyl-2,12-dimethyl-4,10-dioxa-3,11-disilatridec-5-yne (148.6 mg, 0.36 mmol) according to the General Procedure B (24 h, eluent: hexanes) in 66% yield (102.1 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, *J* = 8.1 Hz, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.60 (s, 1H), 7.38 - 7.31 (m, 1H), 7.31 - 7.27 (m, 1H), 7.09 (s, 1H), 3.76 (t, *J* = 6.3 Hz, 2H), 2.87 (t, *J* = 7.6 Hz, 2H), 1.98 - 1.88 (m, 2H), 1.45 - 1.35 (m, 3H), 1.15 (d, *J* = 7.5 Hz, 18H), 1.11 - 1.01 (m, 21H).

¹³C NMR (101 MHz, CDCl₃) δ 153.0, 134.3, 133.2, 129.0, 128.3, 127.0, 126.0, 125.1, 123.4, 112.4, 62.9, 33.0, 27.5, 18.2, 18.1, 13.1, 12.1.

IR (thin film) 3056, 2950, 2864, 2724, 1633, 1599, 1462, 1255 cm⁻¹.

HRMS (CI) Calcd for C₃₁H₅₄O₂Si₂ (M⁺): 514.3662, Found: 514.3666.



3j

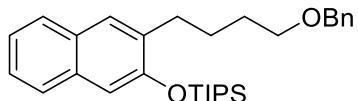
Triisopropyl((3-octylnaphthalen-2-yl)oxy)silane (3j) was prepared as a colorless oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (65.5 mg, 0.3 mmol) and (dec-1-yn-1-yloxy)triisopropylsilane (111.8 mg, 0.36 mmol) according to the General Procedure B (12 h, eluent: hexanes) in 71% yield (88.5 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.56 (s, 1H), 7.37 - 7.31 (m, 1H), 7.31 - 7.26 (m, 1H), 7.09 (s, 1H), 2.76 (t, *J* = 7.6 Hz, 2H), 1.73 - 1.61 (m, 2H), 1.45 - 1.36 (m, 5H), 1.34 - 1.24 (m, 8H), 1.15 (d, *J* = 7.4 Hz, 21H), 0.87 (t, *J* = 6.8 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 153.0, 135.0, 133.2, 129.1, 128.2, 127.0, 126.0, 125.1, 123.4, 112.4, 31.9, 31.5, 30.1, 29.7, 29.6, 29.3, 22.7, 18.2, 14.1, 13.1.

IR (thin film) 3056, 2932, 2860, 2722, 1633, 1599, 1461, 1255 cm⁻¹.

HRMS (CI) Calcd for C₂₇H₄₄OSi (M⁺): 412.3161, Found: 412.3154.



3k

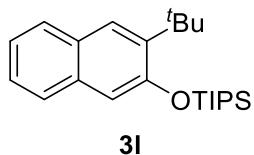
((3-(4-(Benzyl)oxy)butyl)naphthalen-2-yl)oxy)triisopropylsilane (3k) was prepared as a yellow oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (65.5 mg, 0.3 mmol) and ((6-(benzyloxy)hex-1-yn-1-yl)oxy)triisopropylsilane (129.8 mg, 0.36 mmol) according to the General Procedure B (12 h, eluent: hexanes) in 71% yield (98.9 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.56 (s, 1H), 7.38 - 7.31 (m, 5H), 7.31 - 7.26 (m, 2H), 7.09 (s, 1H), 4.50 (s, 2H), 3.51 (t, *J* = 6.1 Hz, 2H), 2.80 (t, *J* = 7.1 Hz, 2H), 1.83 - 1.67 (m, 4H), 1.45 - 1.34 (m, 3H), 1.14 (d, *J* = 7.5 Hz, 18H).

¹³C NMR (101 MHz, CDCl₃) δ 152.9, 138.7, 134.5, 133.3, 129.0, 128.31, 128.29, 127.6, 127.4, 127.0, 126.0, 125.2, 123.4, 112.4, 72.8, 70.4, 31.2, 29.8, 26.5, 18.1, 13.1.

IR (thin film) 3058, 2944, 2862, 2720, 1633, 1600, 1461, 1256 cm⁻¹.

HRMS (CI) Calcd for C₃₀H₄₂O₂Si (M⁺): 462.2954, Found: 462.2946.



3l

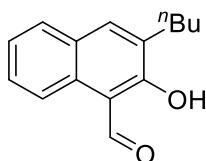
((3-(*tert*-Butyl)naphthalen-2-yl)oxy)triisopropylsilane (3l) was prepared as a colorless oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (87.3 mg, 0.4 mmol) and ((3,3-dimethylbut-1-yn-1-yl)oxy)triisopropylsilane (122.2 mg, 0.48 mmol) according to the General Procedure B (24 h, eluent: hexanes) in 34% yield (48.4 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.74 (d, *J* = 8.2 Hz, 1H), 7.73 (s, 1H), 7.61 (d, *J* = 8.1 Hz, 1H), 7.40 - 7.34 (m, 1H), 7.33 - 7.27 (m, 1H), 7.11 (s, 1H), 1.57 - 1.44 (m, 12H), 1.21 (d, *J* = 7.5 Hz, 18H).

¹³C NMR (101 MHz, CDCl₃) δ 153.7, 140.9, 132.9, 128.7, 127.5, 125.7, 125.44, 125.40, 123.4, 113.3, 35.3, 29.9, 18.3, 13.4.

IR (thin film) 3057, 2957, 2869, 2720, 1632, 1595, 1456, 1252 cm⁻¹.

HRMS (CI) Calcd for C₂₃H₃₆OSi (M⁺): 356.2535, Found: 356.2541.



4a

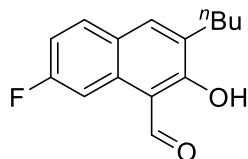
3-Butyl-2-hydroxy-1-naphthaldehyde (4a) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (81.1 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure C (12 h, eluent: hexanes/EtOAc = 100:1) in 78% yield (88.8 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.60 (s, 1H), 10.81 (s, 1H), 8.32 (d, *J* = 8.5 Hz, 1H), 7.82 (s, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.60 - 7.50 (m, 1H), 7.45 - 7.36 (m, 1H), 2.80 (t, *J* = 7.6 Hz, 2H), 1.75 - 1.64 (m, 2H), 1.49 - 1.38 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 193.4, 164.5, 137.6, 132.6, 131.8, 128.8, 128.1, 127.6, 124.3, 118.2, 110.8, 31.3, 29.3, 22.6, 14.0.

IR (thin film) 3449, 2955, 2865, 1635, 748 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₁₇O₂ (M+H⁺): 229.1229, Found: 229.1223.



4b

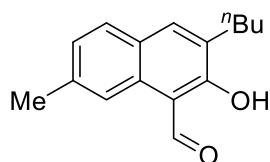
3-Butyl-7-fluoro-2-hydroxy-1-naphthaldehyde (4b) was prepared as a yellow solid from 6-fluoro-1-methoxy-1*H*-isochromene **1h** (72.1 mg, 0.4 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (122.2 mg, 0.48 mmol) according to the General Procedure C (12 h, eluent: hexanes/DCM = 10:1) in 53% yield (52.6 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.60 (s, 1H), 10.66 (s, 1H), 7.95 - 7.87 (m, 1H), 7.79 (s, 1H), 7.76 - 7.69 (m, 1H), 7.20 - 7.12 (m, 1H), 2.77 (t, *J* = 7.6 Hz, 2H), 1.73 - 1.62 (m, 2H), 1.48 - 1.36 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 192.8, 165.2, 162.4 (d, *J* = 247.6 Hz), 137.2, 133.3 (d, *J* = 9.7 Hz), 131.7 (d, *J* = 2.7 Hz), 131.2 (d, *J* = 9.8 Hz), 124.5, 113.9 (d, *J* = 24.6 Hz), 110.6 (d, *J* = 5.0 Hz), 103.1 (d, *J* = 23.5 Hz), 31.3, 29.2, 22.6, 14.0.

IR (thin film) 3443, 2956, 2930, 2865, 1631, 913, 741 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₁₆FO₂ (M+H⁺): 247.1134, Found: 247.1137.



4c

3-Butyl-2-hydroxy-7-methyl-1-naphthaldehyde (4c) was prepared as a yellow solid from 1-methoxy-6-methyl-1*H*-isochromene **1i** (88.1 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure C

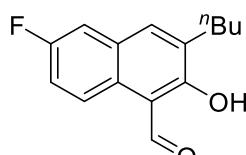
(12 h, eluent: hexanes/EtOAc = 100:1) in 78% yield (94.9 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.59 (s, 1H), 10.79 (s, 1H), 8.09 (s, 1H), 7.76 (s, 1H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 1H), 2.77 (t, *J* = 7.6 Hz, 2H), 2.54 (s, 3H), 1.74 - 1.61 (m, 2H), 1.47 - 1.36 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 193.3, 164.5, 138.2, 137.5, 132.1, 131.4, 128.7, 126.3, 125.8, 117.7, 110.5, 31.3, 29.2, 22.6, 22.3, 14.0.

IR (thin film) 3449, 2957, 2929, 2866, 1635, 1307, 803 cm⁻¹.

HRMS (CI) Calcd for C₁₆H₁₈O₂ (M⁺): 242.1307, Found: 242.1308.



4d

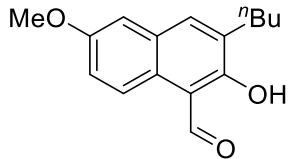
3-Butyl-6-fluoro-2-hydroxy-1-naphthaldehyde (4d) was prepared as a pale yellow solid from 7-fluoro-1-methoxy-1*H*-isochromene **1j** (72.1 mg, 0.4 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (122.2 mg, 0.48 mmol) according to the General Procedure C (16 h, eluent: hexanes/EtOAc = 100:1) in 67% yield (66.4 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.48 (s, 1H), 10.77 (s, 1H), 8.34 - 8.23 (m, 1H), 7.75 (s, 1H), 7.42 - 7.37 (m, 1H), 7.37 - 7.30 (m, 1H), 2.79 (t, *J* = 7.6 Hz, 2H), 1.73 - 1.61 (m, 2H), 1.49 - 1.37 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 193.2, 163.8, 159.6 (d, *J* = 244.5 Hz), 136.5 (d, *J* = 4.4 Hz), 134.2, 128.6 (d, *J* = 8.6 Hz), 128.4 (d, *J* = 1.4 Hz), 120.4 (d, *J* = 8.4 Hz), 117.6 (d, *J* = 24.3 Hz), 112.4 (d, *J* = 20.4 Hz), 110.9, 31.2, 29.4, 22.6, 13.9.

IR (thin film) 3452, 2954, 2930, 1640, 1454, 1412, 742 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₁₆FO₂ (M+H⁺): 247.1134, Found: 247.1131.



4e

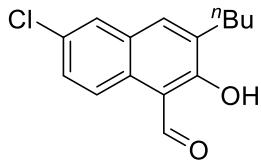
3-Butyl-2-hydroxy-6-methoxy-1-naphthaldehyde (4e) was prepared as a yellow solid from 1,7-dimethoxy-1*H*-isochromene **1k** (69.8 mg, 0.36 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (111.0 mg, 0.44 mmol) according to the General Procedure C (12 h, eluent: hexanes/EtOAc = 50:1) in 61% yield (57.7 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.32 (s, 1H), 10.76 (s, 1H), 8.22 (d, *J* = 9.2 Hz, 1H), 7.74 (s, 1H), 7.25 - 7.19 (m, 1H), 7.13 - 7.05 (m, 1H), 3.91 (s, 3H), 2.78 (t, *J* = 7.6 Hz, 2H), 1.75 - 1.63 (m, 2H), 1.49 - 1.36 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 193.4, 162.6, 156.5, 136.6, 133.1, 128.8, 126.5, 119.8, 119.7, 111.1, 107.9, 55.4, 31.3, 29.4, 22.6, 14.0.

IR (thin film) 3444, 2958, 2933, 2871, 1622, 1307, 750 cm⁻¹.

HRMS (CI) Calcd for C₁₆H₁₉O₃ (M+H⁺): 259.1334, Found: 259.1333.



4f

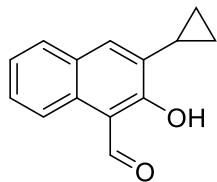
3-Butyl-6-chloro-2-hydroxy-1-naphthaldehyde (4f) was prepared as a yellow solid from 7-chloro-1-methoxy-1*H*-isochromene **1l** (98.3 mg, 0.5 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (152.7 mg, 0.6 mmol) according to the General Procedure C (12 h, eluent: hexanes/EtOAc = 100:1) in 56% yield (74.1 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.54 (s, 1H), 10.75 (s, 1H), 8.24 (d, *J* = 9.0 Hz, 1H), 7.76 - 7.66 (m, 2H), 7.56 - 7.39 (m, 1H), 2.79 (t, *J* = 7.6 Hz, 2H), 1.72 - 1.62 (m, 2H), 1.50 - 1.36 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 193.1, 164.4, 136.3, 134.1, 130.04, 130.00, 128.6, 128.5, 127.5, 119.9, 110.7, 31.2, 29.3, 22.5, 13.9.

IR (thin film) 3444, 2959, 2932, 2869, 1637, 913 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₁₆ClO₂ (M+H⁺): 263.0839, Found: 263.0850.



4g

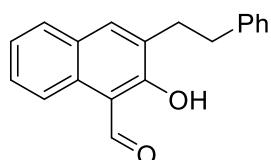
3-Cyclopropyl-2-hydroxy-1-naphthaldehyde (4g) was prepared as an orange solid from 1-methoxy-1*H*-isochromene **1g** (64.9 mg, 0.4 mmol) and ((cyclopropylethynyl)oxy) triisopropylsilane (114.5 mg, 0.48 mmol) according to the General Procedure C (19 h, eluent: hexanes/EtOAc = 200:1) in 85% yield (72.4 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.71 (s, 1H), 10.81 (s, 1H), 8.30 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.58 - 7.51 (m, 2H), 7.43 - 7.35 (m, 1H), 2.36 - 2.23 (m, 1H), 1.12 - 1.01 (m, 2H), 0.83 - 0.75 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 193.2, 164.8, 133.4, 133.1, 131.3, 128.7, 127.9, 127.4, 124.3, 118.1, 110.4, 9.3, 7.6.

IR (thin film) 3450, 3028, 2989, 2937, 2807, 1625, 1301 cm⁻¹.

HRMS (CI) Calcd for C₁₄H₁₃O₂ (M+H⁺): 213.0916, Found: 213.0919.



4h

2-Hydroxy-3-phenethyl-1-naphthaldehyde (4h) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (64.9 mg, 0.4 mmol) and triisopropyl((4-phenylbut-1-yn-1-yl)oxy)silane (145.2 mg, 0.48 mmol) according to the General Procedure C (19 h, eluent: hexanes/EtOAc = 200:1) in 74% yield (81.3 mg).

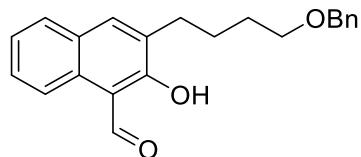
¹H NMR (400 MHz, CDCl₃) δ 13.63 (s, 1H), 10.83 (s, 1H), 8.33 (d, *J* = 8.5 Hz, 1H), 7.74 (s, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.60 - 7.53 (m, 1H), 7.45 - 7.37 (m, 1H), 7.33 - 7.27 (m,

2H), 7.25 - 7.17 (m, 3H), 3.16 - 3.06 (m, 2H), 3.06 - 2.95 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 193.4, 164.3, 141.6, 138.0, 131.9, 131.3, 128.9, 128.5, 128.4, 128.3, 127.5, 126.0, 124.4, 118.2, 110.9, 35.4, 31.9.

IR (thin film) 3068, 3028, 2931, 2866, 1631, 1308 cm⁻¹.

HRMS (CI) Calcd for C₁₉H₁₇O₂ (M+H⁺): 277.1229, Found: 277.1230.



4i

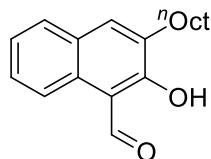
3-(4-(BenzylOxy)butyl)-2-hydroxy-1-naphthaldehyde (4i) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (48.7 mg, 0.3 mmol) and ((6-(benzyloxy)hex-1-yn-1-yl)oxy)triisopropylsilane (129.8 mg, 0.36 mmol) according to the General Procedure C (16 h, eluent: hexanes/EtOAc = 60:1) in 71% yield (71.1 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.60 (s, 1H), 10.78 (s, 1H), 8.29 (d, *J* = 8.4 Hz, 1H), 7.80 (s, 1H), 7.73 (d, *J* = 7.9 Hz, 1H), 7.59 - 7.52 (m, 1H), 7.44 - 7.38 (m, 1H), 7.38 - 7.34 (m, 4H), 7.32 - 7.27 (m, 1H), 4.53 (s, 2H), 3.55 (t, *J* = 6.3 Hz, 2H), 2.83 (t, *J* = 7.4 Hz, 2H), 1.90 - 1.79 (m, 2H), 1.79 - 1.68 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 193.3, 164.3, 138.6, 137.6, 132.0, 131.8, 128.8, 128.3, 128.1, 127.6, 127.53, 127.45, 124.3, 118.1, 110.8, 72.9, 70.1, 29.5, 29.3, 25.7.

IR (thin film) 3455, 3067, 3031, 2936, 2860, 1631, 1308 cm⁻¹.

HRMS (CI) Calcd for C₂₂H₂₂O₃ (M⁺): 334.1569, Found: 334.1556.



4j

2-Hydroxy-3-octyl-1-naphthaldehyde (4j) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (64.9 mg, 0.4 mmol) and (dec-1-yn-1-

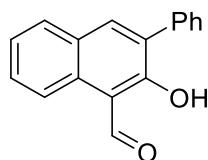
yloxy)triisopropylsilane (149.1 mg, 0.48 mmol) according to the General Procedure C (19 h, eluent: hexanes/EtOAc = 100:1) in 65% yield (74.0 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.59 (s, 1H), 10.81 (s, 1H), 8.31 (d, *J* = 8.5 Hz, 1H), 7.81 (s, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.59 - 7.52 (m, 1H), 7.44 - 7.37 (m, 1H), 2.78 (t, *J* = 7.6 Hz, 2H), 1.75 - 1.65 (m, 2H), 1.45 - 1.22 (m, 10H), 0.88 (t, *J* = 6.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 193.3, 164.4, 137.5, 132.5, 131.7, 128.8, 128.0, 127.5, 124.3, 118.1, 110.7, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 22.6, 14.1.

IR (thin film) 2925, 2858, 1632, 1450, 1308 cm⁻¹.

HRMS (CI) Calcd for C₁₉H₂₅O₂ (M+H⁺): 285.1855, Found: 285.1848.



4k

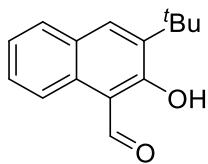
2-Hydroxy-3-phenyl-1-naphthaldehyde (4k) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (64.9 mg, 0.4 mmol) and triisopropyl((phenylethynyl)oxy)silane (131.8 mg, 0.48 mmol) according to the General Procedure C (12 h, eluent: hexanes/EtOAc = 200:1) in 59% yield (58.8 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.81 (s, 1H), 10.86 (s, 1H), 8.35 (d, *J* = 8.5 Hz, 1H), 8.04 (s, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.71 - 7.66 (m, 2H), 7.66 - 7.60 (m, 1H), 7.54 - 7.41 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 193.5, 163.2, 139.0, 136.1, 132.5, 131.7, 129.5, 129.4, 129.0, 128.3, 128.0, 127.7, 124.7, 118.2, 111.3.

IR (thin film) 3454, 3061, 2950, 2873, 1628, 1305 cm⁻¹.

HRMS (CI) Calcd for C₁₇H₁₃O₂ (M+H⁺): 249.0916, Found: 249.0910.



4l

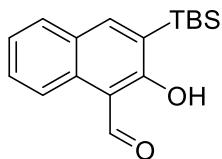
3-(*tert*-Butyl)-2-hydroxy-1-naphthaldehyde (4l) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (64.9 mg, 0.4 mmol) and ((3,3-dimethylbut-1-yn-1-yl)oxy)triisopropylsilane (122.2 mg, 0.48 mmol) according to the General Procedure C (12 h, eluent: hexanes/EtOAc = 200:1) in 26% yield (24.0 mg), together with **3l** in 62% yield (89.8 mg).

¹H NMR (400 MHz, CDCl₃) δ 14.16 (s, 1H), 10.81 (s, 1H), 8.28 (d, *J* = 8.5 Hz, 1H), 7.93 (s, 1H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.59 - 7.50 (m, 1H), 7.45 - 7.36 (m, 1H), 1.52 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 193.6, 166.0, 139.2, 135.4, 131.9, 129.4, 128.3, 127.4, 124.3, 117.8, 111.3, 35.2, 29.4.

IR (thin film) 3453, 3067, 2960, 2873, 1625, 1307 cm⁻¹.

HRMS (CI) Calcd for C₁₅H₁₇O₂ (M+H⁺): 229.1229, Found: 229.1232.



4m

3-(*tert*-Butyldimethylsilyl)-2-hydroxy-1-naphthaldehyde (4m) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (64.9 mg, 0.4 mmol) and *tert*-butyldimethyl(((triisopropylsilyl)oxy)ethynyl)silane (142.4 mg, 0.48 mmol) according to the General Procedure C (12 h, eluent: hexanes/EtOAc = 200:1) in 36% yield (40.9 mg), together with **3m** in 49% yield (81.8 mg).

¹H NMR (400 MHz, CDCl₃) δ 13.60 (s, 1H), 10.81 (s, 1H), 8.32 (d, *J* = 8.5 Hz, 1H), 8.09 (s, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.64 - 7.54 (m, 1H), 7.46 - 7.37 (m, 1H), 0.96 (s, 9H), 0.41 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 193.3, 169.6, 147.5, 133.7, 129.7, 129.3, 129.1, 127.5, 124.2,

118.1, 110.0, 27.1, 17.5, -4.8.

IR (thin film) 3453, 3069, 2942, 2859, 1627, 1251 cm⁻¹.

HRMS (CI) Calcd for C₁₇H₂₃O₂Si (M+H⁺): 287.1467, Found: 287.1465.

IV. Synthesis of β -Naphthyl Thioethers from Thioalkynes

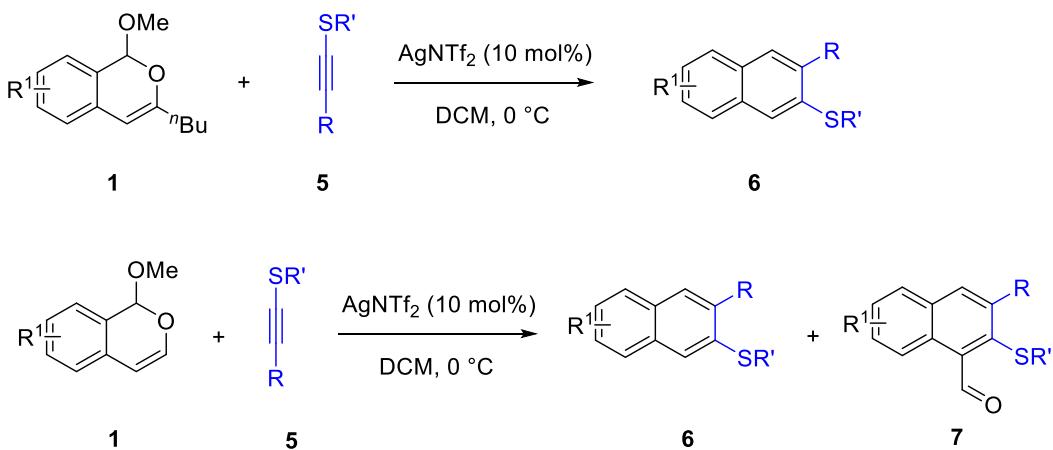
Table S2. Condition Optimization for the Synthesis of **6a.**

entry	catalyst	yield ^a (%)
1	TMSOTf	73
2	TMSOTf (1.0 equiv)	65
3	TiCl ₄	3
4	TiCl ₄ (1.0 equiv)	32
5	BF ₃ •OEt ₂	70
6	BF ₃ •OEt ₂ (0.2 equiv)	72 (68 ^b)
7	BF ₃ •OEt ₂ (1.0 equiv)	80
8	HNTf ₂	20
9	CH ₃ SO ₃ H	7
10	Sc(OTf) ₃	88
11	Au(PPh ₃)Cl	<5
12	AgOTf	80
13	AgNTf ₂	89 (83^b)

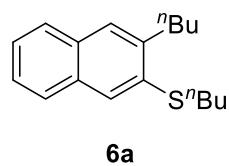
[a] Reaction scale: **1a** (0.05 mmol), **6a** (0.06 mmol), catalyst (10 mol%), DCM (0.5 mL).

Yield is based on ¹H NMR analysis of the crude product using CH₂Br₂ as the internal standard. [b] Yield in parentheses is isolated yield.

General Procedure D.



At room temperature, under N_2 , an oven-dried 25 mL Schlenk tube was charged with AgNTf_2 (0.03 mmol, 0.1 equiv) and CH_2Cl_2 (3.0 mL). The reaction mixture was cooled to 0°C , followed by addition of the starting materials **1** (0.3 mmol, 1.0 equiv) and **5** (0.36 mmol, 1.2 equiv). The reaction mixture was then warmed to room temperature slowly and stirred overnight. Upon completion, the reaction was quenched by addition of 5 mL saturated NaHCO_3 solution. The organic layer was separated and extracted by 5 mL DCM for 3 times. Washed with 5 mL water for 3 times and dried with Na_2SO_4 . Filtered and concentrated. The crude product was purified by silica gel to afford the desired product **6** (or **7**).



Butyl(3-butylnaphthalen-2-yl)sulfane (6a**)** was prepared as a colorless solid from 3-butyl-1-methoxy-1*H*-isochromene **1a** (65.5 mg, 0.3 mmol) and butyl(hex-1-yn-1-yl)sulfane (61.3 mg, 0.36 mmol) according to the General Procedure D (12 h, eluent: hexanes) in 83% yield (67.8 mg).

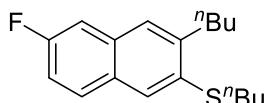
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.76 - 7.68 (m, 2H), 7.63 (s, 1H), 7.59 (s, 1H), 7.43 - 7.35 (m, 2H), 3.04 (t, $J = 7.2$ Hz, 2H), 2.86 (t, $J = 7.6$ Hz, 2H), 1.80 - 1.66 (m, 4H), 1.59 - 1.40

(m, 4H), 1.02 - 0.95 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 139.5, 135.7, 132.4, 131.5, 127.1, 127.0, 126.4, 125.5, 125.1, 124.7, 33.5, 32.7, 32.4, 30.7, 22.7, 22.2, 14.0, 13.7.

IR (thin film) 3053, 2954, 2928, 2864, 2359, 1452 cm⁻¹.

HRMS (CI) Calcd for C₁₈H₂₄S (M⁺): 272.1599, Found: 272.1608.



6b

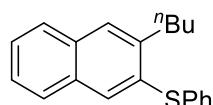
Butyl(3-butyl-6-fluoronaphthalen-2-yl)sulfane (6b) was prepared as a colorless oil from 3-butyl-7-fluoro-1-methoxy-1*H*-isochromene **1c** (70.9 mg, 0.3 mmol) and butyl(hex-1-yn-1-yl)sulfane (61.3 mg, 0.36 mmol) according to the General Procedure D (37 h, eluent: hexanes) in 76% yield (66.6 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.73 - 7.66 (m, 1H), 7.62 (s, 1H), 7.53 (s, 1H), 7.37 - 7.31 (m, 1H), 7.21 - 7.13 (m, 1H), 3.01 (t, *J* = 7.2 Hz, 2H), 2.85 (t, *J* = 7.6 Hz, 2H), 1.77 - 1.64 (m, 4H), 1.57 - 1.40 (m, 4H), 1.02 - 0.93 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 160.3 (d, *J* = 244.8 Hz), 140.9, 134.9 (d, *J* = 2.8 Hz), 132.1 (d, *J* = 9.1 Hz), 129.4, 128.7 (d, *J* = 8.9 Hz), 126.4 (d, *J* = 5.3 Hz), 125.1, 115.7 (d, *J* = 25.4 Hz), 110.1 (d, *J* = 20.5 Hz), 33.5, 32.9, 32.3, 30.7, 22.7, 22.2, 14.0, 13.7.

IR (thin film) 3062, 2955, 2865, 2359, 1495, 1203 cm⁻¹.

HRMS (CI) Calcd for C₁₈H₂₃FS (M⁺): 290.1504, Found: 290.1499.



6c

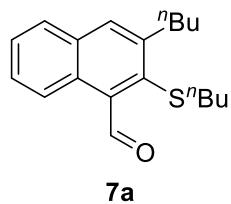
(3-Butylnaphthalen-2-yl)(phenyl)sulfane (6c) was prepared as a colorless solid from 3-butyl-1-methoxy-1*H*-isochromene **1a** (65.5 mg, 0.3 mmol) and hex-1-yn-1-yl(phenyl)sulfane (68.5 mg, 0.36 mmol) according to the General Procedure D (19 h, eluent: hexanes) in 46% yield (40.3 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.79 (s, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.70 (s, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.47 - 7.37 (m, 2H), 7.31 - 7.19 (m, 5H), 2.88 (t, *J* = 7.6 Hz, 2H), 1.72 - 1.61 (m, 2H), 1.46 - 1.34 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 141.4, 136.7, 133.1, 132.7, 132.5, 132.4, 129.9, 129.2, 128.8, 127.8, 127.1, 126.5, 126.3, 125.6, 33.8, 32.9, 22.6, 14.0.

IR (thin film) 3057, 2952, 2863, 2359, 1583, 1475, 1446 cm⁻¹.

HRMS (CI) Calcd for C₂₀H₂₀S (M⁺): 292.1286, Found: 292.1290.



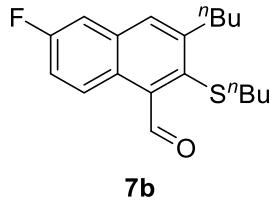
3-Butyl-2-(butylthio)-1-naphthaldehyde (7a) was prepared as a yellow solid from 1-methoxy-1*H*-isochromene **1g** (48.7 mg, 0.3 mmol) and butyl(hex-1-yn-1-yl)sulfane (61.3 mg, 0.36 mmol) according to the General Procedure D (12 h, eluent: hexanes/EtOAc = 100:1) in 78% yield (70.5 mg), together with **6a** in 10% yield (8.3 mg).

¹H NMR (400 MHz, CDCl₃) δ 11.00 (s, 1H), 8.82 (d, *J* = 8.4 Hz, 1H), 7.88 (s, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.59 - 7.48 (m, 2H), 3.07 (t, *J* = 7.6 Hz, 2H), 2.76 (t, *J* = 7.4 Hz, 2H), 1.74 - 1.63 (m, 2H), 1.53 - 1.42 (m, 4H), 1.42 - 1.32 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H), 0.86 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 195.4, 143.2, 141.0, 135.5, 133.3, 132.6, 129.6, 127.8, 127.6, 127.0, 124.6, 39.6, 33.8, 33.7, 31.7, 22.6, 21.9, 14.0, 13.5.

IR (thin film) 3059, 2957, 2928, 2863, 1685, 1455 cm⁻¹.

HRMS (CI) Calcd for C₁₉H₂₄OS (M⁺): 300.1548, Found: 300.1548.



7b

3-Butyl-2-(butylthio)-6-fluoro-1-naphthaldehyde (7b) was prepared as a yellow solid from 7-fluoro-1-methoxy-1*H*-isochromene **1j** (54.1 mg, 0.3 mmol) and butyl(hex-1-yn-1-yl)sulfane (61.3 mg, 0.36 mmol) according to the General Procedure D (19 h, eluent: hexanes) in 59% yield (56.0 mg), together with **6b** in 28% yield (24.2 mg).

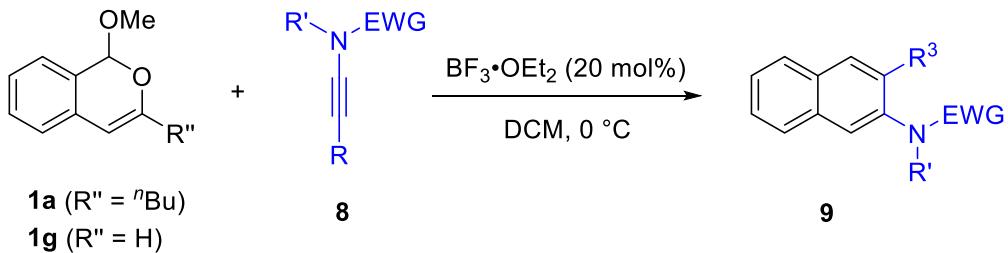
¹H NMR (400 MHz, CDCl₃) δ 10.98 (s, 1H), 8.90 - 8.80 (m, 1H), 7.81 (s, 1H), 7.41 - 7.35 (m, 1H), 7.34 - 7.28 (m, 1H), 3.05 (t, *J* = 7.6 Hz, 2H), 2.75 (t, *J* = 7.2 Hz, 2H), 1.73 - 1.62 (m, 2H), 1.56 - 1.50 (m, 2H), 1.50 - 1.42 (m, 2H), 1.42 - 1.33 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H), 0.87 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 195.3, 161.1 (d, *J* = 249.0 Hz), 144.6, 140.3 (d, *J* = 2.7 Hz), 135.6, 134.5 (d, *J* = 9.2 Hz), 131.8 (d, *J* = 5.2 Hz), 127.7 (d, *J* = 8.8 Hz), 126.6, 117.9 (d, *J* = 24.5 Hz), 110.6 (d, *J* = 20.4 Hz), 39.7, 33.8, 33.6, 31.7, 22.6, 21.9, 14.0, 13.6.

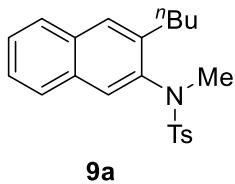
IR (thin film) 2958, 2929, 2864, 1683, 1630, 1495 cm⁻¹.

HRMS (CI) Calcd for C₁₉H₂₃FOS (M⁺): 318.1454, Found: 318.1448.

V. Synthesis of β -Naphthylamines from Ynamides



The products **9a-9d** were prepared according to the General Procedure B, except that the ynamide **8** was used in place of the siloxy alkyne **2**.



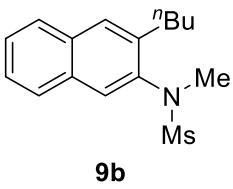
N-(3-Butylnaphthalen-2-yl)-N,4-dimethylbenzenesulfonamide (9a) was prepared as a colorless solid from 3-butyl-1-methoxy-1*H*-isochromene **1a** (54.6 mg, 0.25 mmol) and *N*-(hex-1-yn-1-yl)-*N*,4-dimethylbenzenesulfonamide (79.6 mg, 0.3 mmol) according to the General Procedure B (30 h, eluent: hexanes/EtOAc = 20:1) in 80% yield (73.2 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.78 (d, $J = 8.2$ Hz, 1H), 7.74 (s, 1H), 7.61 (d, $J = 8.2$ Hz, 2H), 7.51 (d, $J = 8.1$ Hz, 1H), 7.49 - 7.42 (m, 1H), 7.40 - 7.34 (m, 1H), 7.32 (d, $J = 8.1$ Hz, 2H), 7.01 (s, 1H), 3.21 (s, 3H), 3.19 - 3.10 (m, 1H), 2.88 - 2.77 (m, 1H), 2.48 (s, 3H), 1.77 - 1.66 (m, 2H), 1.50 - 1.39 (m, 2H), 0.98 (t, $J = 7.3$ Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 143.6, 141.0, 139.4, 134.5, 133.1, 131.7, 129.4, 128.5, 128.4, 127.22, 127.18, 126.6, 126.0, 125.5, 40.1, 32.6, 31.3, 22.8, 21.6, 14.1.

IR (thin film) 3058, 2956, 2866, 2440, 1348, 1162 cm⁻¹.

HRMS (CI) Calcd for C₂₂H₂₅NO₂S (M⁺): 367.1606, Found: 367.1602.



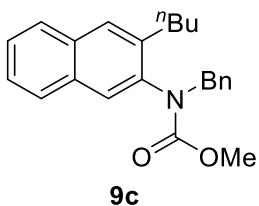
N-(3-Butylnaphthalen-2-yl)-N-methylmethanesulfonamide (9b) was prepared as a colorless solid from 3-butyl-1-methoxy-1*H*-isochromene **1a** (54.6 mg, 0.25 mmol) and *N*-(hex-1-yn-1-yl)-*N*-methylmethanesulfonamide (56.8 mg, 0.3 mmol) according to the General Procedure B (30 h, eluent: hexanes/EtOAc = 10:1) in 56% yield (41.1 mg).

¹H NMR (400 MHz, CDCl₃) δ 7.84 - 7.71 (m, 4H), 7.53 - 7.40 (m, 2H), 3.34 (s, 3H), 3.13 - 2.97 (m, 4H), 2.91 - 2.75 (m, 1H), 1.78 - 1.66 (m, 2H), 1.51 - 1.40 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 140.5, 139.1, 133.2, 132.0, 128.9, 127.3, 127.2, 126.8, 125.9, 125.8, 39.6, 36.2, 32.5, 31.2, 22.7, 14.0.

IR (thin film) 3056, 3018, 2955, 2866, 1340, 1151 cm⁻¹.

HRMS (CI) Calcd for C₁₆H₂₁NO₂S (M⁺): 291.1293, Found: 291.1300.



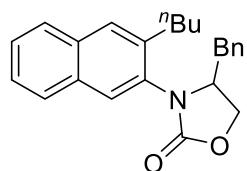
Methyl benzyl(3-butylnaphthalen-2-yl)carbamate (9c) was prepared as a colorless oil from 3-butyl-1-methoxy-1*H*-isochromene **1a** (54.6 mg, 0.25 mmol) and methyl benzyl(hex-1-yn-1-yl)carbamate (73.6 mg, 0.3 mmol) according to the General Procedure B (12 h, eluent: hexanes/EtOAc = 40:1) in 85% yield (73.7 mg).

¹H NMR (400 MHz, C₆D₆) δ 7.61 - 7.54 (m, 2H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.32 (s, 1H), 7.28 - 7.18 (m, 3H), 7.15 - 7.11 (m, 1H), 7.09 - 7.03 (m, 3H), 5.09 (d, *J* = 14.3 Hz, 1H), 4.51 (d, *J* = 14.4 Hz, 1H), 3.46 (s, 3H), 2.69 - 2.55 (m, 1H), 2.55 - 2.40 (m, 1H), 1.69 - 1.55 (m, 1H), 1.54 - 1.40 (m, 1H), 1.35 - 1.23 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 156.8, 138.7, 138.5, 137.5, 132.8, 131.9, 129.2, 128.4, 127.8, 127.6, 127.5, 127.2, 127.1, 126.2, 125.4, 54.8, 52.9, 32.0, 30.3, 22.8, 14.0.

IR (thin film) 3057, 3028, 2953, 2864, 1705, 1452 cm⁻¹.

HRMS (CI) Calcd for C₂₃H₂₅NO₂ (M⁺): 347.1885, Found: 347.1899.



9d

4-Benzyl-3-(3-butynaphthalen-2-yl)oxazolidin-2-one (9d) was prepared as a colorless solid from 3-butyl-1-methoxy-1*H*-isochromene **1a** (54.6 mg, 0.25 mmol) and methyl benzyl(hex-1-yn-1-yl)carbamate (77.2 mg, 0.3 mmol) according to the General Procedure B (12 h, eluent: hexanes/EtOAc = 10:1) in 74% yield (66.4 mg).

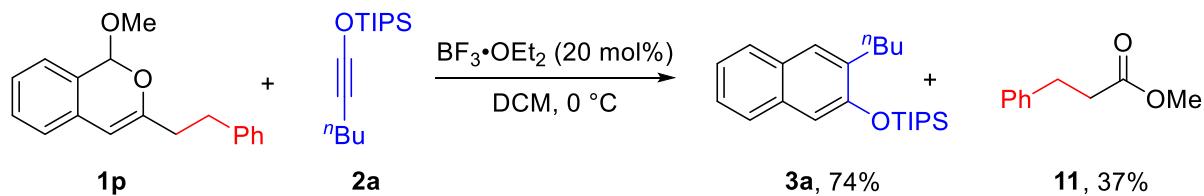
¹H NMR (400 MHz, C₆D₆) δ 7.67 - 7.61 (m, 3H), 7.57 (s, 1H), 7.34 - 7.23 (m, 2H), 7.00 - 6.94 (m, 3H), 6.66 - 6.60 (m, 2H), 4.14 - 4.01 (m, 1H), 3.91 - 3.77 (m, 2H), 2.82 (t, *J* = 7.6 Hz, 2H), 2.75 - 2.64 (m, 1H), 2.43 - 2.33 (m, 1H), 1.80 - 1.65 (m, 2H), 1.45 - 1.35 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CD₂Cl₂) δ 157.3, 139.5, 136.1, 133.6, 132.5, 130.1, 129.3, 129.2, 128.8, 127.9, 127.6, 127.4, 127.3, 127.1, 126.2, 68.1, 67.8, 39.4, 32.7, 31.1, 23.2, 14.2.

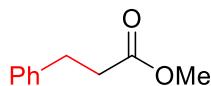
IR (thin film) 3058, 2928, 2863, 2360, 1756, 1403 cm⁻¹.

HRMS (CI) Calcd for C₂₄H₂₆NO₂ (M⁺): 360.1964, Found: 360.1956.

VI. Mechanistic Study



The reaction between 1-methoxy-3-phenethyl-1*H*-isochromene **1p** (106.5 mg, 0.4 mmol) and (hex-1-yn-1-yloxy)triisopropylsilane (122.2 mg, 0.48 mmol) according to the General Procedure B (16 h, eluent: hexanes/EtOAc = 50:1) resulted in formation of **3a** in 74% yield (105.0 mg) and ester **11** in 37% yield (24.3 mg).



11

¹H NMR (400 MHz, CDCl₃) δ 7.33 - 7.27 (m, 2H), 7.24 - 7.17 (m, 3H), 3.67 (s, 3H), 3.01 - 2.90 (m, 2H), 2.69 - 2.59 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 173.3, 140.5, 128.5, 128.3, 126.3, 51.6, 35.7, 30.9.

IR (thin film) 3069, 3029, 2952, 2864, 1738, 1640, 1445 cm⁻¹.

HRMS (CI) Calcd for C₁₀H₁₂O₂ (M⁺): 164.0837, Found: 164.0842.

VII. Product Structure Determination

We have determined the structures of products **4a** and **4b** by X-ray crystallography. The X-ray data have been deposited at the Cambridge Crystallographic Data Center (CCDC 1995829 for **4a**, CCDC 1995830 for **4b**).

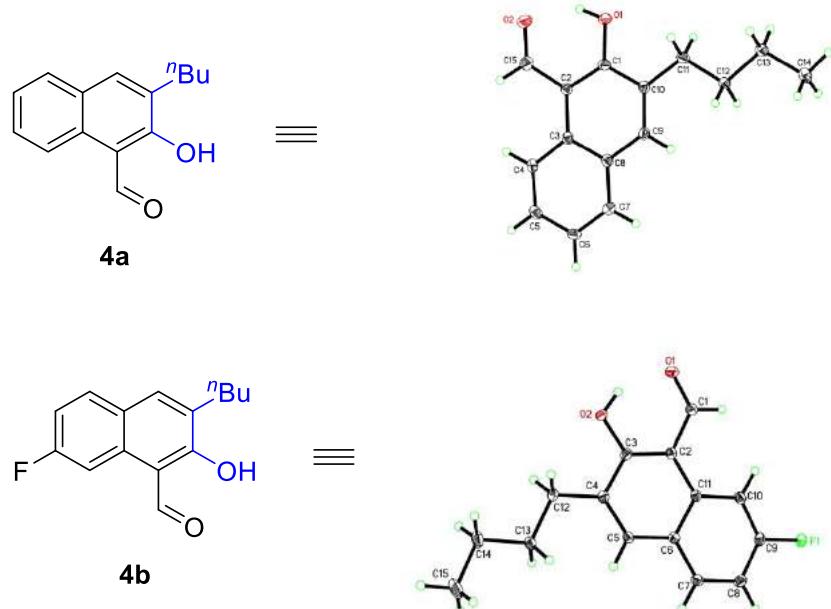


Table S3. Crystal data and structure refinement for **4a.**

	4a
Identification code	
Empirical formula	C ₁₅ H ₁₆ O ₂
Formula weight	228.28
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	12.3890(5)
b/Å	12.5456(4)
c/Å	7.5297(3)
α/°	90
β/°	99.928(4)
γ/°	90
Volume/Å ³	1152.81(8)
Z	4
Q _{calc} g/cm ³	1.315
μ/mm ⁻¹	0.086

F(000)	488.0
Crystal size/mm ³	0.38 × 0.18 × 0.15
Radiation	MoK α ($\lambda = 0.71073$)
2 Θ range for data collection/ $^\circ$	6.382 to 51.992
Index ranges	-13 ≤ h ≤ 15, -15 ≤ k ≤ 9, -9 ≤ l ≤ 8
Reflections collected	4219
Independent reflections	2233 [$R_{\text{int}} = 0.0250$, $R_{\text{sigma}} = 0.0392$]
Data/restraints/parameters	2233/0/156
Completeness to theta = 25.0°	98.4%
Goodness-of-fit on F ²	1.003
Final R indexes [$I >= 2\sigma(I)$]	$R_1 = 0.0434$, $wR_2 = 0.1029$
Final R indexes [all data]	$R_1 = 0.0634$, $wR_2 = 0.1124$
Largest diff. peak/hole / e Å ⁻³	0.19/-0.25

Table S4. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å²×10³) for 4a. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	z	U(eq)
O1	219.8(8)	3695.2(8)	1738.0(14)	20.4(3)
O2	-926.9(8)	2120.5(9)	2350.5(14)	24.8(3)
C1	1004.4(11)	3000.5(12)	1500.2(17)	15.6(3)
C2	842.5(11)	1901.6(12)	1605.2(18)	15.3(3)
C3	1695.7(11)	1184.9(12)	1288.5(18)	15.4(3)
C4	1594.4(12)	60.0(12)	1264.9(18)	18.1(3)
C5	2444.2(12)	-577.1(12)	976.8(18)	20.4(4)
C6	3433.6(12)	-132.6(13)	679.5(18)	20.6(4)
C7	3548.8(12)	947.0(13)	654.2(18)	19.7(4)
C8	2695.1(11)	1629.3(12)	955.7(17)	15.6(3)
C9	2813.2(11)	2757.9(11)	907.7(18)	15.9(3)
C10	2009.0(11)	3449.1(12)	1164.3(18)	15.4(3)
C11	2106.3(11)	4644.5(12)	1114.1(19)	17.4(3)
C12	3187.1(12)	5076.9(12)	745.7(18)	16.5(3)
C13	3200.8(12)	6291.1(12)	708(2)	20.6(4)
C14	4258.4(12)	6737.7(13)	245(2)	25.0(4)
C15	-163.4(12)	1531.8(13)	2116.6(19)	19.7(4)

Table S5. Anisotropic Displacement Parameters (Å²×10³) for 4a. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^{*}b^{*}U_{12}+\dots]$.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
O1	16.0(5)	16.2(6)	30.7(6)	-1.6(5)	8.4(5)	1.1(5)
O2	18.6(6)	23.3(6)	34.2(6)	-1.4(5)	8.9(5)	0.6(5)
C1	16.7(7)	16.4(8)	13.7(7)	-0.1(6)	2.9(6)	3.9(7)
C2	17.4(7)	15.1(8)	14.1(7)	0.5(6)	4.1(6)	0.0(7)
C3	18.0(8)	16.0(8)	12.1(7)	1.1(6)	2.6(6)	-0.5(6)
C4	19.9(8)	16.4(8)	18.2(7)	1.0(6)	3.7(6)	-3.3(7)

C5	29.0(8)	12.6(8)	19.5(8)	-1.3(6)	3.8(6)	0.8(7)
C6	22.1(8)	17.7(8)	23.0(8)	-1.2(7)	6.6(6)	5.4(7)
C7	18.9(8)	21.1(8)	20.1(8)	-0.3(7)	6.2(6)	0.7(7)
C8	18.3(7)	15.4(8)	13.2(7)	0.1(6)	3.0(6)	-0.2(7)
C9	16.2(7)	14.9(8)	17.2(7)	0.1(6)	4.9(6)	-2.4(7)
C10	18.2(7)	15.0(8)	13.3(7)	0.4(6)	3.3(6)	-1.6(7)
C11	19.3(8)	14.9(8)	18.3(7)	-0.6(6)	3.7(6)	1.7(7)
C12	17.2(8)	13.2(8)	19.5(7)	-0.1(6)	4.6(6)	0.1(6)
C13	21.8(8)	13.9(8)	27.1(8)	-0.3(6)	6.8(6)	0.0(7)
C14	27.0(9)	16.1(8)	32.8(9)	1.6(7)	7.7(7)	-2.5(7)
C15	19.0(8)	18.8(8)	21.1(8)	-1.9(6)	3.5(6)	-2.5(7)

Table S6. Bond Lengths for 4a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C1	1.3405(16)	C5	C6	1.399(2)
O2	C15	1.2365(17)	C6	C7	1.362(2)
C1	C2	1.397(2)	C7	C8	1.409(2)
C1	C10	1.4277(19)	C8	C9	1.424(2)
C2	C3	1.4387(19)	C9	C10	1.3596(19)
C2	C15	1.4429(18)	C10	C11	1.5055(19)
C3	C4	1.417(2)	C11	C12	1.5140(19)
C3	C8	1.4190(19)	C12	C13	1.5238(19)
C4	C5	1.369(2)	C13	C14	1.5199(19)

Table S7. Bond Angles for 4a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	C1	C2	121.30(12)	C6	C7	C8	121.22(14)
O1	C1	C10	116.21(13)	C3	C8	C9	119.45(13)
C2	C1	C10	122.48(13)	C7	C8	C3	119.45(14)
C1	C2	C3	119.37(12)	C7	C8	C9	121.10(13)
C1	C2	C15	118.12(13)	C10	C9	C8	123.32(13)
C3	C2	C15	122.44(13)	C1	C10	C11	118.24(12)
C4	C3	C2	123.96(12)	C9	C10	C1	117.15(13)
C4	C3	C8	117.86(13)	C9	C10	C11	124.61(13)
C8	C3	C2	118.17(13)	C10	C11	C12	116.02(12)
C5	C4	C3	120.98(13)	C11	C12	C13	112.00(12)
C4	C5	C6	120.78(14)	C14	C13	C12	112.68(12)
C7	C6	C5	119.68(14)	O2	C15	C2	124.27(14)

Table S8. Hydrogen Bonds for 4a.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O1	H1	O2	0.84	1.77	2.5218(14)	148.1

Table S9. Torsion Angles for 4a.

A	B	C	D	Angle/ [°]	A	B	C	D	Angle/ [°]
O1	C1	C2	C3	178.19(11)	C4	C3	C8	C9	177.87(13)
O1	C1	C2	C15	-4.7(2)	C4	C5	C6	C7	-0.8(2)
O1	C1	C10	C9	-179.58(11)	C5	C6	C7	C8	1.2(2)
O1	C1	C10	C11	0.29(18)	C6	C7	C8	C3	-0.1(2)
C1	C2	C3	C4	-176.44(13)	C6	C7	C8	C9	-179.26(13)
C1	C2	C3	C8	2.70(19)	C7	C8	C9	C10	179.04(13)
C1	C2	C15	O2	5.1(2)	C8	C3	C4	C5	1.7(2)
C1	C10	C11	C12	-179.77(12)	C8	C9	C10	C1	0.1(2)
C2	C1	C10	C9	1.3(2)	C8	C9	C10	C11	-179.72(13)
C2	C1	C10	C11	-178.80(13)	C9	C10	C11	C12	0.1(2)
C2	C3	C4	C5	-179.19(12)	C10	C1	C2	C3	-2.8(2)
C2	C3	C8	C7	179.53(12)	C10	C1	C2	C15	174.30(12)
C2	C3	C8	C9	-1.32(19)	C10	C11	C12	C13	179.58(12)
C3	C2	C15	O2	-177.88(13)	C11	C12	C13	C14	-177.05(12)
C3	C4	C5	C6	-0.6(2)	C15	C2	C3	C4	6.6(2)
C3	C8	C9	C10	-0.1(2)	C15	C2	C3	C8	-174.24(12)
C4	C3	C8	C7	-1.28(19)					

Table S10. Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 4a.

Atom	x	y	z	U(eq)
H1	-323	3362	1986	31
H4	927	-257	1452	22
H5	2361	-1330	979	25
H6	4022	-582	496	25
H7	4217	1245	429	24
H9	3488	3042	685	19
H11A	1980	4931	2286	21
H11B	1514	4920	174	21
H12A	3322	4801	-428	20
H12B	3786	4820	1692	20
H13A	2576	6547	-192	25
H13B	3103	6565	1903	25
H14A	4347	6490	-956	38
H14B	4880	6493	1138	38
H14C	4230	7518	253	38
H15	-244	787	2285	24

Table S11. Crystal data and structure refinement for 4b.

Identification code	4b
Empirical formula	C ₁₅ H ₁₅ FO ₂
Formula weight	246.27
Temperature/K	100.01(10)
Crystal system	monoclinic
Space group	P2 ₁ /n
a/Å	7.37471(14)
b/Å	19.0645(3)
c/Å	9.32895(18)
α/°	90
β/°	111.872(2)
γ/°	90
Volume/Å ³	1217.19(4)
Z	4
Q _{calcg} /cm ³	1.344
μ/mm ⁻¹	0.810
F(000)	520.0
Crystal size/mm ³	0.25 × 0.1 × 0.08
Radiation	CuKα ($\lambda = 1.54184$)
2Θ range for data collection/°	9.278 to 133.95
Index ranges	-5 ≤ h ≤ 8, -22 ≤ k ≤ 21, -11 ≤ l ≤ 9
Reflections collected	3701
Independent reflections	2115 [R _{int} = 0.0121, R _{sigma} = 0.0172]
Data/restraints/parameters	2115/0/165
Completeness to theta = 66.5°	97.3%
Goodness-of-fit on F ²	1.001
Final R indexes [I>=2σ (I)]	R ₁ = 0.0307, wR ₂ = 0.0798
Final R indexes [all data]	R ₁ = 0.0325, wR ₂ = 0.0814
Largest diff. peak/hole / e Å ⁻³	0.19/-0.21

Table S2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å $^2 \times 10^3$) for 4b. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	z	U(eq)
F1	-1615.6(8)	6535.8(3)	780.5(6)	22.27(17)
O1	4661.9(11)	6960.2(4)	7640.6(8)	22.56(19)
O2	5464.4(10)	5678.6(4)	8367.1(8)	19.74(18)
C1	3508.0(14)	6761.5(5)	6364.1(11)	18.0(2)
C2	3149.0(13)	6034.0(5)	5909.1(10)	14.7(2)
C3	4185.2(14)	5515.7(5)	6956.1(11)	14.9(2)
C4	3962.1(14)	4786.8(5)	6593.8(11)	15.0(2)
C5	2693.0(14)	4608.3(5)	5149.0(11)	15.3(2)
C6	1595.4(13)	5106.2(5)	4027.6(11)	15.1(2)
C7	311.0(14)	4883.4(5)	2552.3(11)	16.7(2)
C8	-766.1(14)	5357.0(5)	1458.0(11)	17.5(2)

C9	-547.0(14)	6065.1(5)	1861.3(11)	16.8(2)
C10	663.8(14)	6315.7(5)	3264.7(11)	15.9(2)
C11	1790.8(13)	5831.8(5)	4396.9(10)	14.0(2)
C12	5129.8(14)	4266.4(5)	7802.9(11)	16.6(2)
C13	4832.9(14)	3499.4(5)	7306.4(11)	17.6(2)
C14	6050.7(16)	3001.6(6)	8576.8(12)	24.4(2)
C15	5911.7(19)	2247.6(6)	8009.6(16)	34.1(3)

Table S13. Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 4b. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + 2hka^{*}b^{*}U_{12} + \dots]$.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
F1	23.6(3)	17.5(3)	17.1(3)	5.2(2)	-2.4(2)	4.3(2)
O1	27.5(4)	15.2(4)	19.2(4)	-3.4(3)	1.9(3)	-2.4(3)
O2	23.5(4)	14.8(3)	13.9(3)	-1.3(3)	-1.1(3)	0.1(3)
C1	20.3(5)	14.6(5)	16.4(5)	0.2(4)	3.6(4)	0.1(4)
C2	15.4(5)	13.2(5)	15.0(5)	1.1(4)	5.1(4)	0.6(4)
C3	13.9(4)	16.7(5)	12.7(4)	-0.3(4)	3.3(4)	-0.9(4)
C4	14.7(4)	14.6(5)	15.5(5)	1.2(4)	5.5(4)	0.8(4)
C5	16.6(5)	11.7(4)	16.8(5)	0.7(4)	5.1(4)	0.6(4)
C6	14.6(4)	14.3(5)	16.0(5)	0.1(4)	5.4(4)	0.0(4)
C7	17.4(5)	14.0(5)	17.3(5)	-1.1(4)	4.8(4)	0.0(4)
C8	16.0(5)	19.6(5)	13.8(4)	-1.5(4)	1.9(4)	-0.7(4)
C9	15.1(4)	17.6(5)	15.6(5)	5.1(4)	3.3(4)	3.7(4)
C10	16.6(5)	12.4(4)	17.6(5)	1.1(4)	5.2(4)	0.6(4)
C11	13.6(4)	14.0(5)	14.3(4)	1.0(4)	5.0(4)	0.4(3)
C12	16.9(5)	15.8(5)	14.4(4)	2.5(4)	2.7(4)	1.7(4)
C13	18.2(5)	14.6(5)	19.3(5)	3.5(4)	6.1(4)	2.1(4)
C14	26.0(5)	19.8(5)	24.9(5)	8.3(4)	6.7(4)	4.9(4)
C15	39.1(7)	17.2(6)	44.0(7)	10.2(5)	12.9(6)	7.9(5)

Table S14. Bond Lengths for 4b.

Atom	Atom	Length/ \AA	Atom	Atom	Length/ \AA
F1	C9	1.3605(11)	C6	C7	1.4144(14)
O1	C1	1.2372(13)	C6	C11	1.4199(14)
O2	C3	1.3393(12)	C7	C8	1.3726(14)
C1	C2	1.4459(14)	C8	C9	1.3946(14)
C2	C3	1.3993(13)	C9	C10	1.3669(14)
C2	C11	1.4454(13)	C10	C11	1.4154(13)
C3	C4	1.4251(14)	C12	C13	1.5249(13)
C4	C5	1.3670(14)	C13	C14	1.5228(13)
C4	C12	1.5081(13)	C14	C15	1.5219(16)
C5	C6	1.4212(14)			

Table S15. Bond Angles for 4b.

Atom	Atom	Atom	Angle/ [°]	Atom	Atom	Atom	Angle/ [°]
O1	C1	C2	124.14(9)	C11	C6	C5	119.58(9)
C3	C2	C1	118.67(9)	C8	C7	C6	121.22(9)
C3	C2	C11	119.52(9)	C7	C8	C9	117.34(9)
C11	C2	C1	121.79(9)	F1	C9	C8	117.49(8)
O2	C3	C2	121.60(9)	F1	C9	C10	118.08(9)
O2	C3	C4	115.84(9)	C10	C9	C8	124.43(9)
C2	C3	C4	122.56(9)	C9	C10	C11	118.65(9)
C3	C4	C12	118.74(9)	C6	C11	C2	117.86(9)
C5	C4	C3	116.92(9)	C10	C11	C2	123.73(9)
C5	C4	C12	124.34(9)	C10	C11	C6	118.40(9)
C4	C5	C6	123.54(9)	C4	C12	C13	115.23(8)
C7	C6	C5	120.46(9)	C14	C13	C12	112.64(8)
C7	C6	C11	119.96(9)	C15	C14	C13	111.97(9)

Table S16. Hydrogen Bonds for 4b.

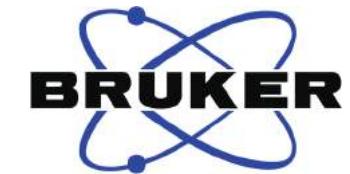
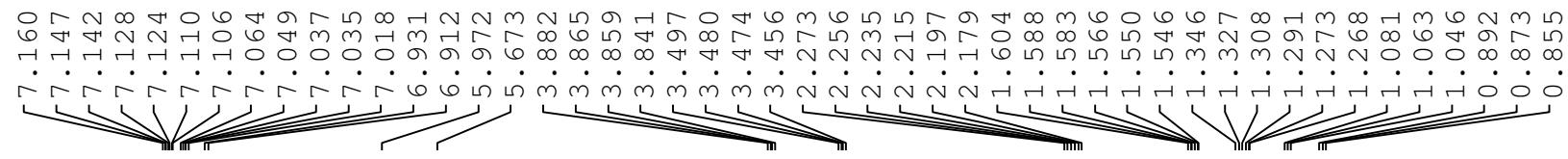
D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/ [°]
O2	H2	O1	0.84	1.80	2.5457(9)	147.6

Table S17. Torsion Angles for 4b.

A	B	C	D	Angle/ [°]	A	B	C	D	Angle/ [°]
F1	C9	C10	C11	-179.70(7)	C5	C4	C12	C13	0.80(14)
O1	C1	C2	C3	-1.14(15)	C5	C6	C7	C8	179.72(8)
O1	C1	C2	C11	-179.63(9)	C5	C6	C11	C2	1.27(13)
O2	C3	C4	C5	-178.74(8)	C5	C6	C11	C10	-179.29(8)
O2	C3	C4	C12	0.89(13)	C6	C7	C8	C9	-0.19(14)
C1	C2	C3	O2	1.19(14)	C7	C6	C11	C2	-178.86(8)
C1	C2	C3	C4	-178.42(8)	C7	C6	C11	C10	0.58(13)
C1	C2	C11	C6	177.29(8)	C7	C8	C9	F1	-179.86(8)
C1	C2	C11	C10	-2.12(14)	C7	C8	C9	C10	0.08(15)
C2	C3	C4	C5	0.89(14)	C8	C9	C10	C11	0.36(15)
C2	C3	C4	C12	-179.49(8)	C9	C10	C11	C2	178.73(8)
C3	C2	C11	C6	-1.18(13)	C9	C10	C11	C6	-0.68(13)
C3	C2	C11	C10	179.41(8)	C11	C2	C3	O2	179.71(8)
C3	C4	C5	C6	-0.81(14)	C11	C2	C3	C4	0.11(14)
C3	C4	C12	C13	-178.80(8)	C11	C6	C7	C8	-0.14(14)
C4	C5	C6	C7	179.86(8)	C12	C4	C5	C6	179.59(8)
C4	C5	C6	C11	-0.27(14)	C12	C13	C14	C15	-173.71(9)
C4	C12	C13	C14	180.00(8)					

Table S18. Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 4b.

Atom	x	y	z	U(eq)
H2	5513	6116	8478	30
H1	2814	7109	5637	22
H5	2536	4125	4880	18
H7	192	4397	2314	20
H8	-1626	5208	466	21
H10	747	6805	3476	19
H12A	4785	4324	8725	20
H12B	6534	4381	8113	20
H13A	3434	3378	7004	21
H13B	5186	3435	6390	21
H14A	7434	3153	8962	29
H14B	5596	3025	9449	29
H15A	6450	2216	7197	51
H15B	4540	2101	7595	51
H15C	6656	1941	8871	51



Current Data Parameters
 NAME wuan-67
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151011
 Time 4.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 8
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 287
 DW 60.800 usec
 DE 6.00 usec
 TE 295.6 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300438 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.04
1.99
1.01

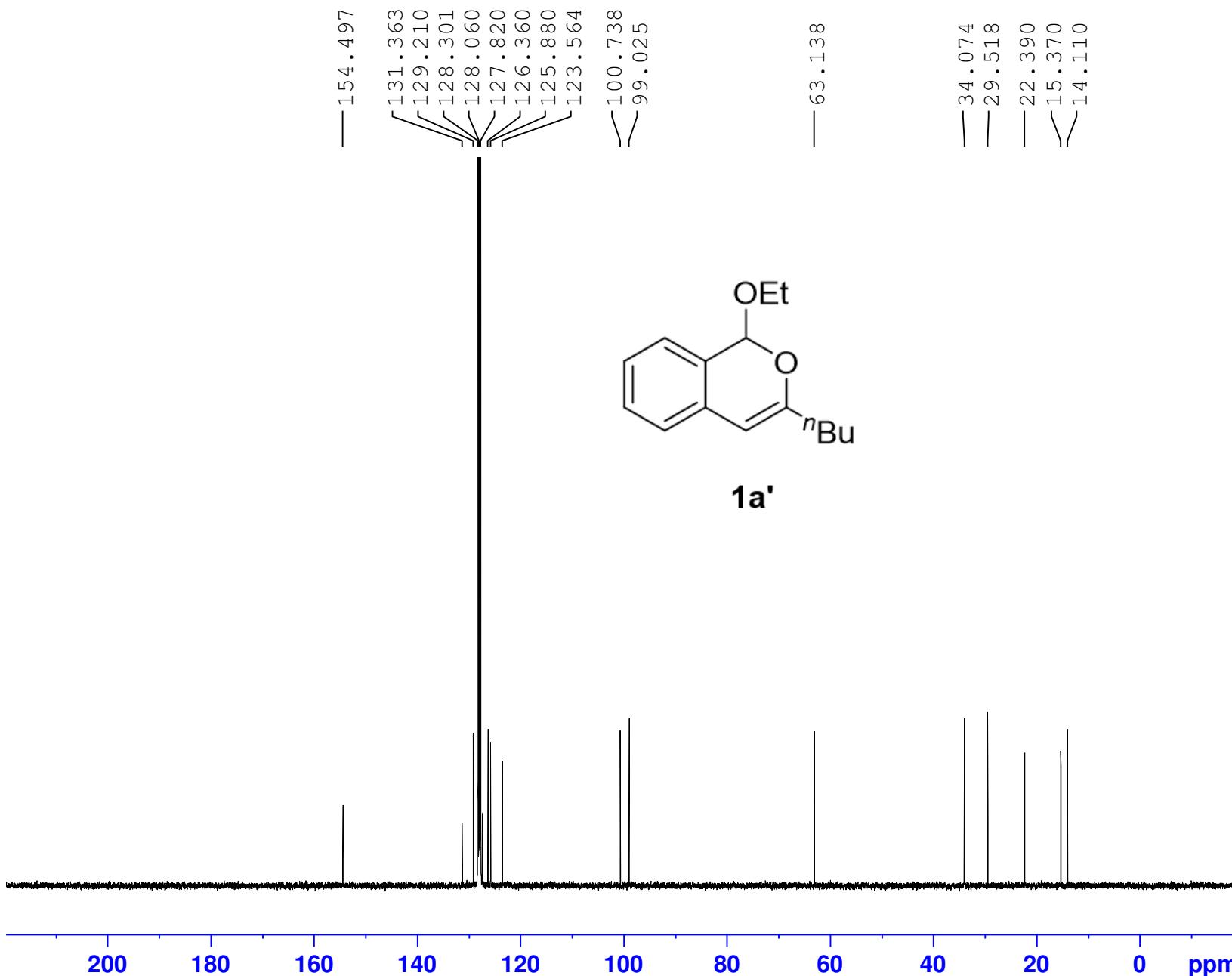
0.98
0.98

1.00
1.01

2.03

2.04
2.18

3.02
3.09



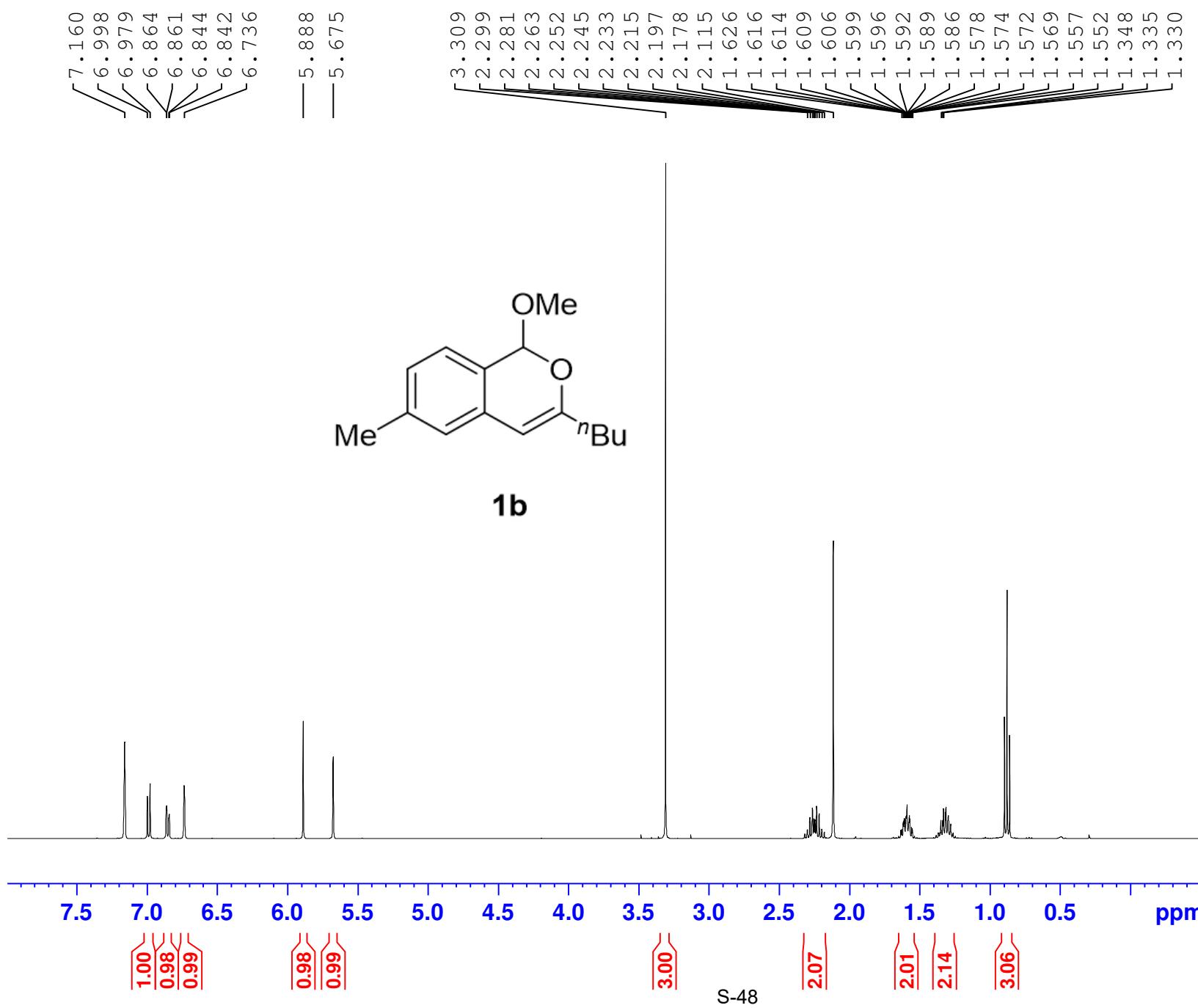
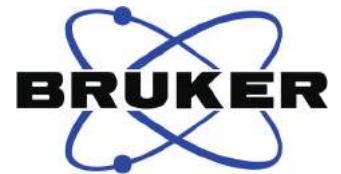
Current Data Parameters
 NAME wuan-67
 EXPNO 2
 PROCNO 1

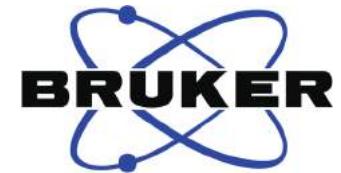
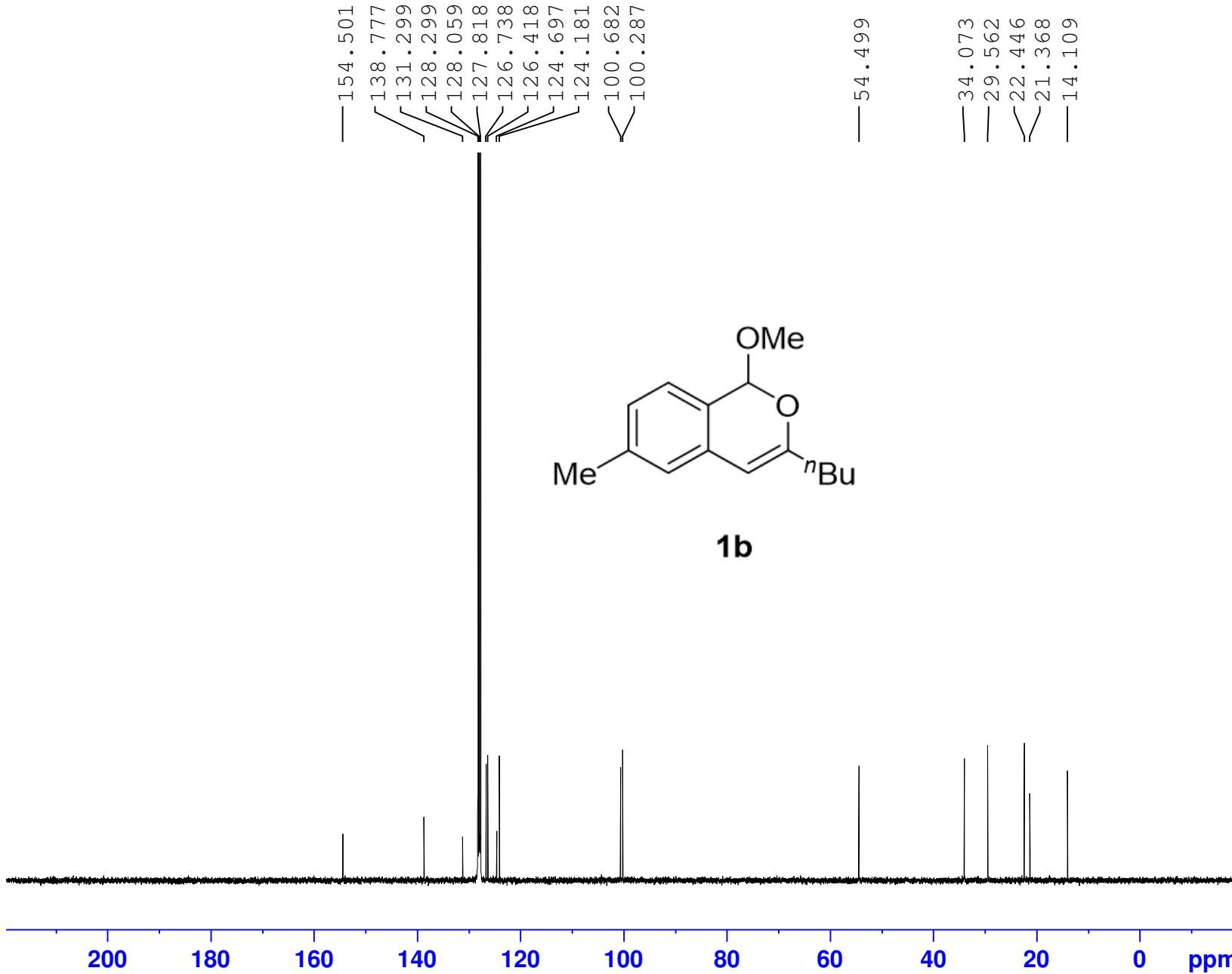
F2 - Acquisition Parameters
 Date_ 20151222
 Time 23.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 150
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 295.7 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.8999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 ^{13}C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 ^1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127430 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





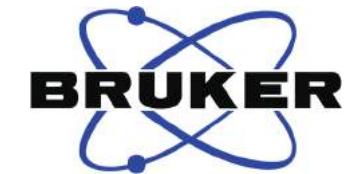
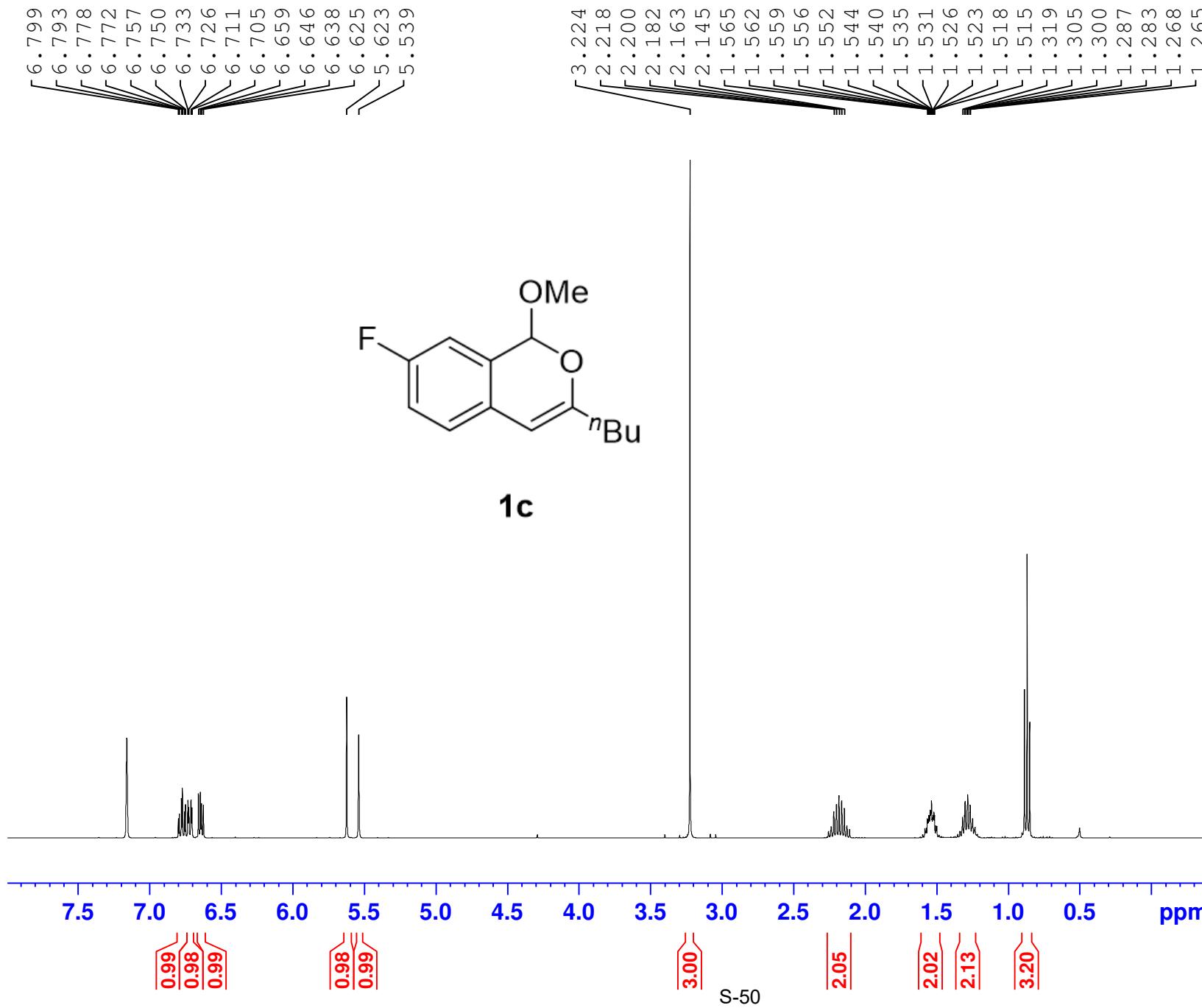
Current Data Parameters
 NAME wuan-106
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 3.26
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 125
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.4 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.8999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127430 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

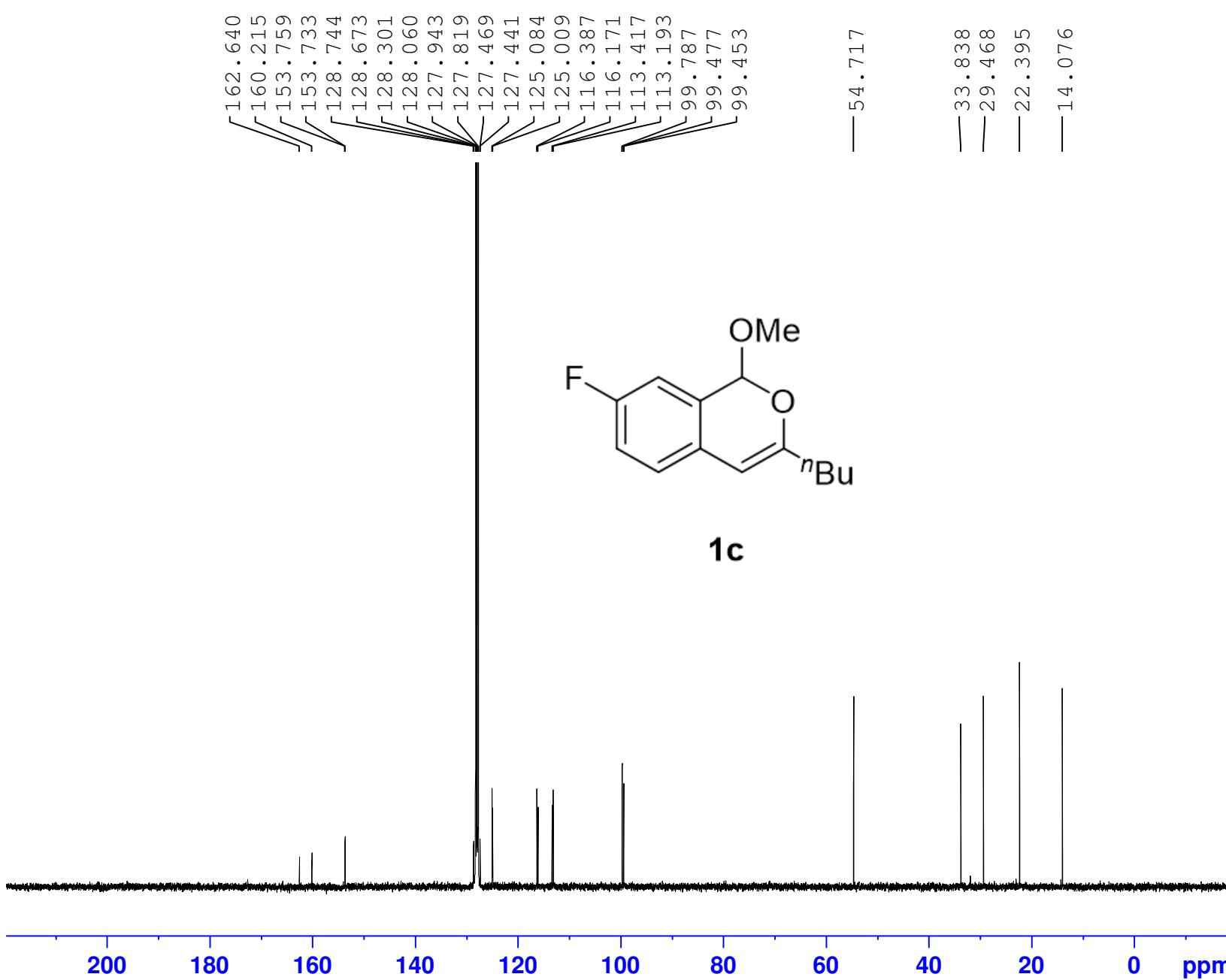


Current Data Parameters
 NAME wuan-107
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151222
 Time 22.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 49.32
 DW 62.400 usec
 DE 6.50 usec
 TE 297.2 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1299965 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



S-51



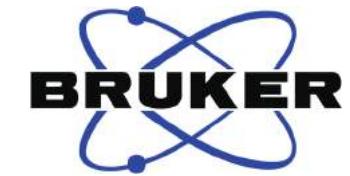
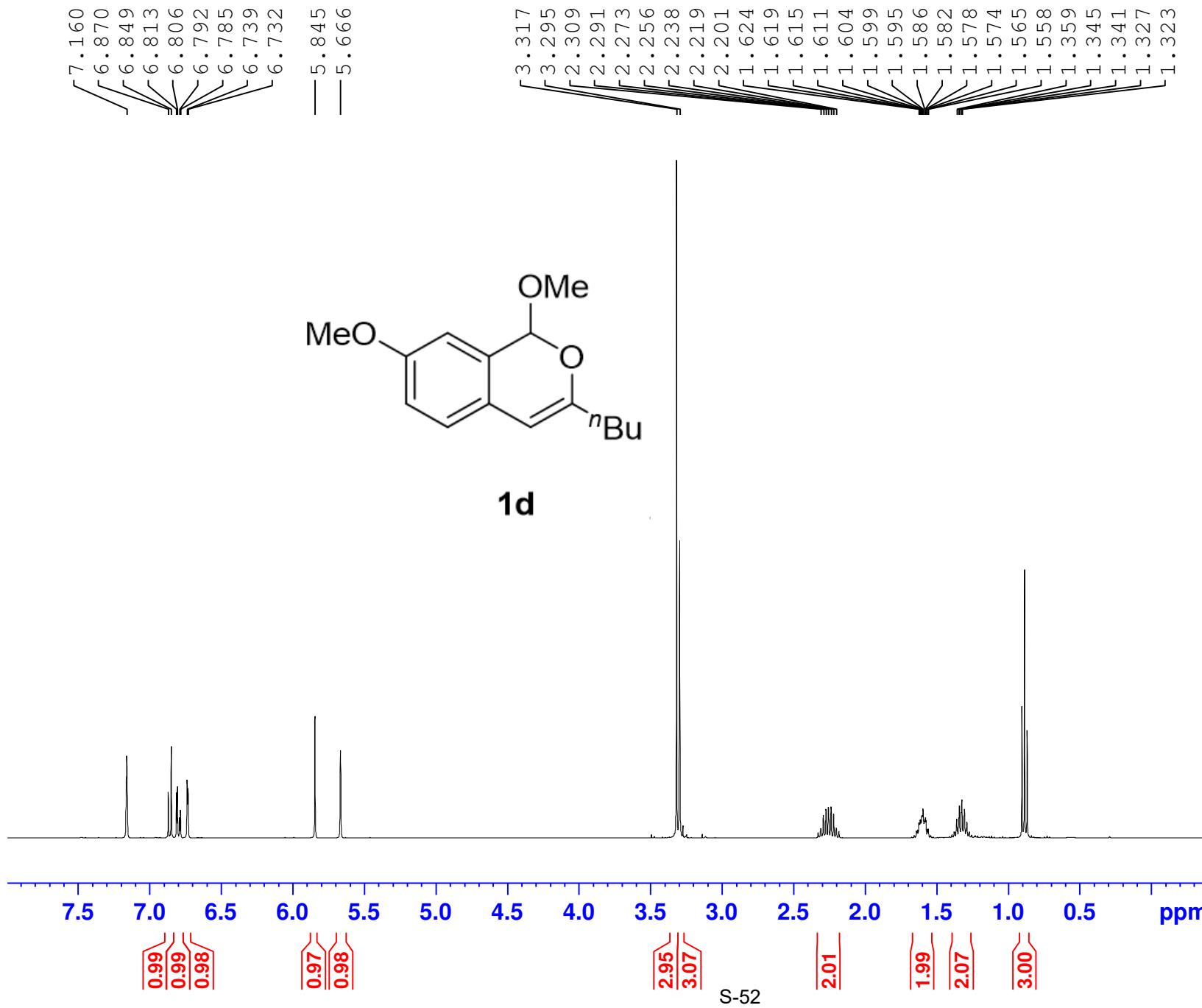
Current Data Parameters
 NAME wuan-107
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151222
 Time 23.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 154
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 295.3 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.8999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127429 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

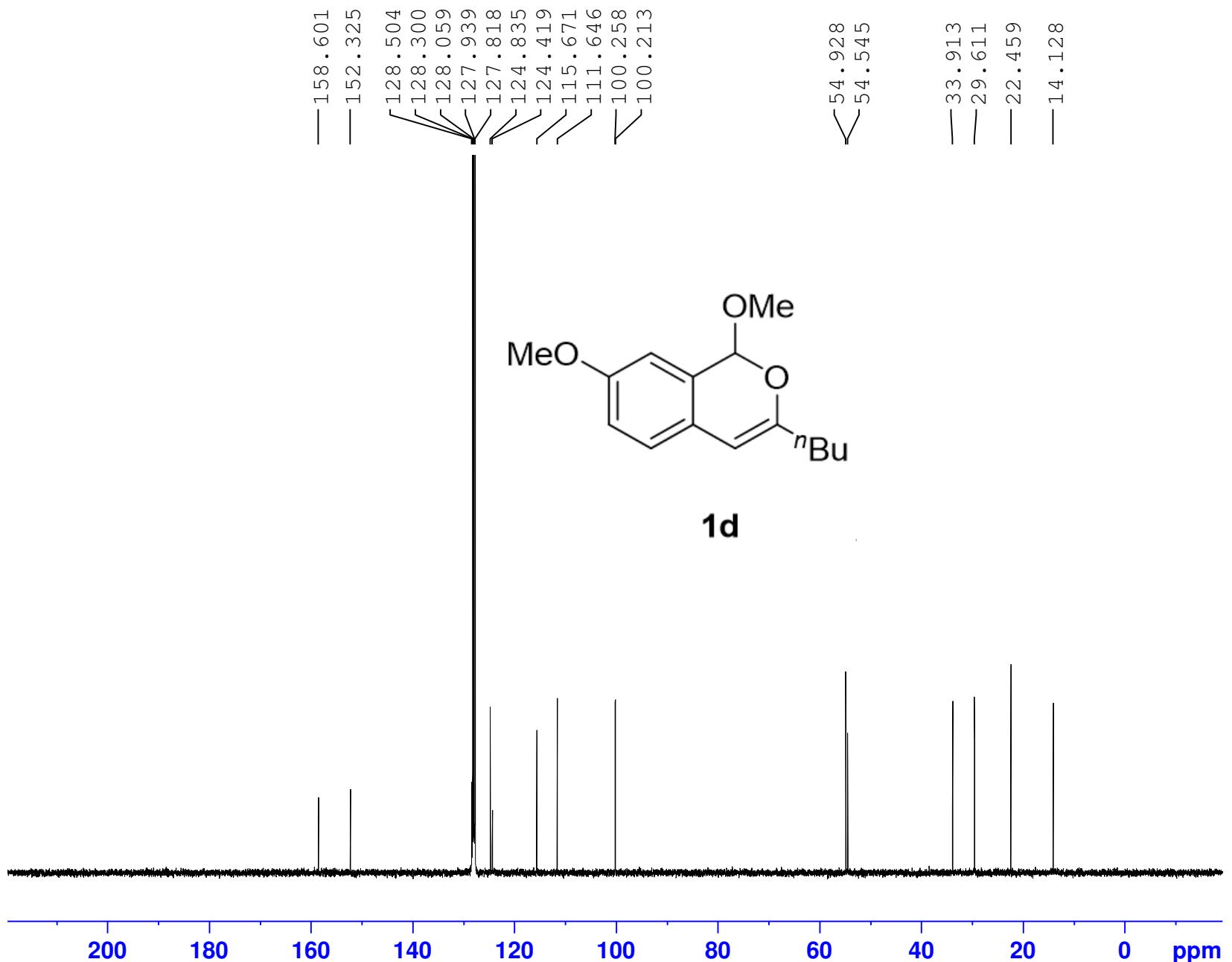


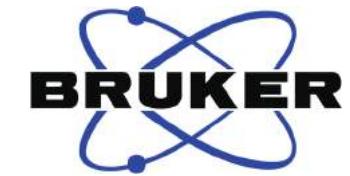
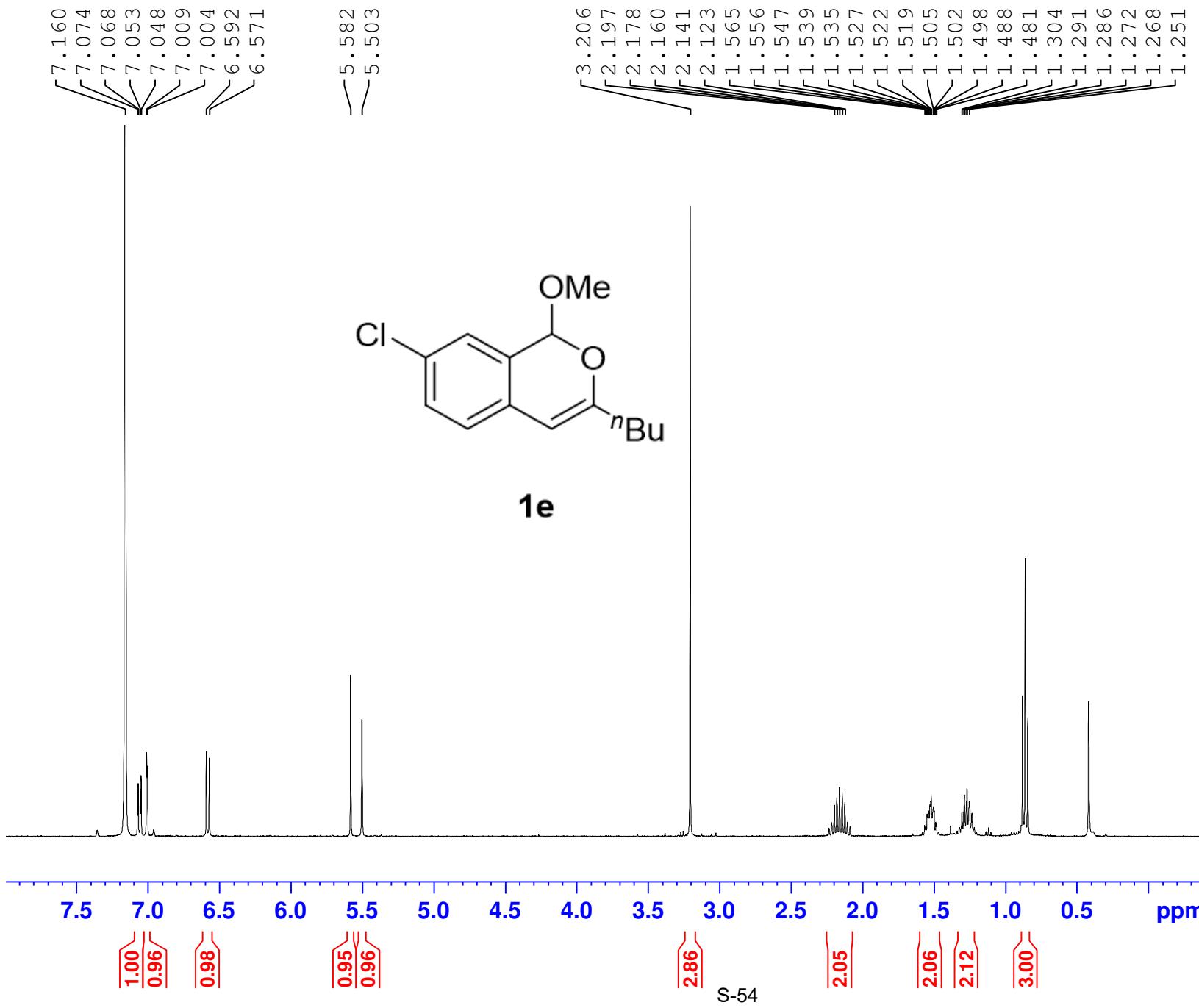
Current Data Parameters
 NAME wuan-108
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 3.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 128
 DW 60.800 usec
 DE 6.00 usec
 TE 295.7 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 ======
 NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300435 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



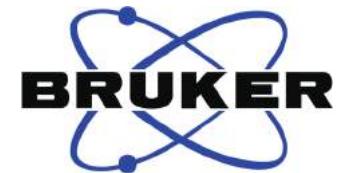
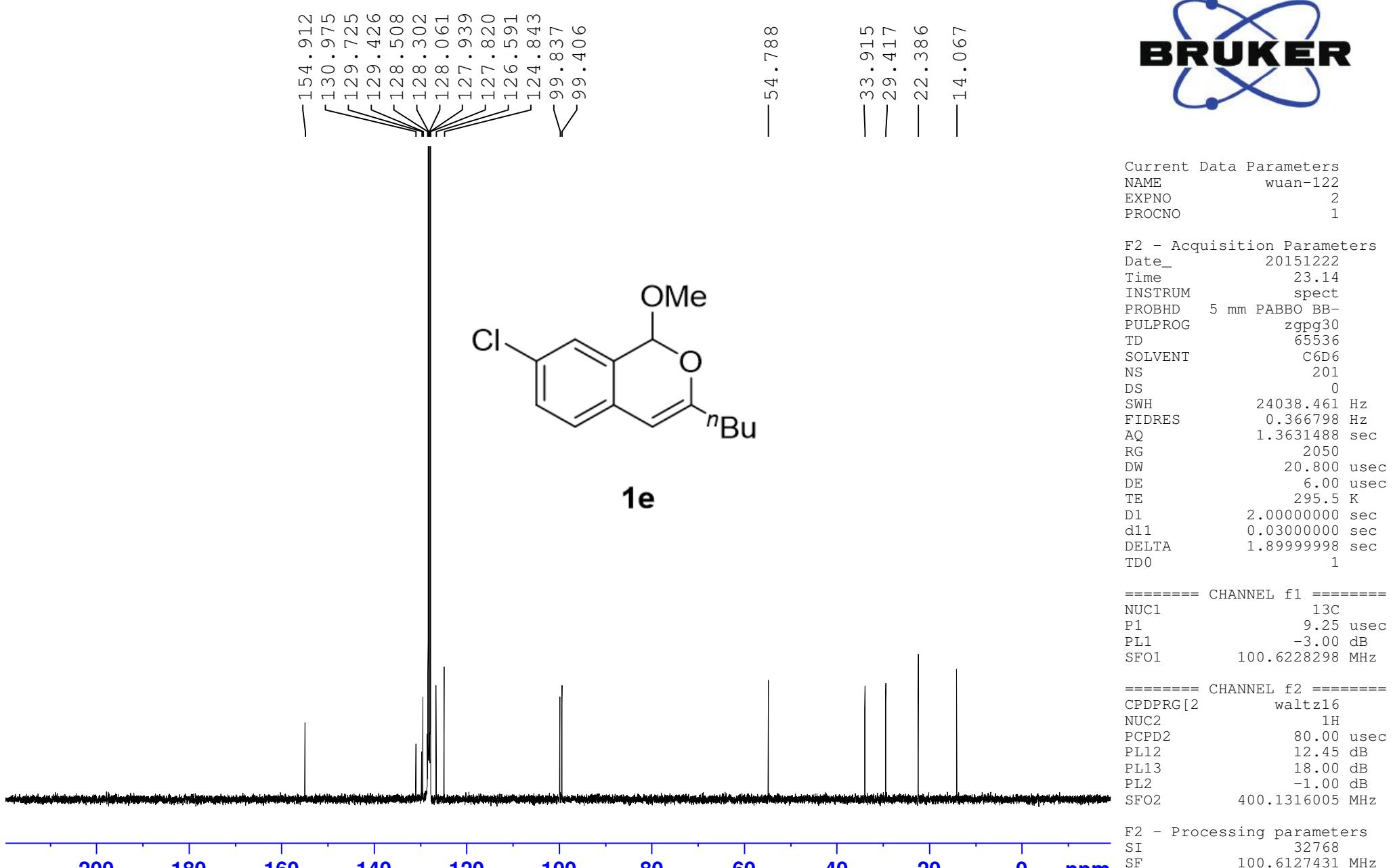


Current Data Parameters
 NAME wuan-122
 EXPNO 1
 PROCNO 1

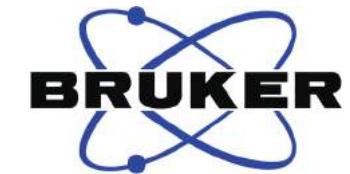
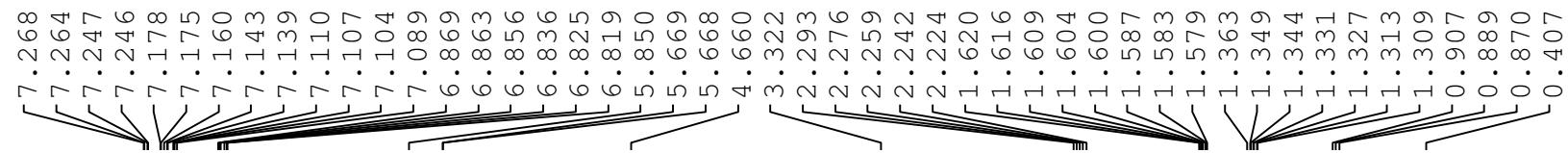
F2 - Acquisition Parameters
 Date_ 20151119
 Time 22.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 456
 DW 60.800 usec
 DE 6.00 usec
 TE 294.5 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 ======
 NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300439 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



200 180 160 140 120 100 80 60 40 20 0 ppm

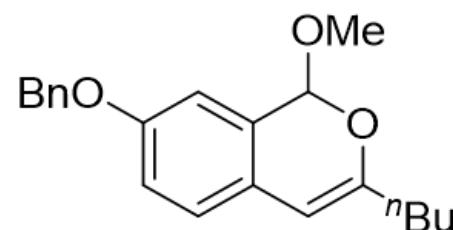


Current Data Parameters
NAME wuan-148-1
EXPNO 3
PROCNO 1

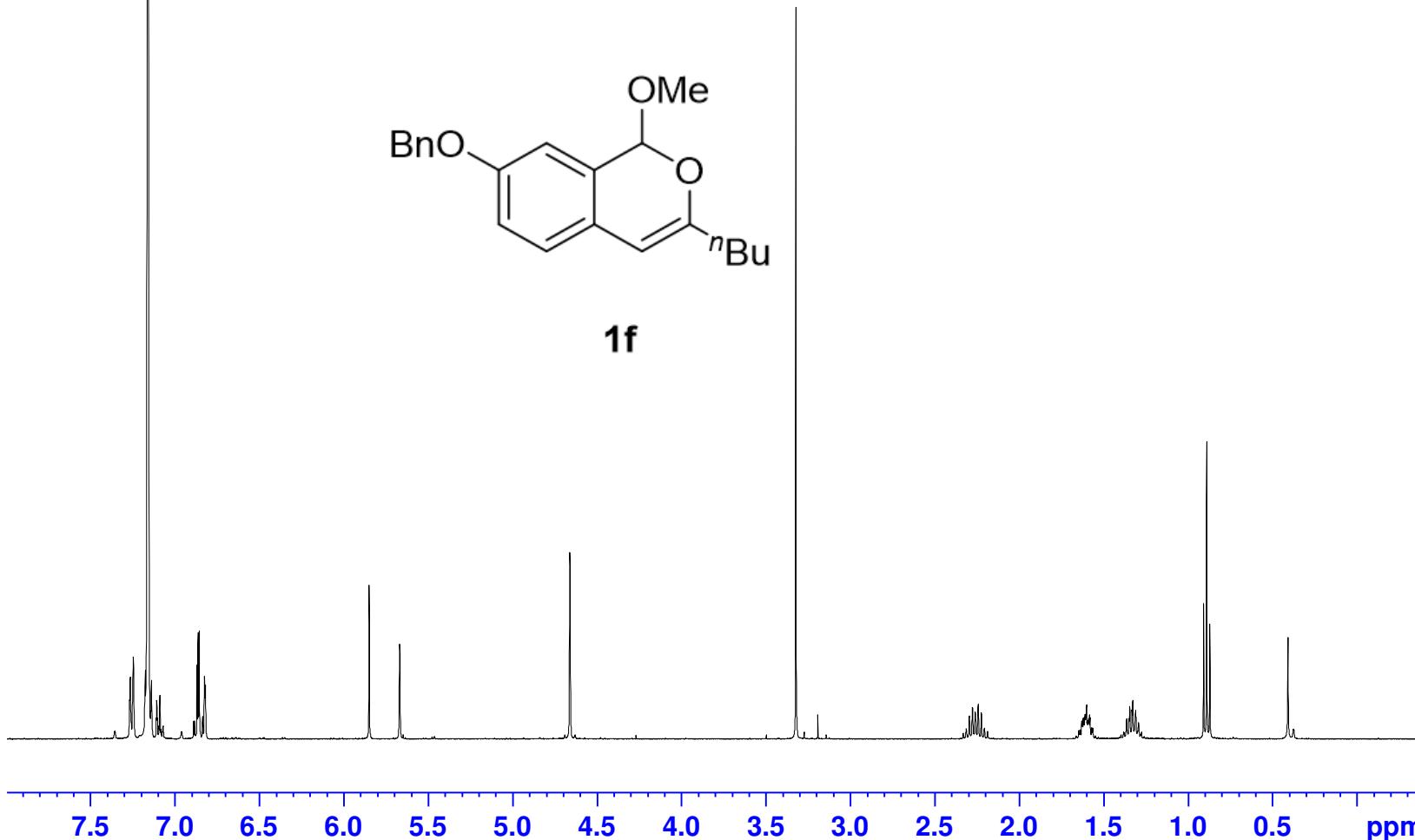
F2 - Acquisition Parameters
Date_ 20151223
Time 7.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 4
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 512
DW 60.800 usec
DE 6.00 usec
TE 296.0 K
D1 1.0000000 sec
TD0 1

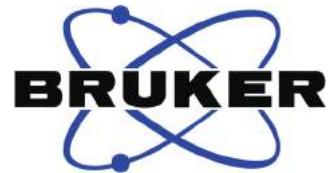
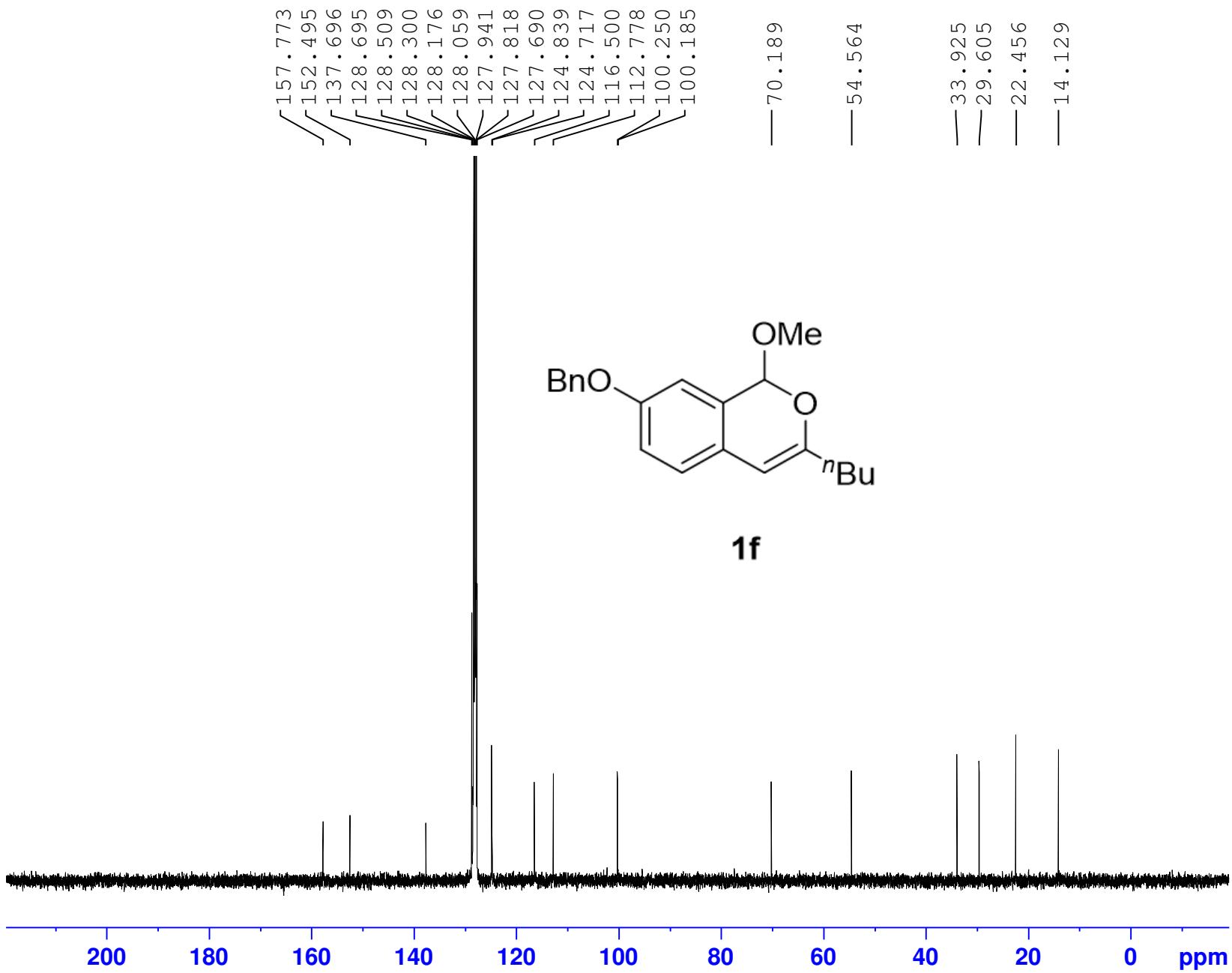
===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300435 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



1f





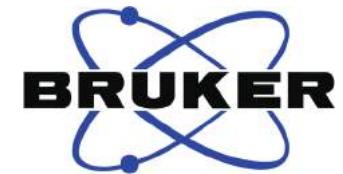
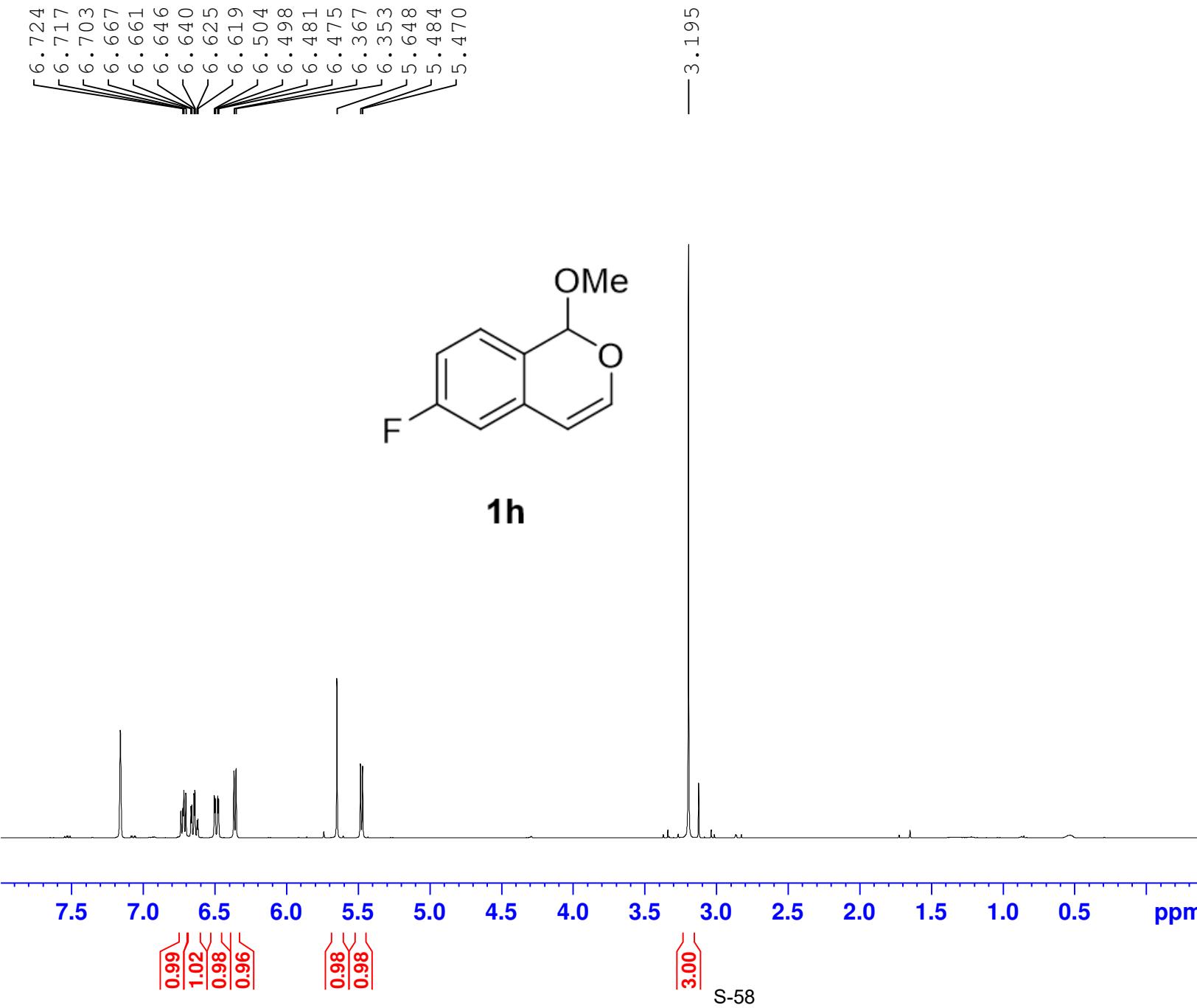
Current Data Parameters
 NAME wuan-148-1
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 8.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 1117
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.7 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127434 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



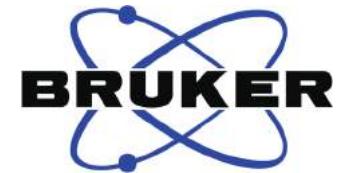
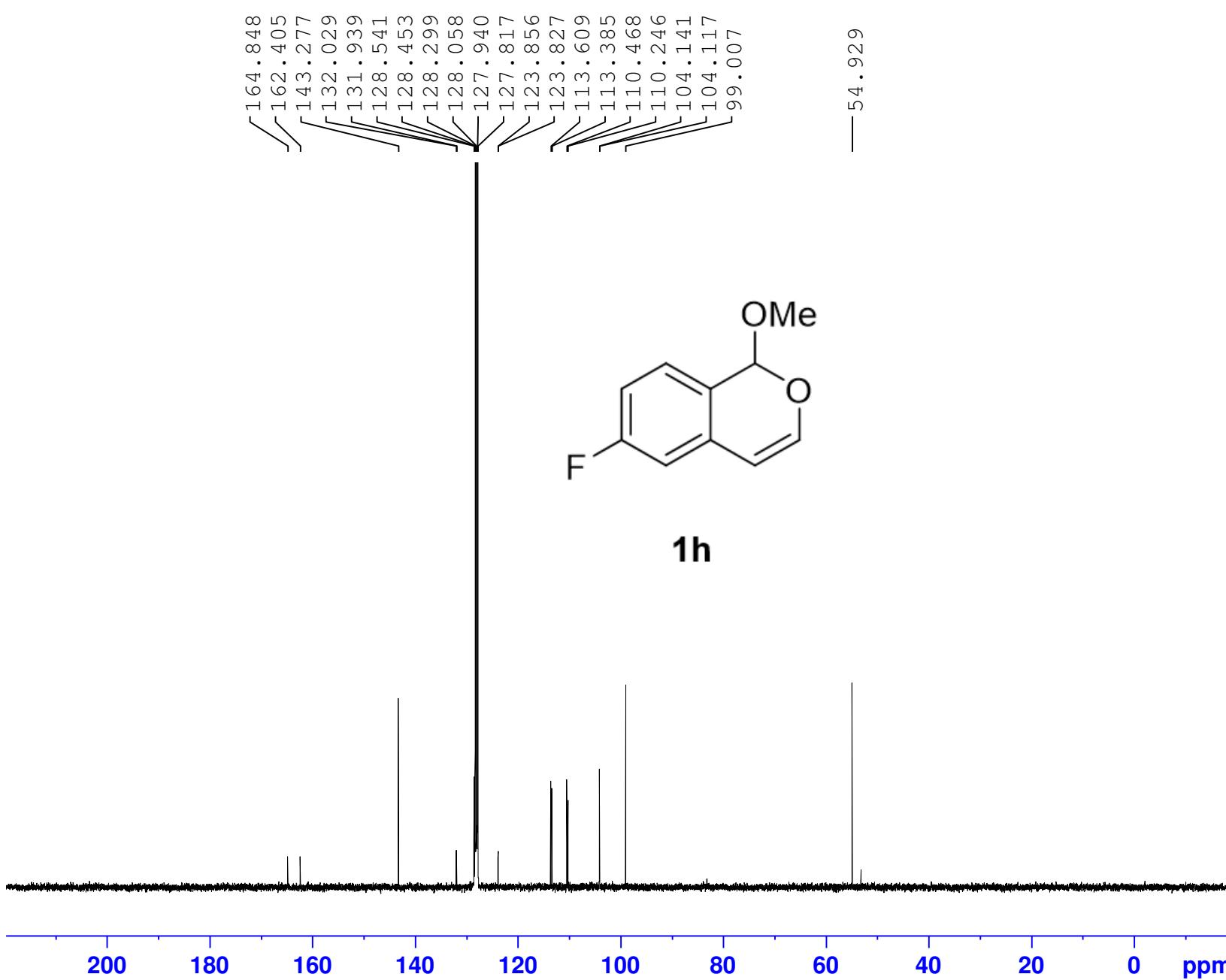
Current Data Parameters
 NAME wuan-85-1
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 0.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 203
 DW 60.800 usec
 DE 6.00 usec
 TE 295.3 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 ======

NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300437 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



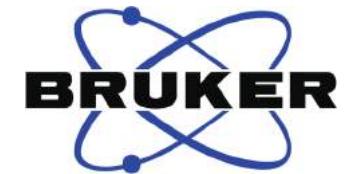
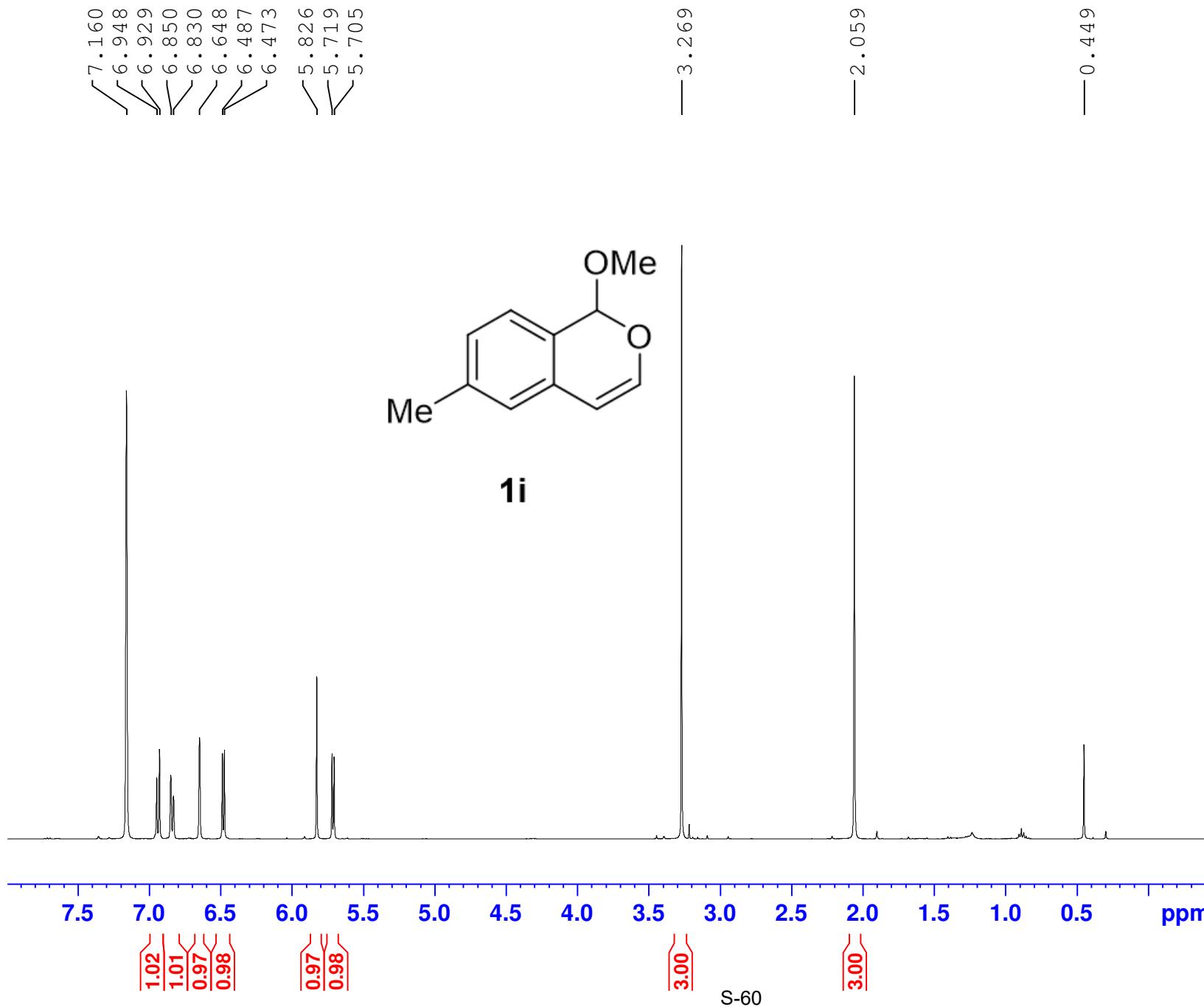
Current Data Parameters
 NAME wuan-85-1
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 0.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 161
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.0 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127435 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

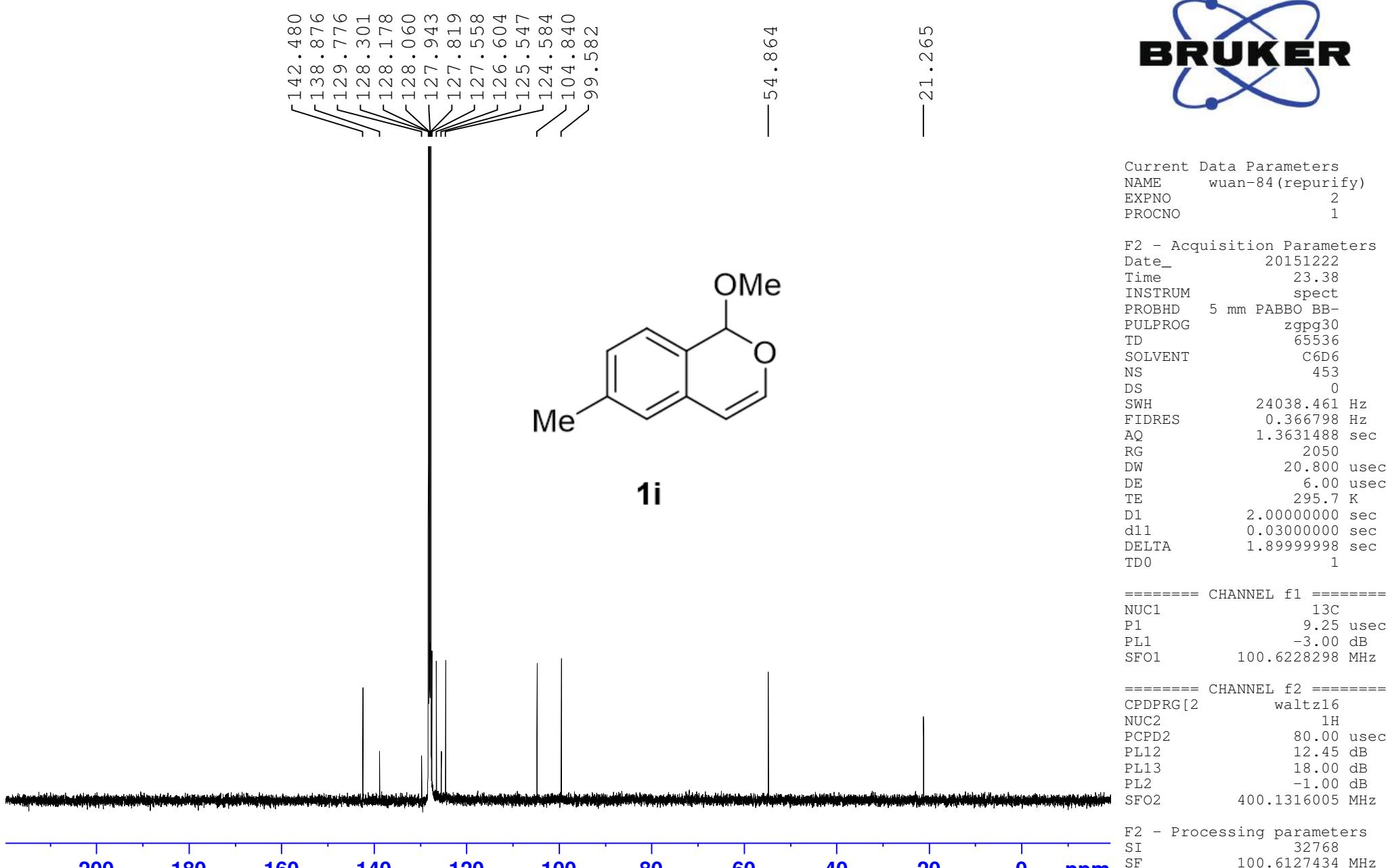


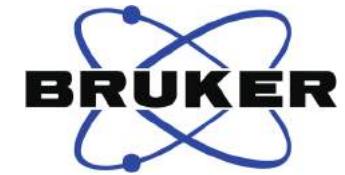
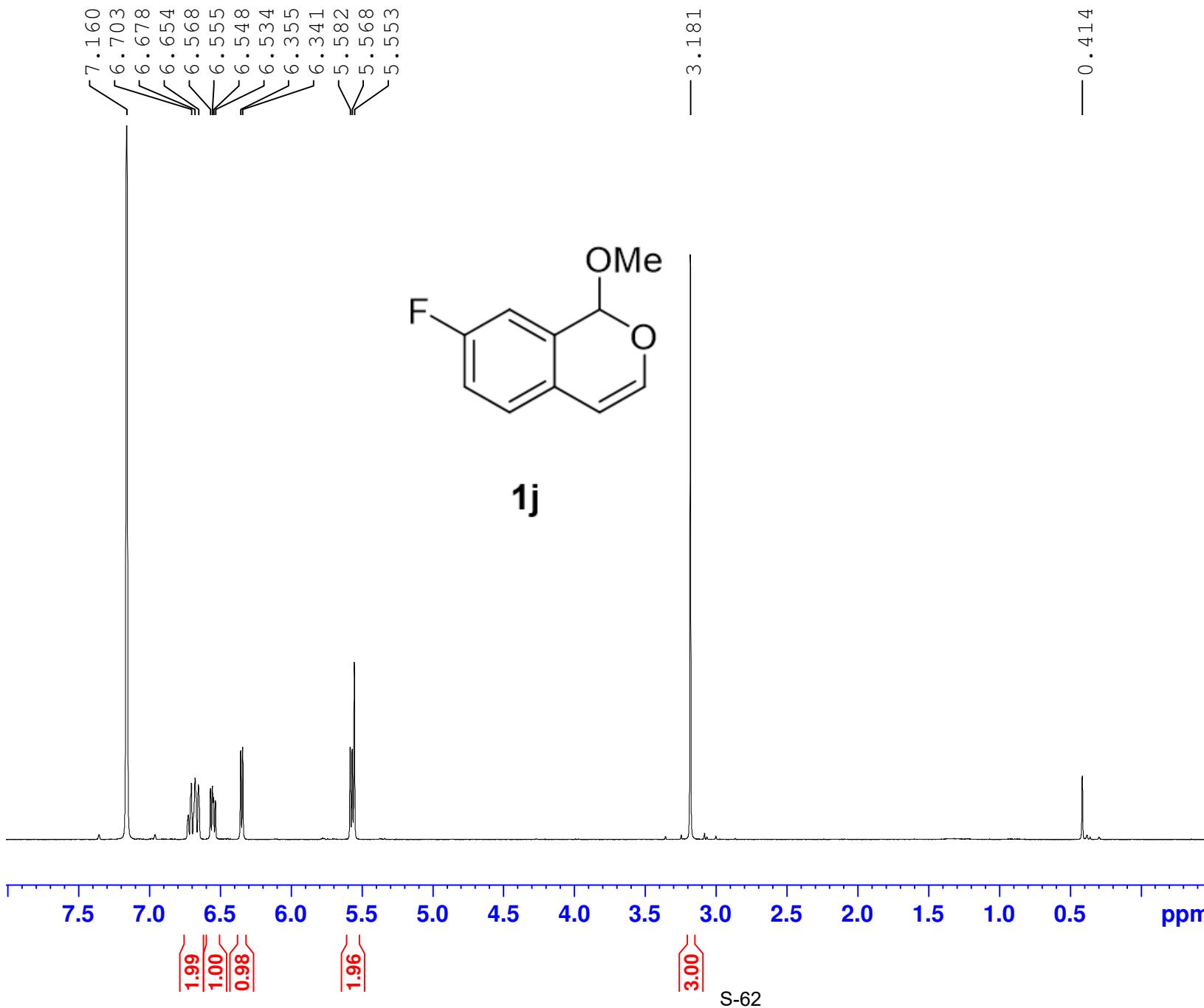
Current Data Parameters
 NAME wuan-84(repurify)
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151028
 Time 23.43
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 103.52
 DW 62.400 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1299965 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



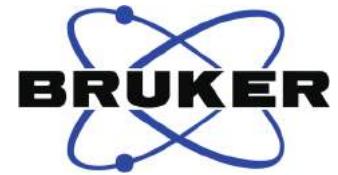
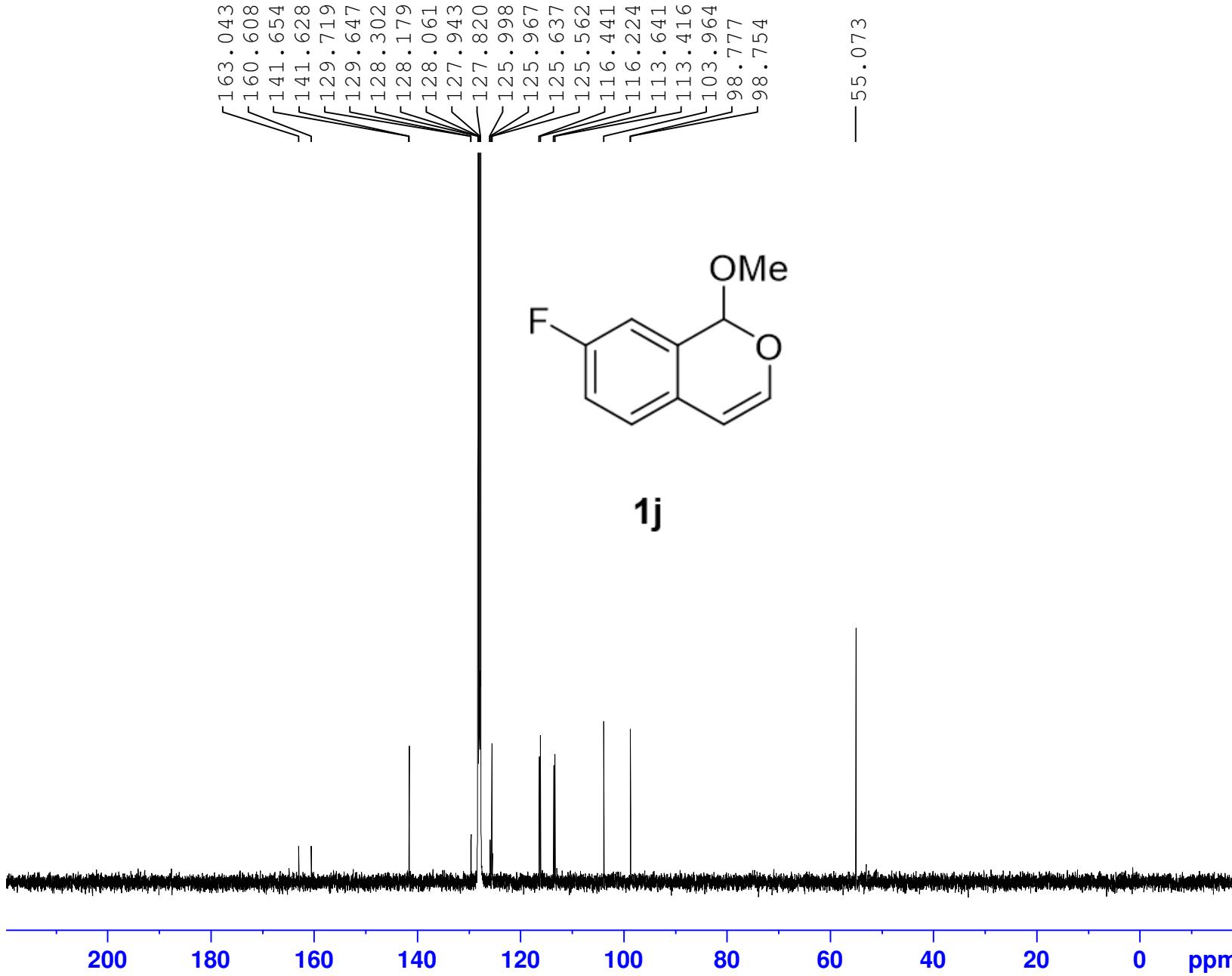


Current Data Parameters
 NAME wuan-165-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160215
 Time 22.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1299962 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



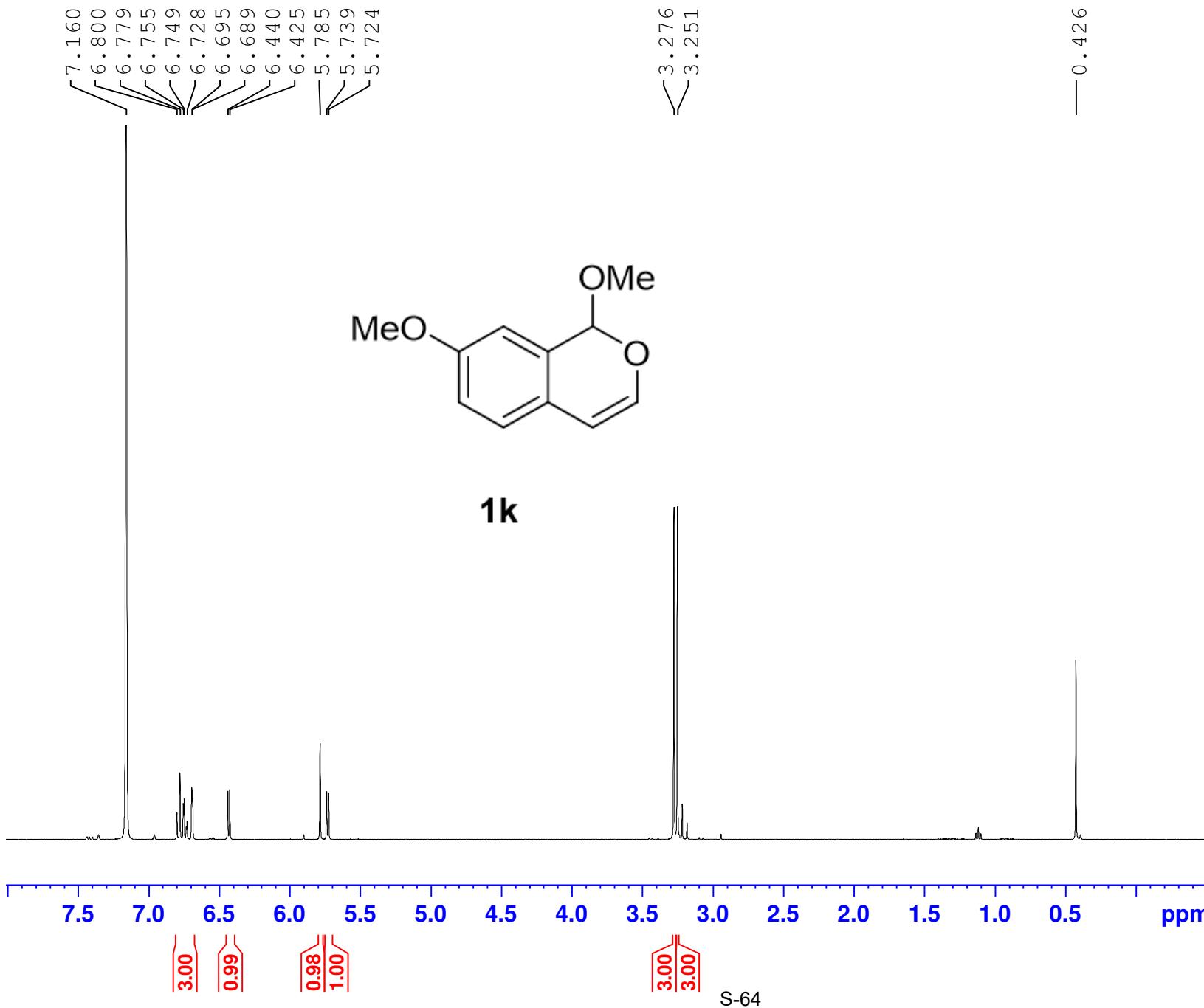
Current Data Parameters
 NAME wuan-86
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 0.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 710
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.1 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127431 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

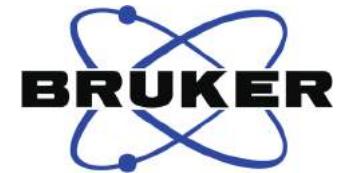
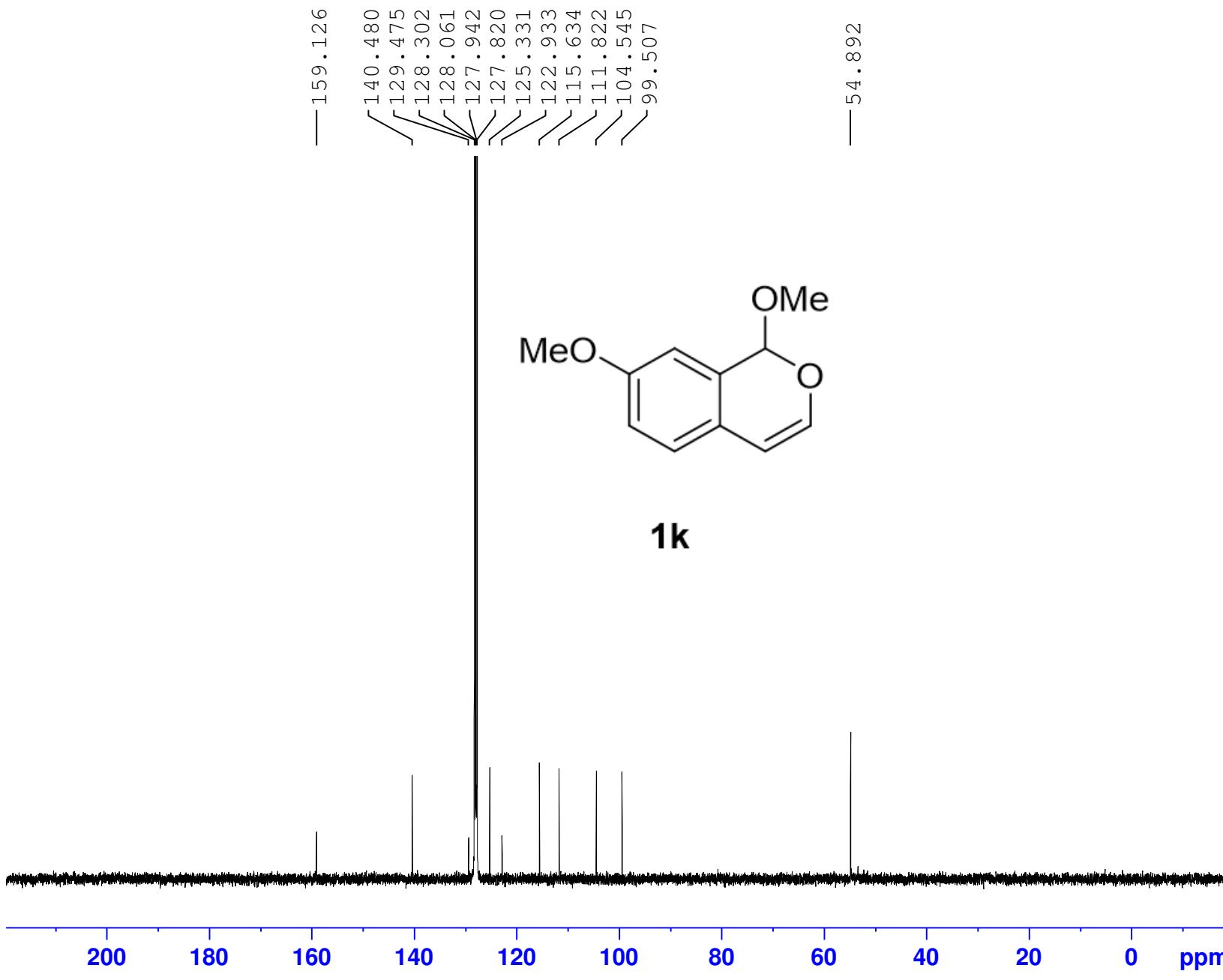


Current Data Parameters
 NAME wuan-87
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151029
 Time 23.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 297.4 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1299965 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



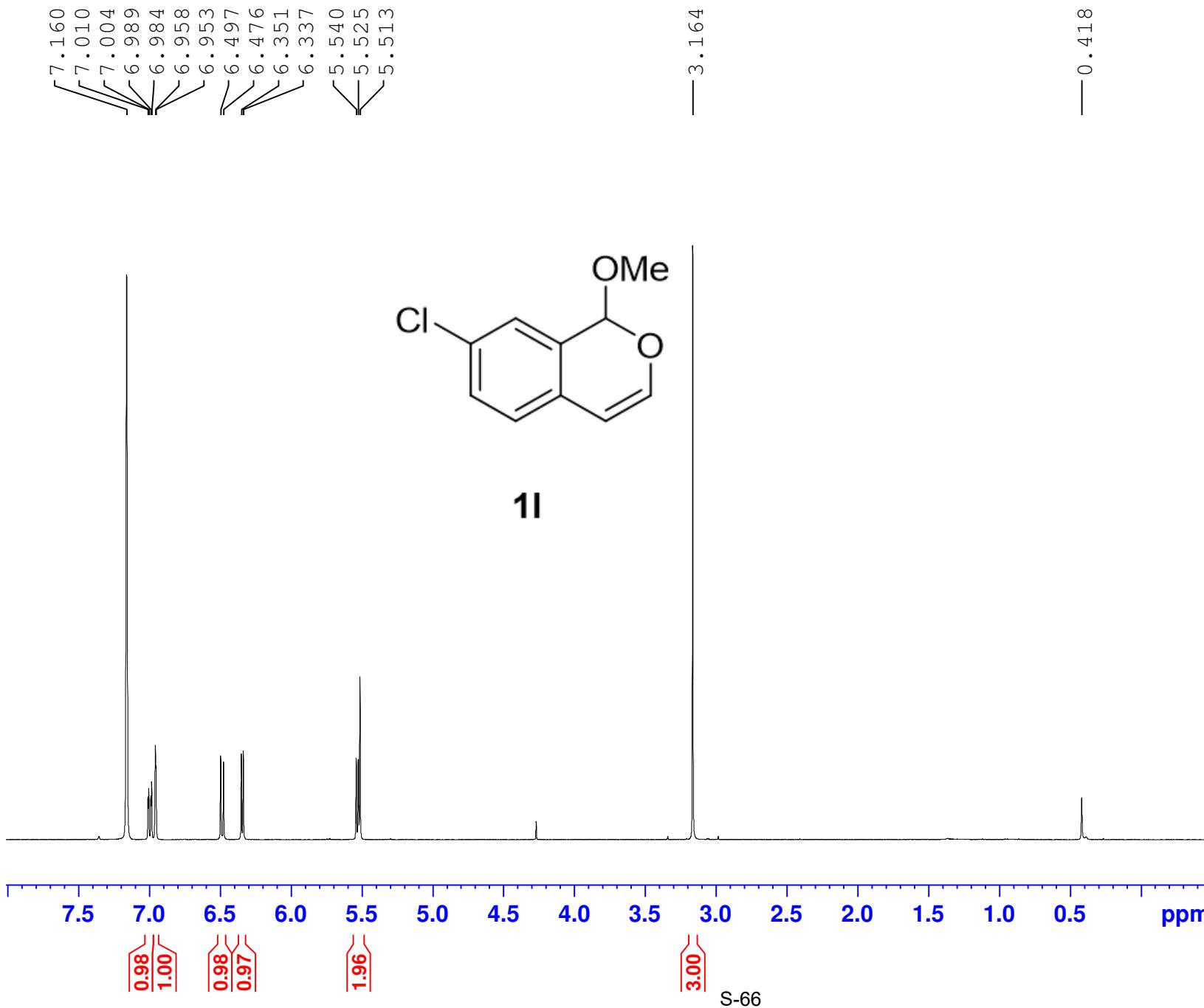
Current Data Parameters
 NAME wuan-87
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 4.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 224
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.4 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.8999999 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127431 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

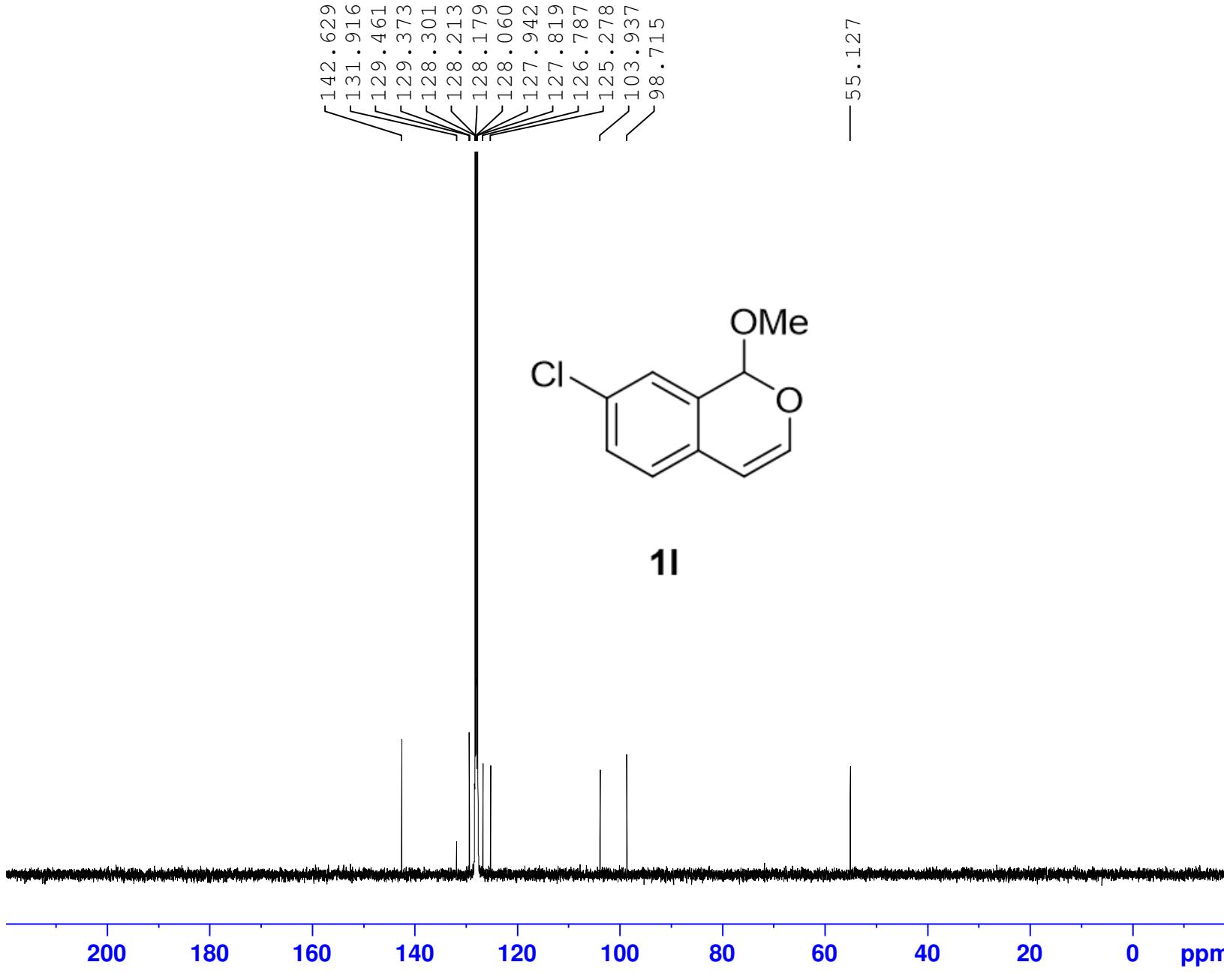


Current Data Parameters
 NAME wuan-121-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151209
 Time 21.15
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.6 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300283 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



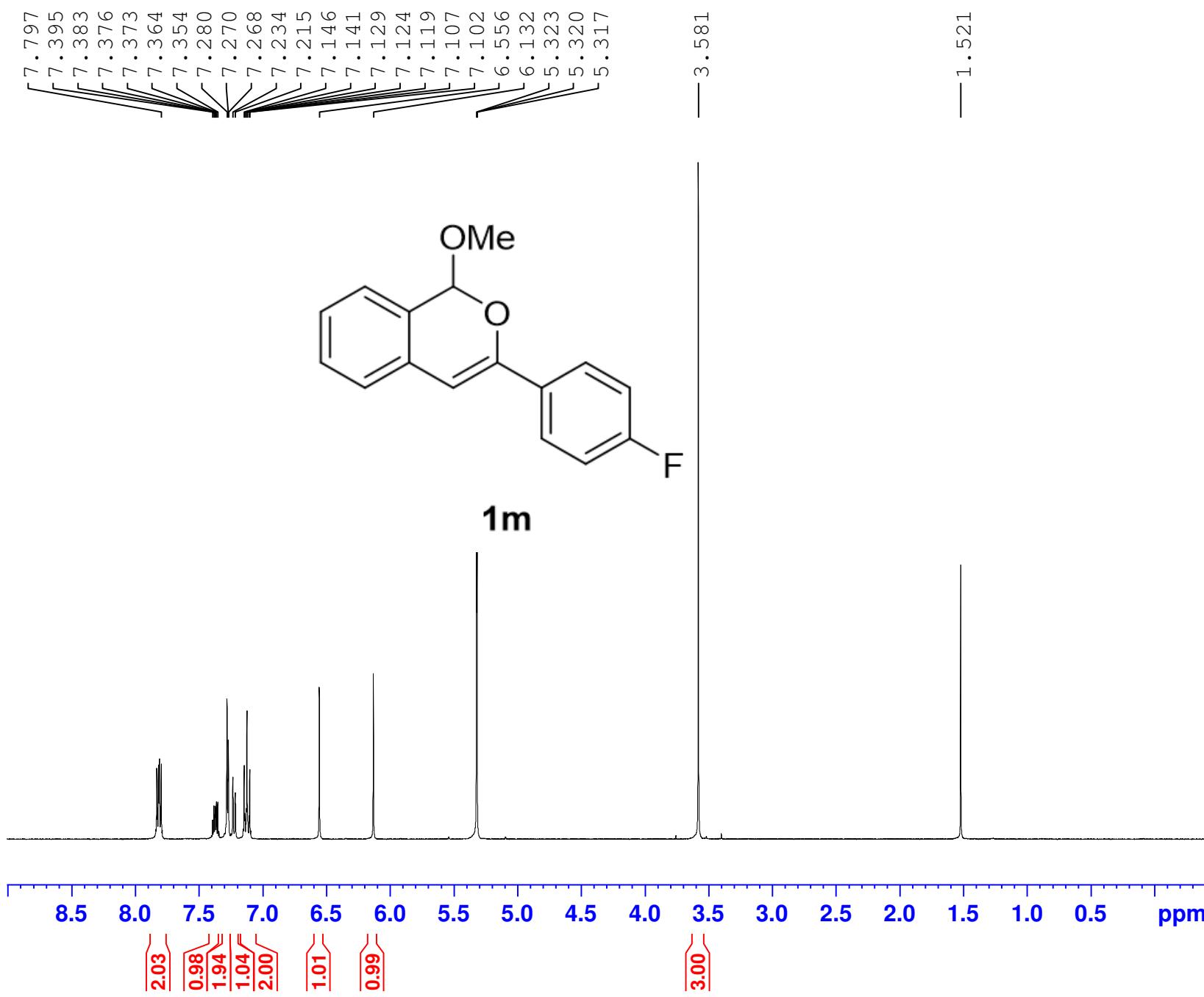
Current Data Parameters
 NAME wuan-121-1
 EXPNO 2
 PROCNO 1

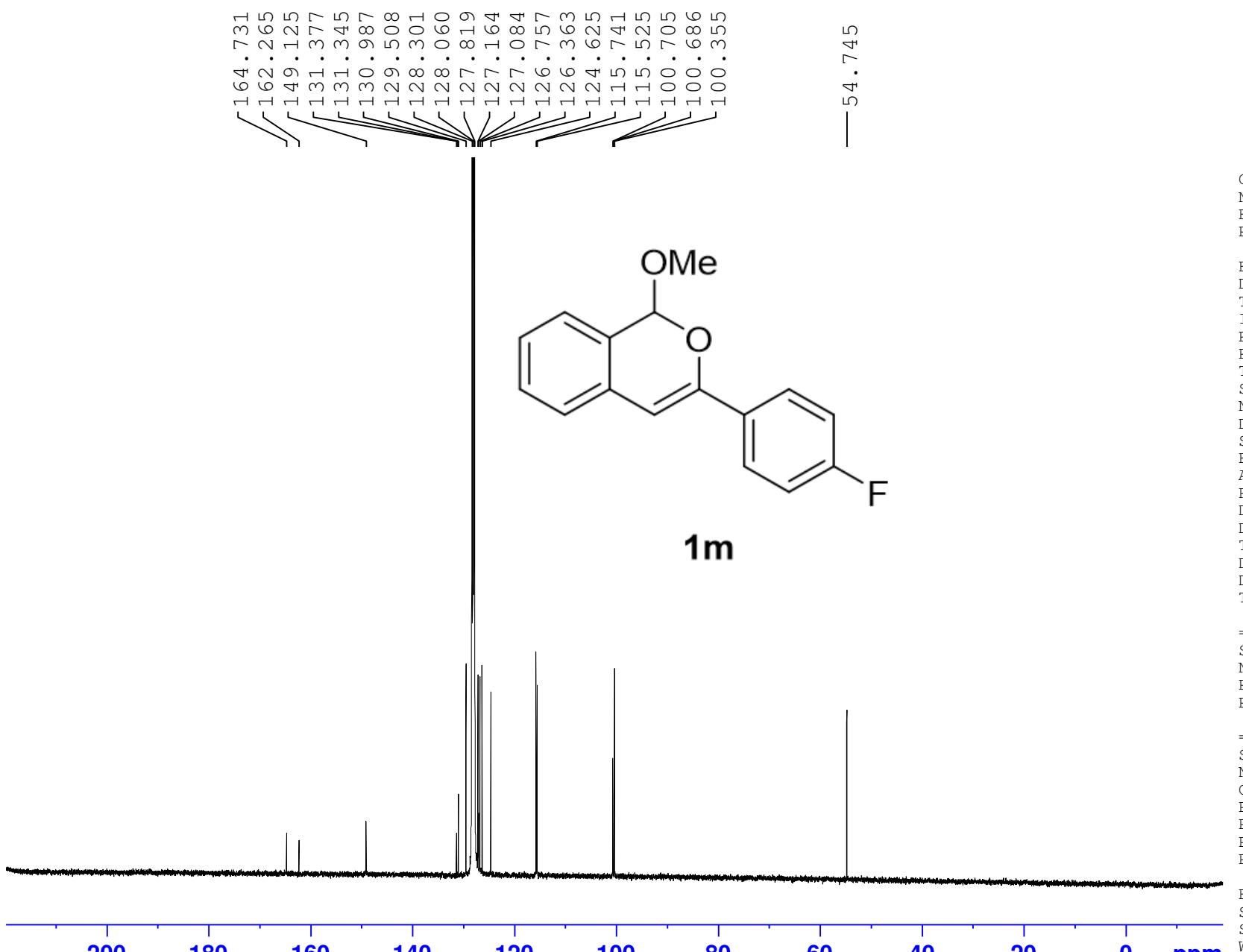
F2 - Acquisition Parameters
 Date_ 20151209
 Time 21.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 325
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.1 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

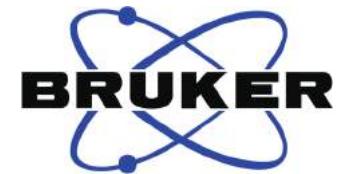
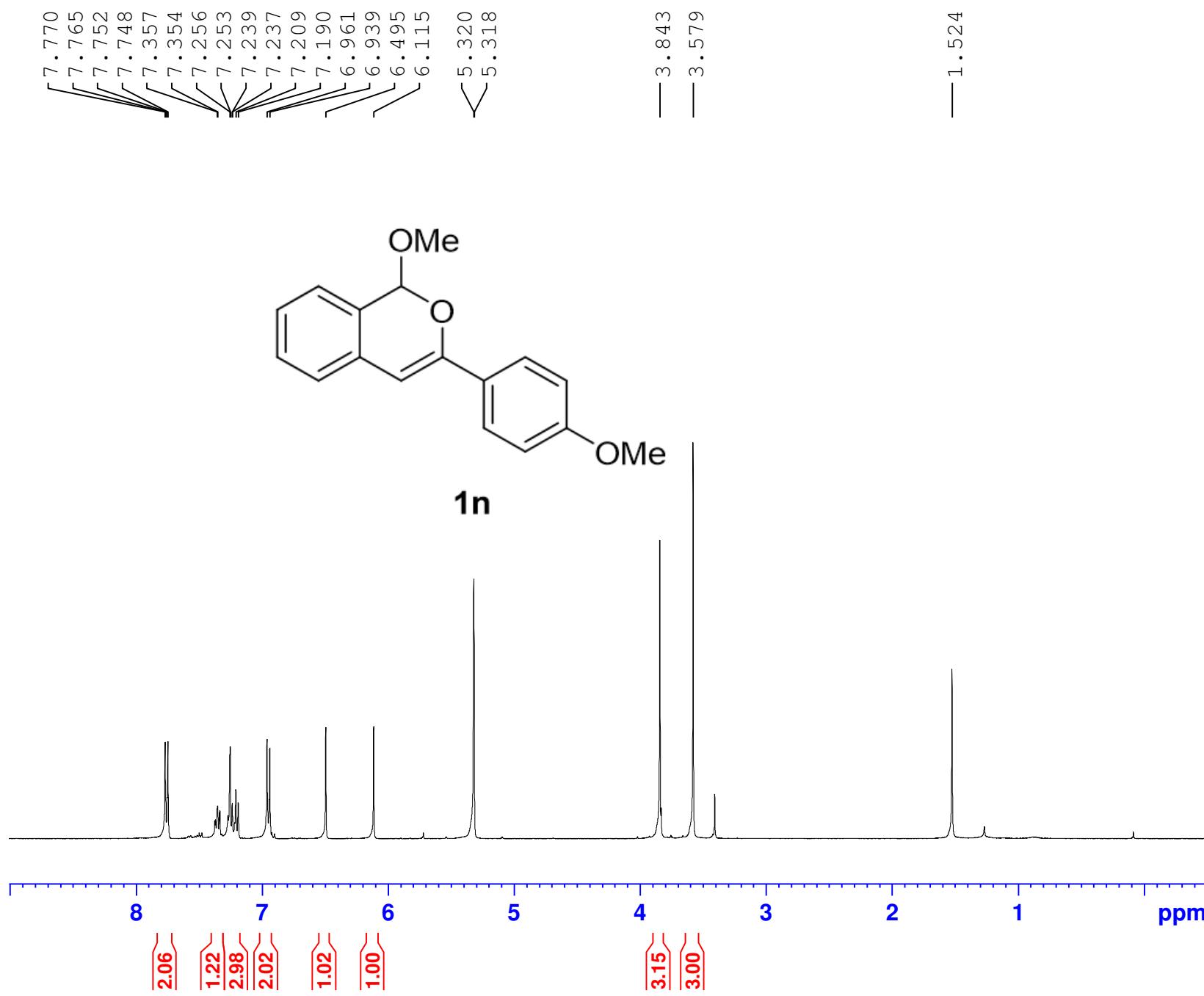
===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127392 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





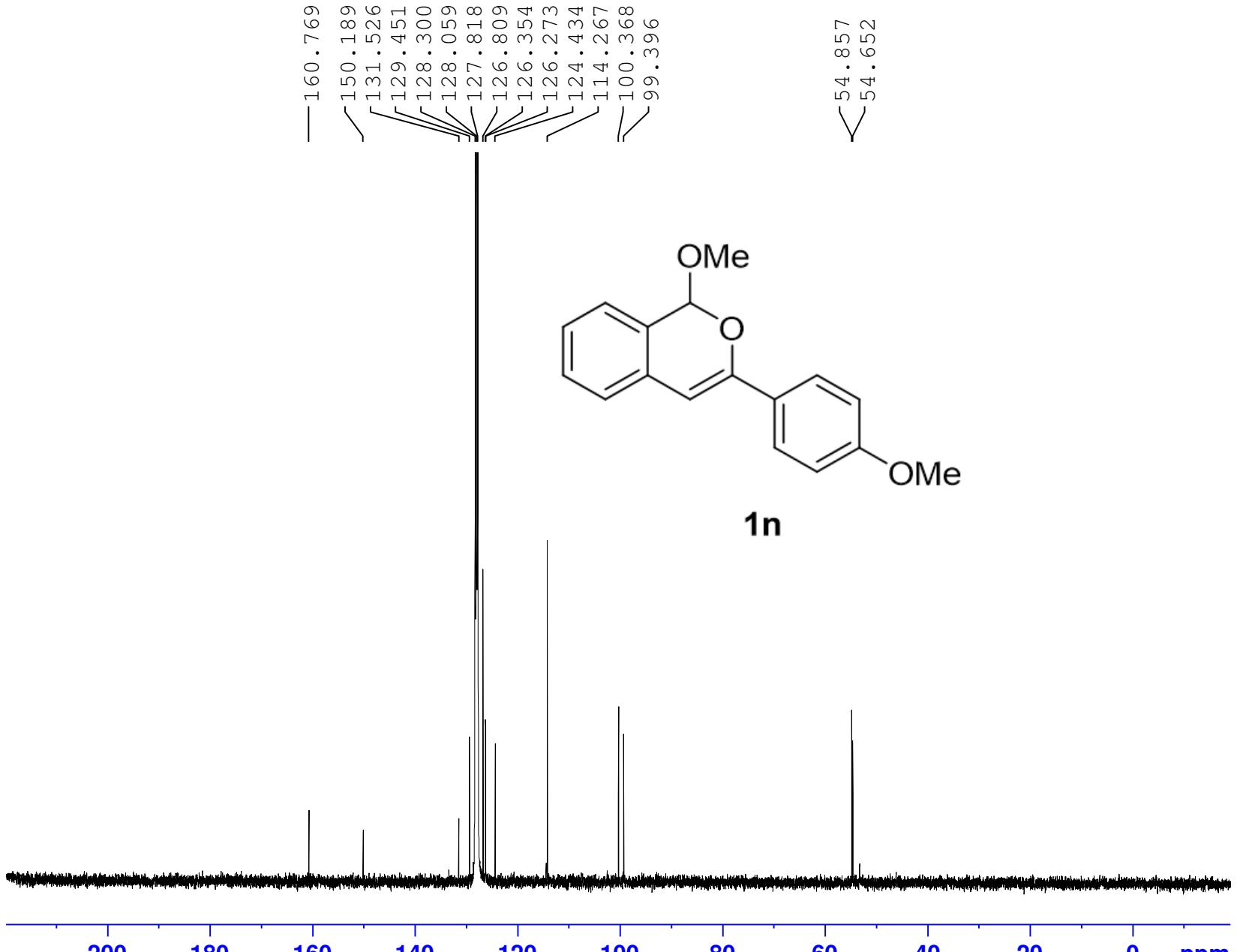


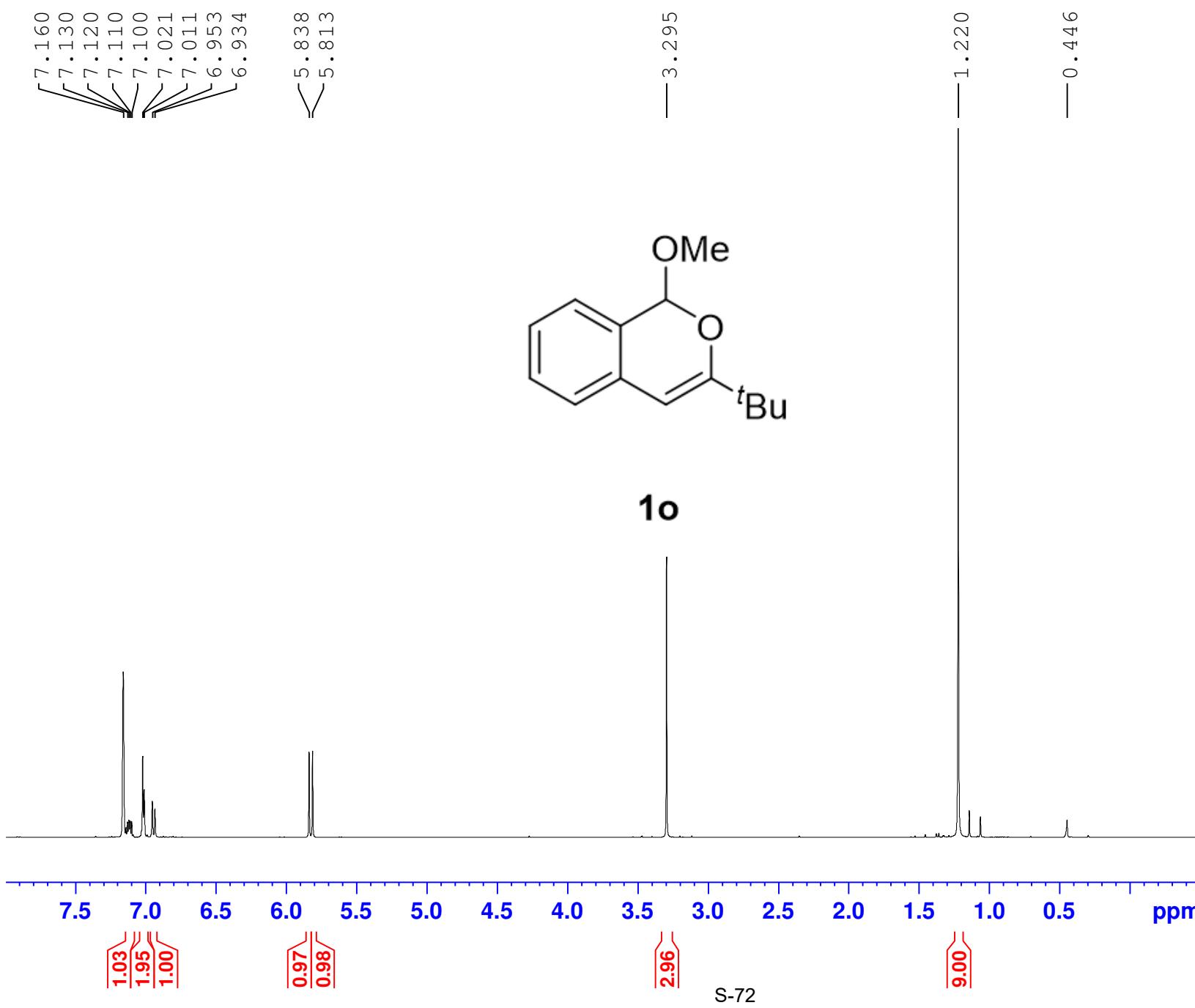
Current Data Parameters
 NAME wuan-188-8
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160901
 Time 23.38
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CD2C12
 NS 16
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 299.7 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300153 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



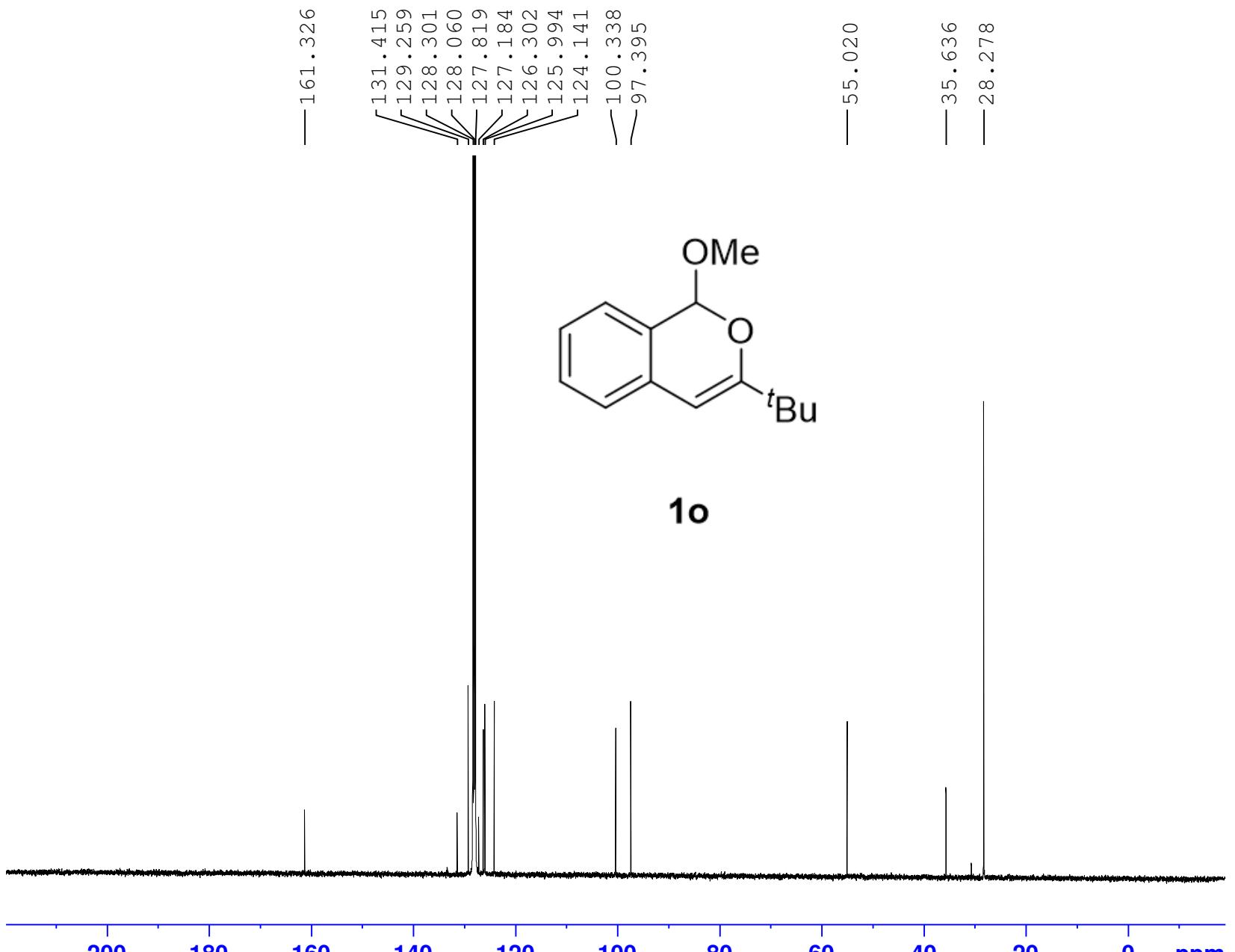


Current Data Parameters
 NAME wuan-189
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160116
 Time 17.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 82.92
 DW 62.400 usec
 DE 6.50 usec
 TE 296.6 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1299965 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



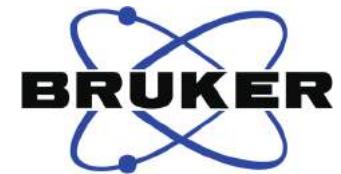
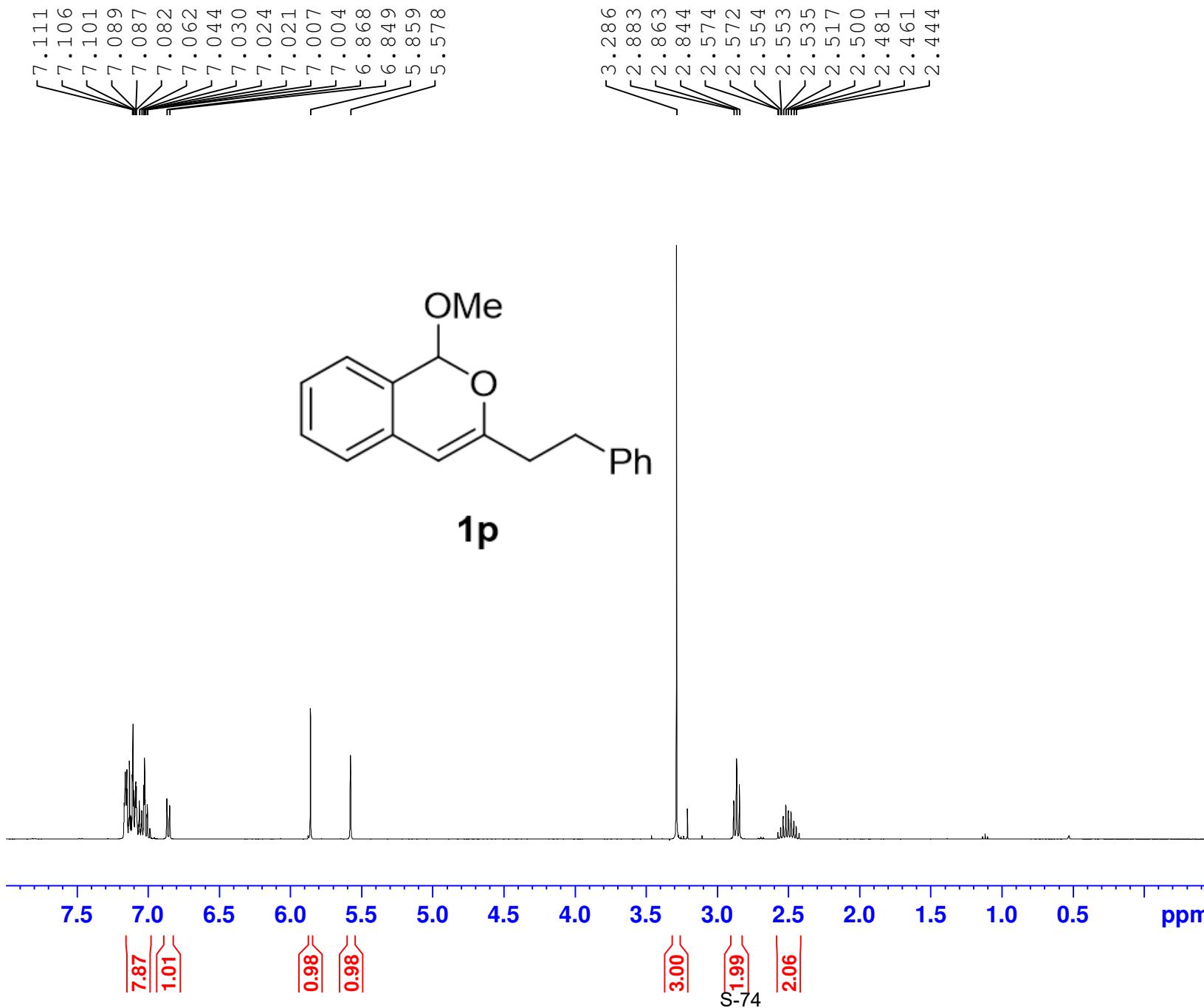
Current Data Parameters
 NAME wuan-189
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160116
 Time 17.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 938
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.8 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127309 MHz
 WDW 0
 SSB EM
 LB 1.00 Hz
 GB 0
 PC 1.40

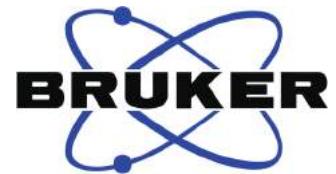
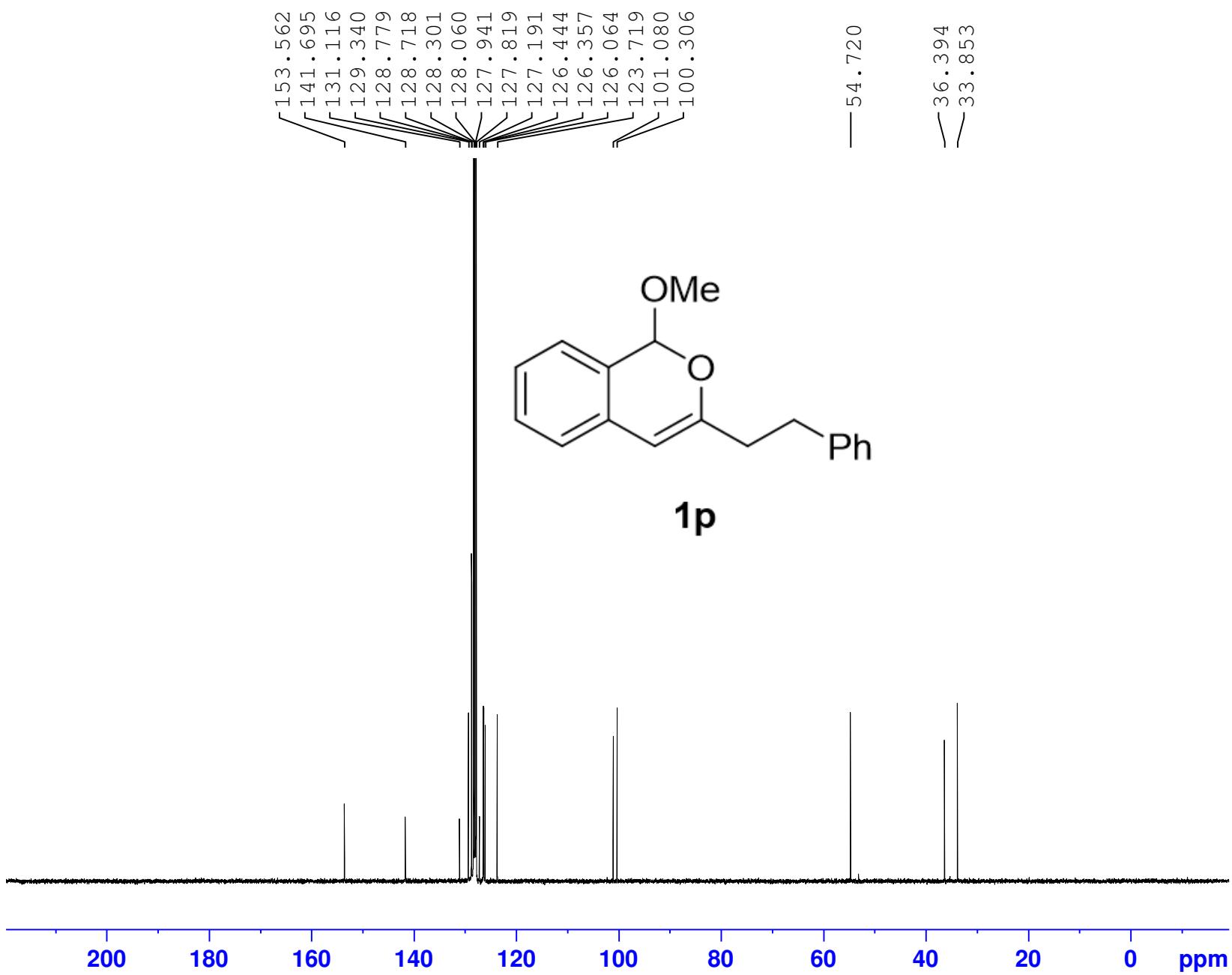


Current Data Parameters
 NAME wuan-159-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 7.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 128
 DW 60.800 usec
 DE 6.00 usec
 TE 295.9 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 ======
 NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300437 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



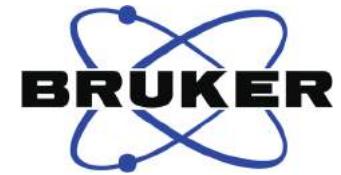
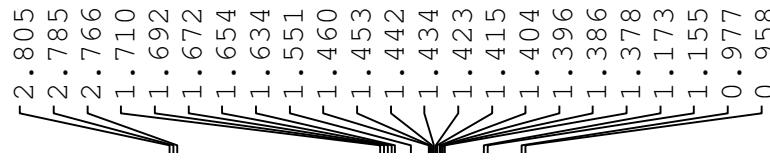
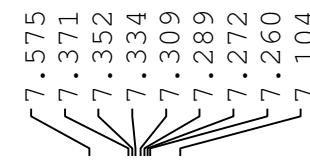
Current Data Parameters
 NAME wuan-159-1
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 8.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT C6D6
 NS 229
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.7 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.8999999 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127441 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

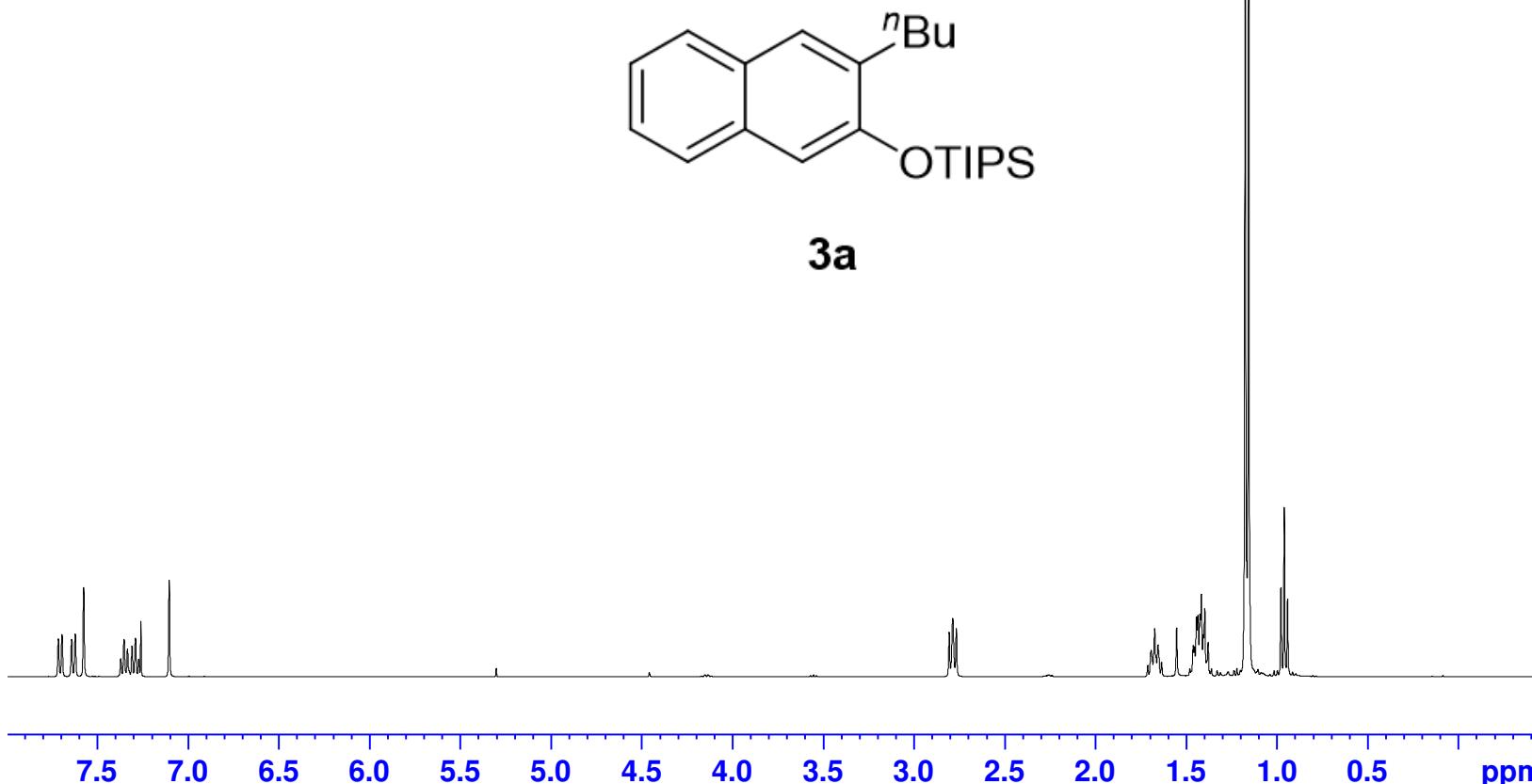


Current Data Parameters
 NAME wuan-55-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151209
 Time 18.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 70.97
 DW 62.400 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

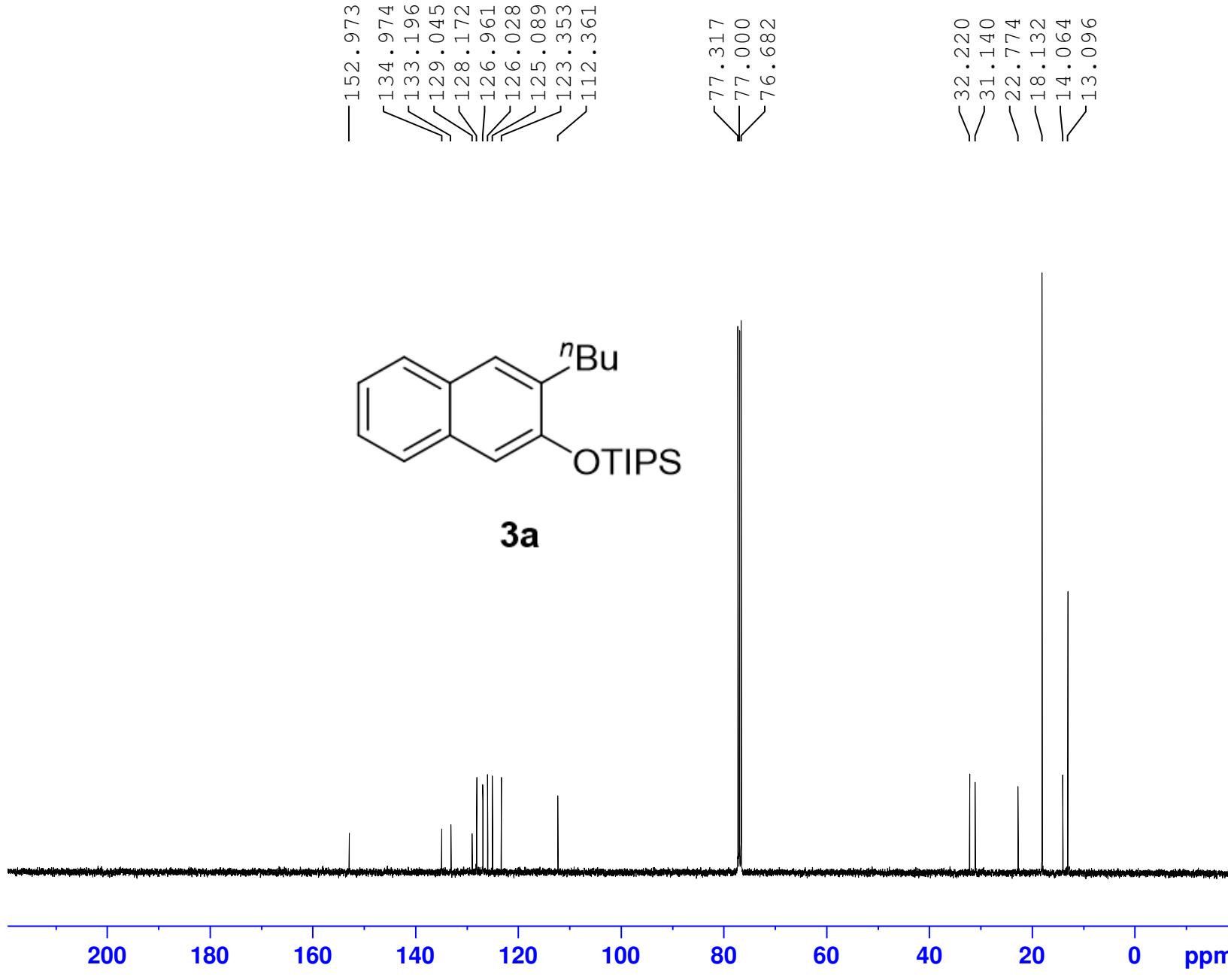
F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



0.99
1.00
1.07
1.02
1.00

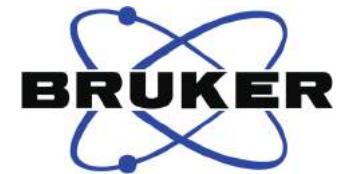
2.01
S.76

2.02
5.08
18.00
3.02



7.583
7.512
7.405
7.260
7.132
7.112
7.108
7.129
7.016

2.770
2.751
2.731
2.460
1.670
1.664
1.656
1.650
1.642
1.631
1.626
1.541
1.441
1.434
1.422
1.415
1.403
1.396
1.385
1.377
1.359
1.159
1.140
0.961

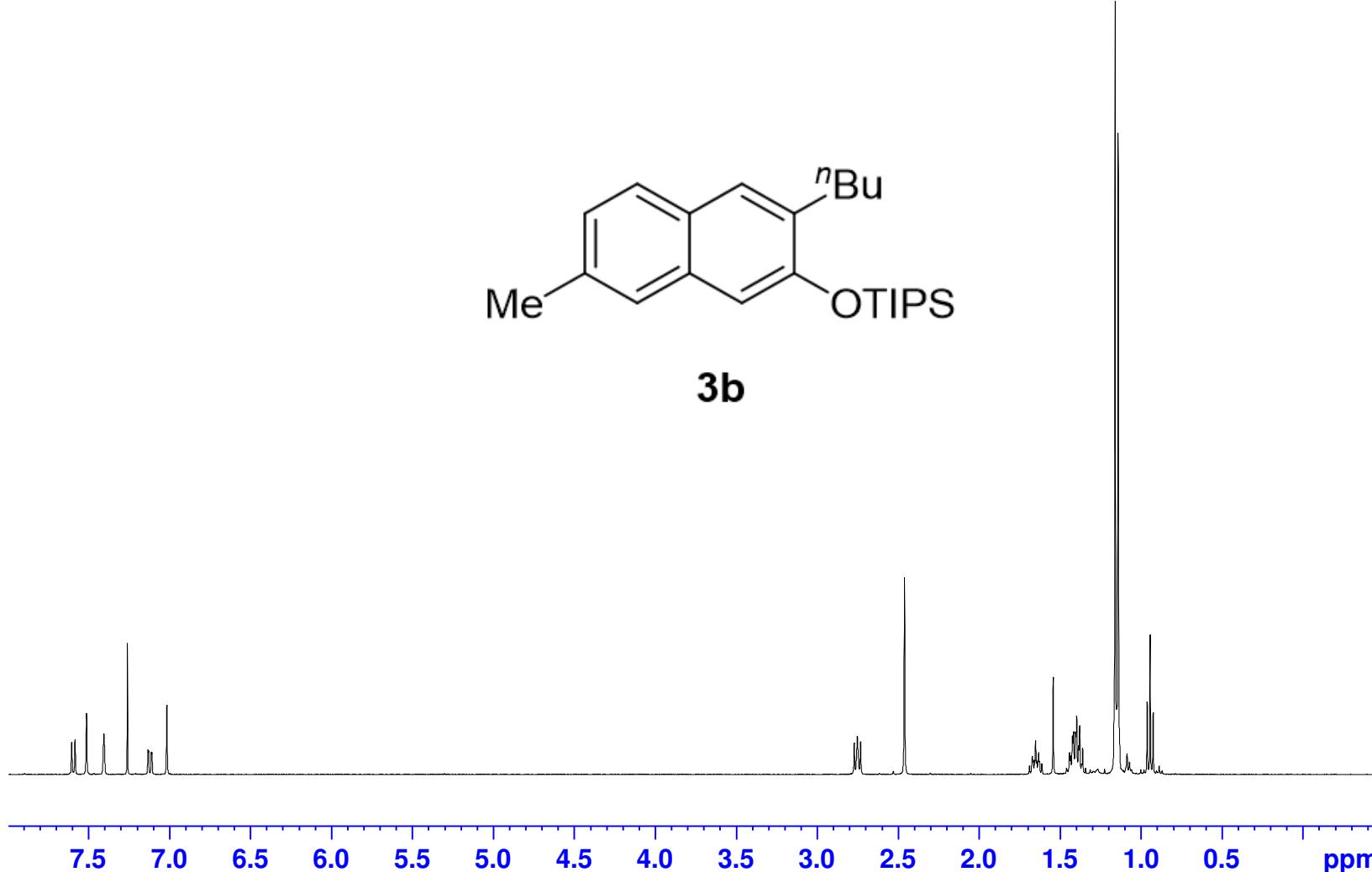


Current Data Parameters
 NAME wuan-119A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151113
 Time 19.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 406
 DW 60.800 usec
 DE 6.00 usec
 TE 293.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

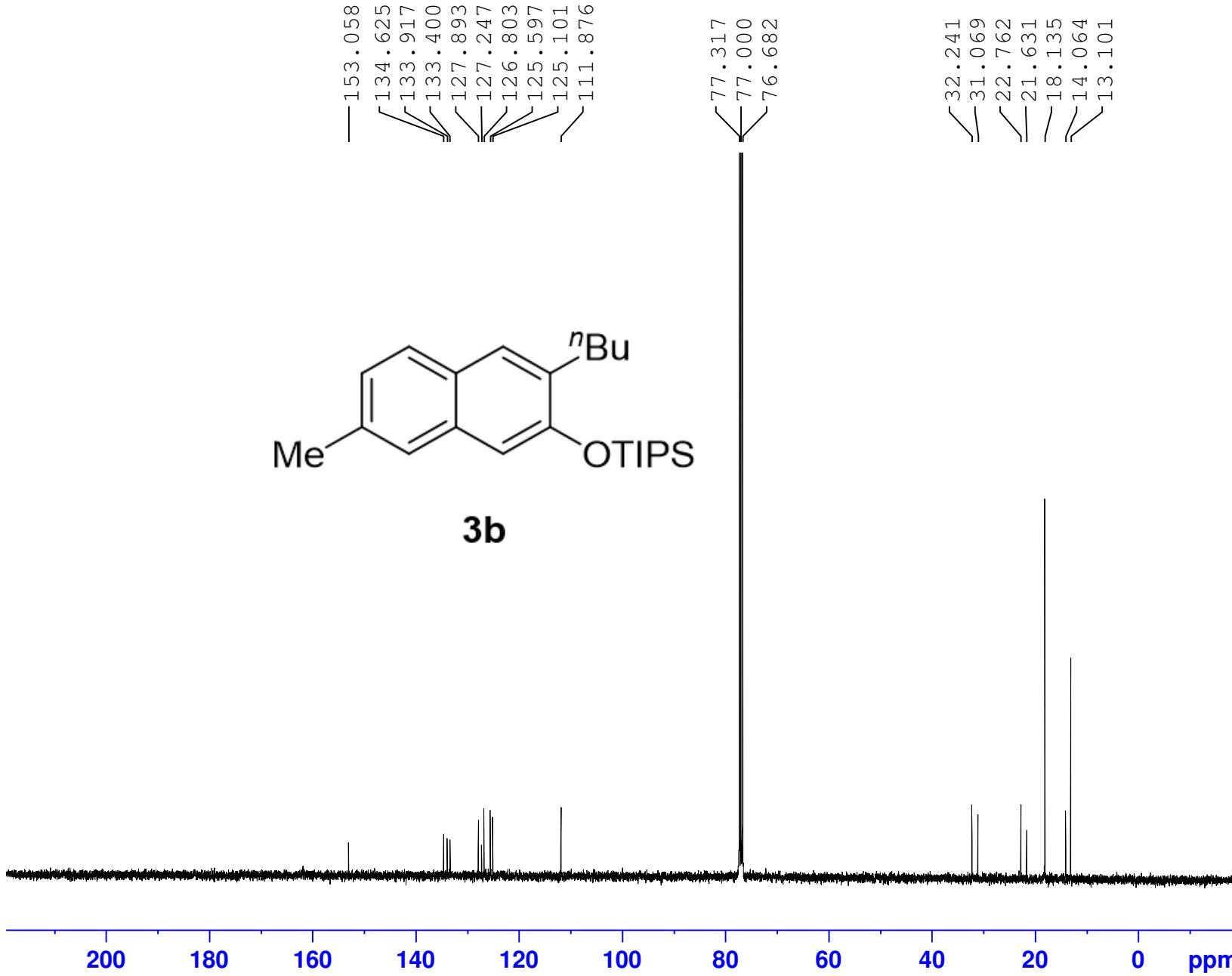
F2 - Processing parameters
 SI 32768
 SF 400.1300054 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



3b

1.01
1.00
0.99
1.00
1.01

0.04
3.00
S-78
2.07
5.06
18.00
3.06



S-79



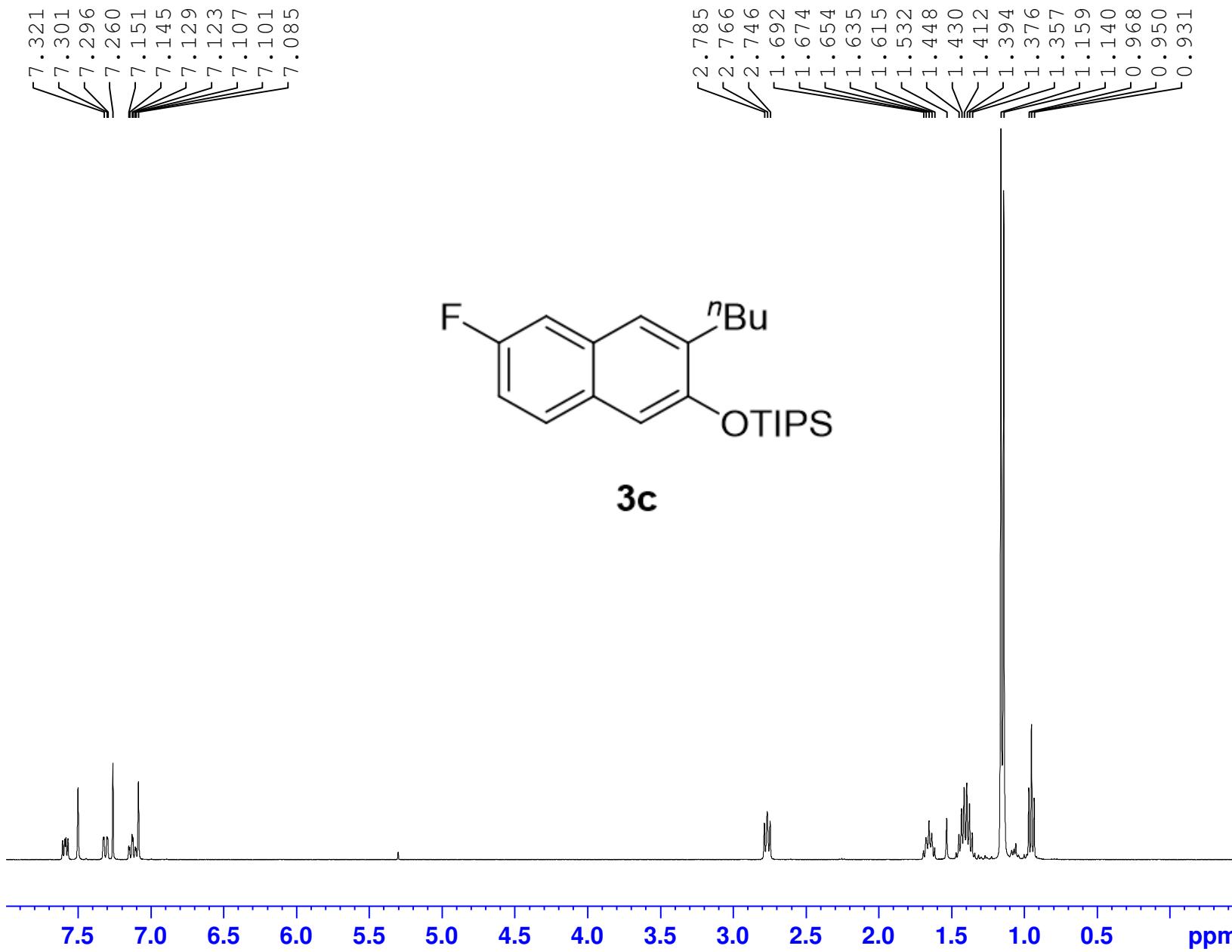
Current Data Parameters
NAME wuan-119A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151114
Time 11.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 441
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127705 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

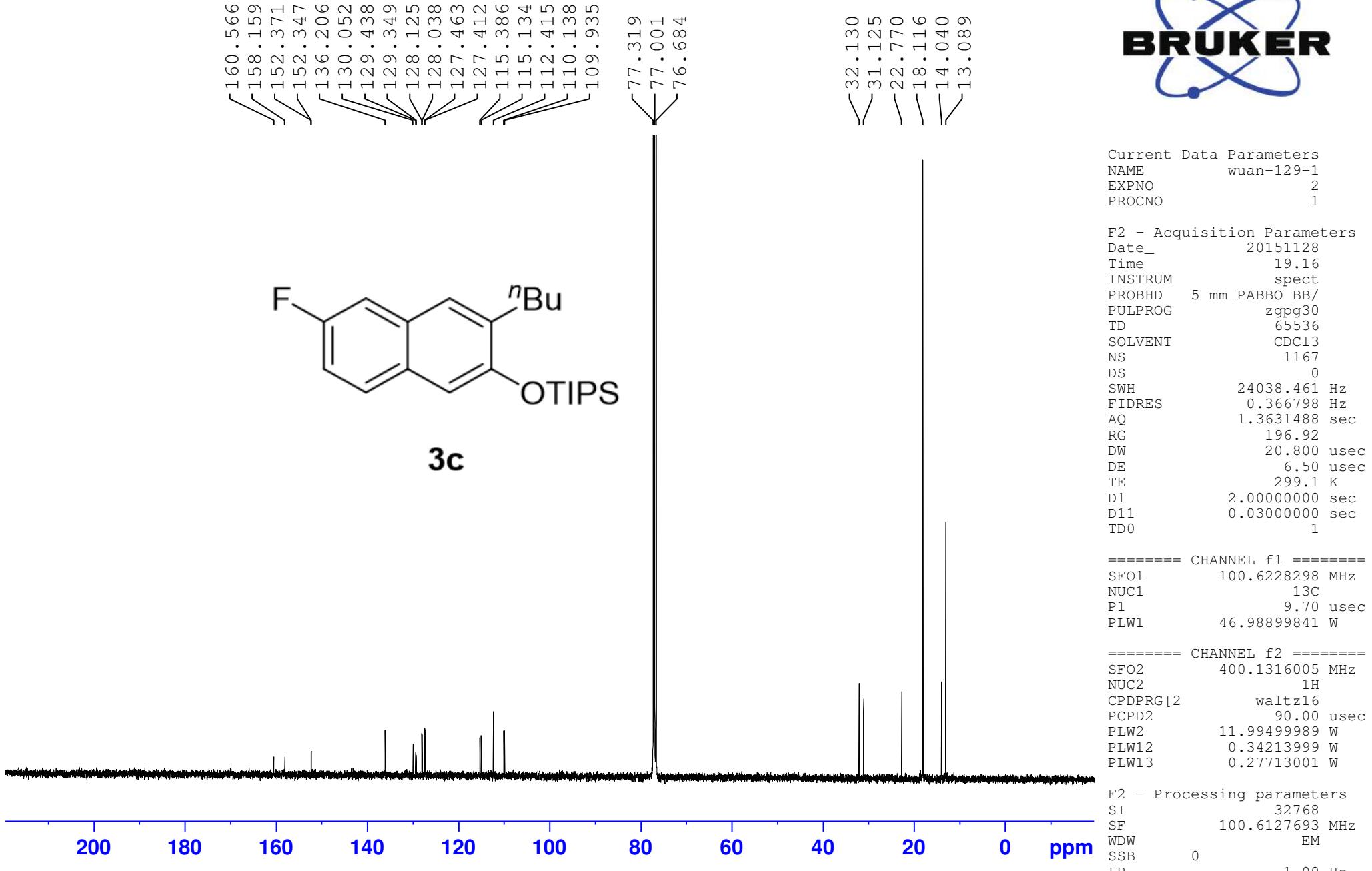


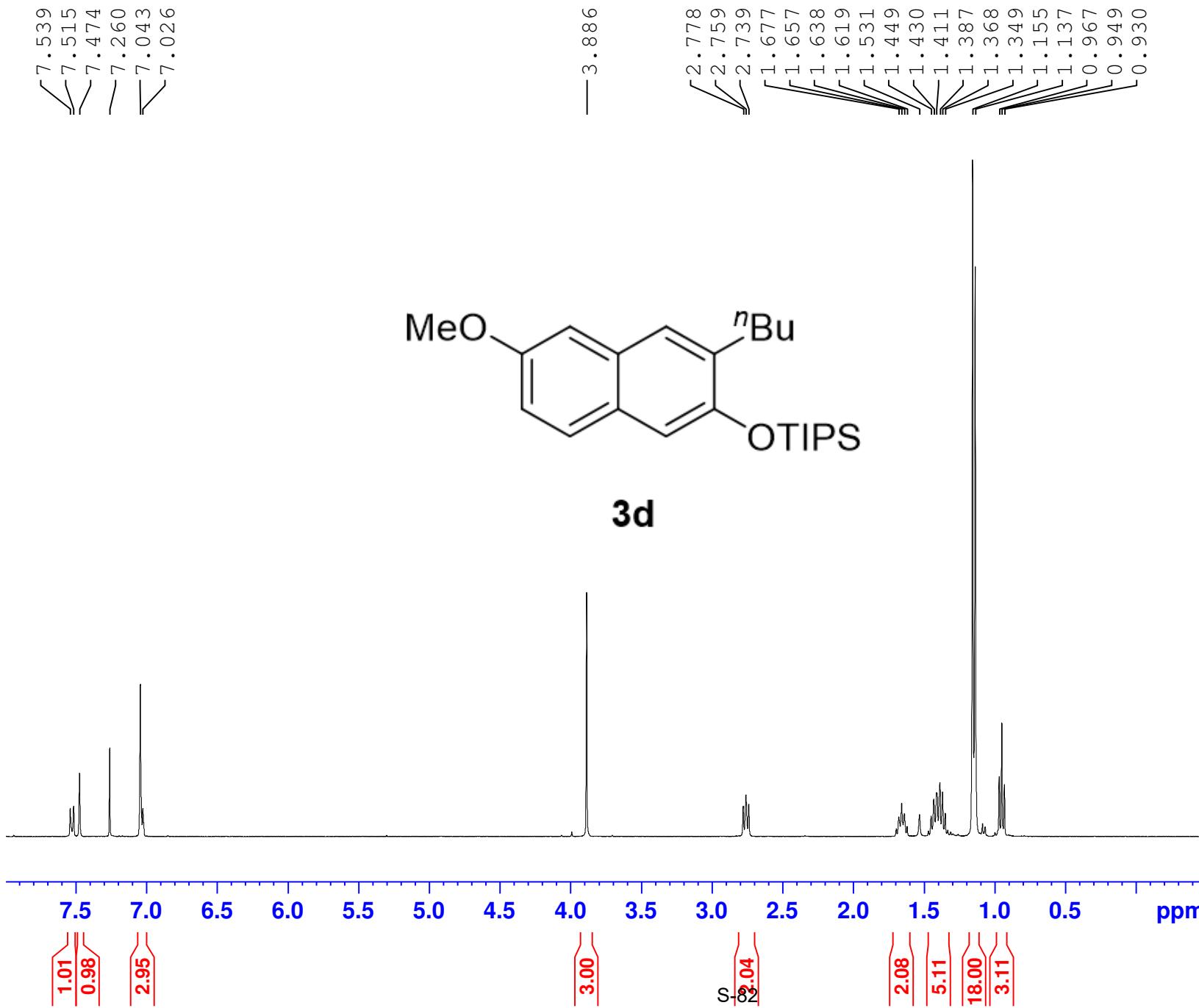
Current Data Parameters
 NAME wuan-129-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151127
 Time 14.47
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 103.52
 DW 62.400 usec
 DE 6.50 usec
 TE 296.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



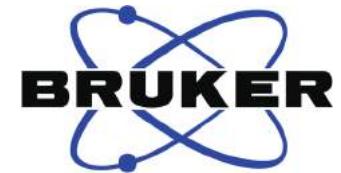
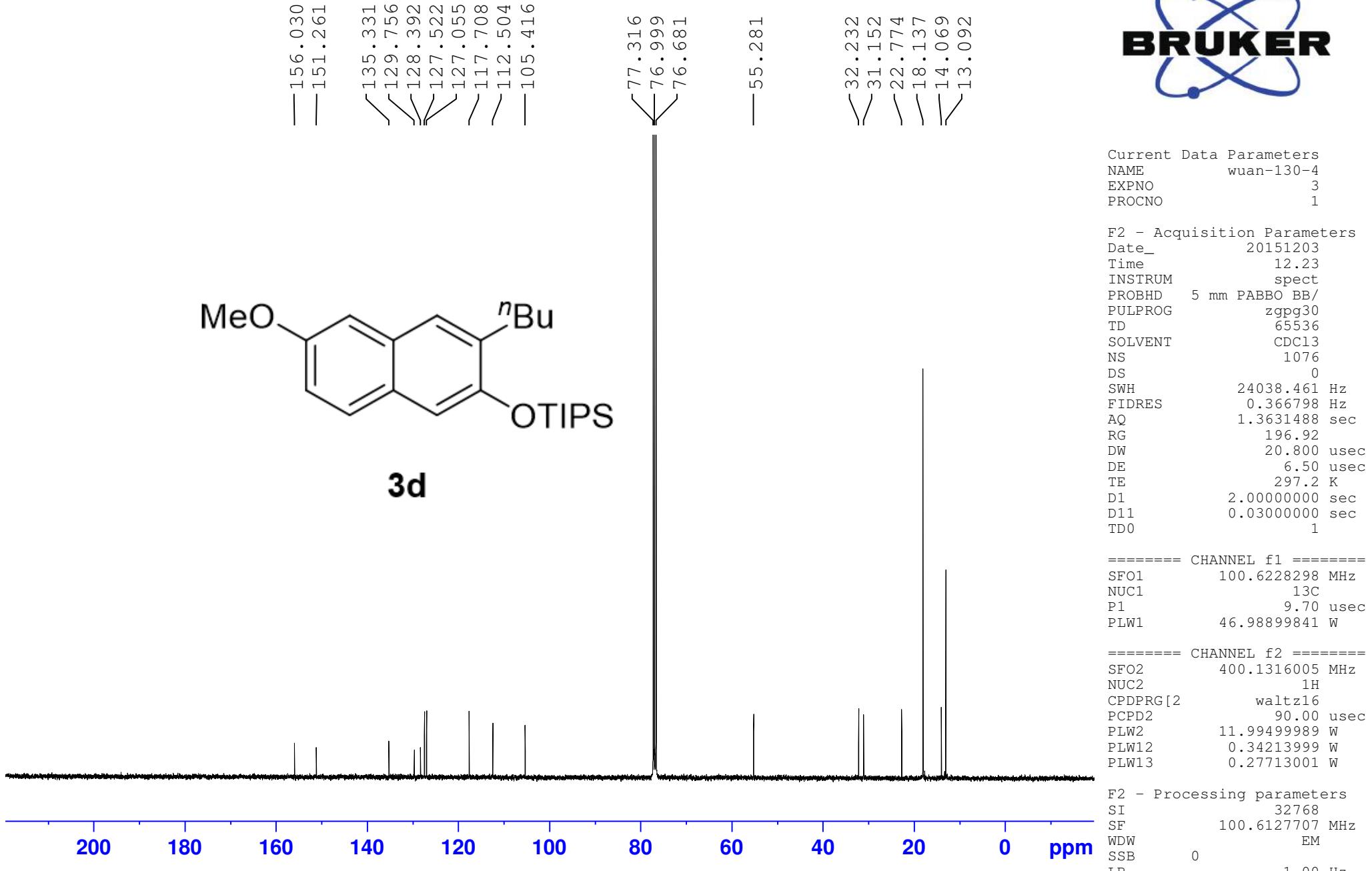


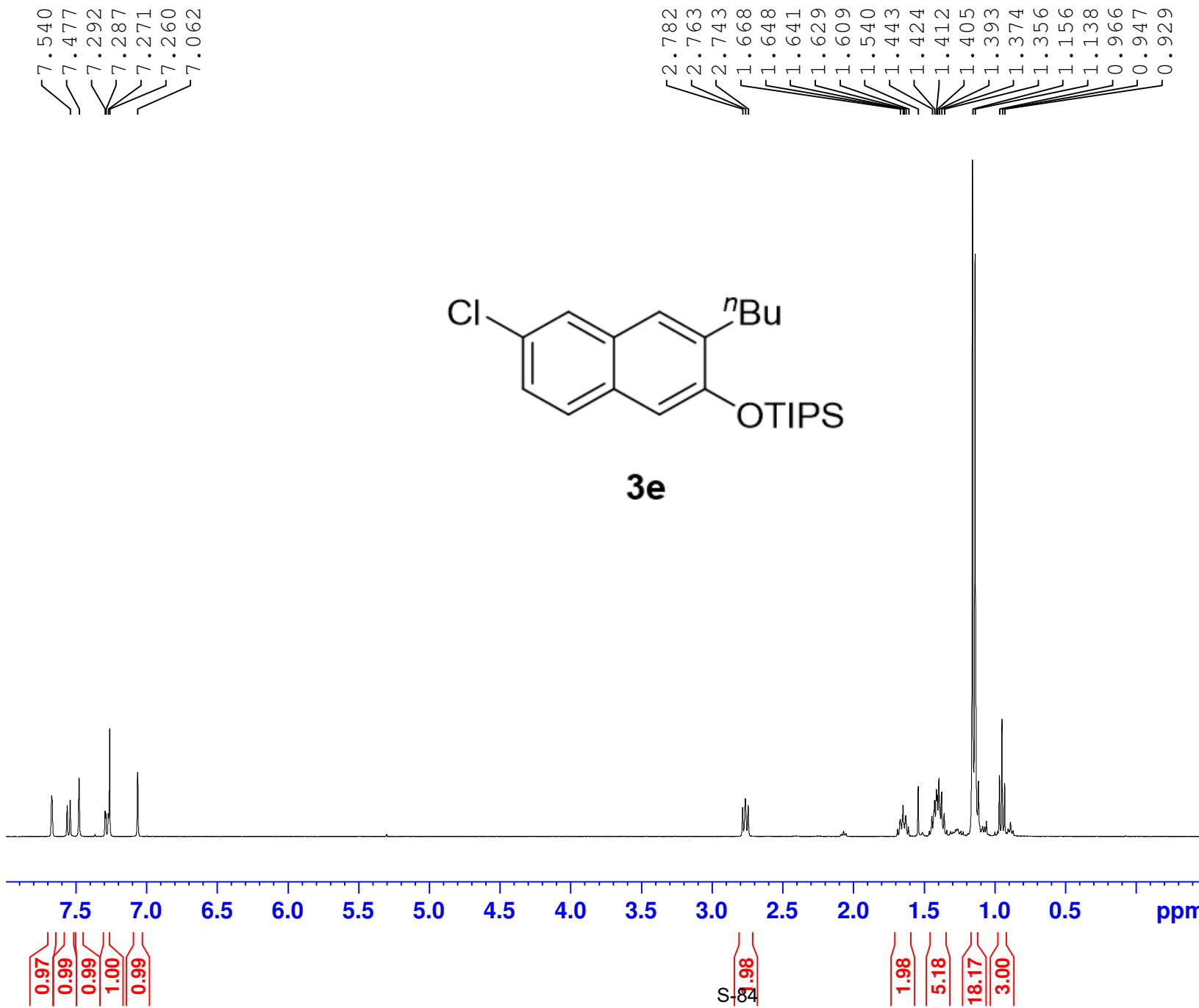
Current Data Parameters
 NAME wuan-130-4
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151127
 Time 14.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 103.52
 DW 62.400 usec
 DE 6.50 usec
 TE 296.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 ======
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



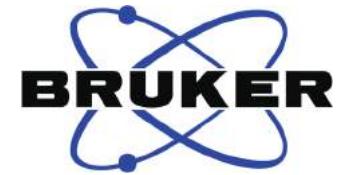
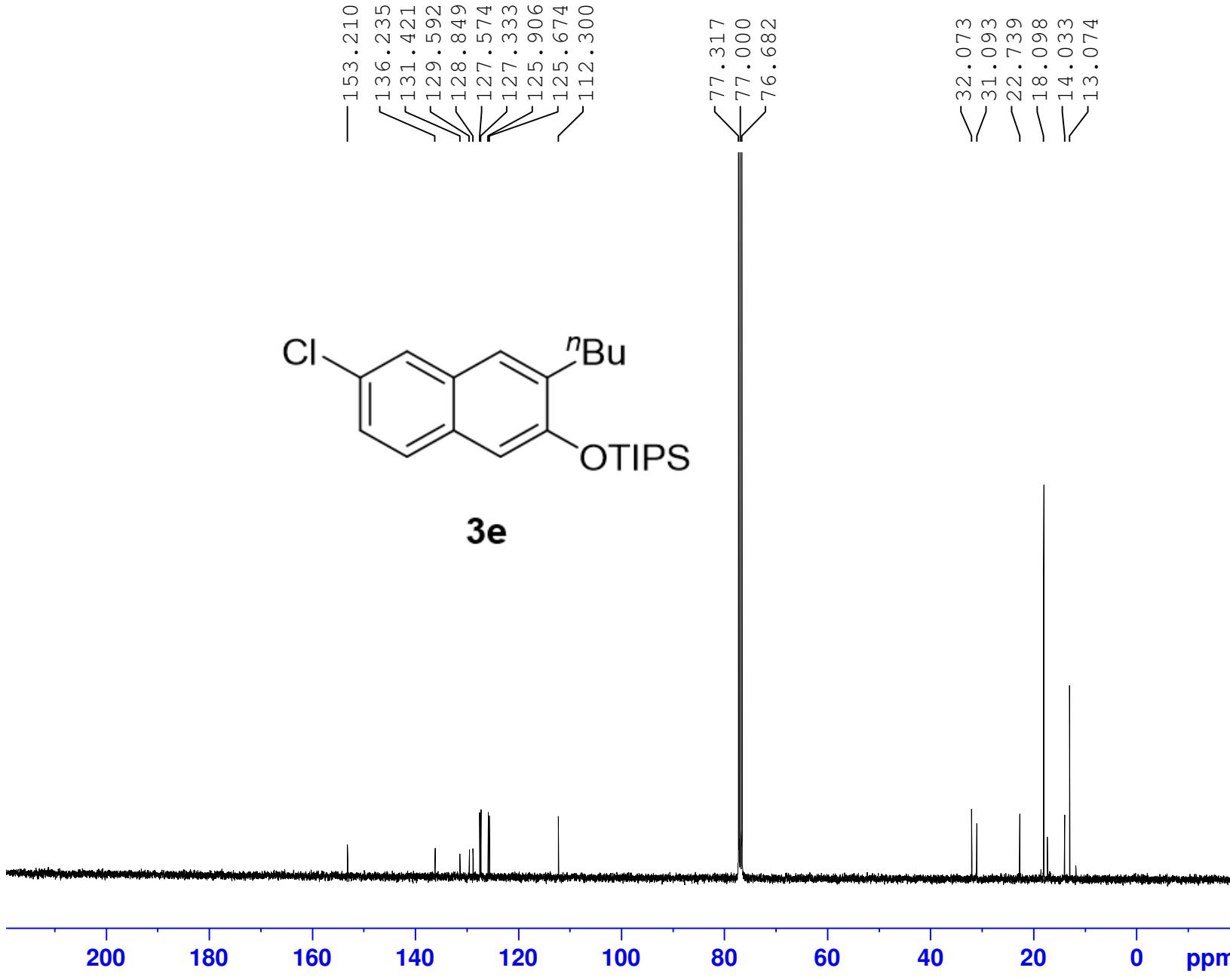


Current Data Parameters
 NAME wuan-133-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151128
 Time 11.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 ======
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



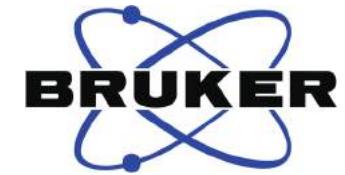
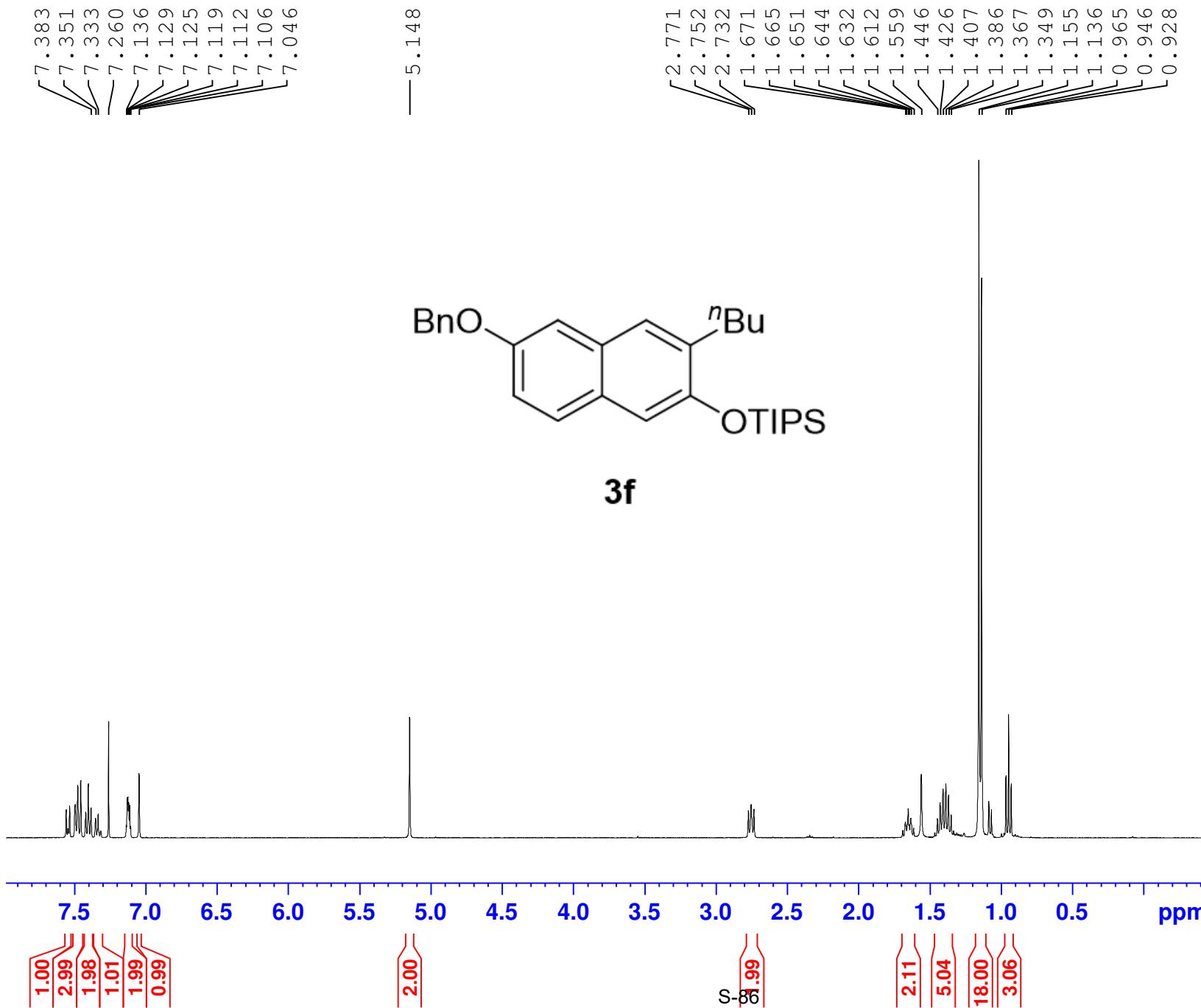
Current Data Parameters
 NAME wuan-133-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151128
 Time 11.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 844
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 298.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127697 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



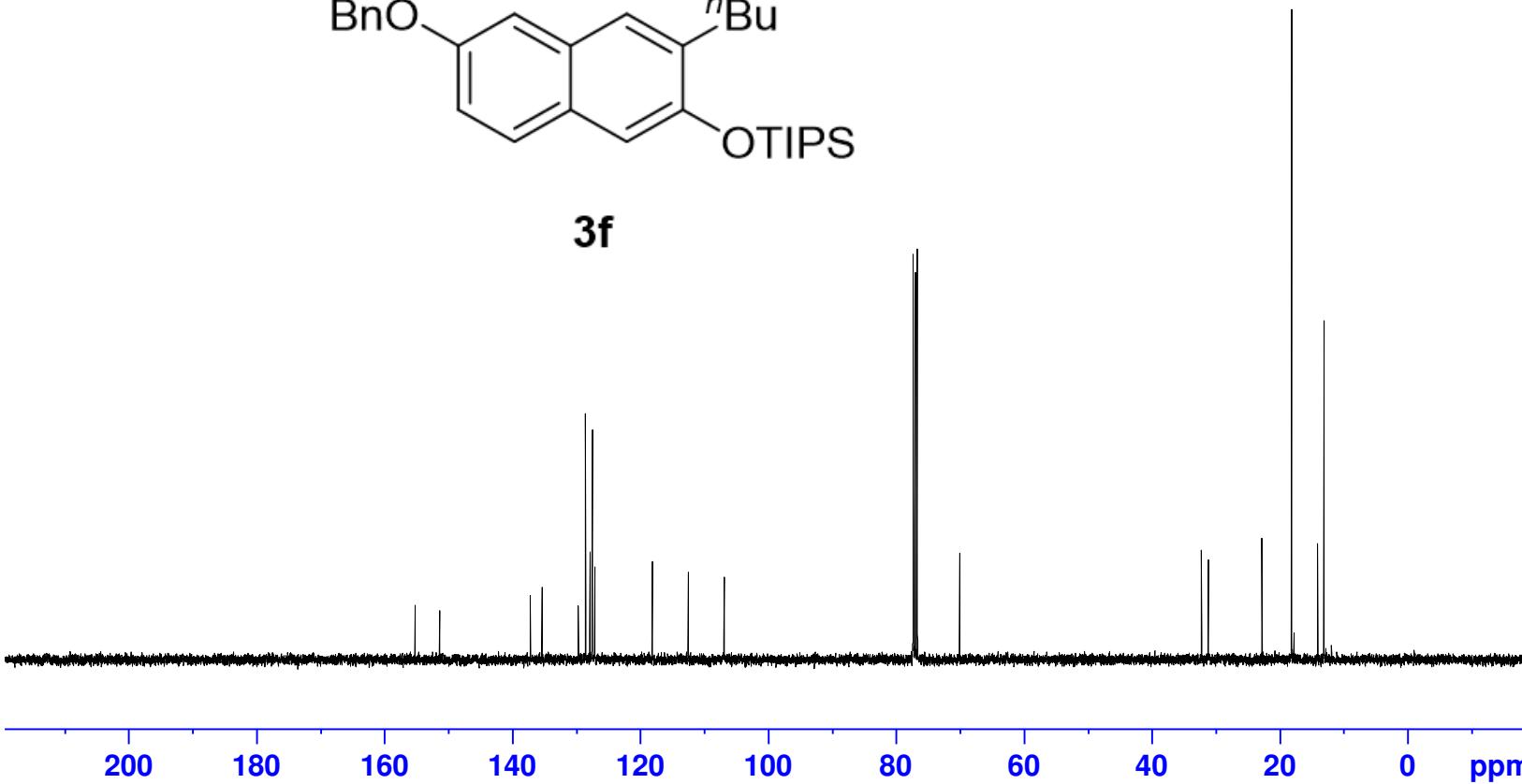
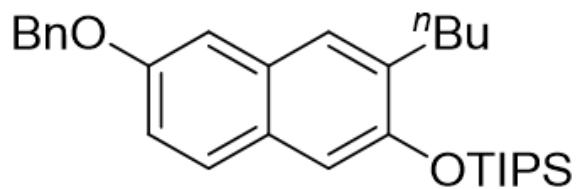
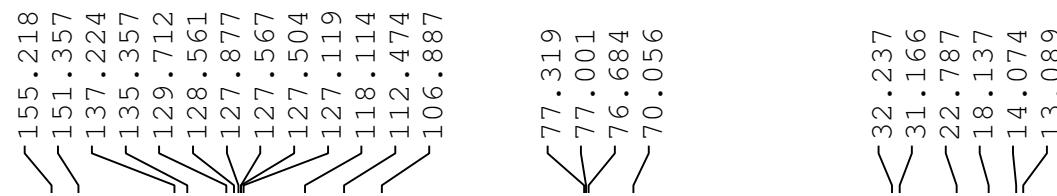
Current Data Parameters
NAME wuan-160
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151218
Time 18.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 293.4 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 ======

NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
 NAME wuan-160
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 3.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 145
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 296.4 K
 D1 2.0000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

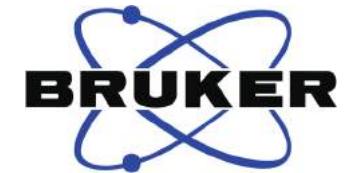
===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 9.25 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG[2] waltz16
 NUC2 ¹H
 PCPD2 80.00 usec
 PL12 12.45 dB
 PL13 18.00 dB
 PL2 -1.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127717 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

7.617
7.358
7.340
7.320
7.290
7.276
7.260
7.119

2.338
2.324
2.317
2.304
2.290
2.283
2.269
1.553
1.459
1.440
1.421
1.403
1.384
1.183
1.164
1.012
0.998
0.978
0.965
0.774
0.761
0.748

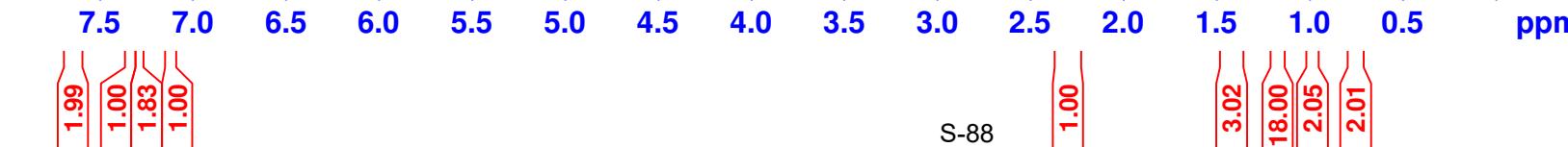
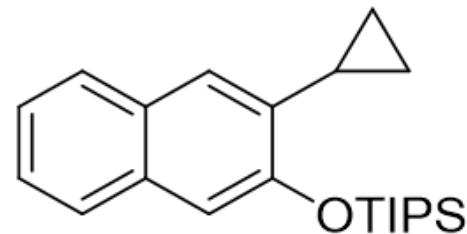


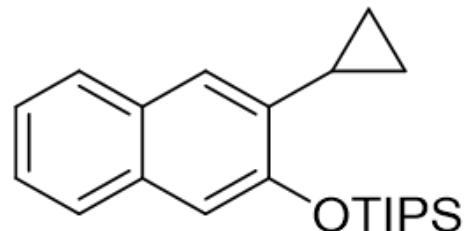
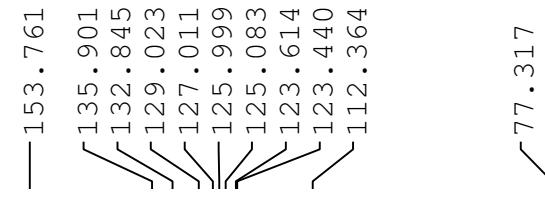
Current Data Parameters
 NAME wuan-197-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160202
 Time 23.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 70.97
 DW 62.400 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.00000000 sec
 TD0 1

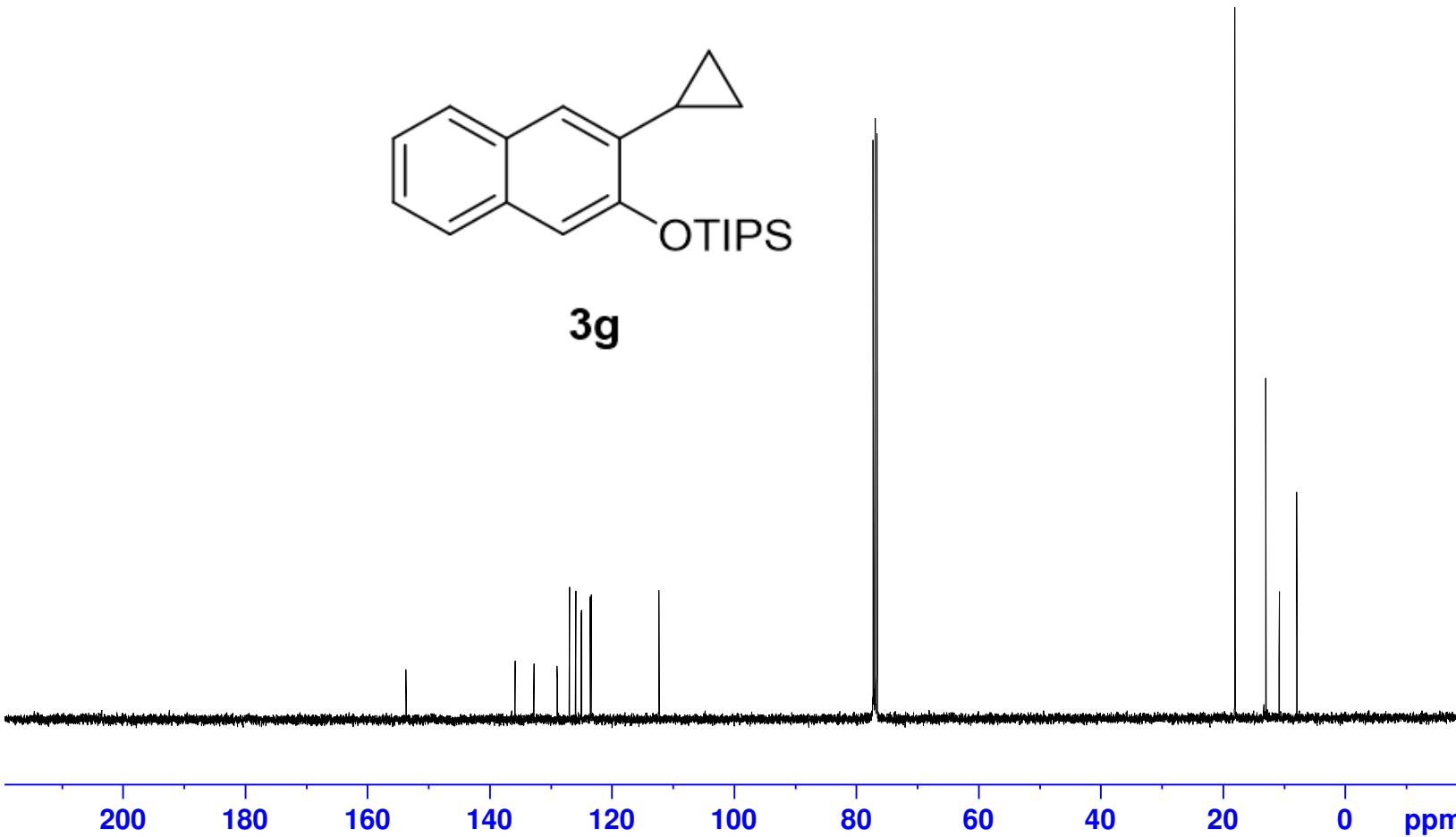
===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





3g



BRUKER

Current	Data	Parameters
NAME	wuan-197-1	
EXPNO		2
PROCNO		1

```

F2 - Acquisition Parameters
Date_           20160202
Time            23.37
INSTRUM         spect
PROBHD         5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT         CDC13
NS              228
DS              0
SWH             24038.461 Hz
FIDRES         0.366798 Hz
AQ              1.3631488 sec
RG              196.92
DW              20.800 usec
DE              6.50 usec
TE              297.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0             1

```

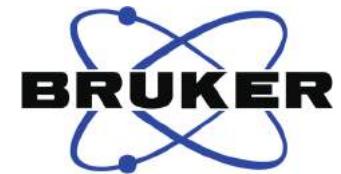
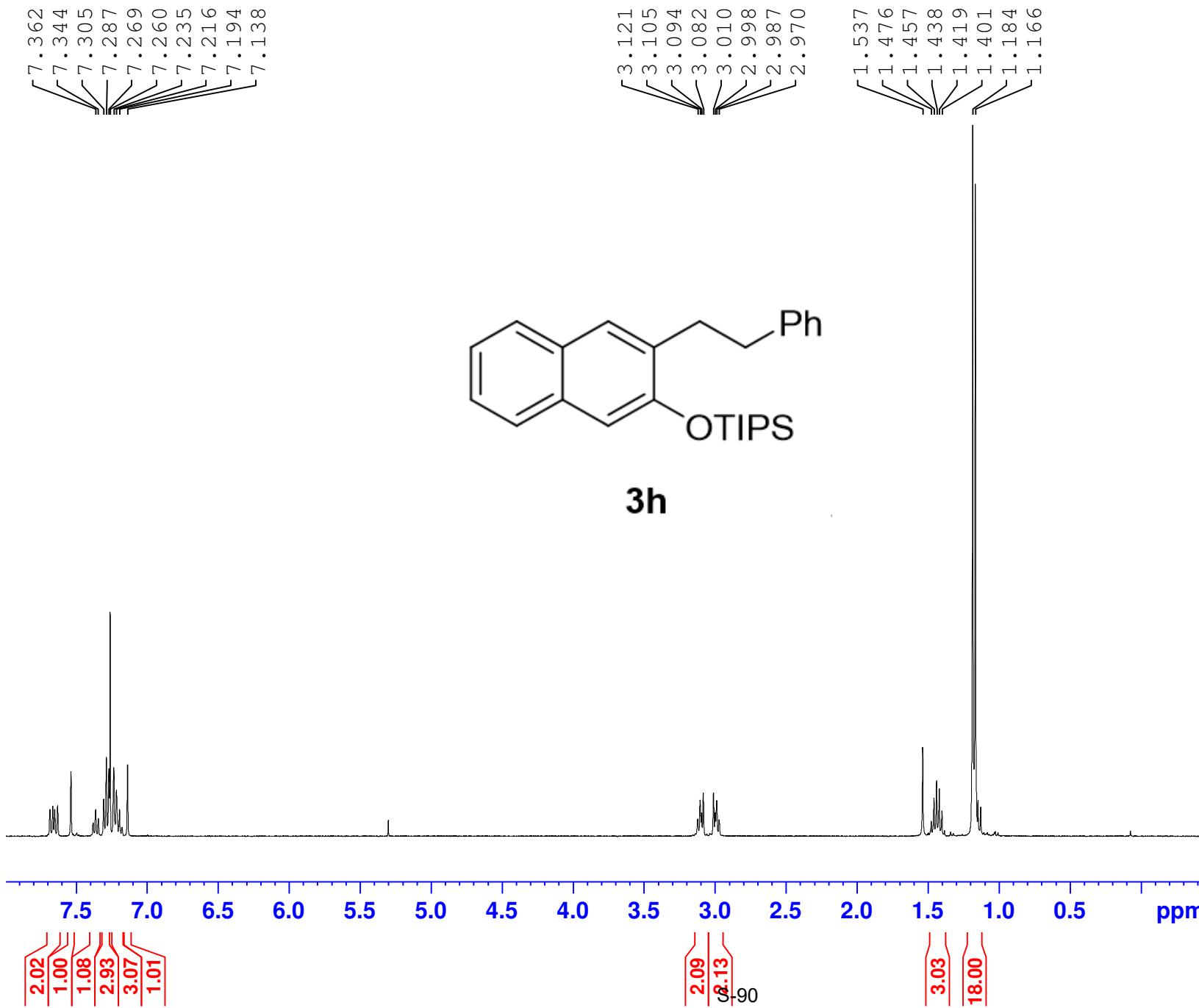
```
===== CHANNEL f1 ======  
SFO1      100.6228298 MHz  
NUC1          13C  
P1            9.70 usec  
PLW1        46.98899841 W
```

```

===== CHANNEL f2 =====
SFO2          400.1316005 MHz
NUC2           1H
CPDPRG[2]      waltz16
PCPD2          90.00 usec
PLW2           11.99499989 W
PLW12          0.34213999 W
PLW13          0.27713001 W

```

F2 - Processing parameters
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
 NAME wuan-195-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160113
 Time 16.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.4 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

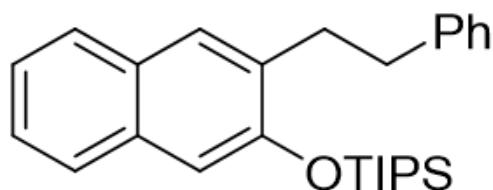
F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0 0.30 Hz
 LB 0 1.00
 GB PC



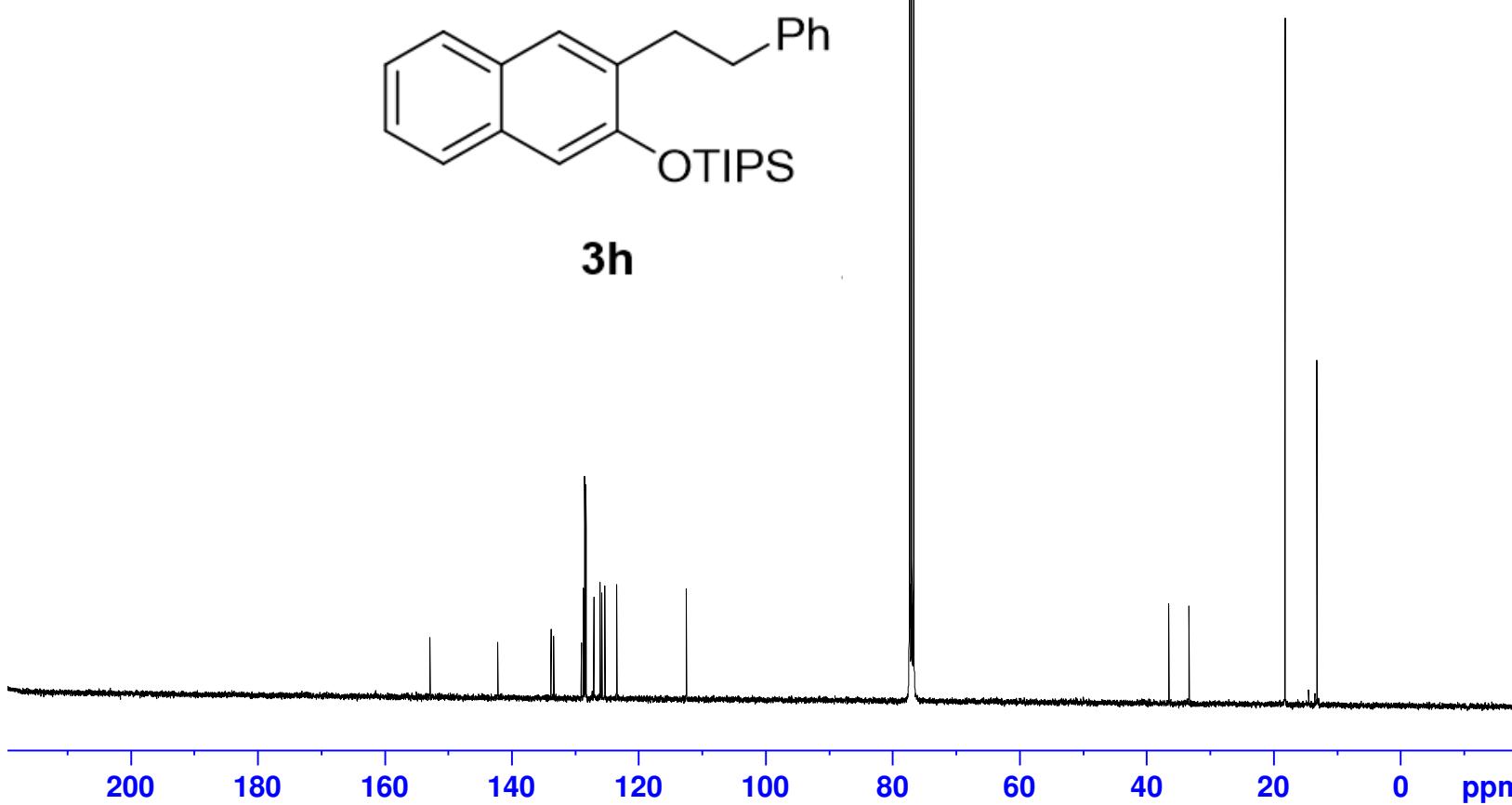
152.867
142.196
133.800
133.370
129.025
128.658
128.468
128.286
127.043
126.057
125.778
125.313
123.460
112.487

77.316
76.999
76.681

36.488
33.310
18.188
13.157



3h



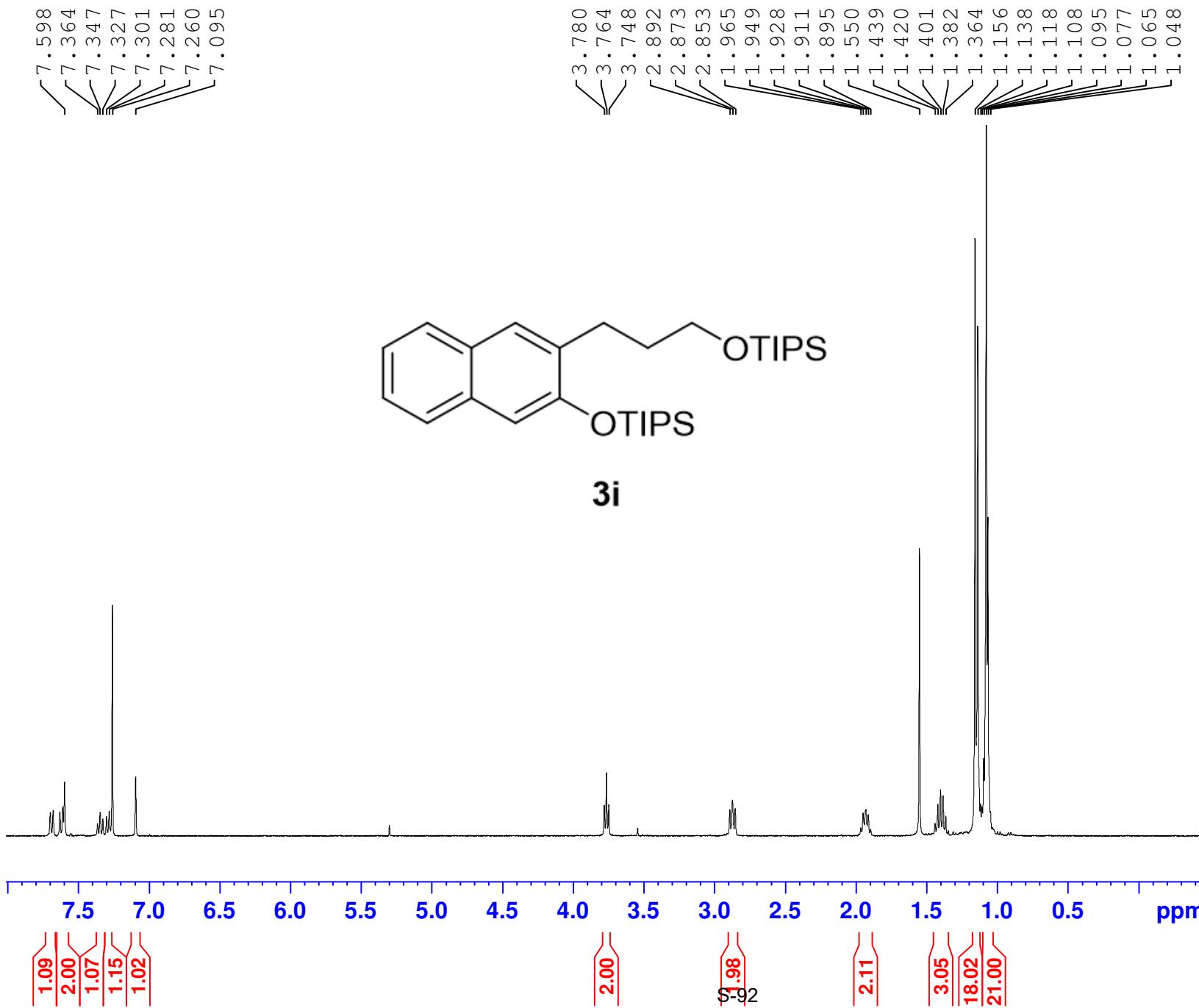
Current Data Parameters
NAME wuan-195-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160116
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12734
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPKG[2] waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127699 MHz
WDW 0 EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

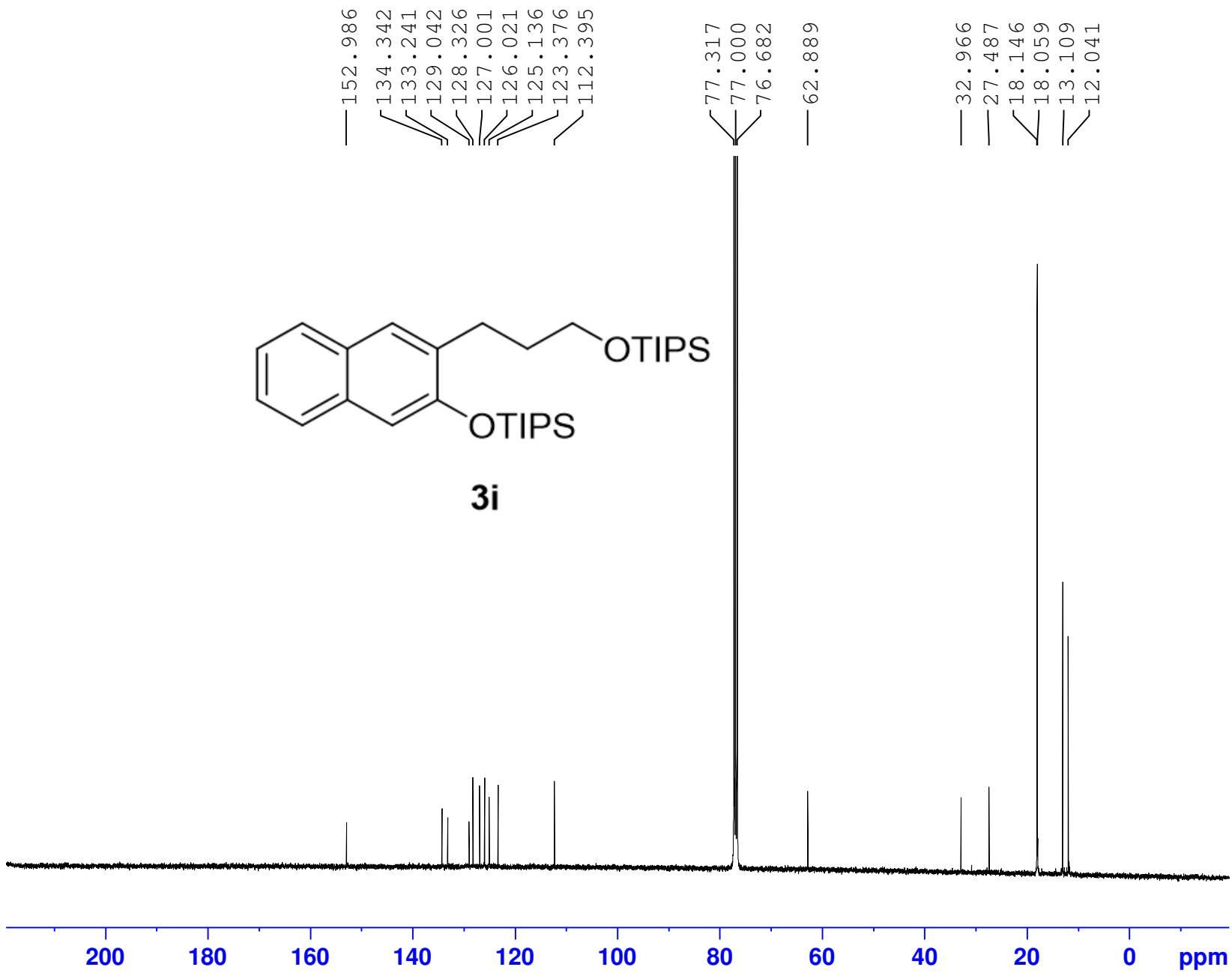


Current Data Parameters
 NAME wuan-221-1
 EXPNO 1
 PROCNO 1

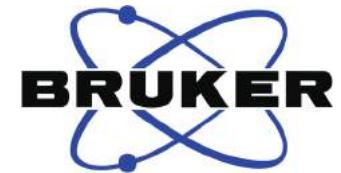
F2 - Acquisition Parameters
 Date_ 20160129
 Time 11.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122268 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300096 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



S-93



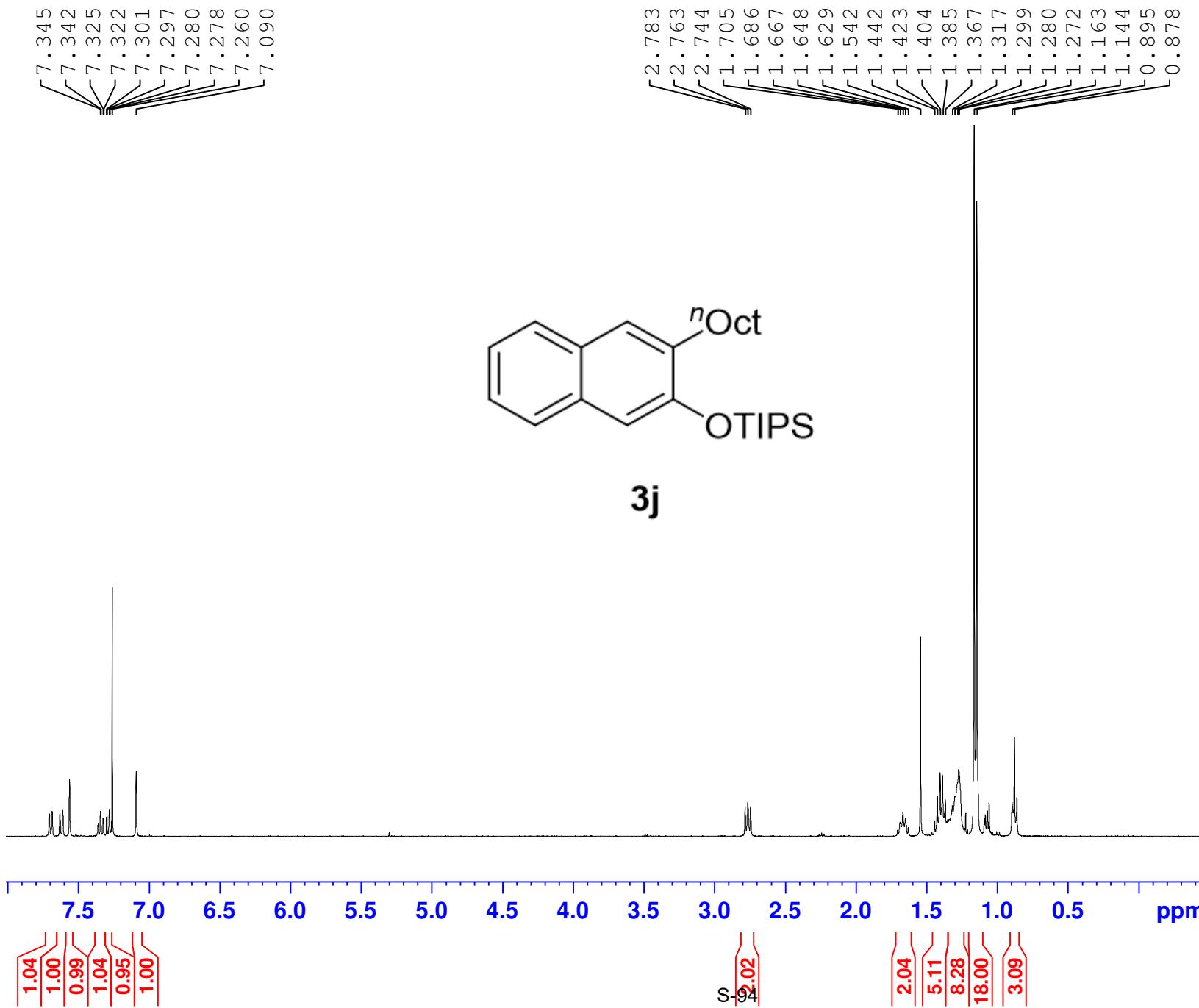
Current Data Parameters
 NAME wuan-174
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160112
 Time 9.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10491
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.3366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 298.8 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127697 MHz
 WDW 0 EM
 SSB 1.00 Hz
 LB 0
 GB 0
 PC 1.40

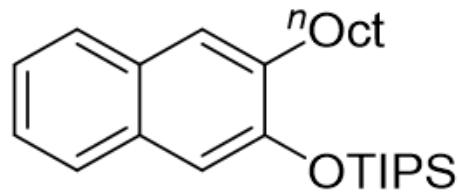
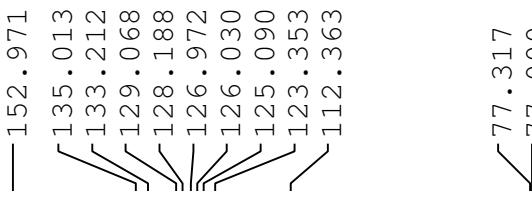


Current Data Parameters
 NAME wuan-176
 EXPNO 1
 PROCNO 1

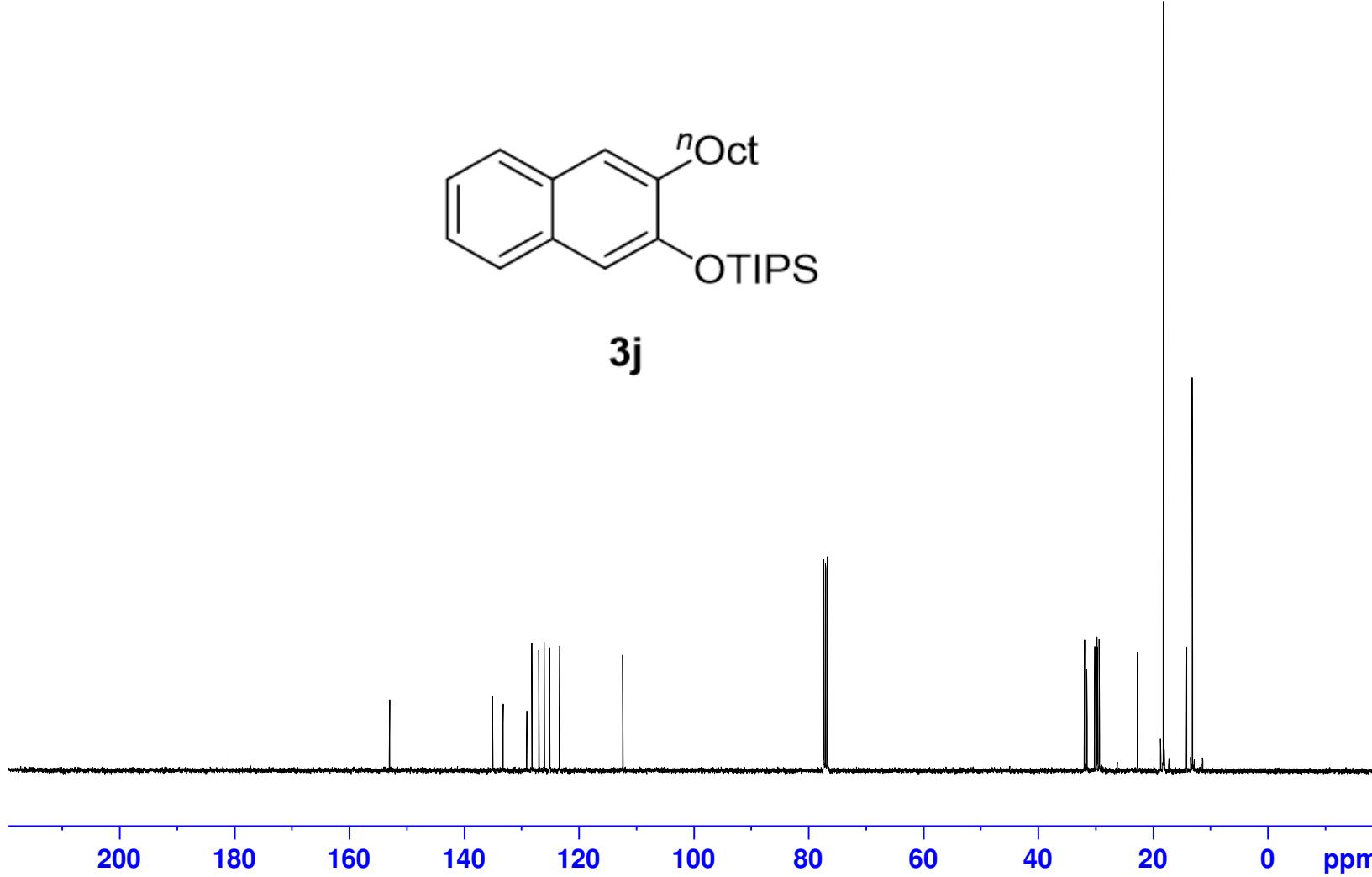
F2 - Acquisition Parameters
 Date_ 20151231
 Time 14.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.4 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



3j





Current Data Parameters	
NAME	wuan-176
EXPNO	2
PROCNO	1

```

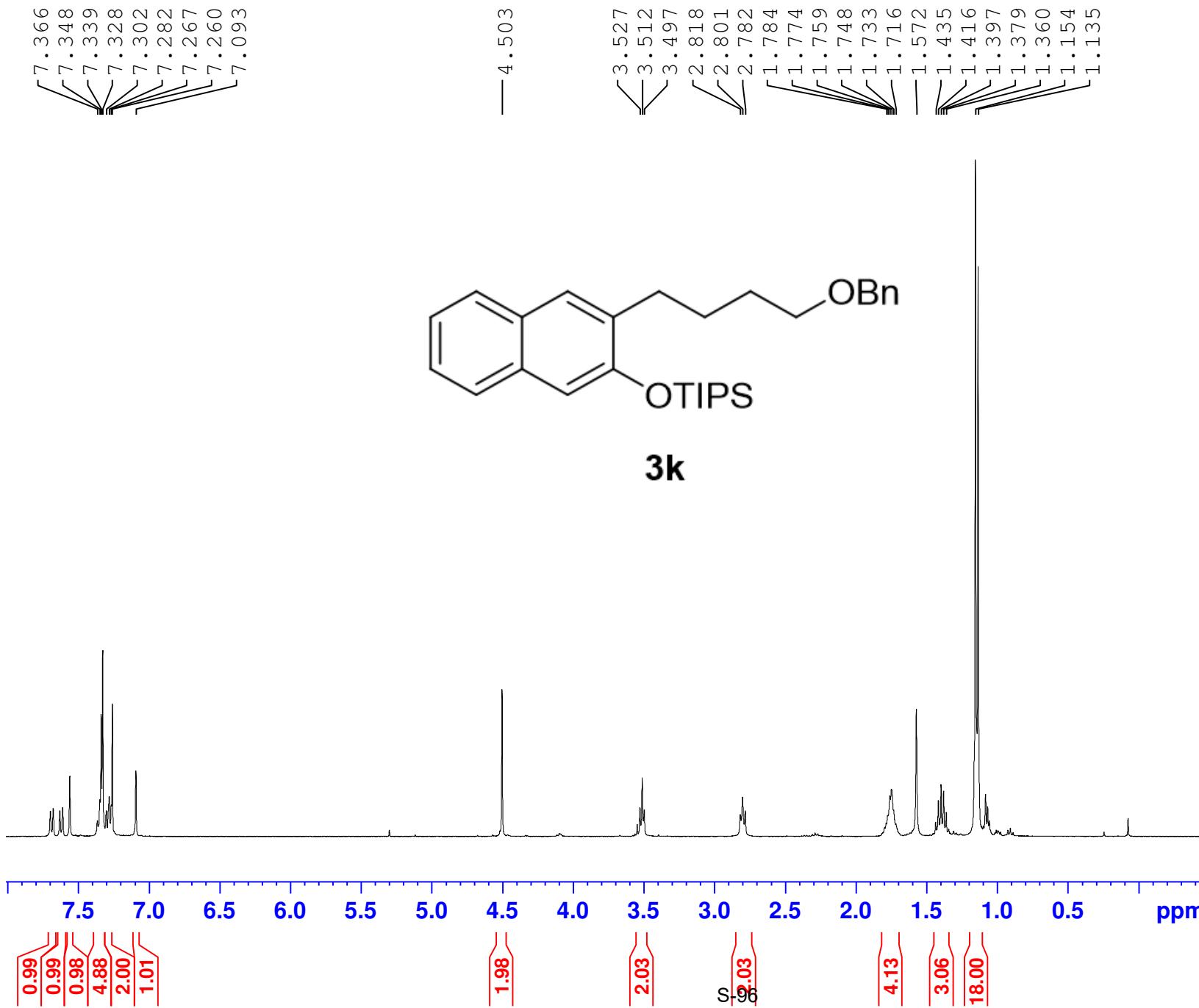
F2 - Acquisition Parameters
Date_           20160202
Time            20.15
INSTRUM         spect
PROBHD         5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT         CDC13
NS              127
DS              0
SWH             24038.461 Hz
FIDRES         0.366798 Hz
AQ              1.3631488 sec
RG              196.92
DW              20.800 usec
DE              6.50 usec
TE              297.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0             1

```

```
===== CHANNEL f1 ======  
SFO1      100.6228298 MHz  
NUC1          13C  
P1            9.70 usec  
PLW1        46.98899841 W
```

```
===== CHANNEL f2 =====
SFO2          400.1316005 MHz
NUC2           1H
CPDPRG[2]      waltz16
PCPD2          90.00 usec
PLW2           11.99499989 W
PLW12          0.34213999 W
PLW13          0.27713001 W
```

F2 - Processing parameters
SI 32768
SF 100.6127733 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

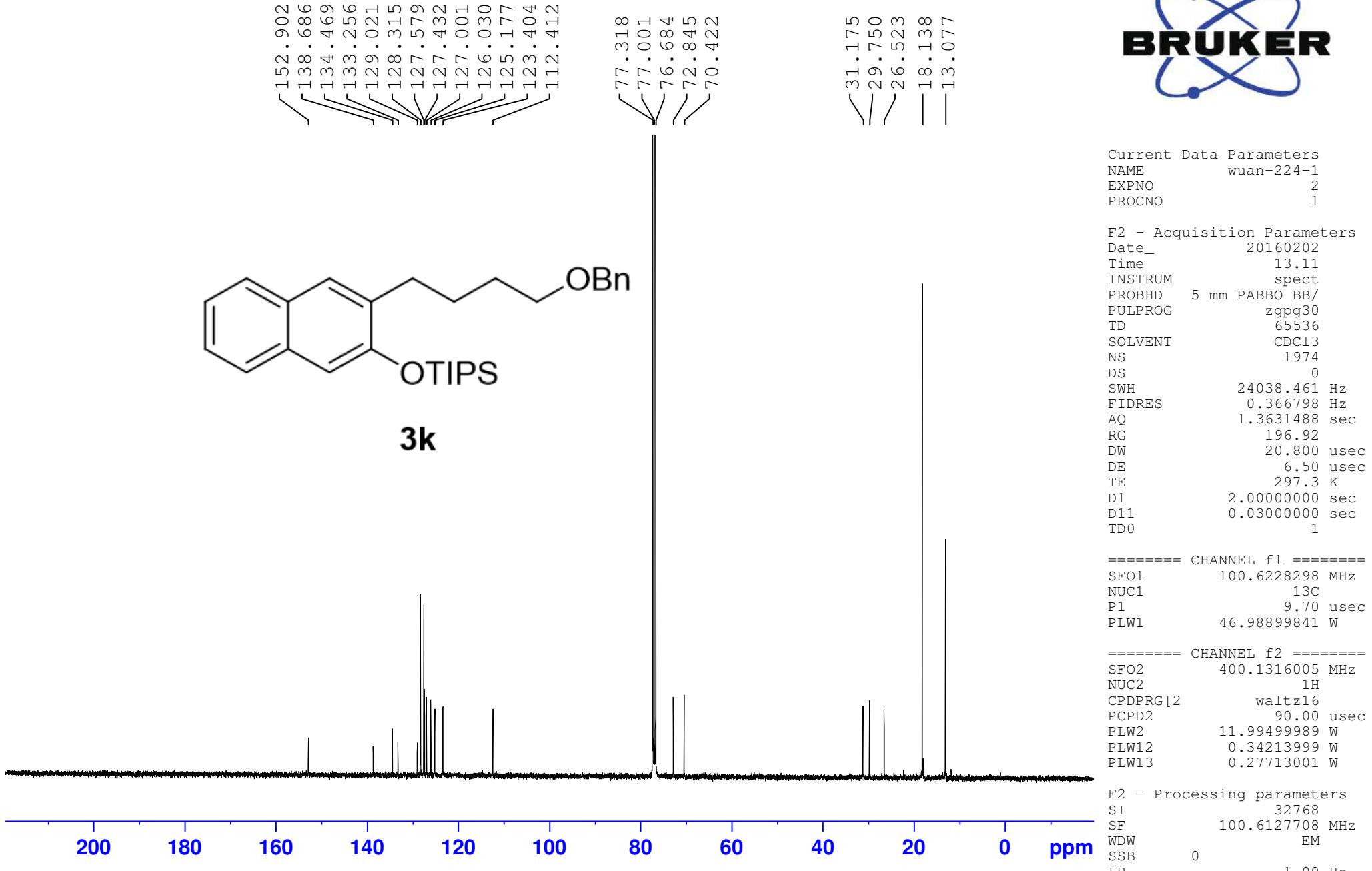


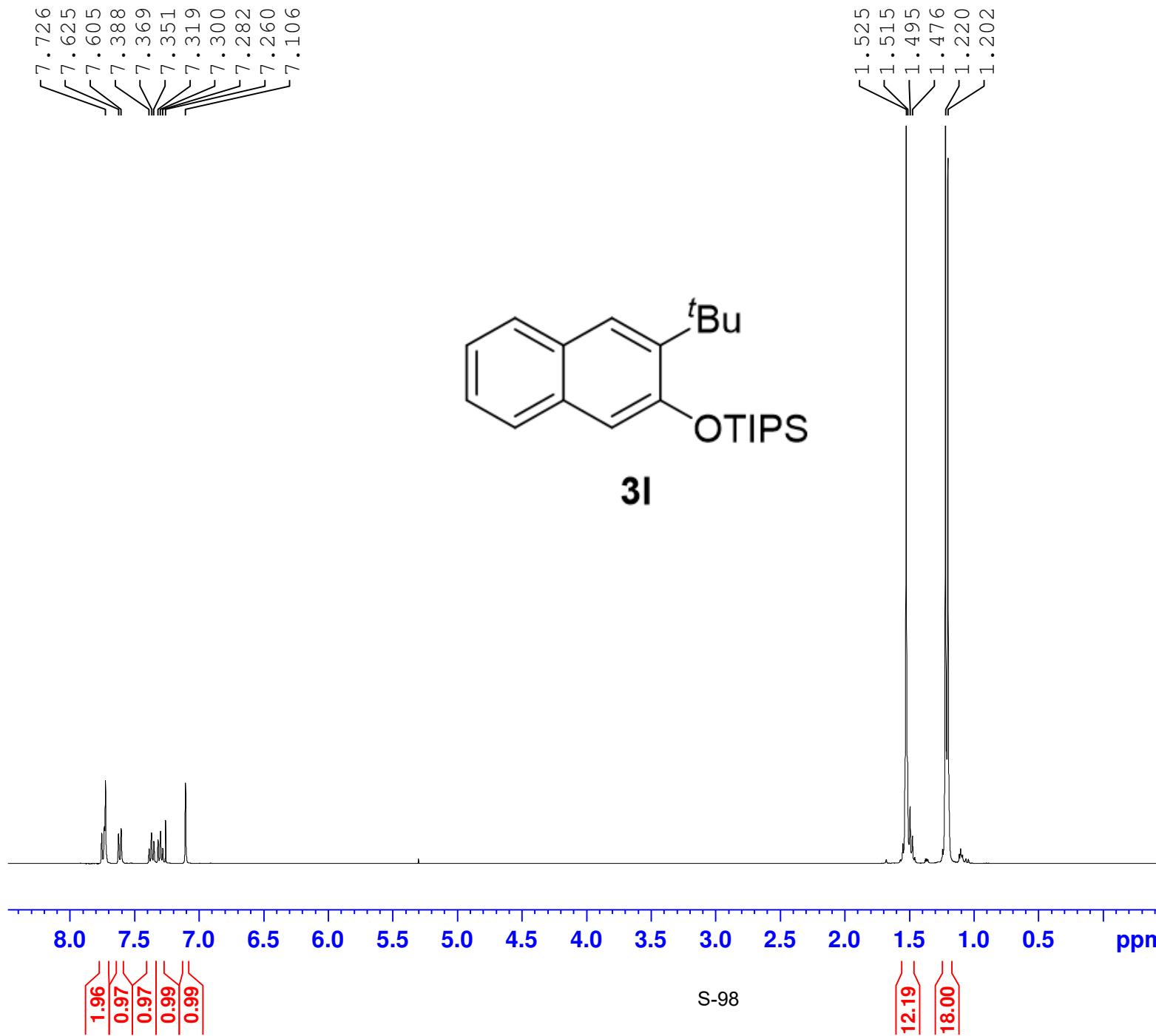
Current Data Parameters
 NAME wuan-224-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160130
 Time 16.47
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 126.97
 DW 62.400 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



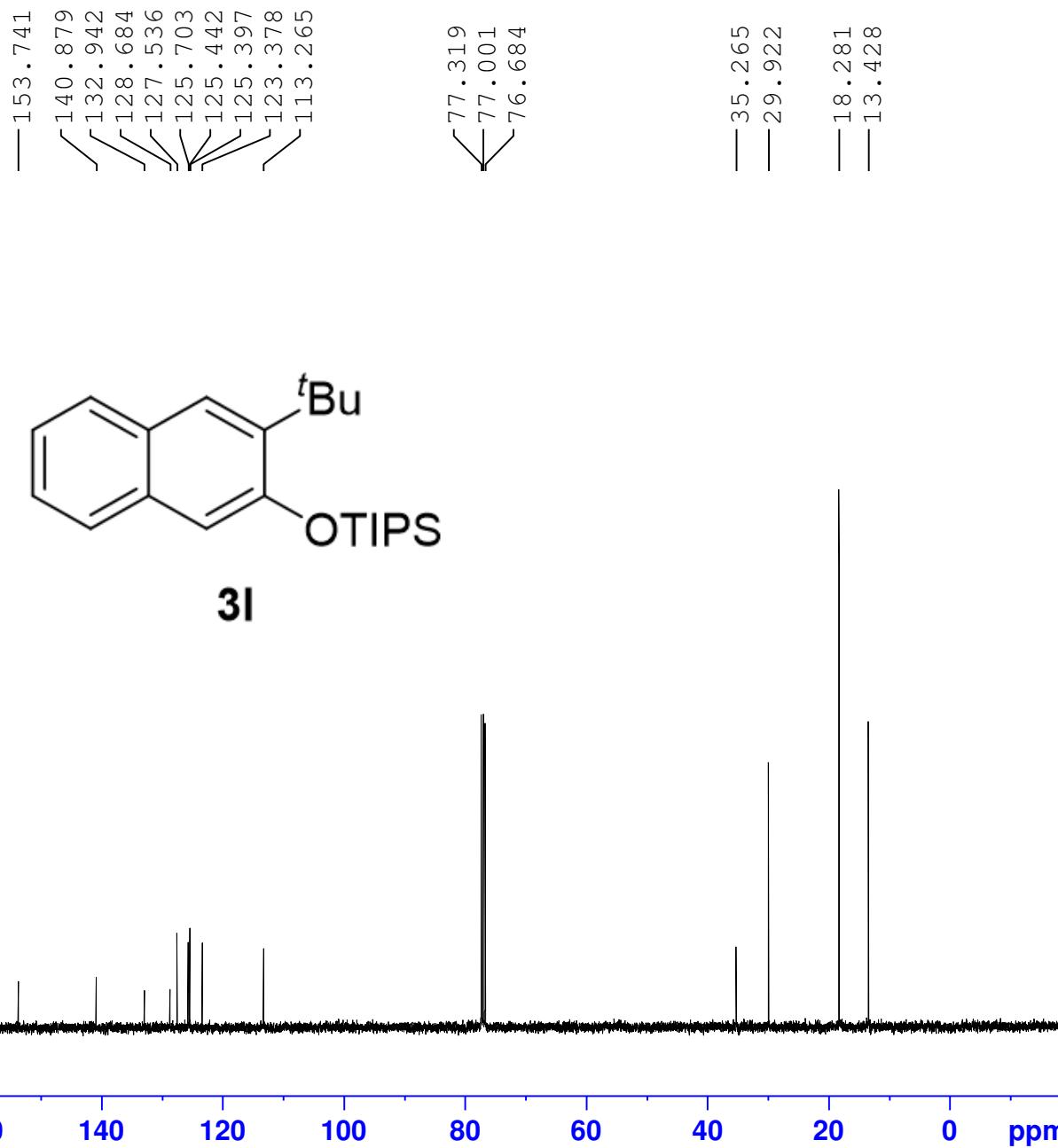
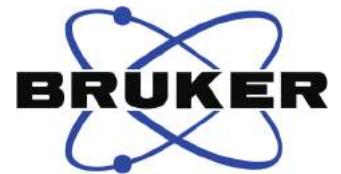


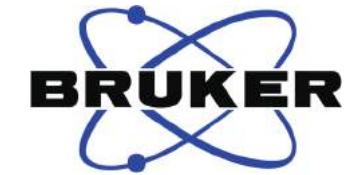
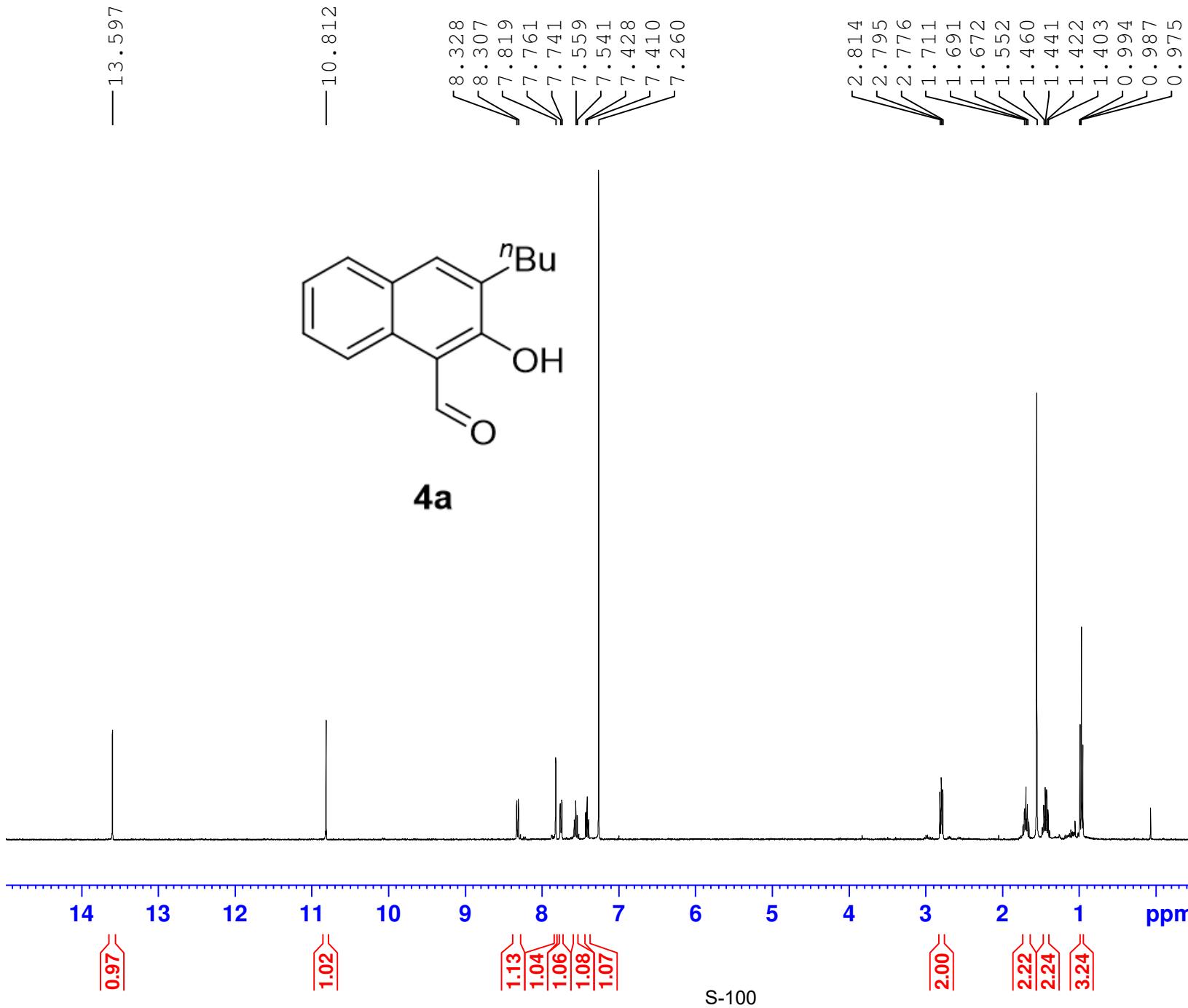
Current Data Parameters
 NAME wuan-239-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160604
 Time 20.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 54.81
 DW 62.400 usec
 DE 6.50 usec
 TE 299.6 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



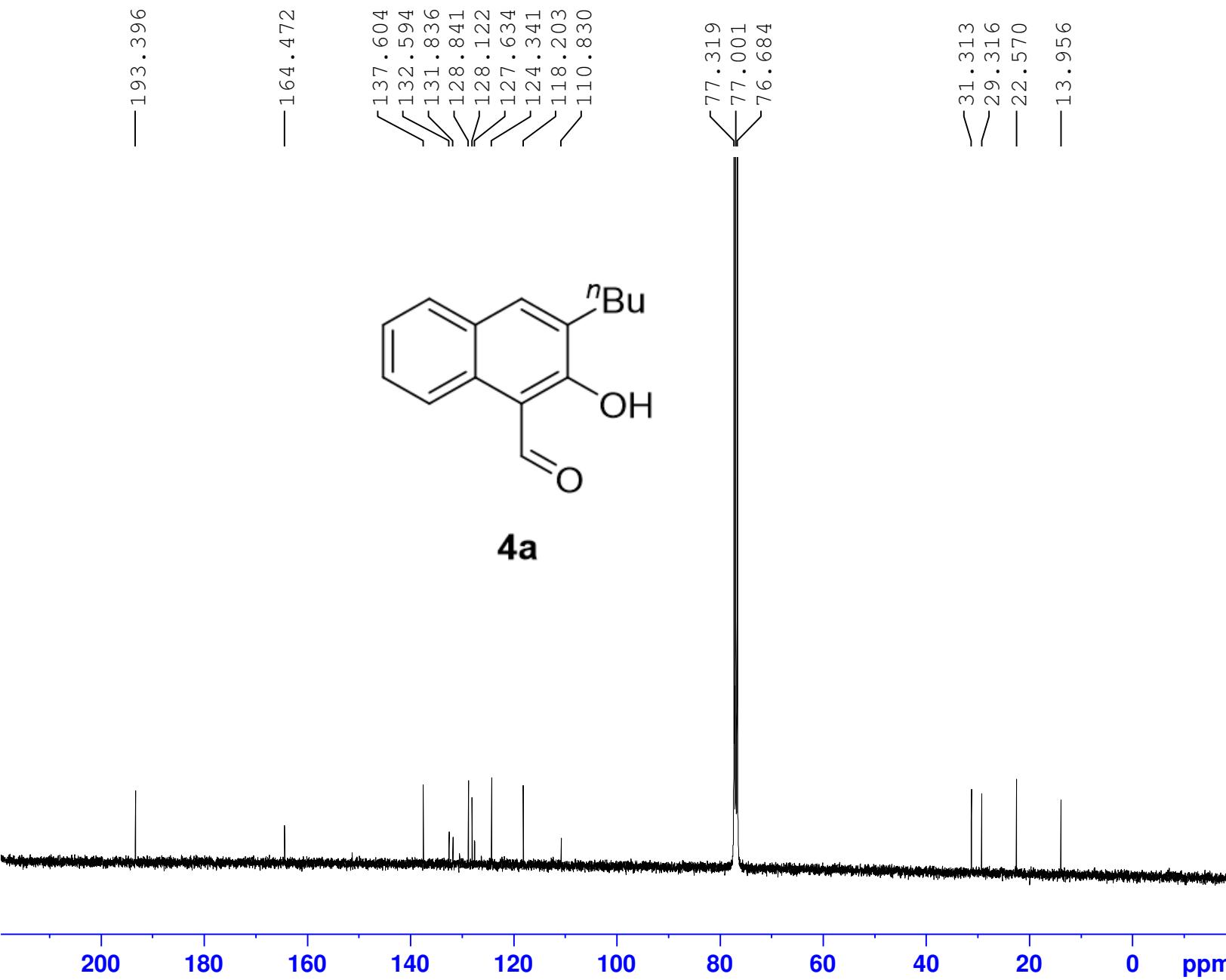


Current Data Parameters
 NAME wuan-112A-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151112
 Time 19.52
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9845889 sec
 RG 812
 DW 60.800 usec
 DE 6.00 usec
 TE 293.4 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 ======
 NUC1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300053 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



S-101



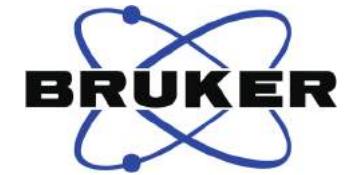
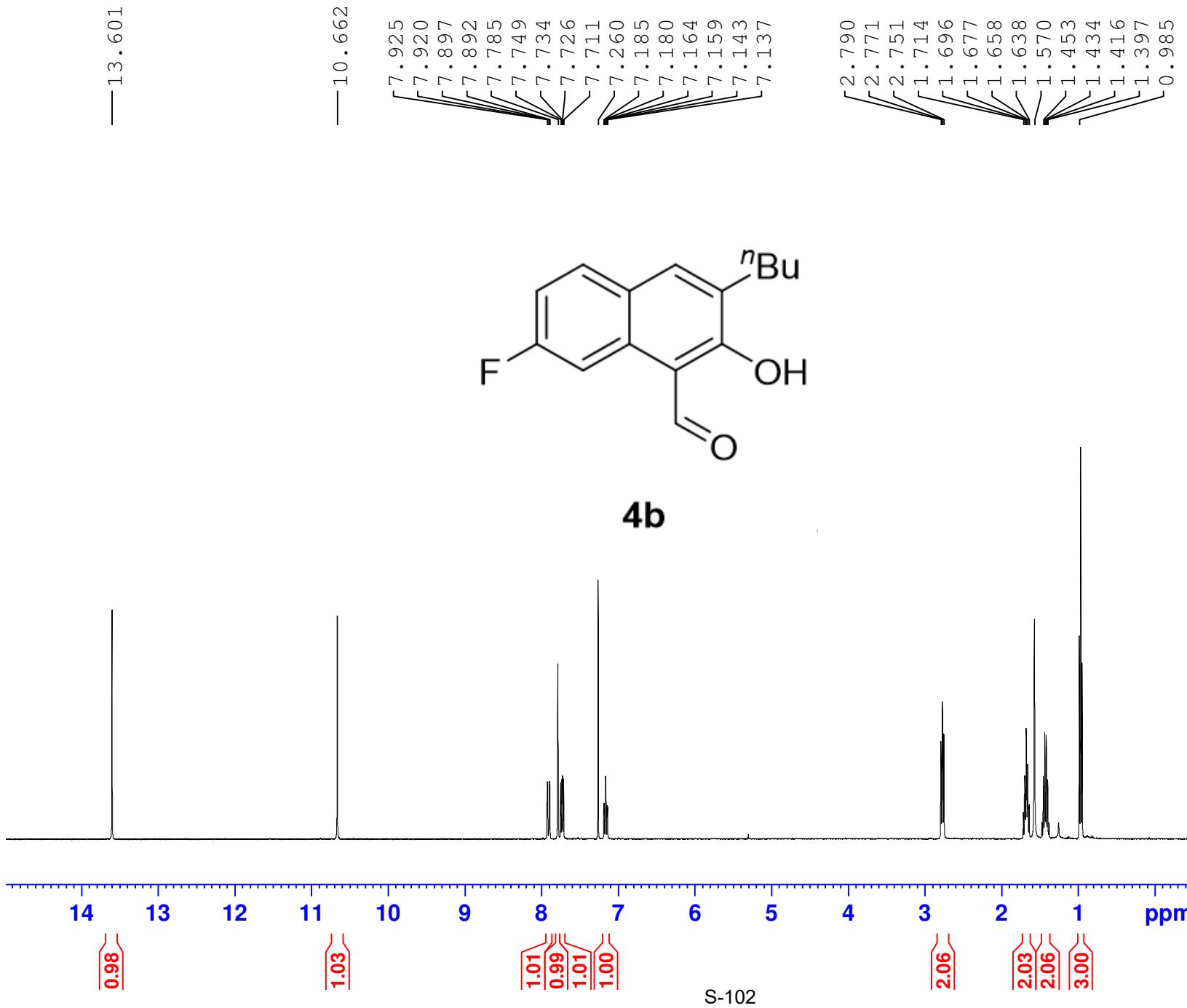
Current Data Parameters
 NAME wuan-112A-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151113
 Time 10.17
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10060
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 298.3 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127693 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME wuan-146-4
 EXPNO 1
 PROCNO 1

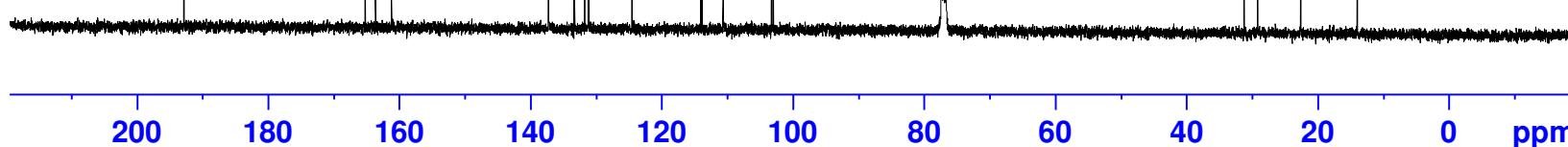
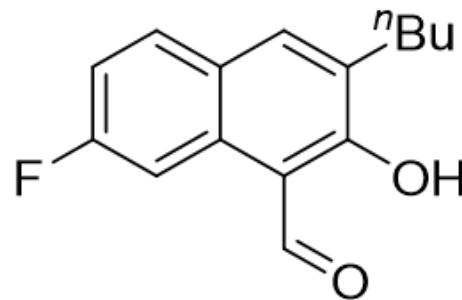
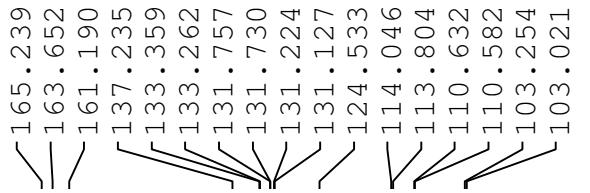
F2 - Acquisition Parameters
 Date_ 20151214
 Time 11.27
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

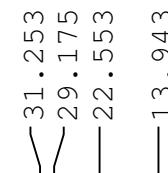
F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



— 192.857



S-103



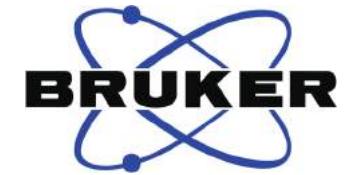
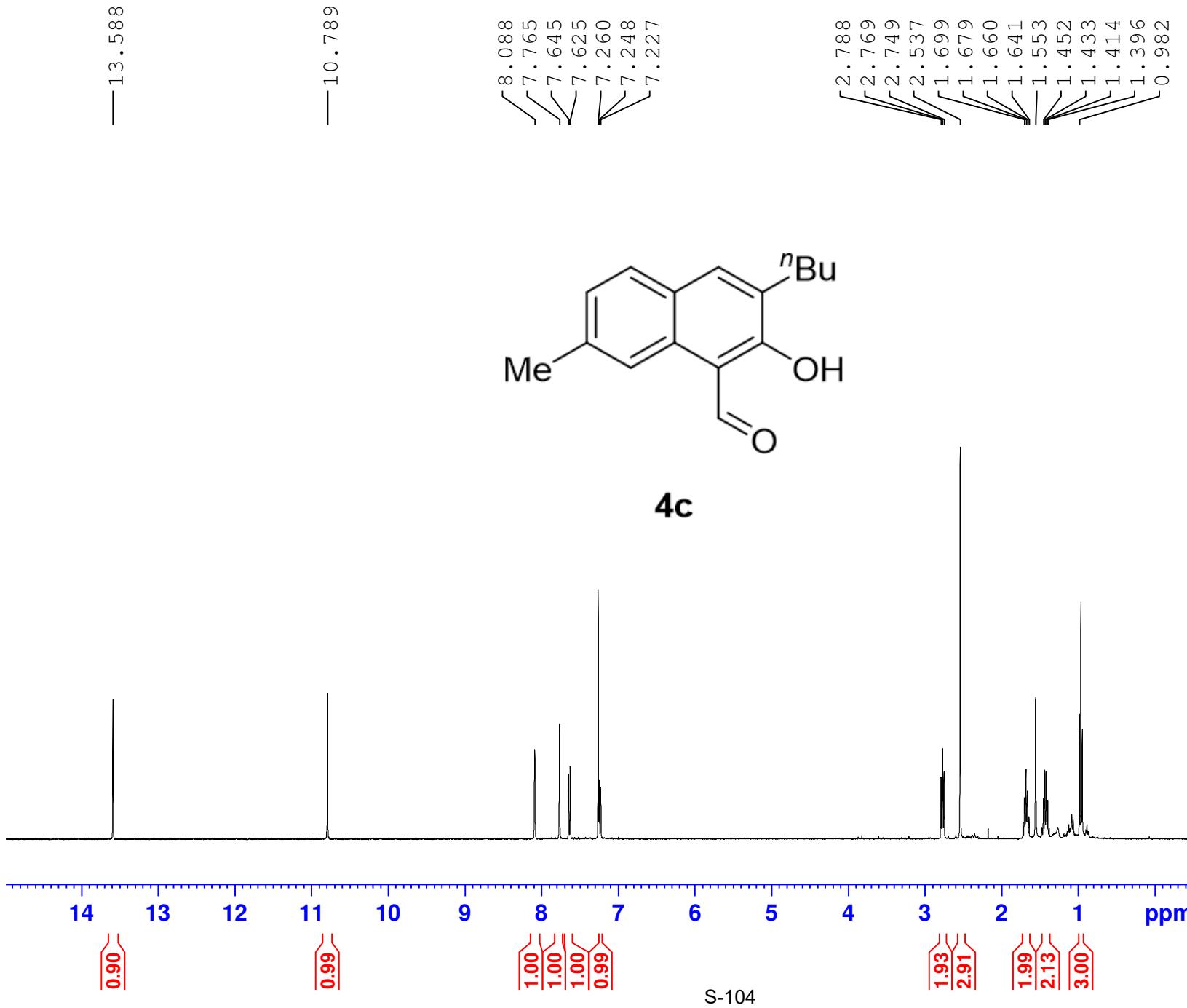
Current Data Parameters
 NAME wuan-146-4
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151214
 Time 13.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1602
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.8 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127701 MHz
 WDW 0
 SSB EM
 LB 1.00 Hz
 GB 0
 PC 1.40

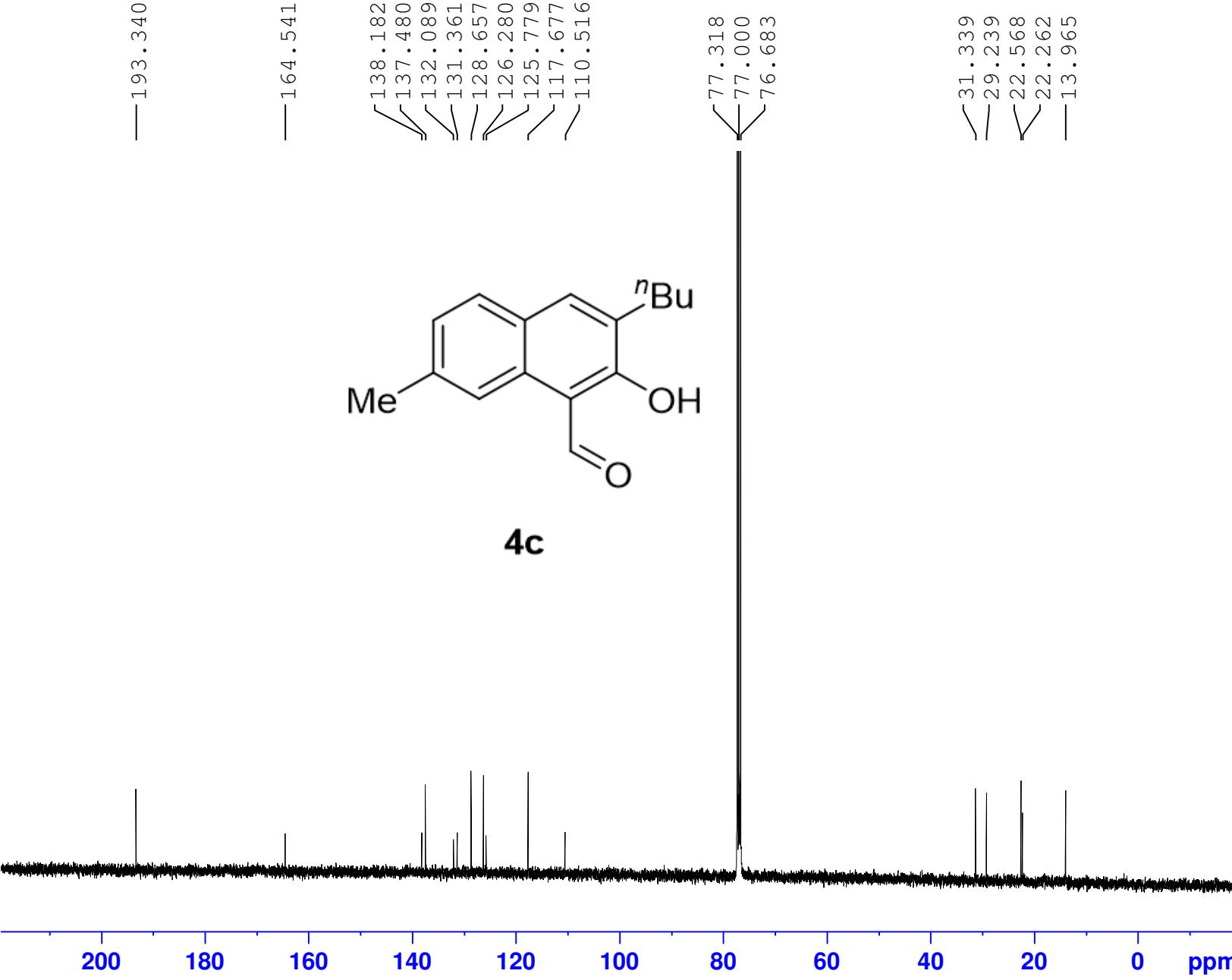


Current Data Parameters
 NAME wuan-118A-3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151114
 Time 17.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



S-105



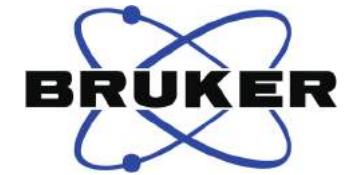
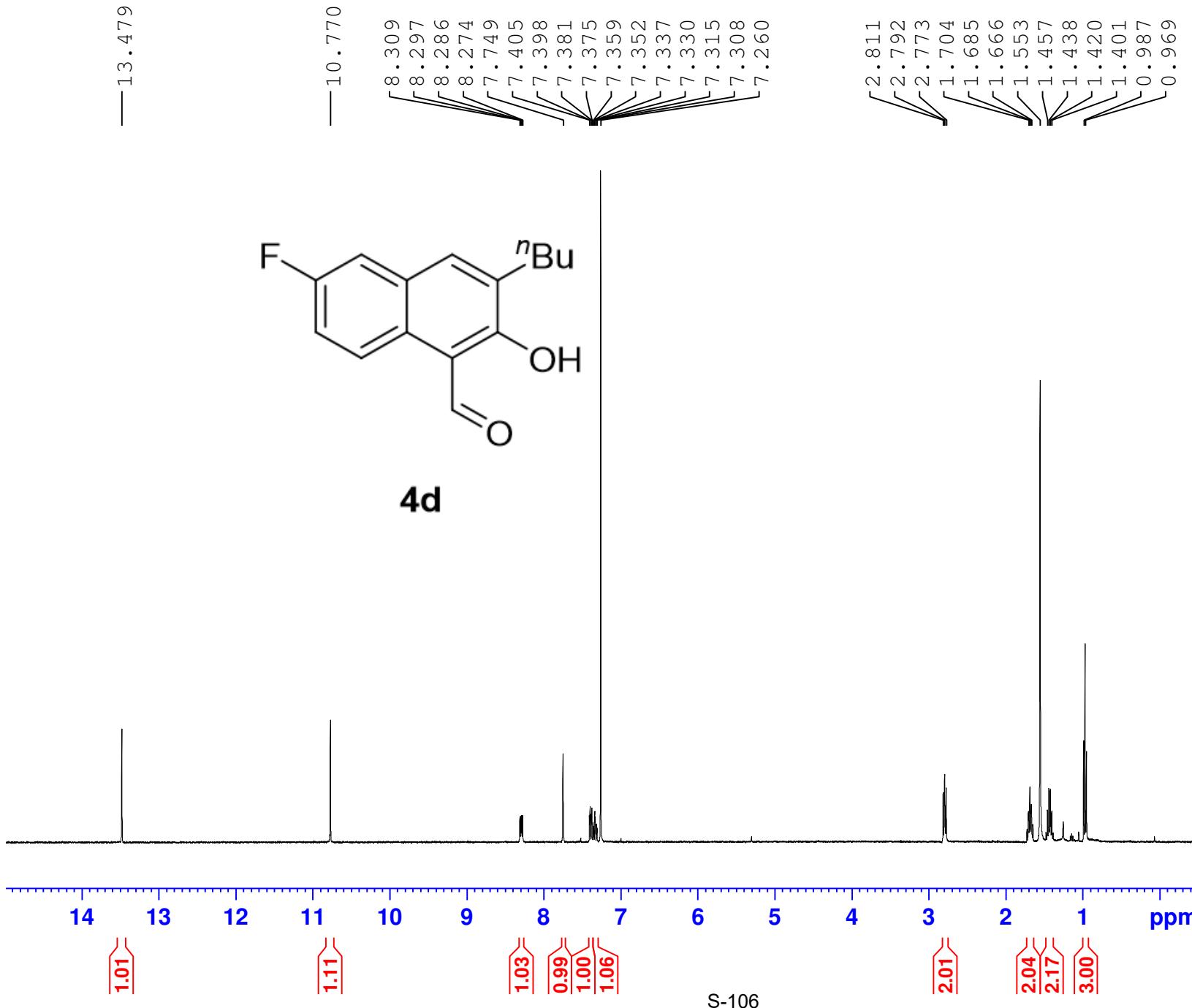
Current Data Parameters
 NAME wuan-118A-3
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151114
 Time 17.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1225
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.8 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127701 MHz
 WDW 0
 SSB EM
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME wuan-136-3
 EXPNO 1
 PROCNO 1

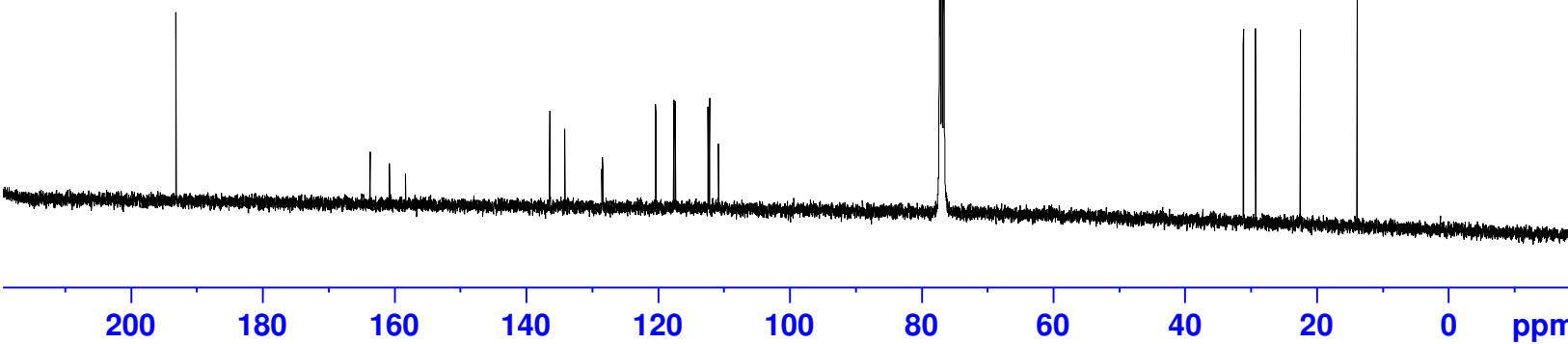
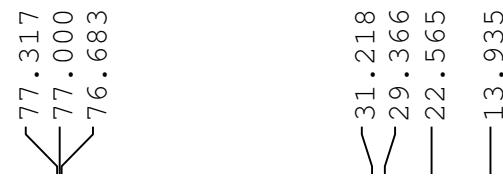
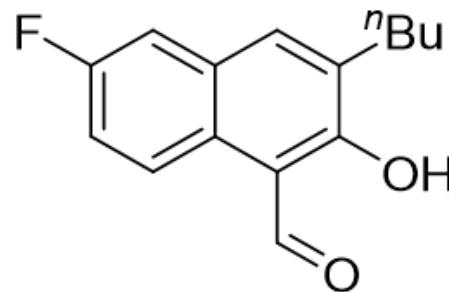
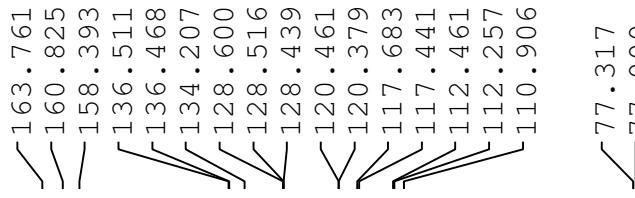
F2 - Acquisition Parameters
 Date_ 20151204
 Time 20.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.5 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



— 193.244



S-107

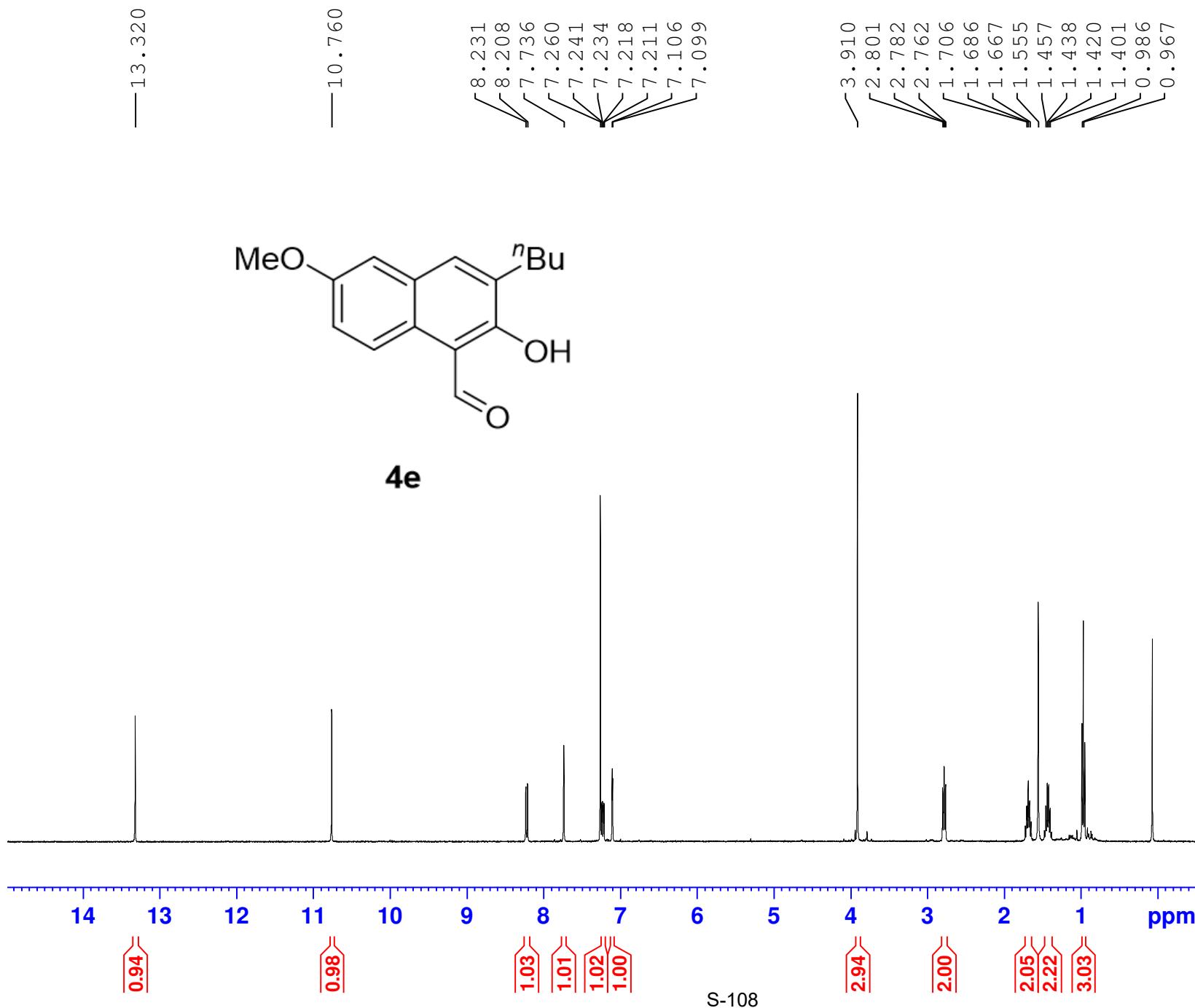
Current Data Parameters
 NAME wuan-136-3
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151206
 Time 23.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10530
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127695 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current	Data	Parameters
NAME	wuan-137-1	
EXPNO		1
PROCNO		1

```

F2 - Acquisition Parameters
Date_           20151204
Time            20.37
INSTRUM        spect
PROBHD         5 mm PABBO BB/
PULPROG        zg30
TD              65536
SOLVENT         CDC13
NS                4
DS                0
SWH             8012.820 Hz
FIDRES        0.122266 Hz
AQ              4.0894465 sec
RG              187.77
DW              62.400 usec
DE                6.50 usec
TE                296.6 K
D1      1.000000000 sec
TD0                  1

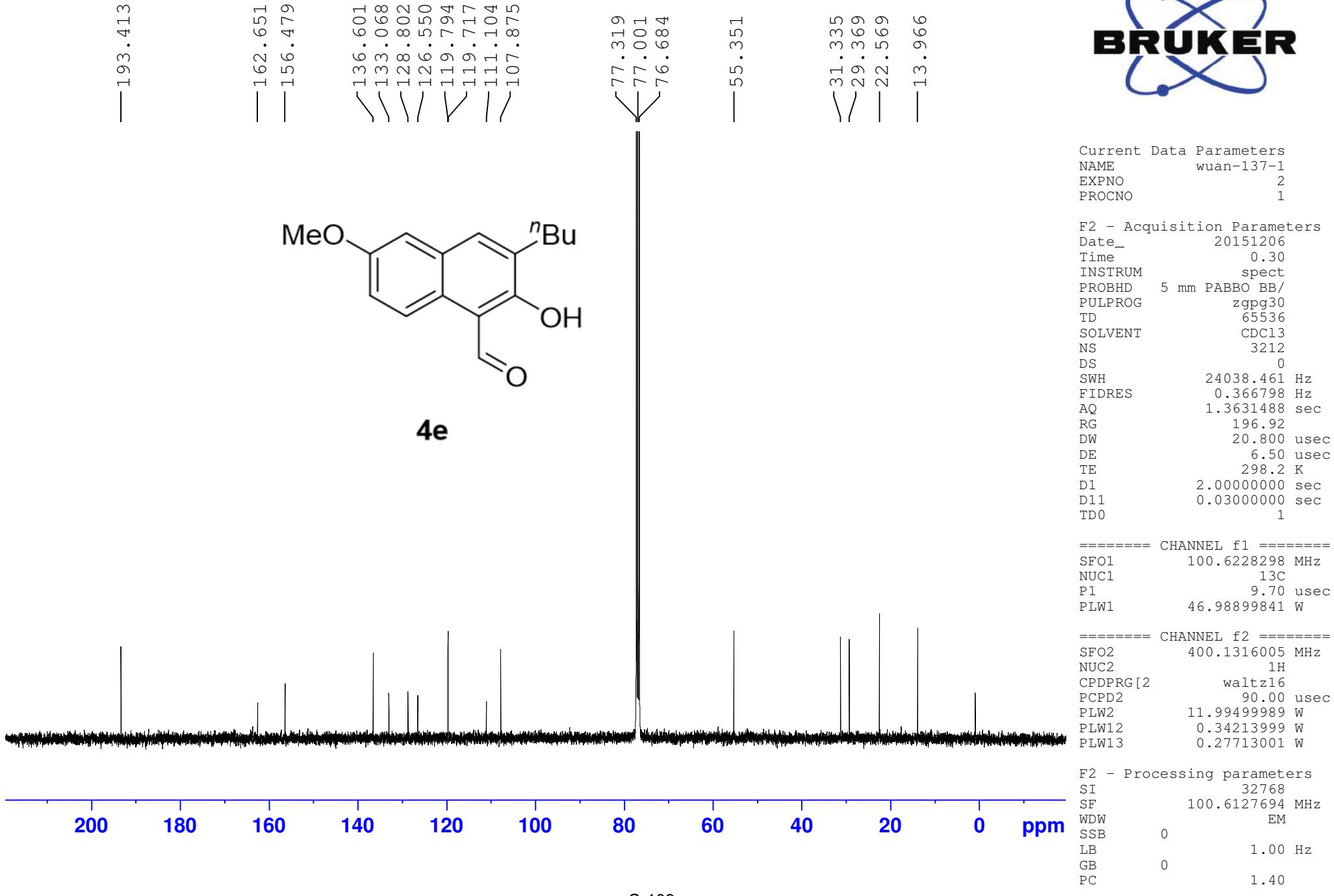
```

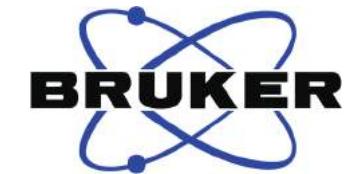
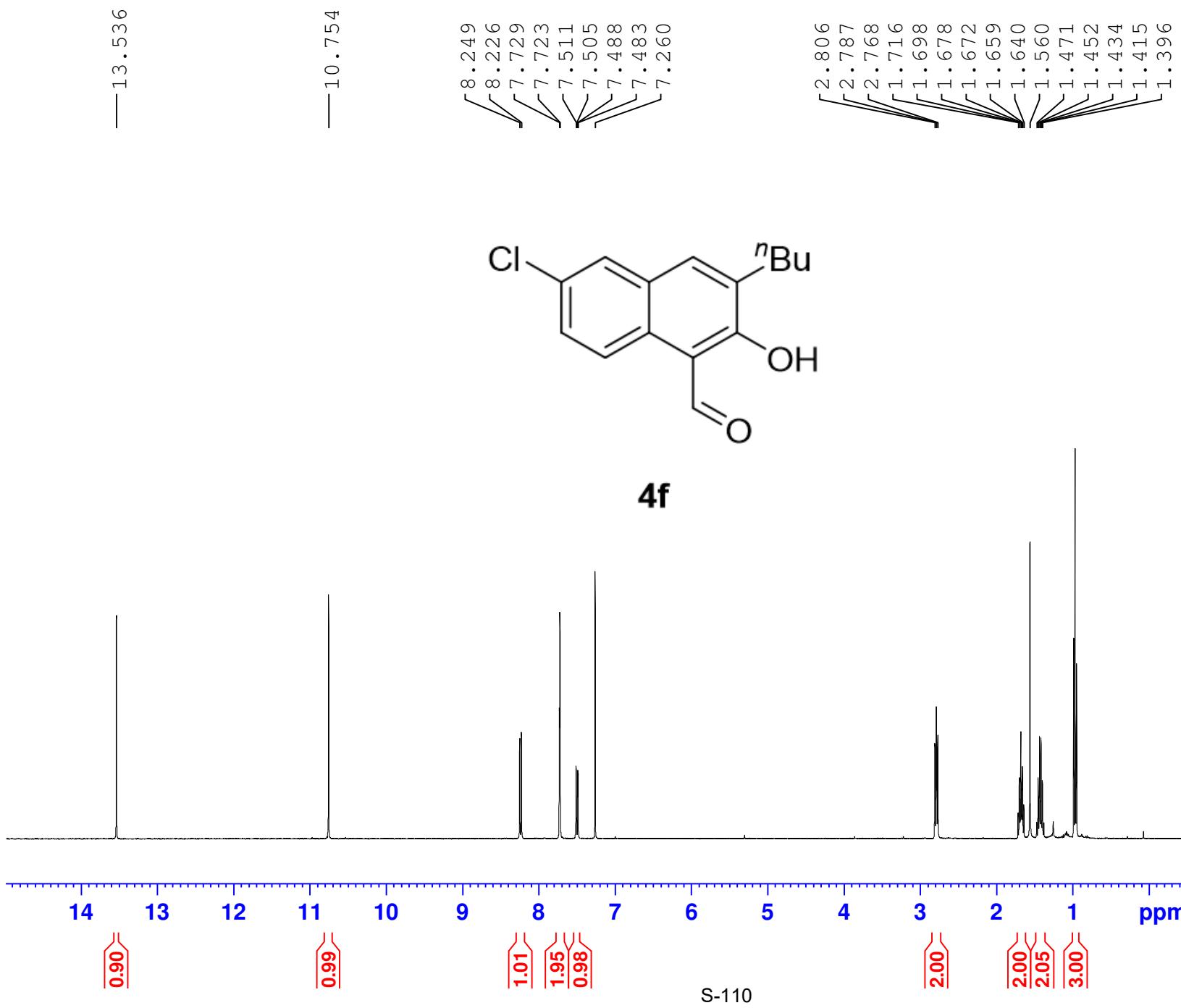
===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PI.W1 11.99499989 W

```

F2 - Processing parameters
SI           65536
SF          400.1300092 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB          0
PC          1.00

```





Current Data Parameters
 NAME wuan-147-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151212
 Time 10.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

 BRUKER

Current	Data	Parameters
NAME	wuan-147-2	
EXPNO		4
PROCNO		1

```

F2 - Acquisition Parameters
Date_           20151212
Time            11.39
INSTRUM        spect
PROBHD         5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT         CDC13
NS              785
DS              0
SWH             24038.461 Hz
FIDRES         0.366798 Hz
AQ              1.3631488 sec
RG              196.92
DW              20.800 usec
DE              6.50 usec
TE              298.0 K
D1              2.0000000 sec
D11             0.03000000 sec
TD0

```

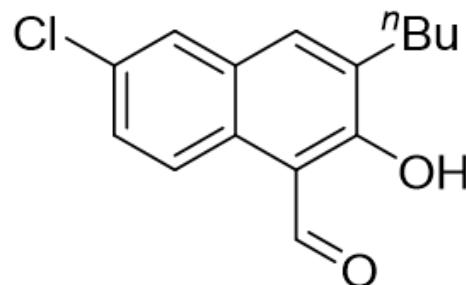
===== CHANNEL f1 ======
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PIW1 46.98899841 W

```

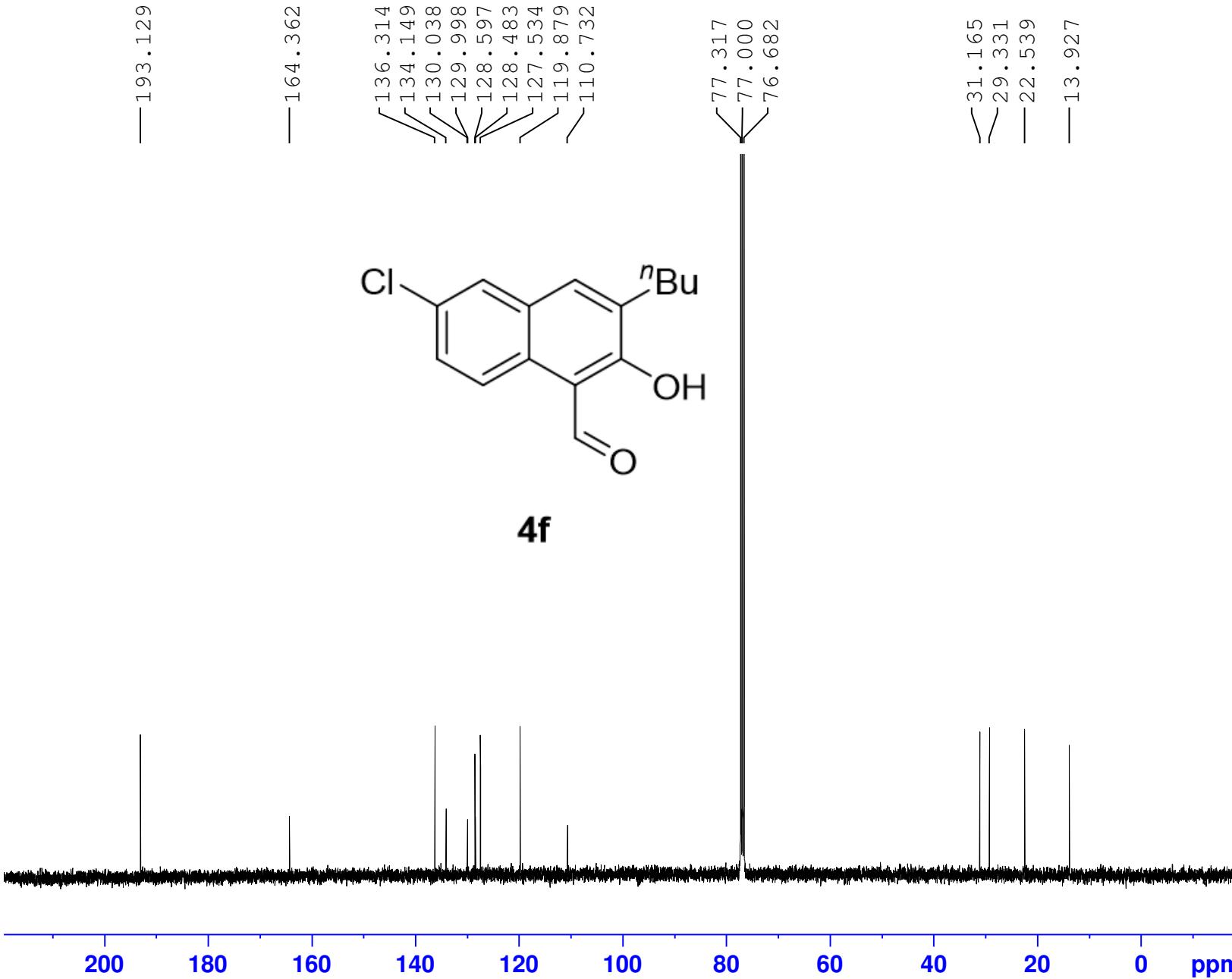
===== CHANNEL f2 =====
SFO2          400.1316005 MHz
NUC2           1H
CPDPRG[2]     waltz16
PCPD2          90.00 usec
PLW2           11.9949998 W
PLW12          0.34213999 W
PLW13          0.27713001 W

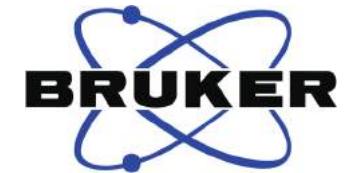
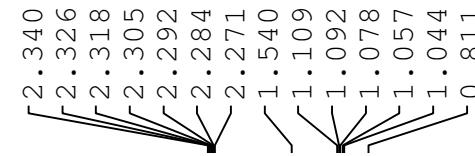
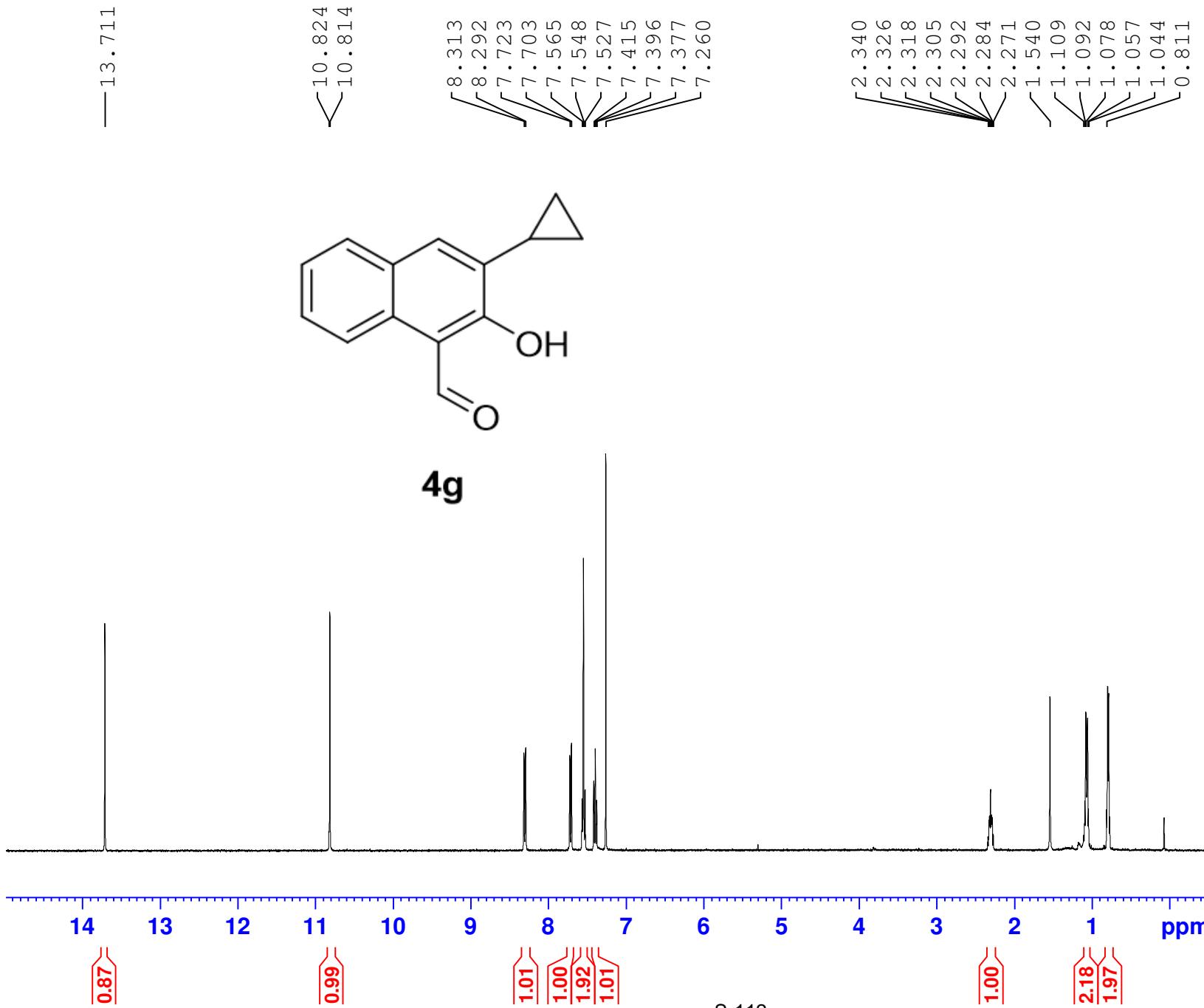
```

F2 - Processing parameters
SI 32768
SF 100.6127700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



4f





Current Data Parameters
 NAME wuan-236-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160216
 Time 21.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 297.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME wuan-236-2
 EXPNO 2
 PROCNO 1

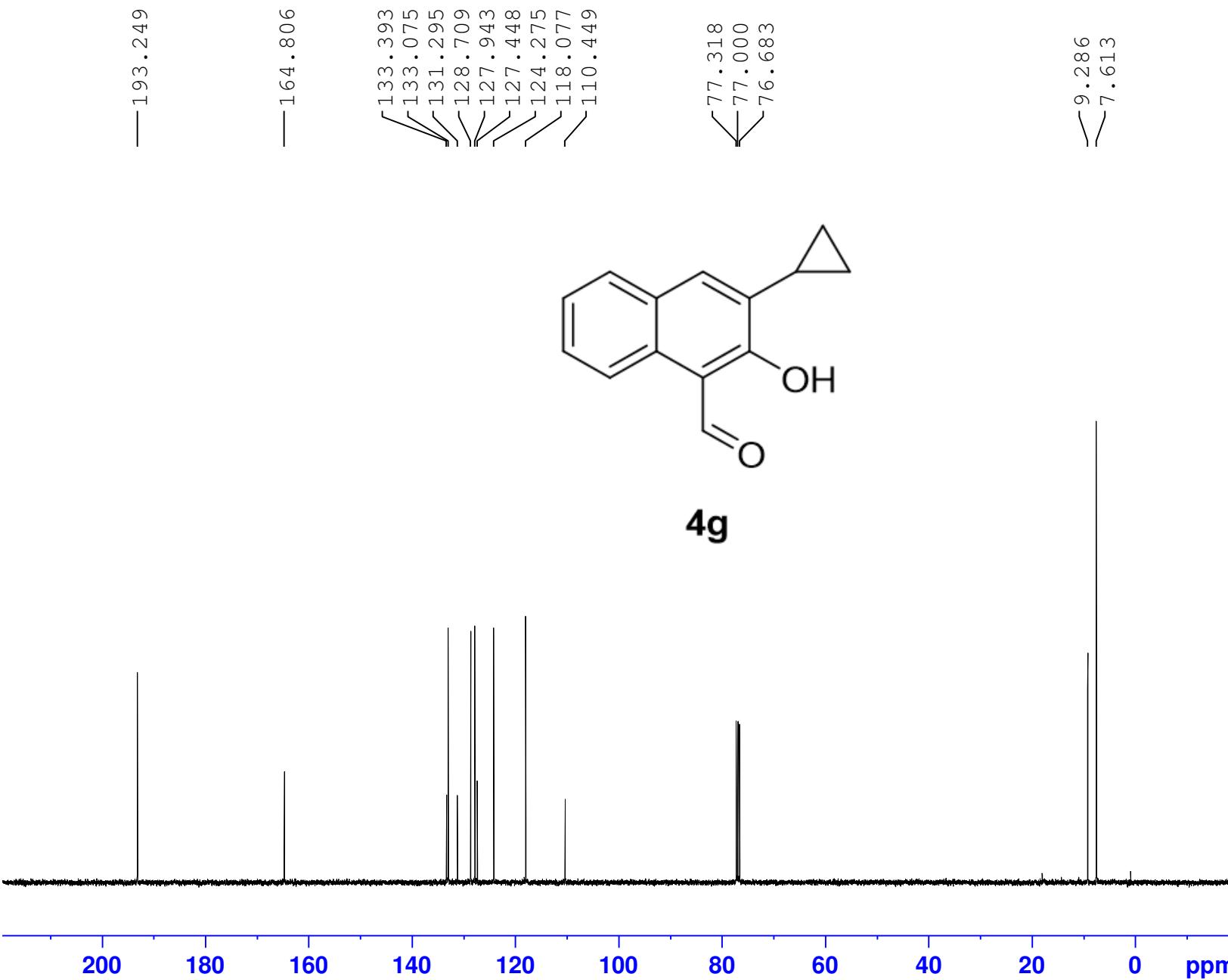
F2 - Acquisition Parameters
 Date_ 20160217
 Time 18.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.5 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

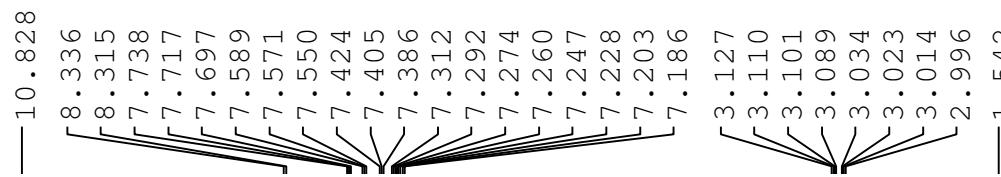
F2 - Processing parameters
 SI 32768
 SF 100.6127797 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

S-113





— 13.632

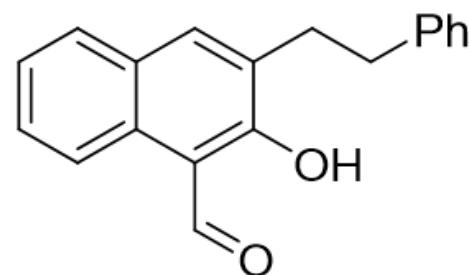


Current Data Parameters
 NAME wuan-238-2
 EXPNO 2
 PROCNO 1

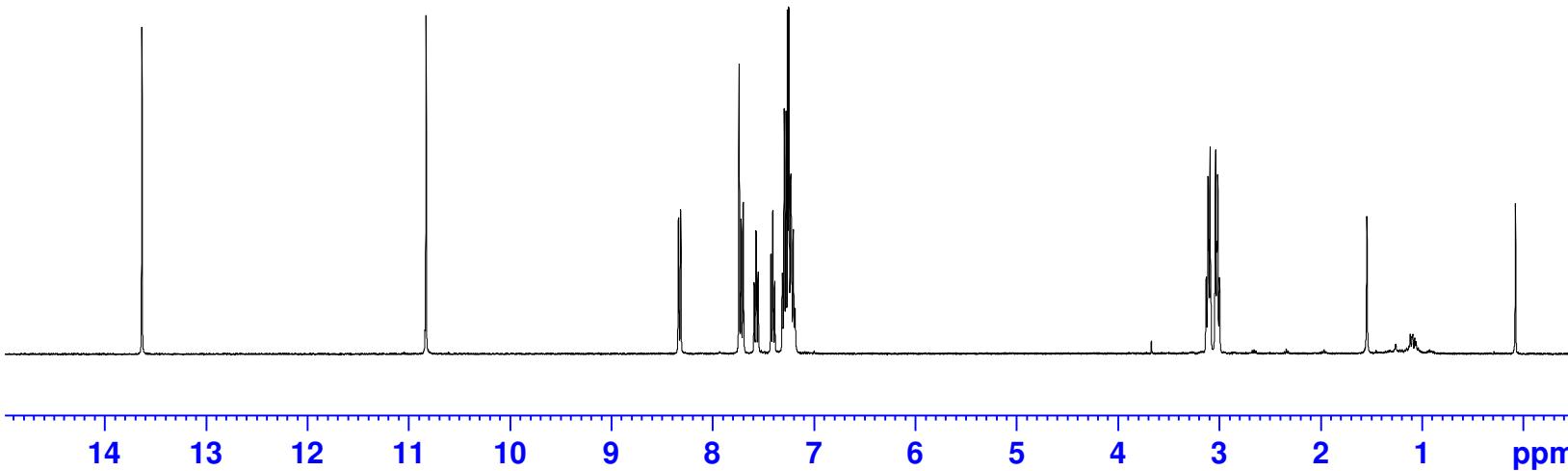
F2 - Acquisition Parameters
 Date_ 20160217
 Time 18.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.8 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



4h

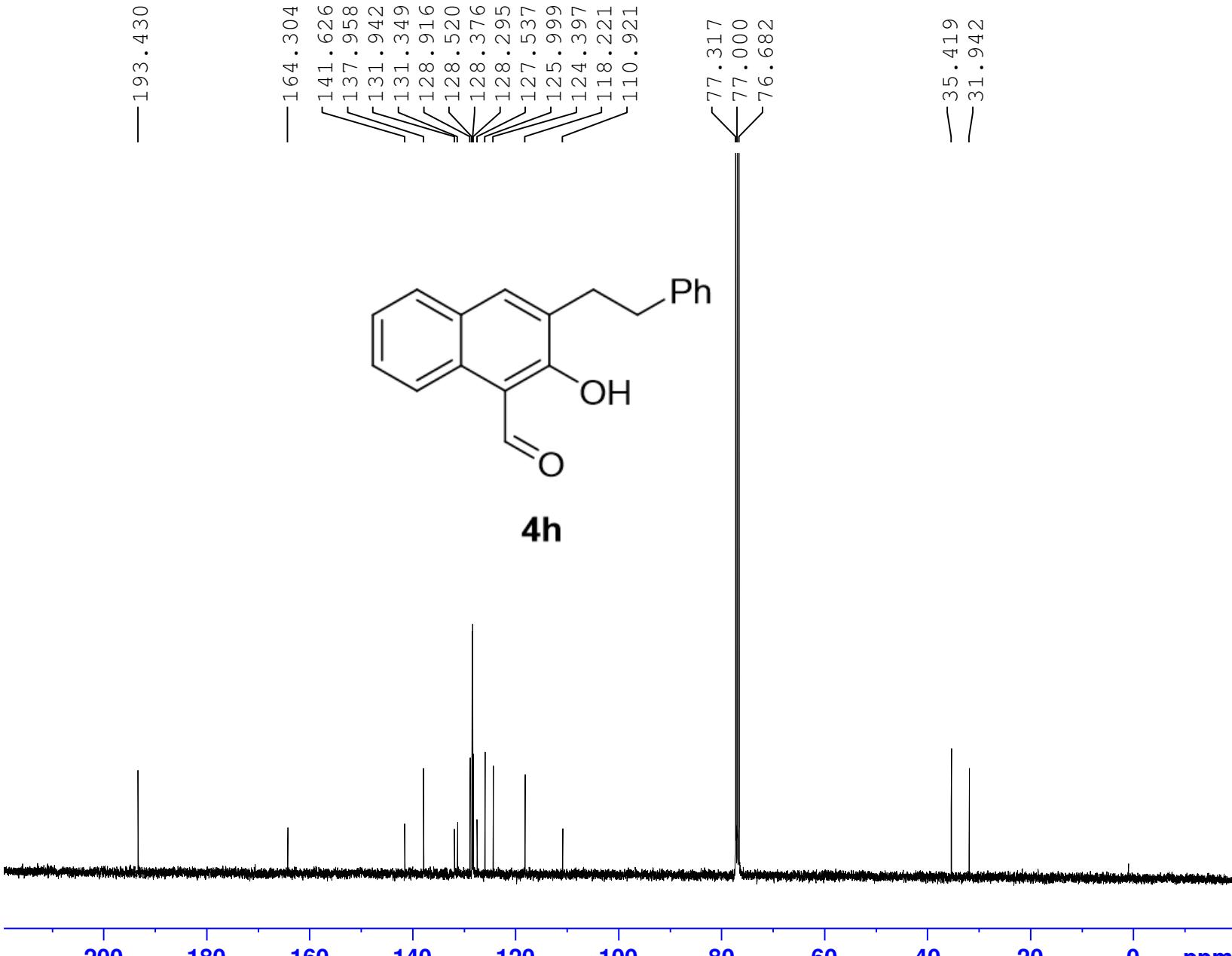


0.95

1.03

1.05
2.01
1.00
1.02
2.02
3.07

2.01
2.00



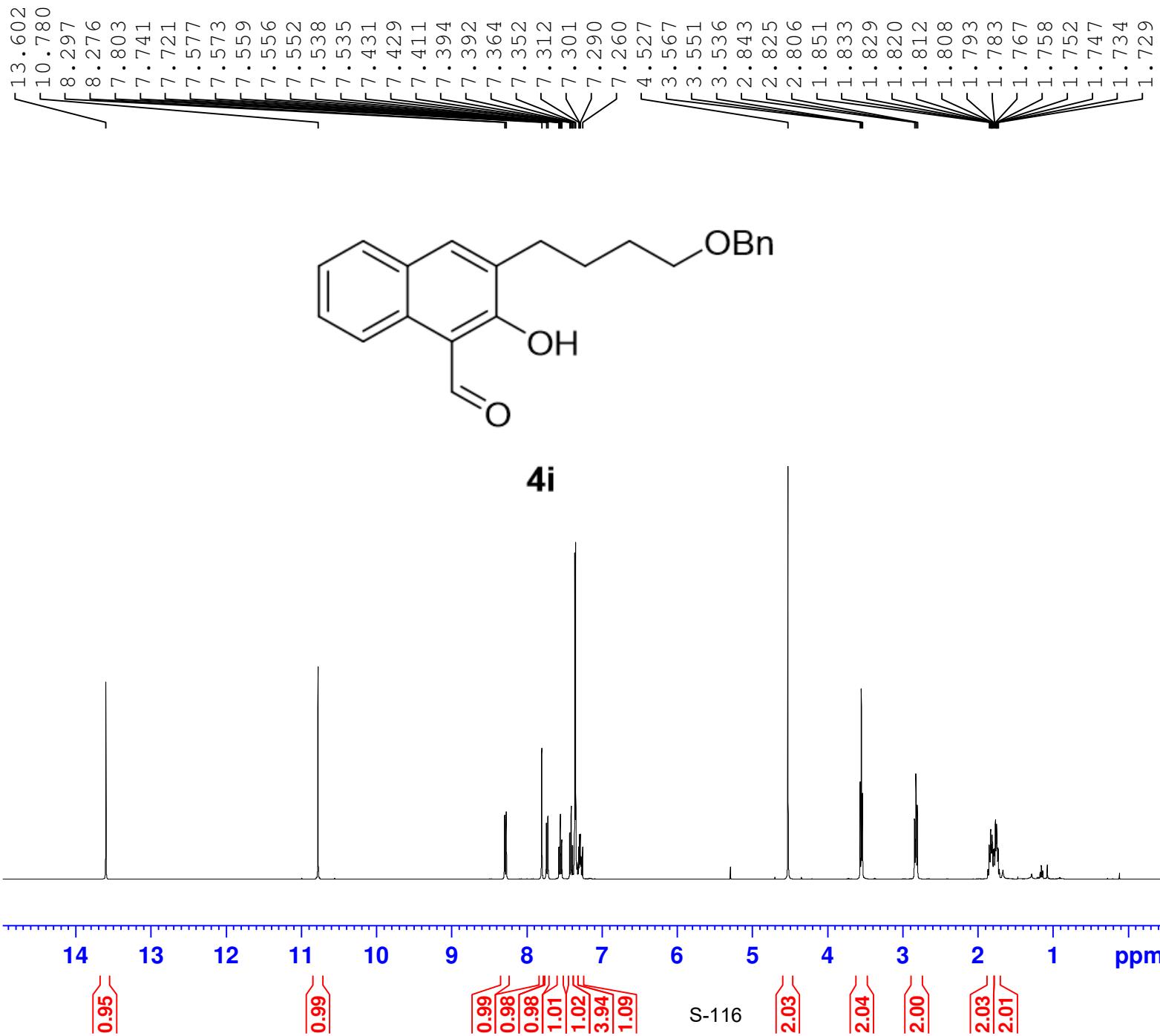
Current Data Parameters
 NAME wuan-238-2
 EXPNO 3
 PROCNO 1

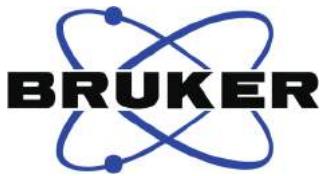
F2 - Acquisition Parameters
 Date_ 20160217
 Time 18.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 956
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.8 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

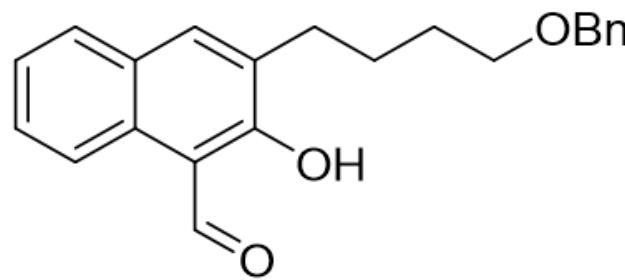
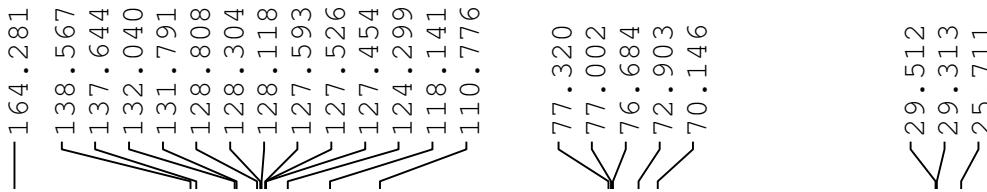
===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127706 MHz
 WDW 0 EM
 SSB 1.00 Hz
 LB 0
 GB 0 1.40
 PC

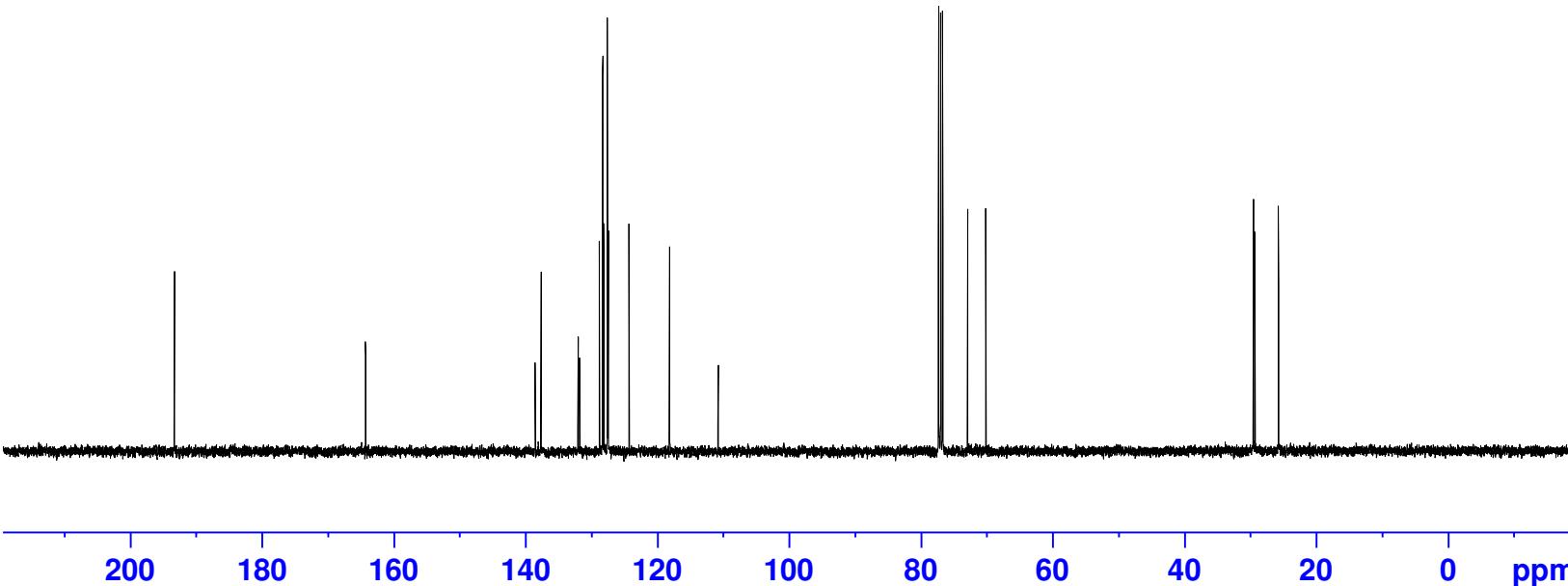




—193.298



4i



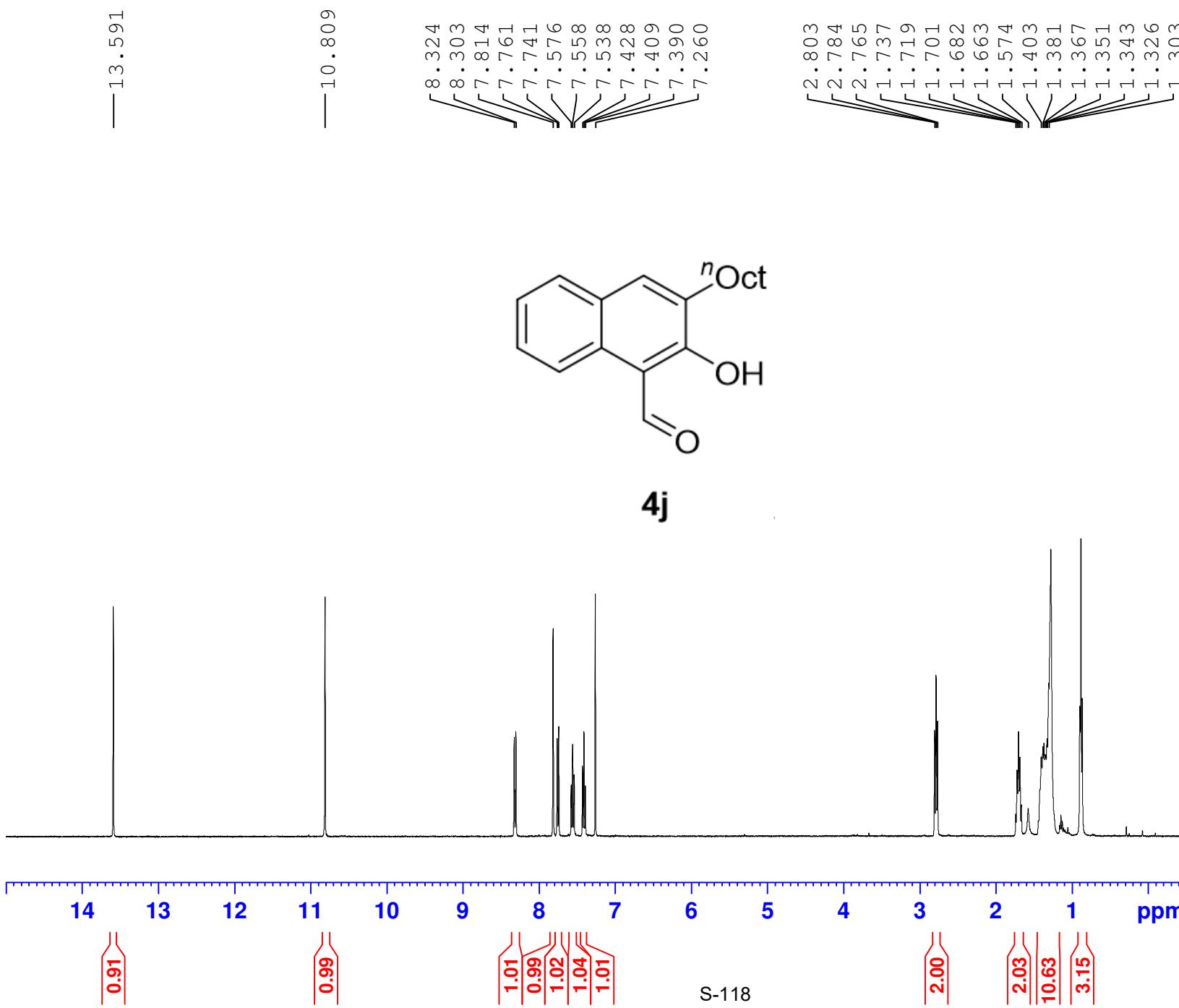
Current Data Parameters
 NAME wuan-237-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160604
 Time 20.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 71
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 300.3 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127774 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME wuan-230-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160216
 Time 21.38
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.9 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



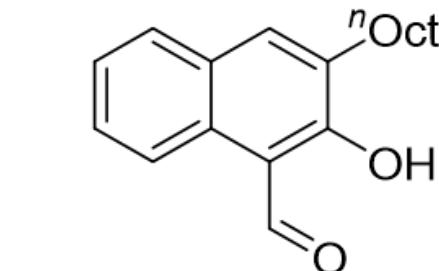
Current Data Parameters
 NAME wuan-230-3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160217
 Time 18.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 123
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 296.7 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

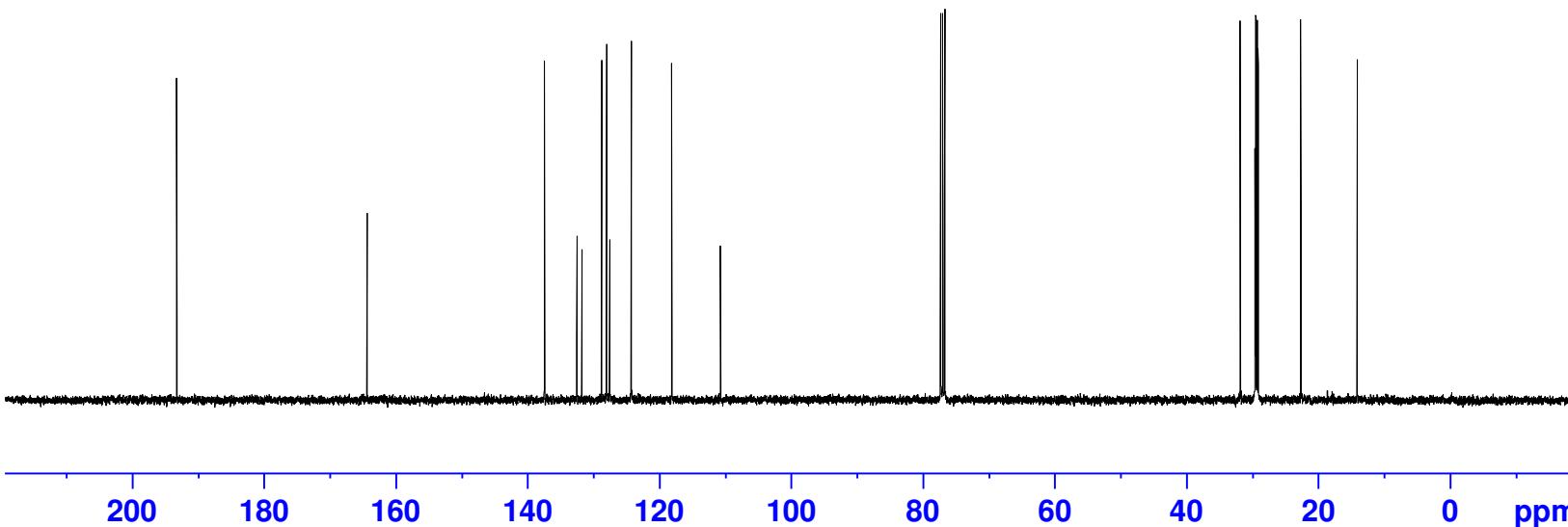
===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

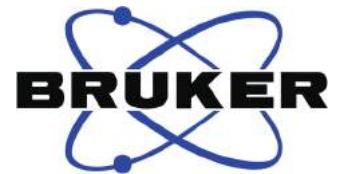
===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127757 MHz
 WDW 0
 SSB EM
 LB 1.00 Hz
 GB 0
 PC 1.40

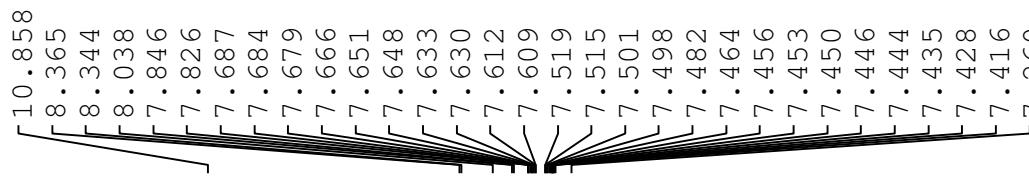


4j





— 13.809

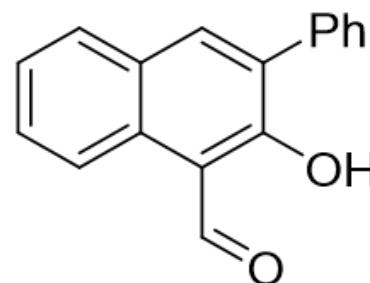


Current Data Parameters
NAME wuan-240-3
EXPNO 1
PROCNO 1

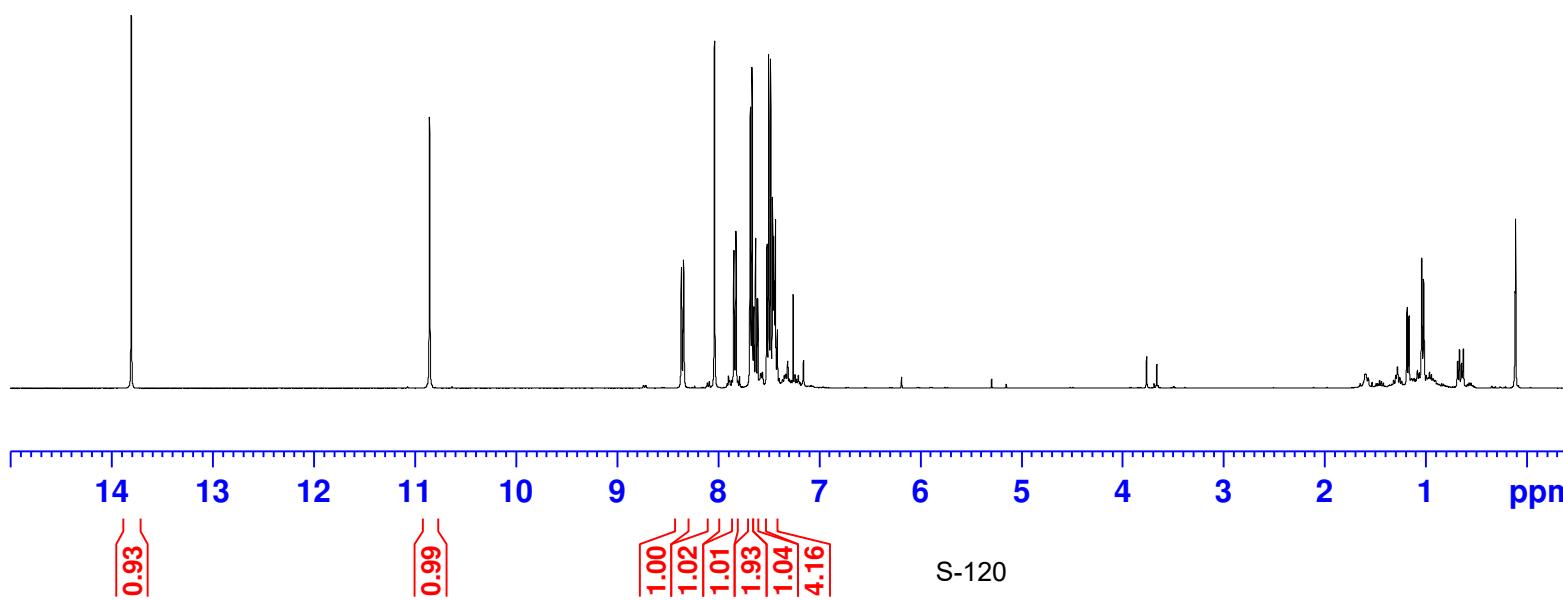
F2 - Acquisition Parameters
Date_ 20160604
Time 20.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 299.5 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

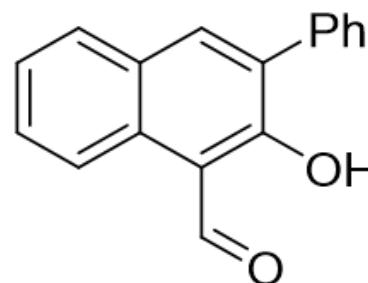
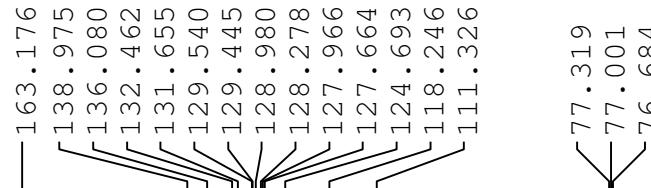


4k

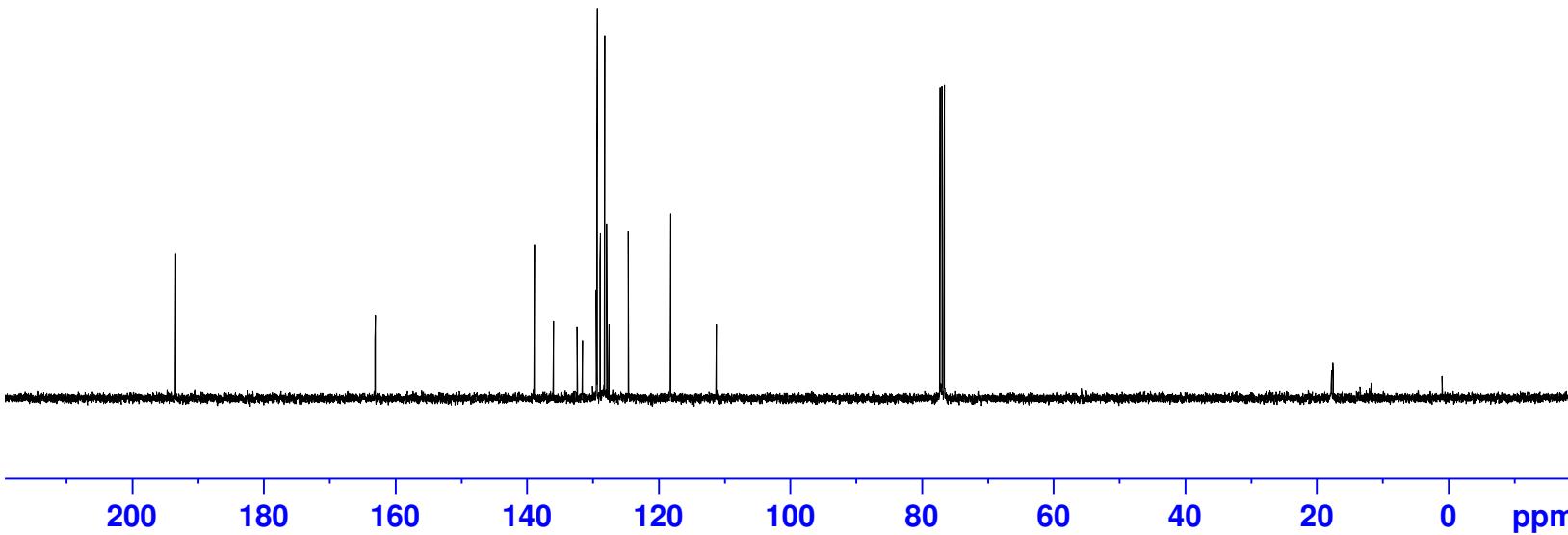




—193.492



4k



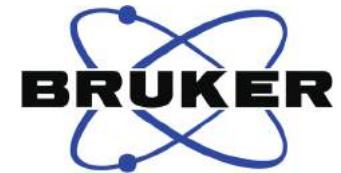
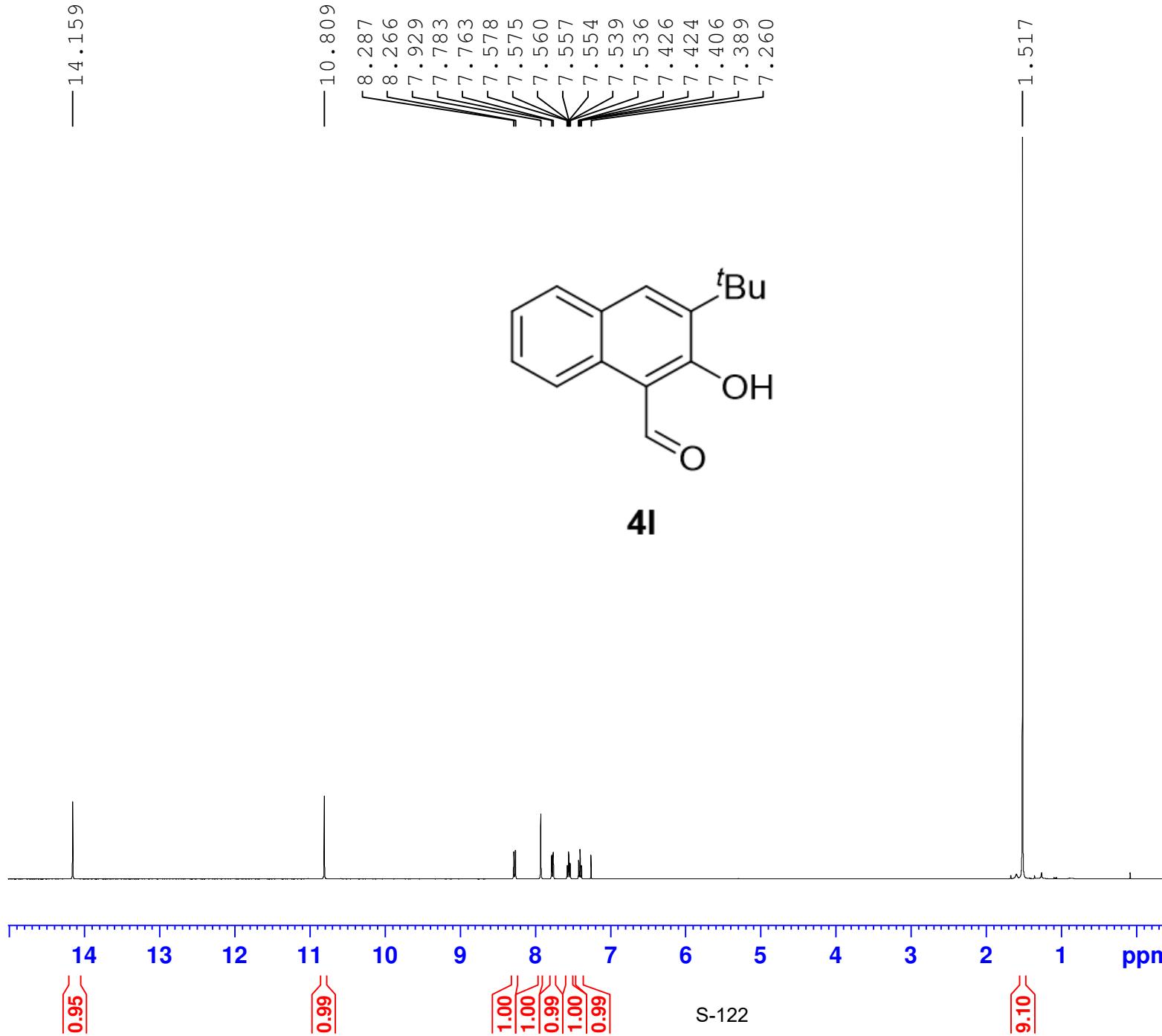
Current Data Parameters
NAME wuan-240-3
EXPNO 2
PROCNO 1

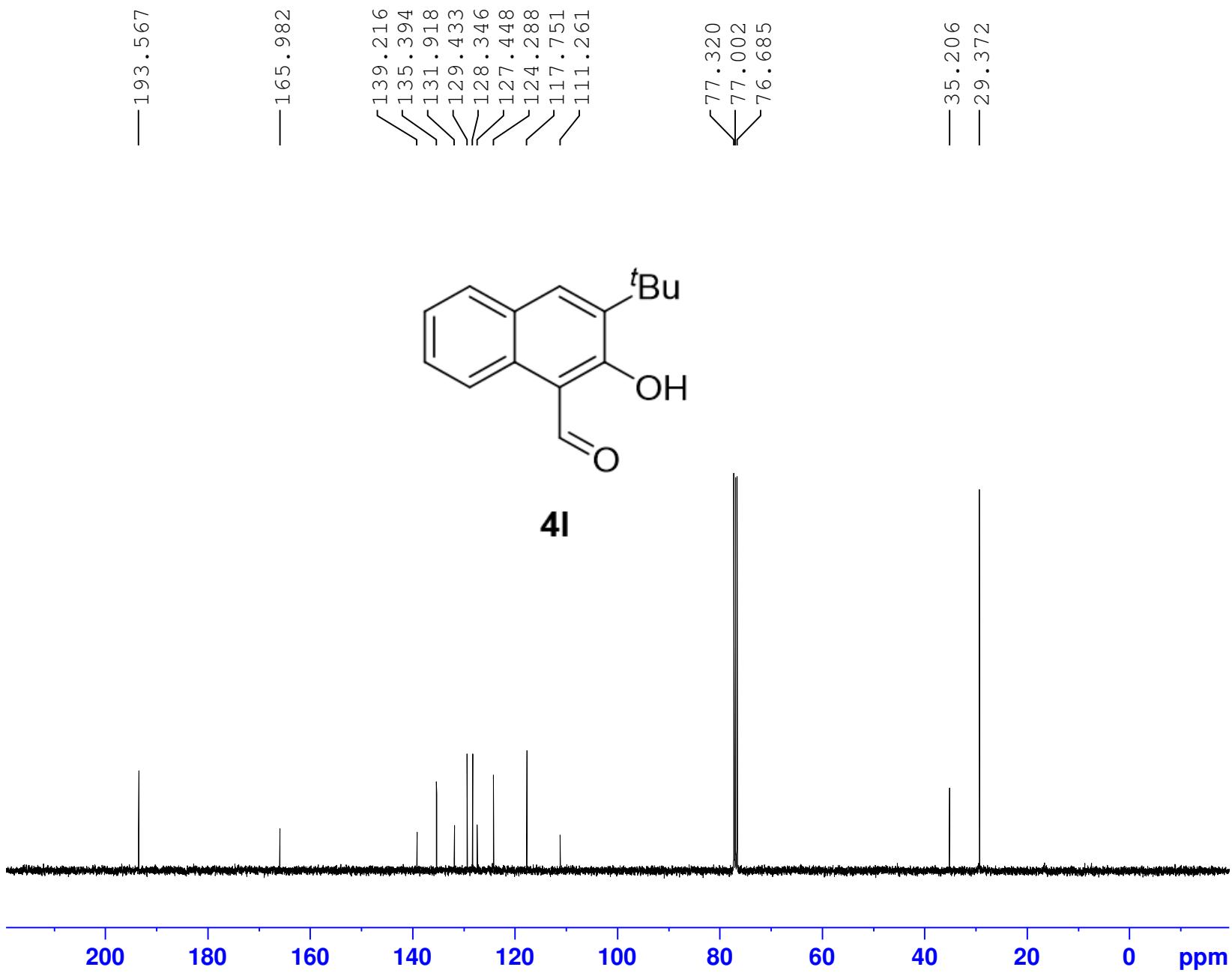
F2 - Acquisition Parameters
Date_ 20160604
Time 20.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPKG[2] waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127752 MHz
WDW 0
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





S-123

Current Data Parameters
 NAME wuan-239-4
 EXPNO 2
 PROCNO 1

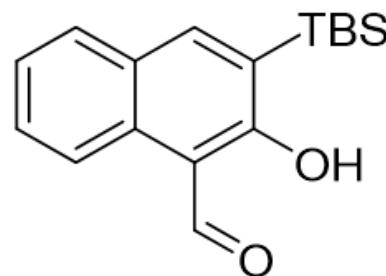
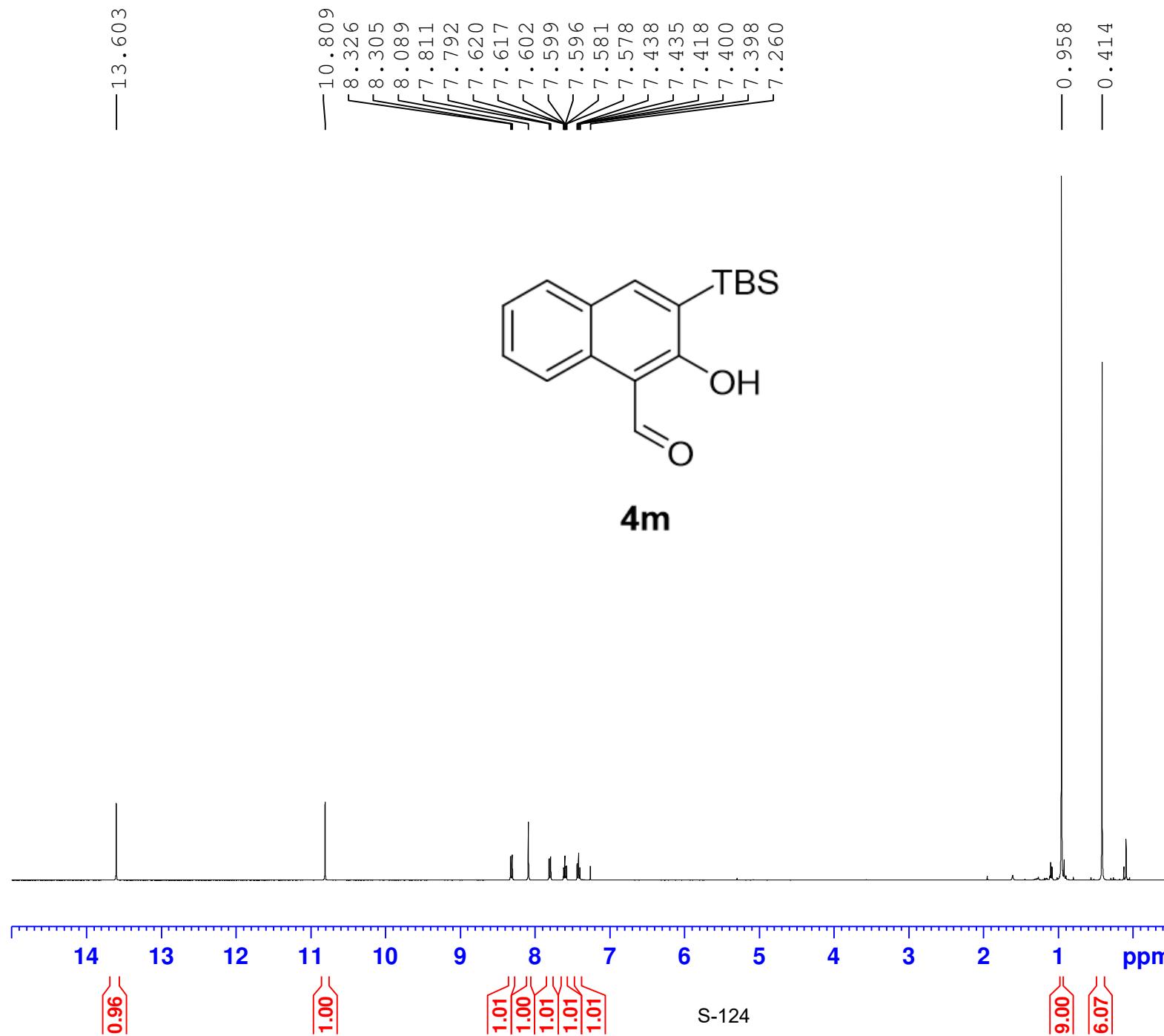
F2 - Acquisition Parameters
 Date_ 20160605
 Time 22.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 125
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 300.8 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 ¹³C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 ¹H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127707 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

— 13.603



4m



Current	Data	Parameters
NAME	wuan-245-3	
EXPNO		1
PROCNO		1

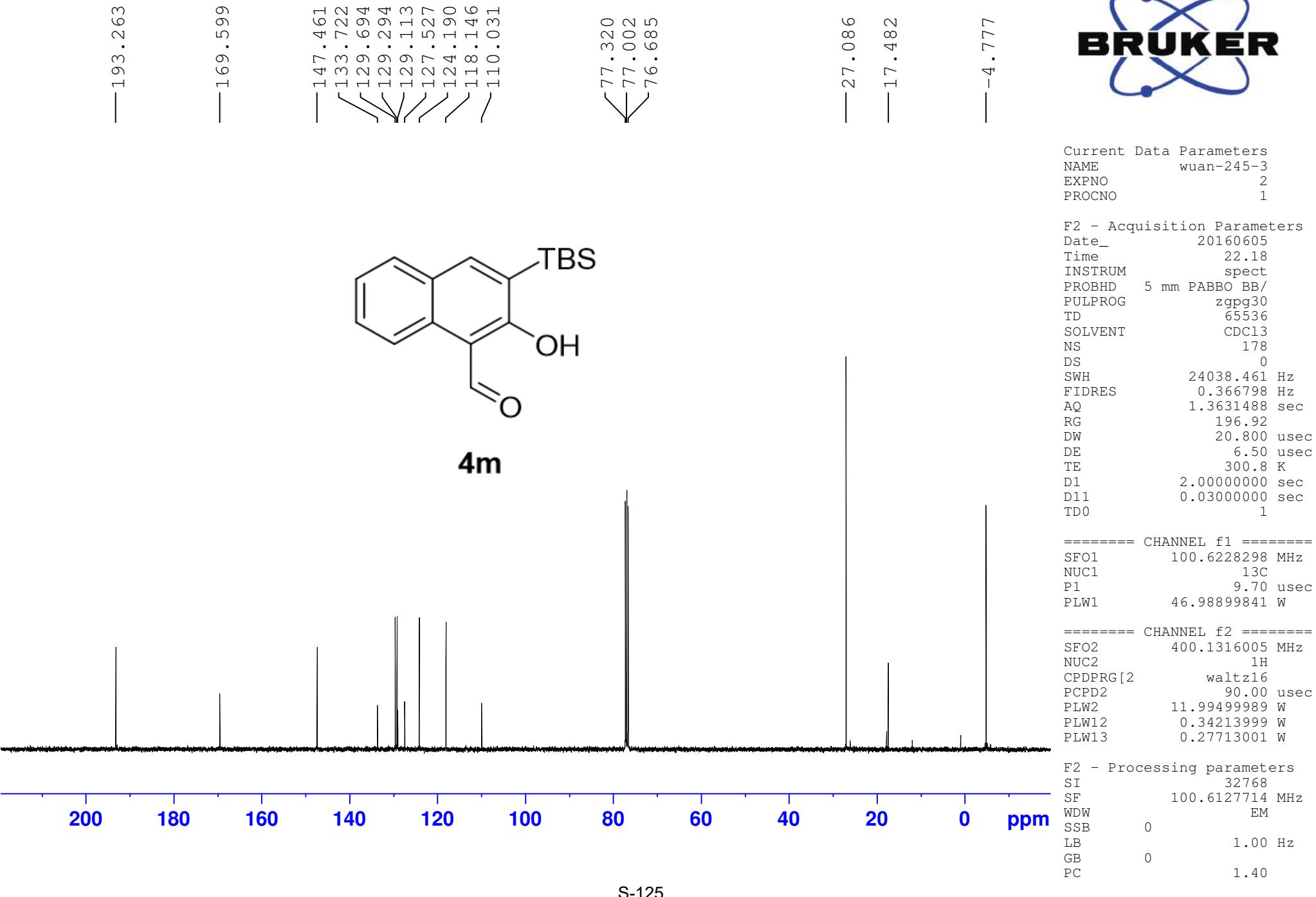
```

F2 - Acquisition Parameters
Date_           20160605
Time            22.10
INSTRUM        spect
PROBHD         5 mm PABBO BB/
PULPROG        zg30
TD              65536
SOLVENT         CDC13
NS              4
DS              0
SWH             8012.820 Hz
FIDRES         0.122266 Hz
AQ              4.0894465 sec
RG              49.32
DW              62.400 usec
DE              6.50 usec
TE              300.0 K
D1              1.00000000 sec
TD0             1

```

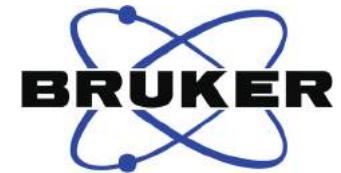
===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PI.W1 11.9949989 W

F2 - Processing parameters
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7.594
 7.419
 7.407
 7.402
 7.399
 7.391
 7.383
 7.375
 7.260

3.055
 3.037
 3.018
 2.881
 2.862
 2.842
 1.782
 1.764
 1.745
 1.727
 1.708
 1.701
 1.689
 1.685
 1.670
 1.579
 1.561
 1.542
 1.523
 1.505
 1.488
 1.470
 1.451
 1.432
 1.003
 0.993

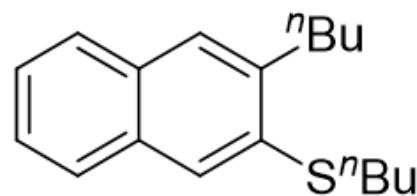


Current Data Parameters
 NAME wuan-179-1
 EXPNO 1
 PROCNO 1

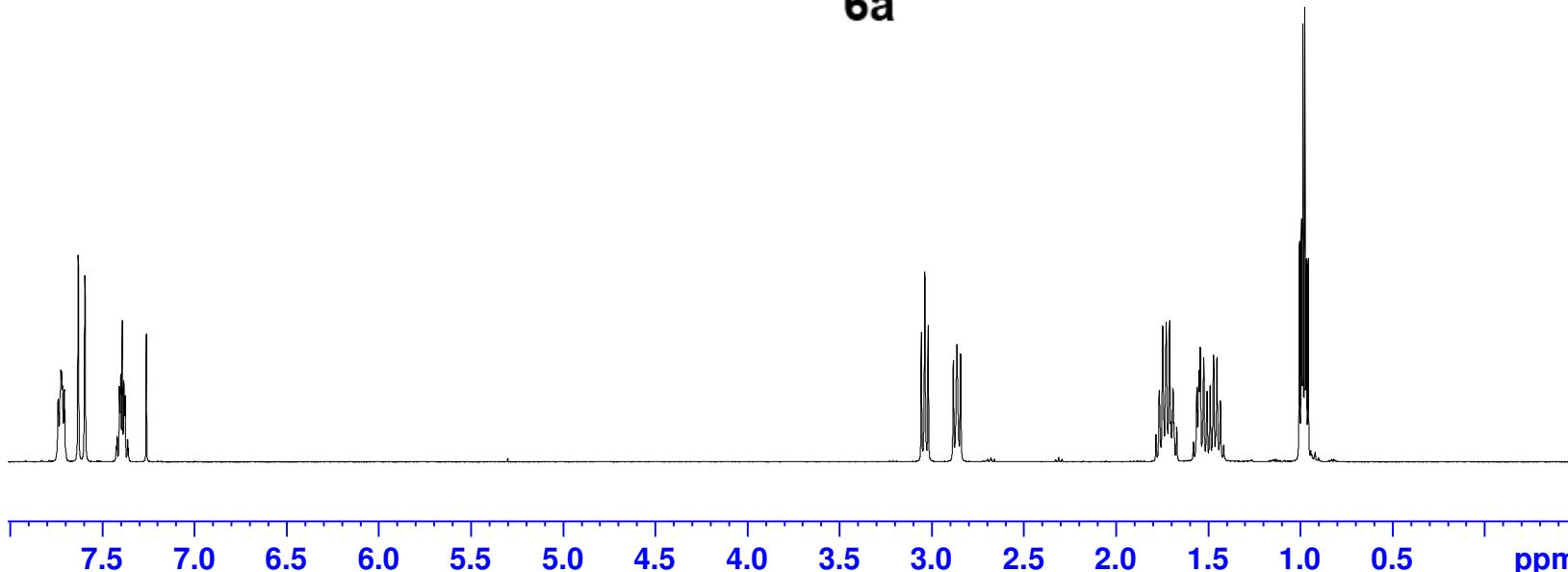
F2 - Acquisition Parameters
 Date_ 20160104
 Time 17.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 82.92
 DW 62.400 usec
 DE 6.50 usec
 TE 296.7 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



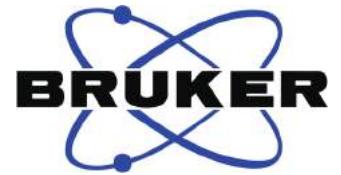
6a



1.97
 0.99
 0.98
 1.97

2.00
 2.00
 S-126

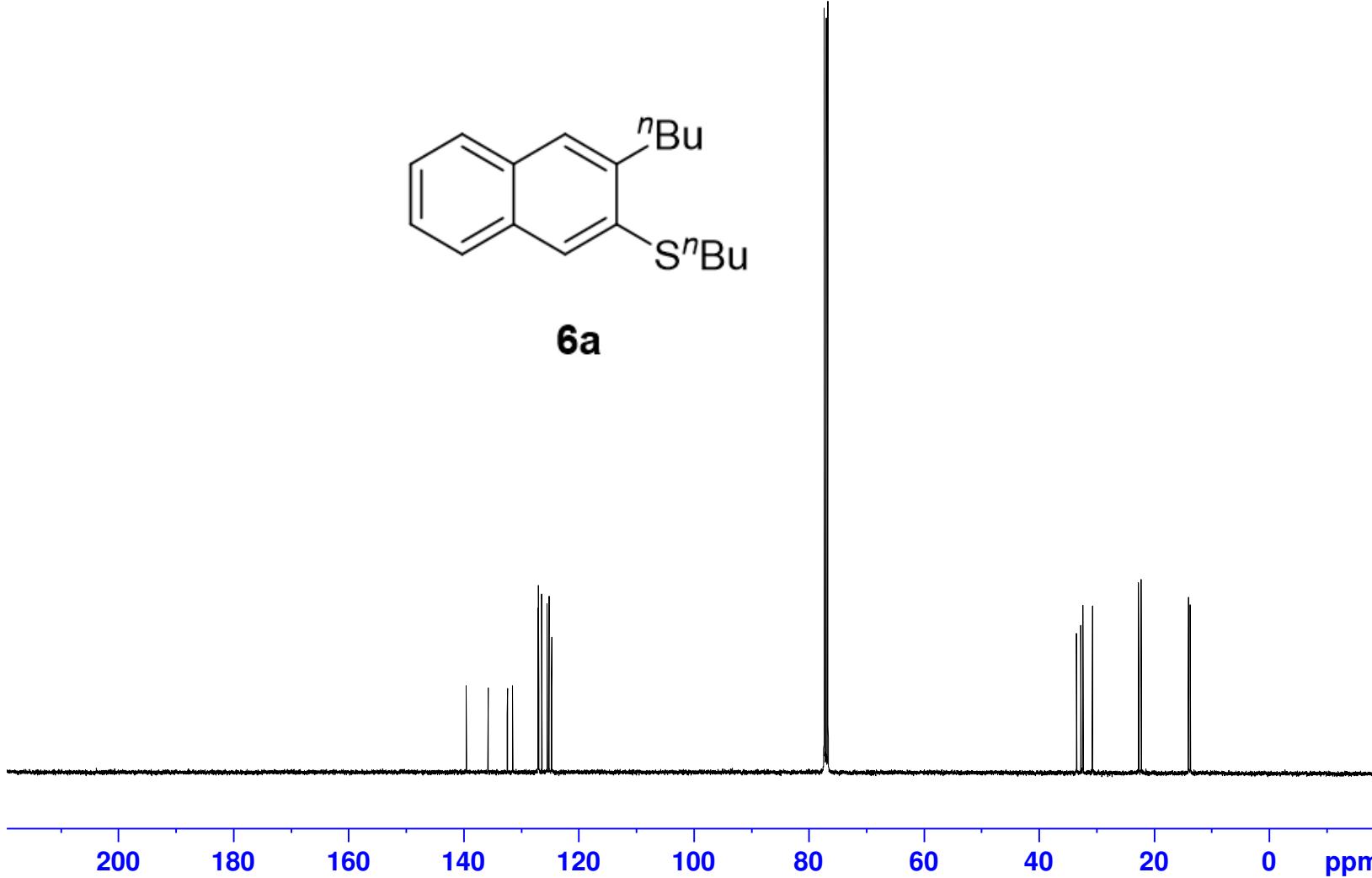
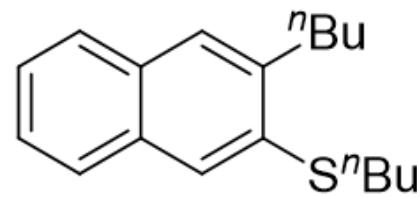
4.01
 4.42
 3.04
 2.92



139.519
135.720
132.381
131.469
127.081
126.955
126.409
125.469
125.125
124.694

77.317
77.000
76.682

33.478
32.720
32.342
30.687
22.675
22.216
14.014
13.706



S-127

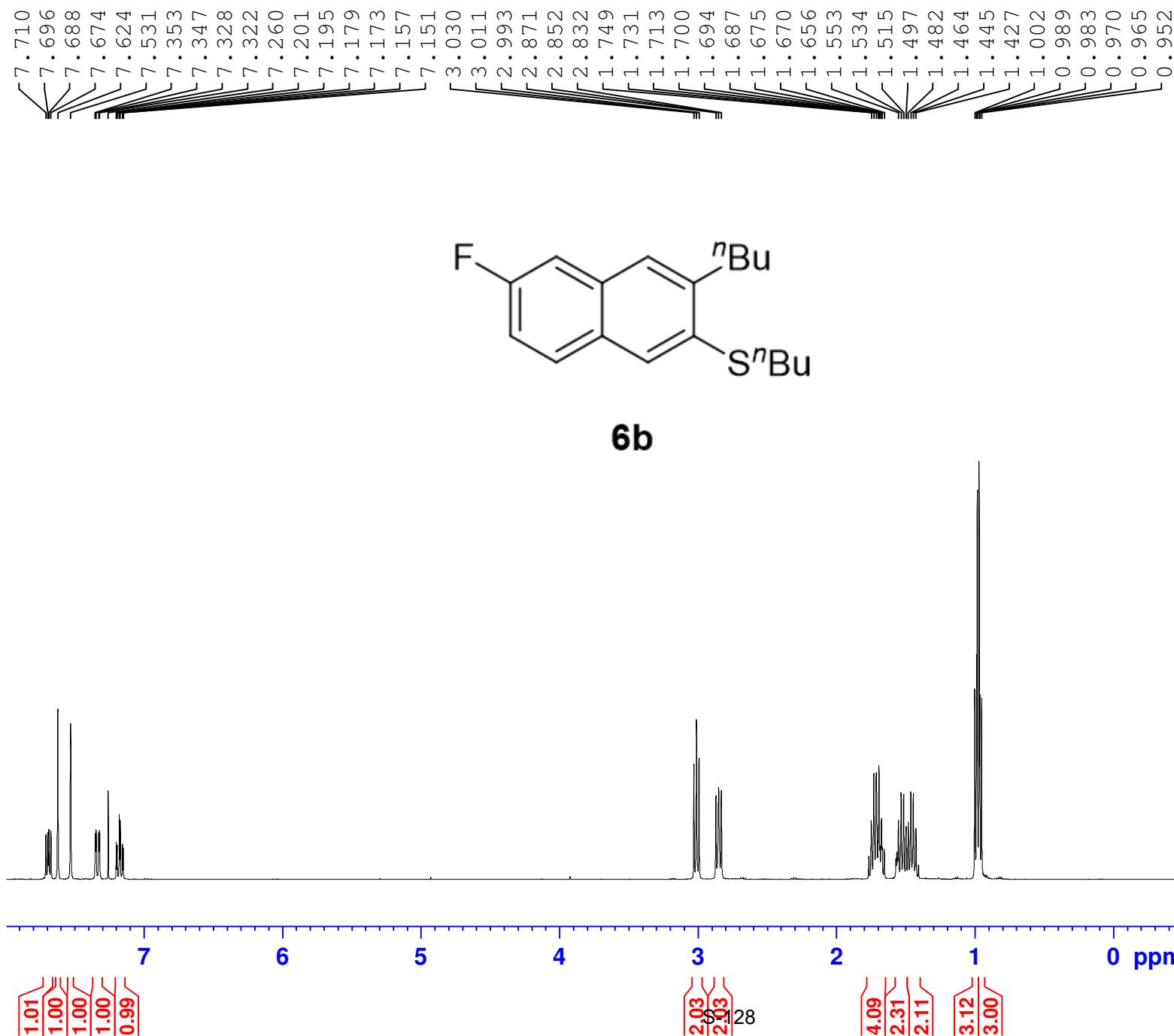
Current Data Parameters
NAME wuan-179-1
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160108
Time 12.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1969
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.60 usec
PLW1 31.98900032 W

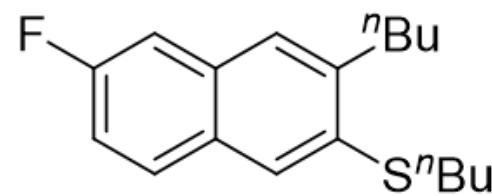
===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPGR[2] waltz16
PCPD2 90.00 usec
PLW2 9.10000038 W
PLW12 0.24608000 W
PLW13 0.19933000 W

F2 - Processing parameters
SI 32768
SF 100.6127719 MHz
WDW 0 EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





Current	Data	Parameters
NAME	wuan-241-1	
EXPNO		1
PROCNO		1



6b

```

F2 - Acquisition Parameters
Date_           20160328
Time            18.38
INSTRUM        spect
PROBHD         5 mm PABBO BB/
PULPROG        zg30
TD              65536
SOLVENT         CDCl3
NS              4
DS              0
SWH             8012.820 Hz
FIDRES         0.122266 Hz
AQ              4.0894465 sec
RG              70.97
DW              62.400 usec
DE              6.50  usec
TE              296.4 K
D1              1.00000000 sec
TD0              1

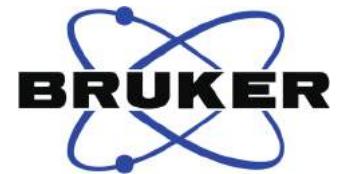
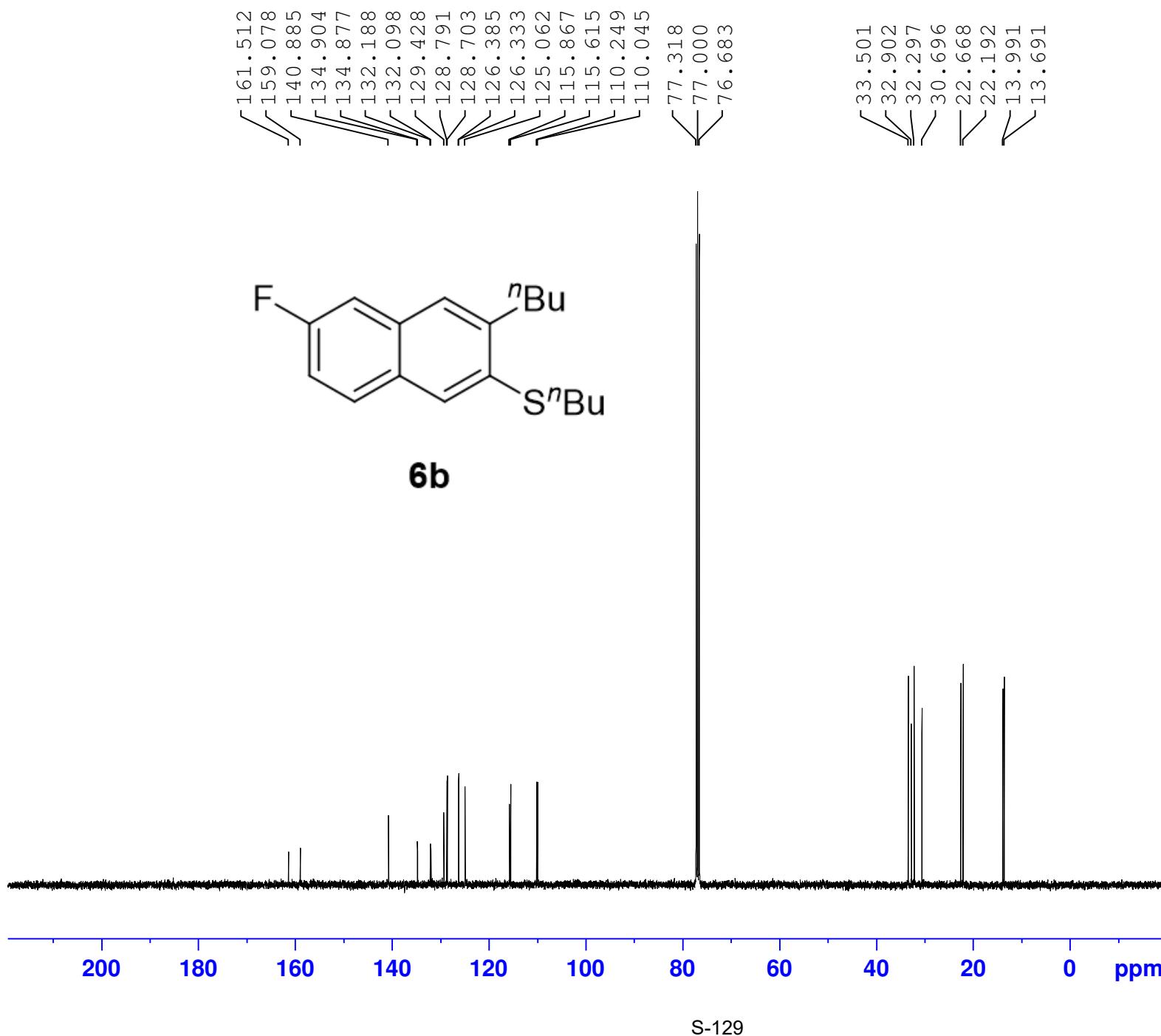
```

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PI.W1 11.9949998 W

```

F2 - Processing parameters
SI           65536
SF          400.1300096 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB          0
PC          1.00

```



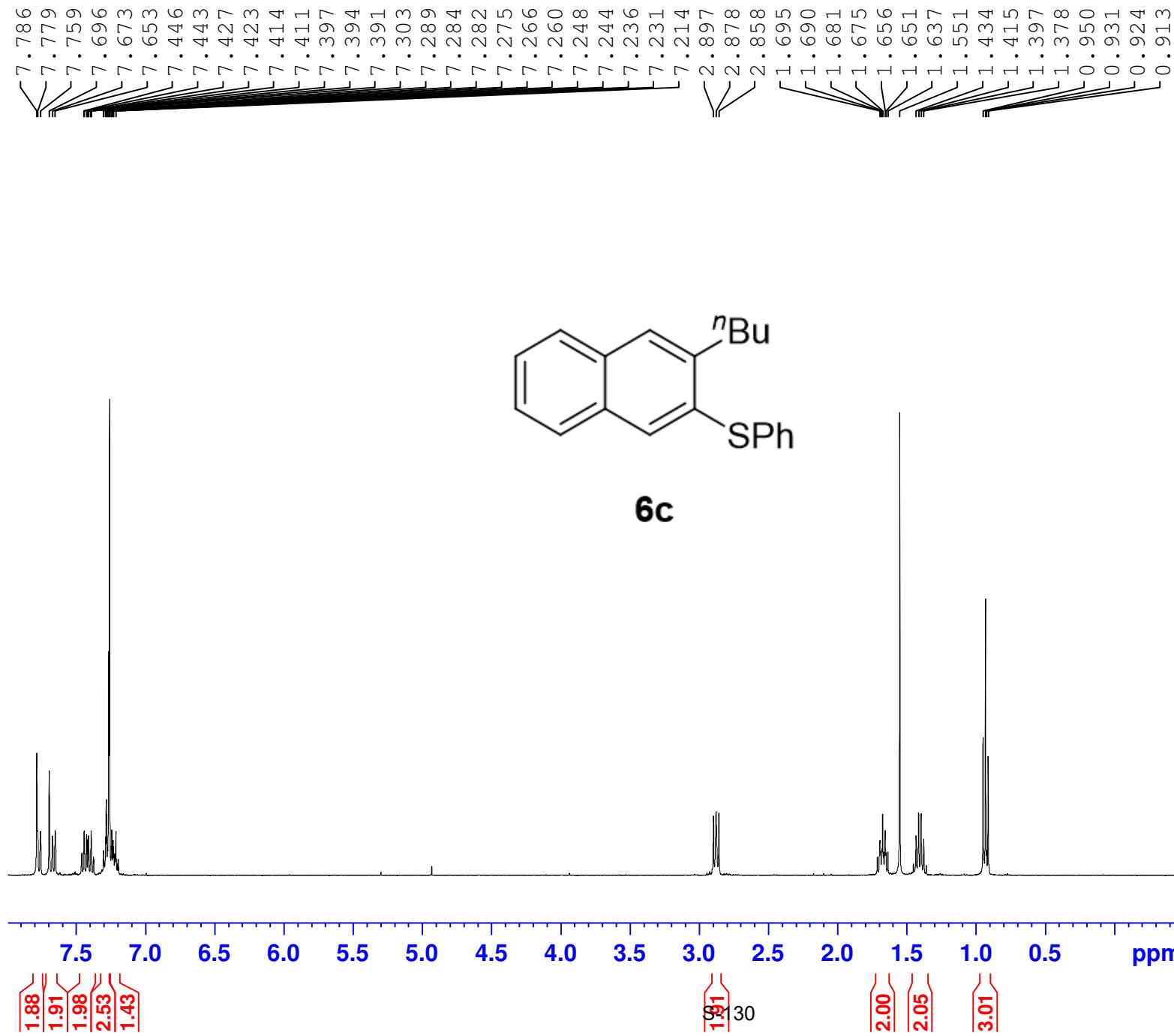
Current Data Parameters
 NAME wuan-241-1
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160328
 Time 19.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 412
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.3 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127714 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

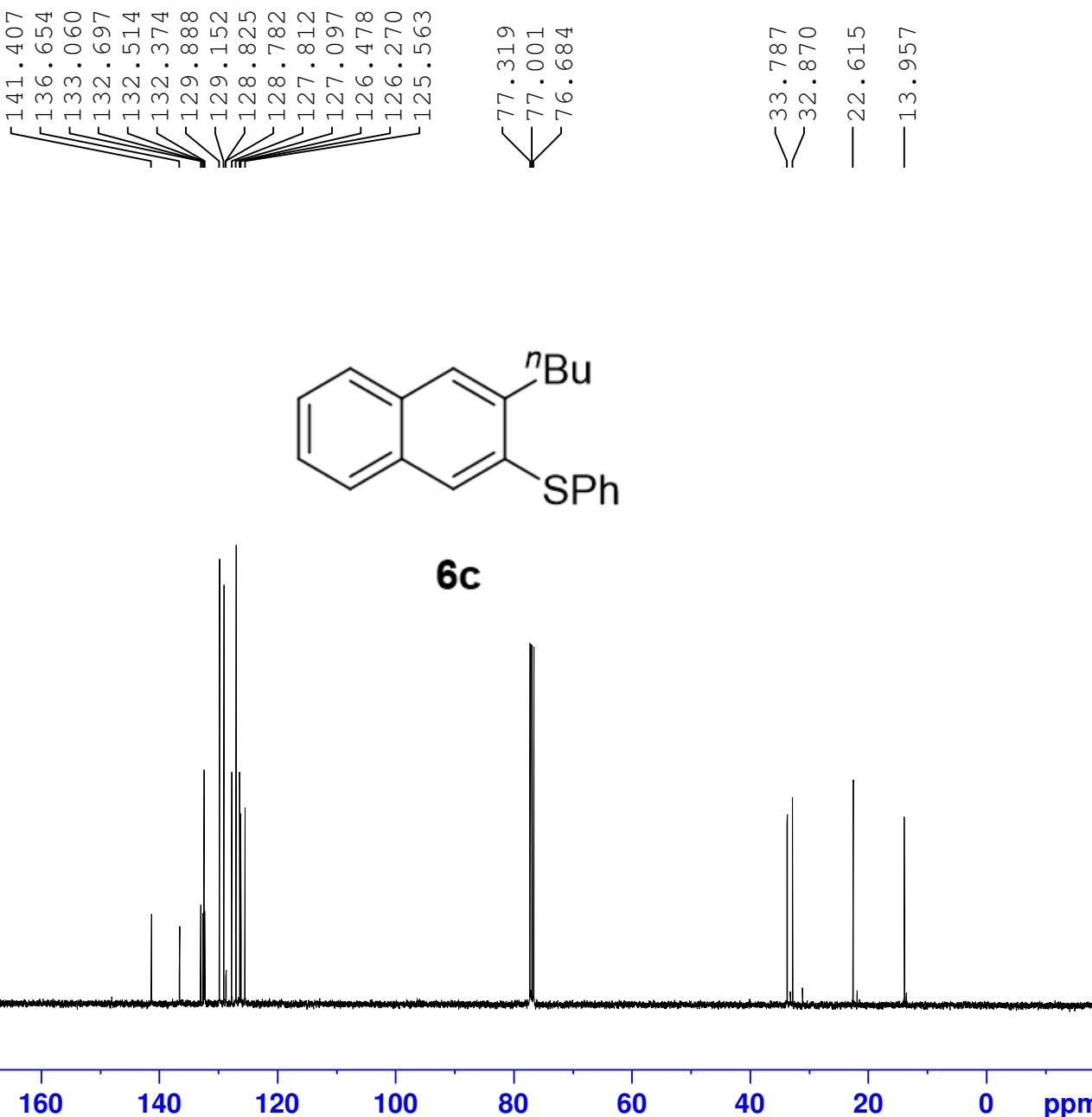


Current Data Parameters
 NAME wuan-295-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160405
 Time 1.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 298.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





— 11.003

8.830
8.809
7.879
7.801
7.781
7.560
7.538
7.519
7.260

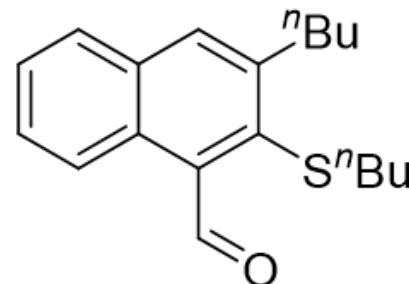
3.085
3.066
3.046
2.778
2.760
2.741
1.706
1.685
1.667
1.542
1.530
1.511
1.491
1.472
1.453
1.435
1.403
1.384
1.366
1.348

Current Data Parameters
 NAME wuan-219-2
 EXPNO 1
 PROCNO 1

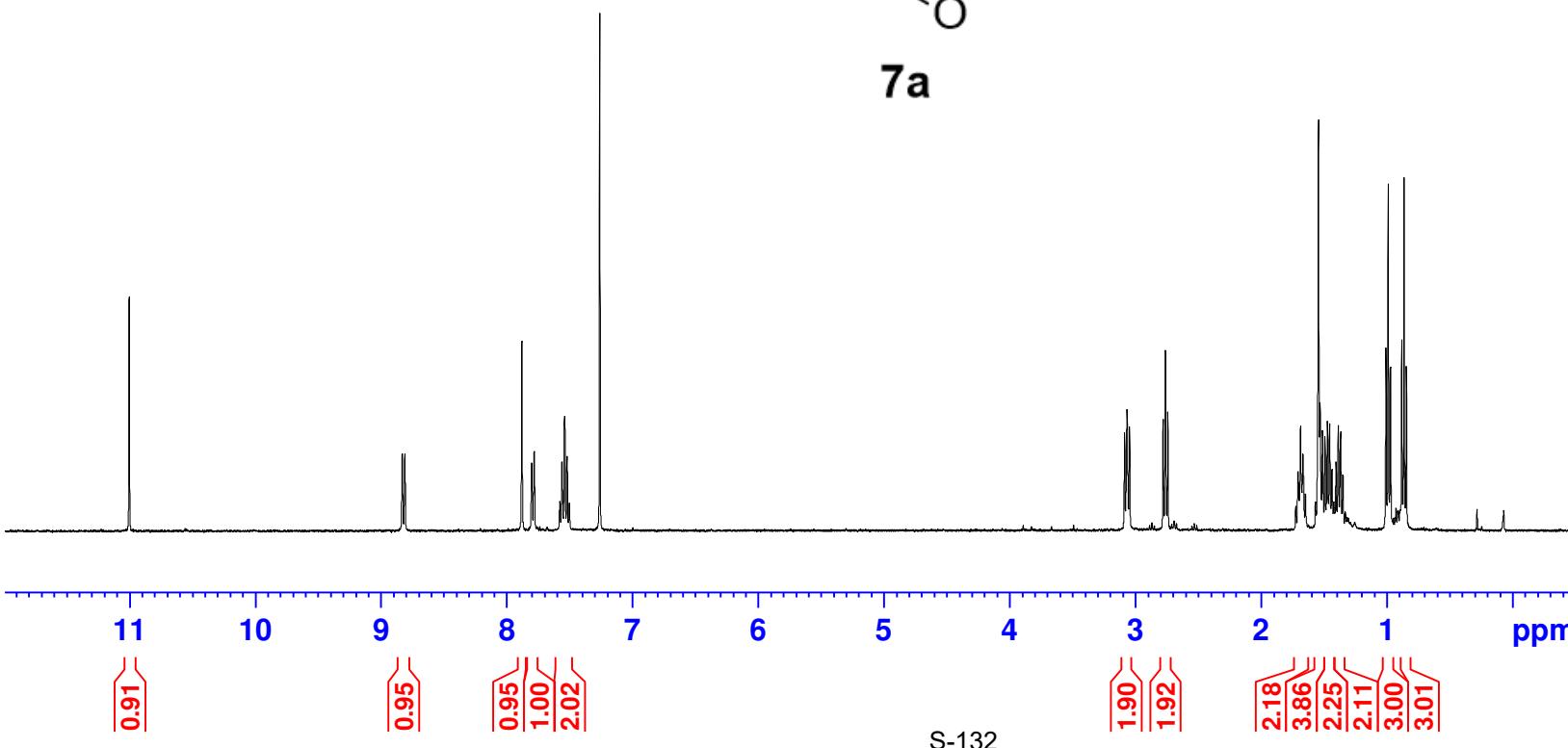
F2 - Acquisition Parameters
 Date_ 20160127
 Time 22.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.3 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



7a



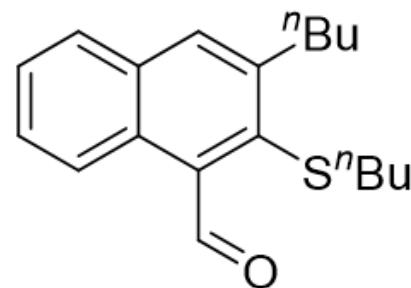
S-132

— 195.422

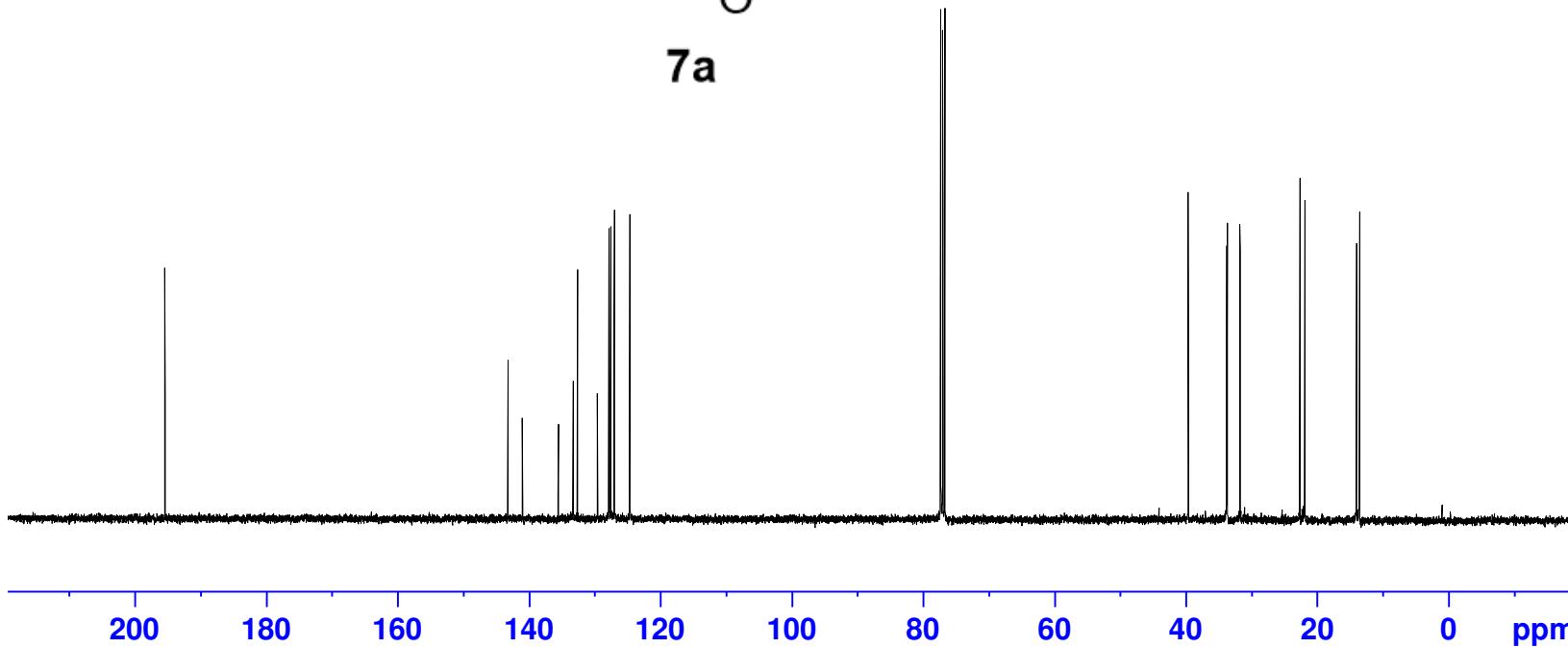
143.216
140.998
135.537
133.261
132.603
129.572
127.839
127.581
127.021
124.637

77.317
77.000
76.682

39.614
33.761
33.662
31.736
22.625
21.846
13.975
13.533



7a



S-133



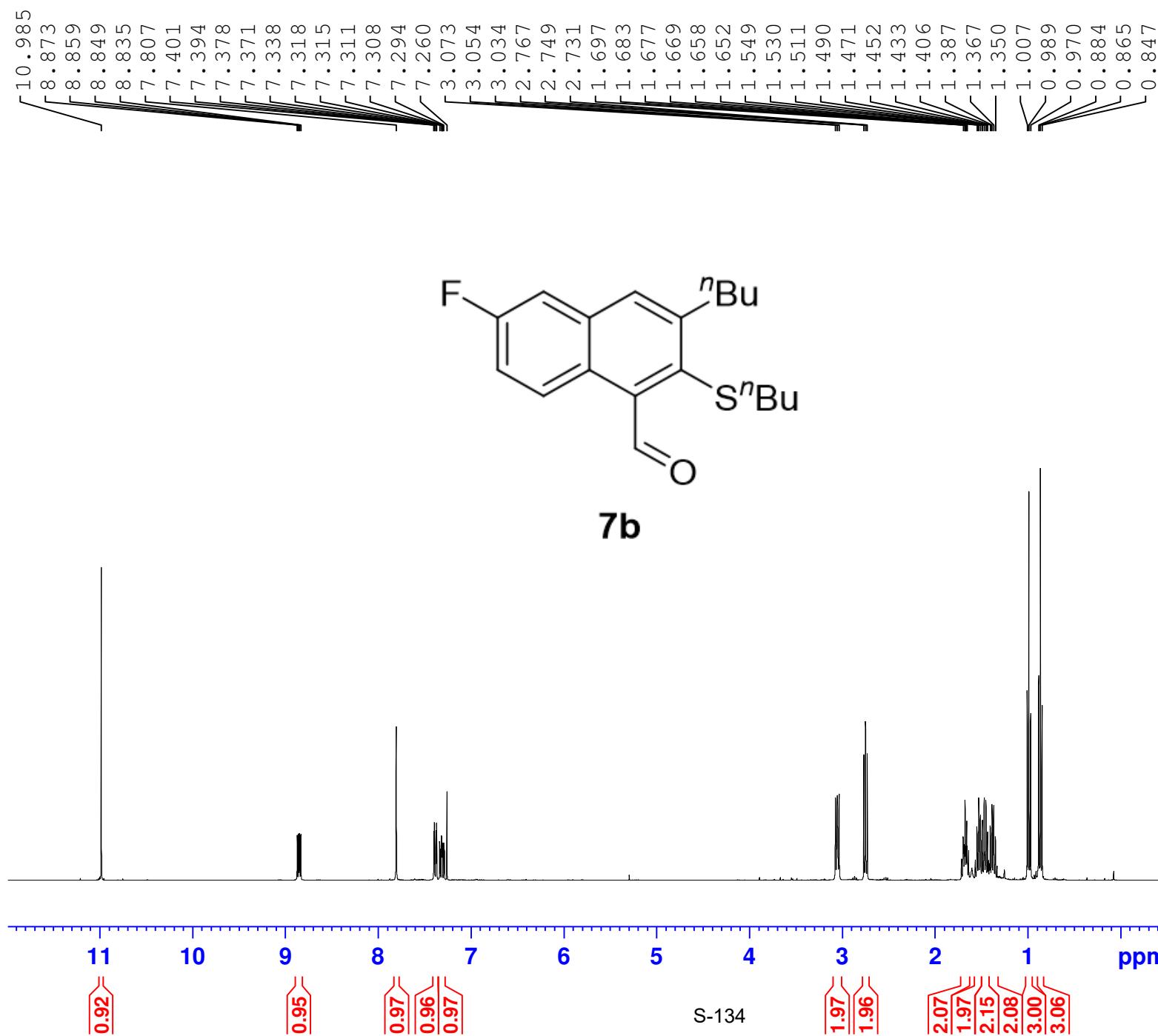
Current Data Parameters
NAME wuan-219-2
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160202
Time 20.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 263
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
PLW1 46.98899841 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPKG[2] waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127740 MHz
WDW 0 EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





Current Data Parameters
NAME wuan-242-2
EXPNO 2
PROCNO 1

```

F2 - Acquisition Parameters
Date_           20160324
Time            13.06
INSTRUM        spect
PROBHD         5 mm PABBO BB/
PULPROG        zg30
TD              65536
SOLVENT         CDCl3
NS              4
DS              0
SWH             8012.820 Hz
FIDRES         0.122266 Hz
AQ              4.0894465 sec
RG              70.97
DW              62.400 usec
DE              6.50  usec
TE              296.9 K
D1              1.00000000 sec
TD0              1

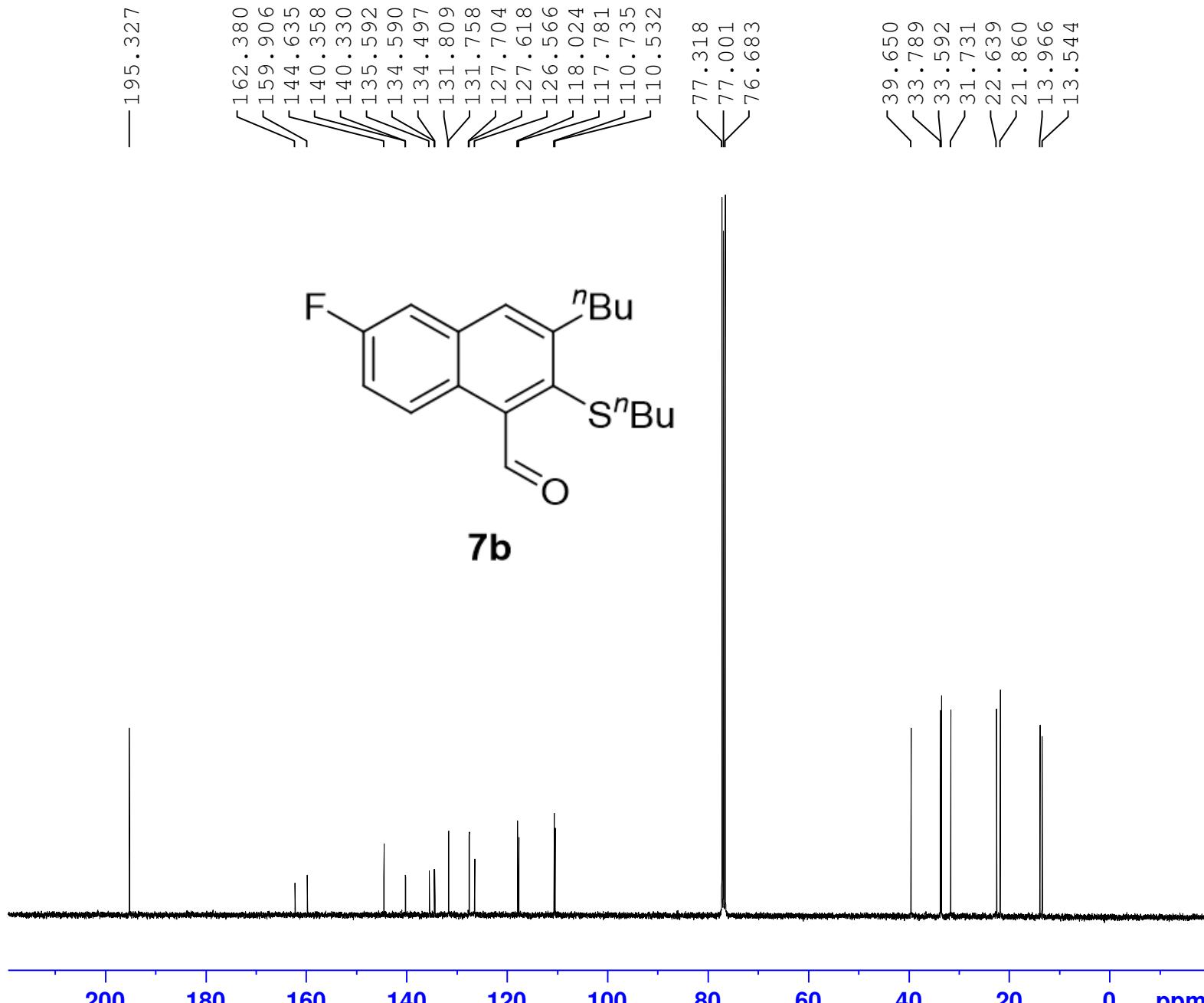
```

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.9949998 W

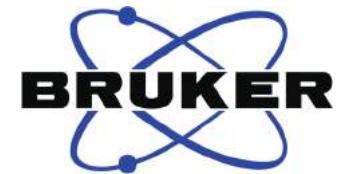
```

F2 - Processing parameters
SI           65536
SF          400.1300095 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB          0
PC          1.00

```



S-135



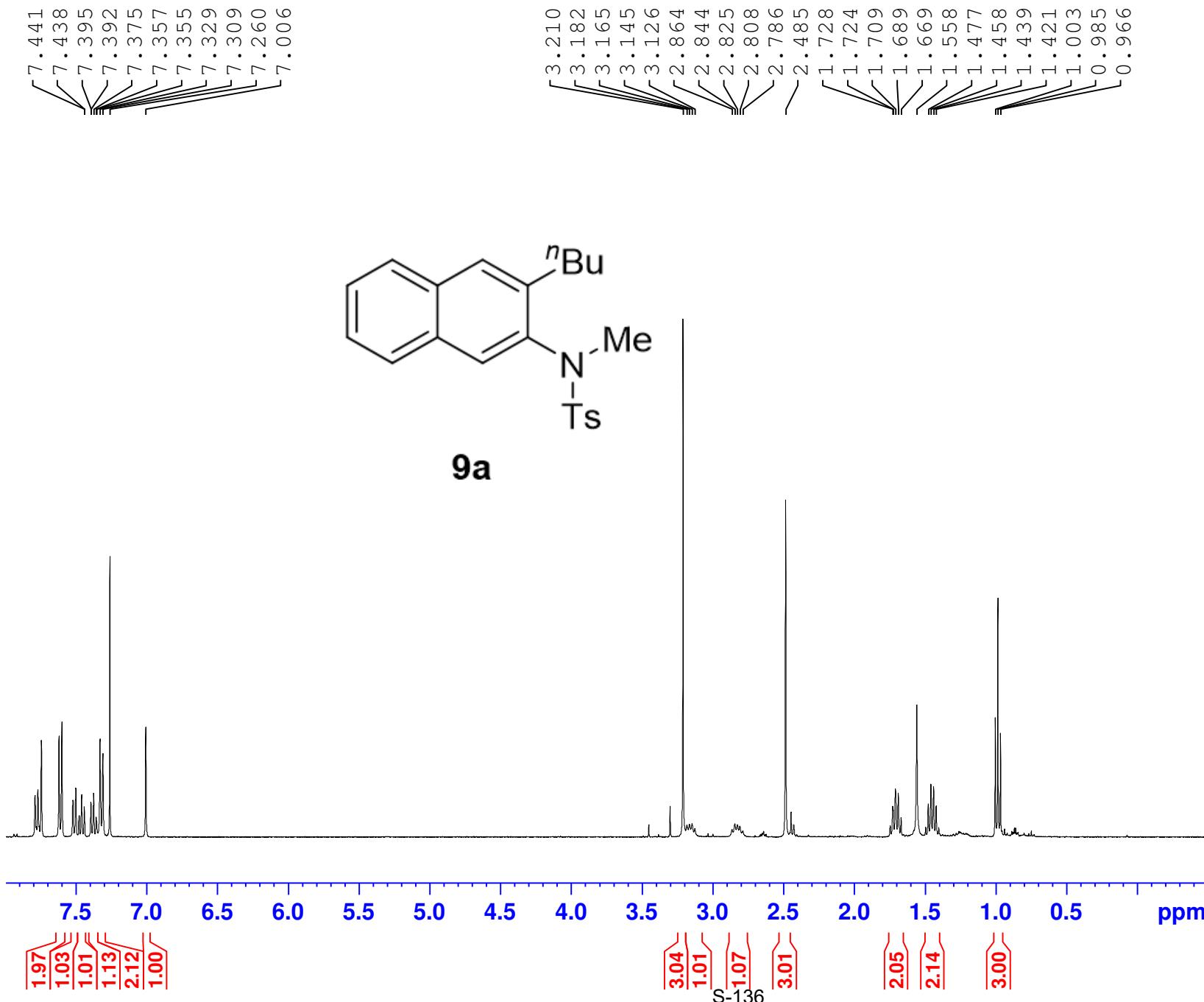
Current Data Parameters
 NAME wuan-242-2
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160324
 Time 13.17
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 718
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.9 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127710 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current	Data	Parameters
NAME	wuan-178-1	
EXPNO		2
PROCNO		1

```

F2 - Acquisition Parameters
Date_           20160102
Time            20.25
INSTRUM        spect
PROBHD         5 mm PABBO BB/
PULPROG        zg30
TD              65536
SOLVENT         CDC13
NS                4
DS                0
SWH             8012.820 Hz
FIDRES        0.122266 Hz
AQ              4.0894465 sec
RG              187.77
DW              62.400 usec
DE                6.50 usec
TE                298.1 K
D1      1.000000000 sec
TD0                  1

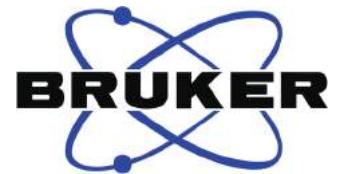
```

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 11.99499989 W

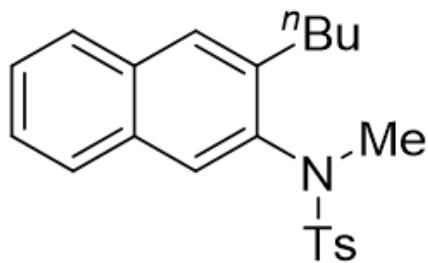
```

F2 - Processing parameters
SI           65536
SF          400.1300094 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB          0
PC          1.00

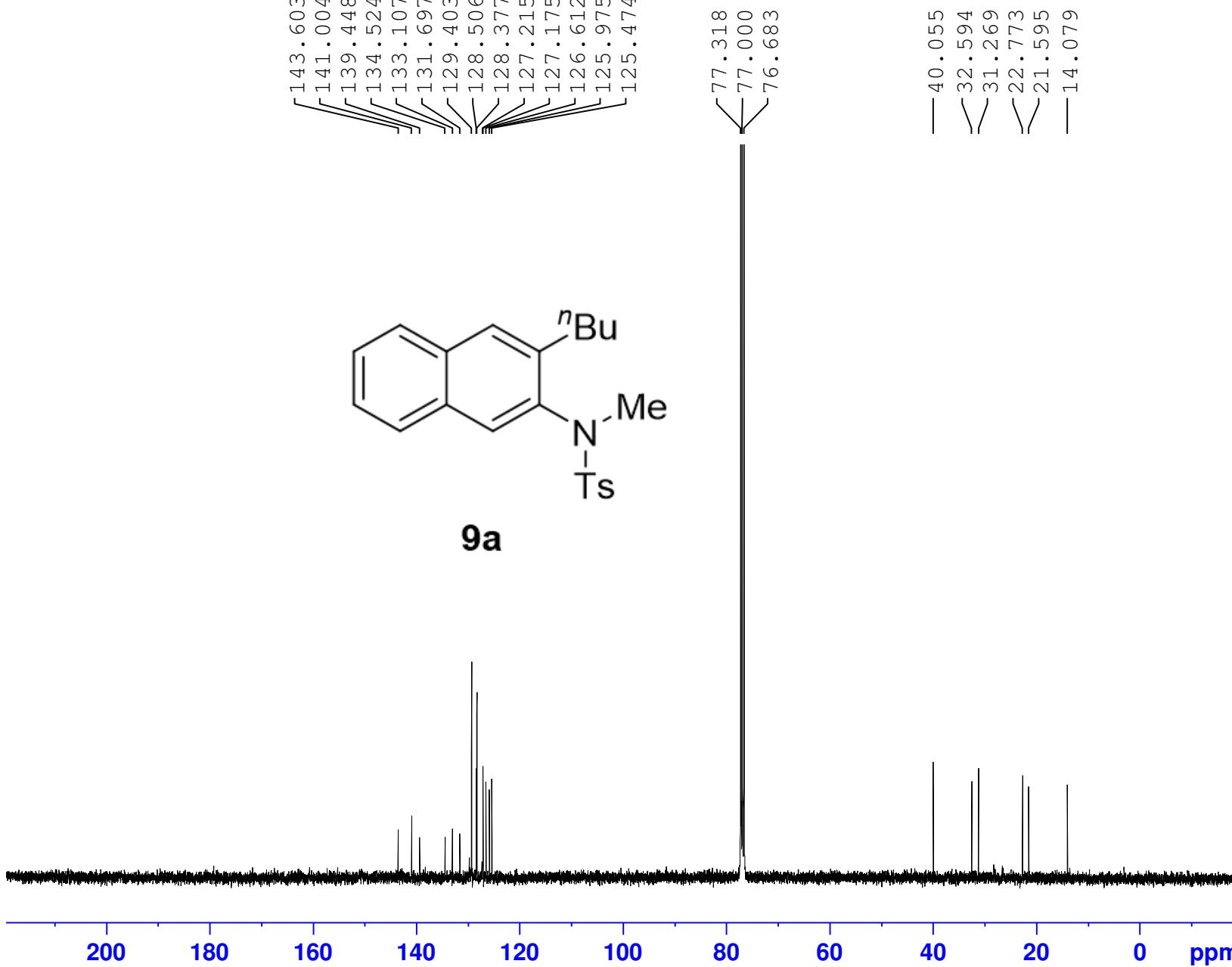
```



143.603
141.004
139.448
134.524
133.107
131.697
129.403
128.506
128.377
127.215
127.175
126.612
125.975
125.474



9a



S-137

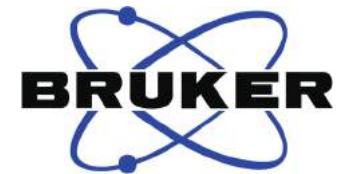
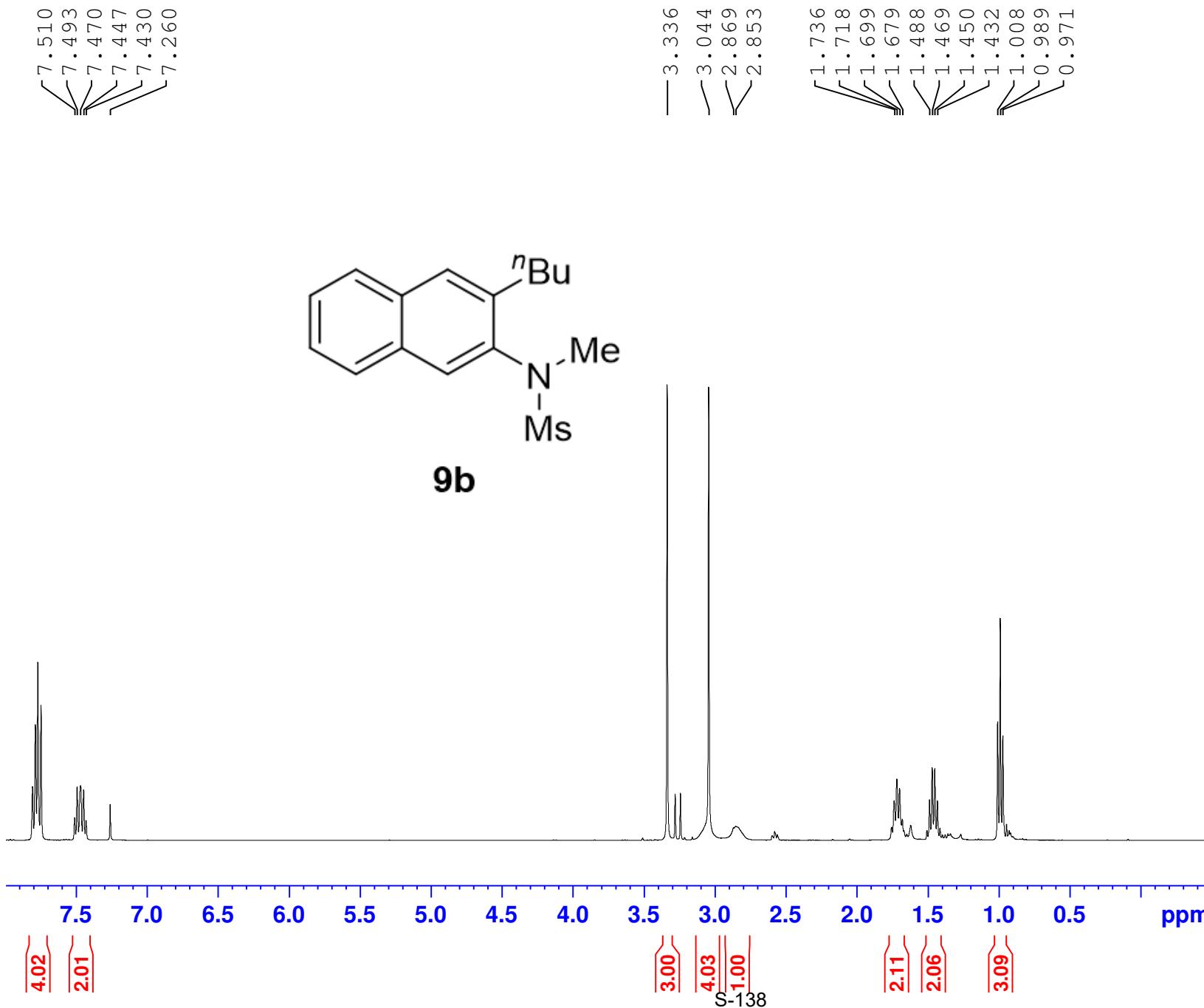
Current Data Parameters
NAME wuan-178-1
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160102
Time 20.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1380
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 ¹³C
P1 9.70 usec
PLW1 46.98899841 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 ¹H
CPDPKG[2] waltz16
PCPD2 90.00 usec
PLW2 11.99499989 W
PLW12 0.34213999 W
PLW13 0.27713001 W

F2 - Processing parameters
SI 32768
SF 100.6127700 MHz
WDW 0 EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

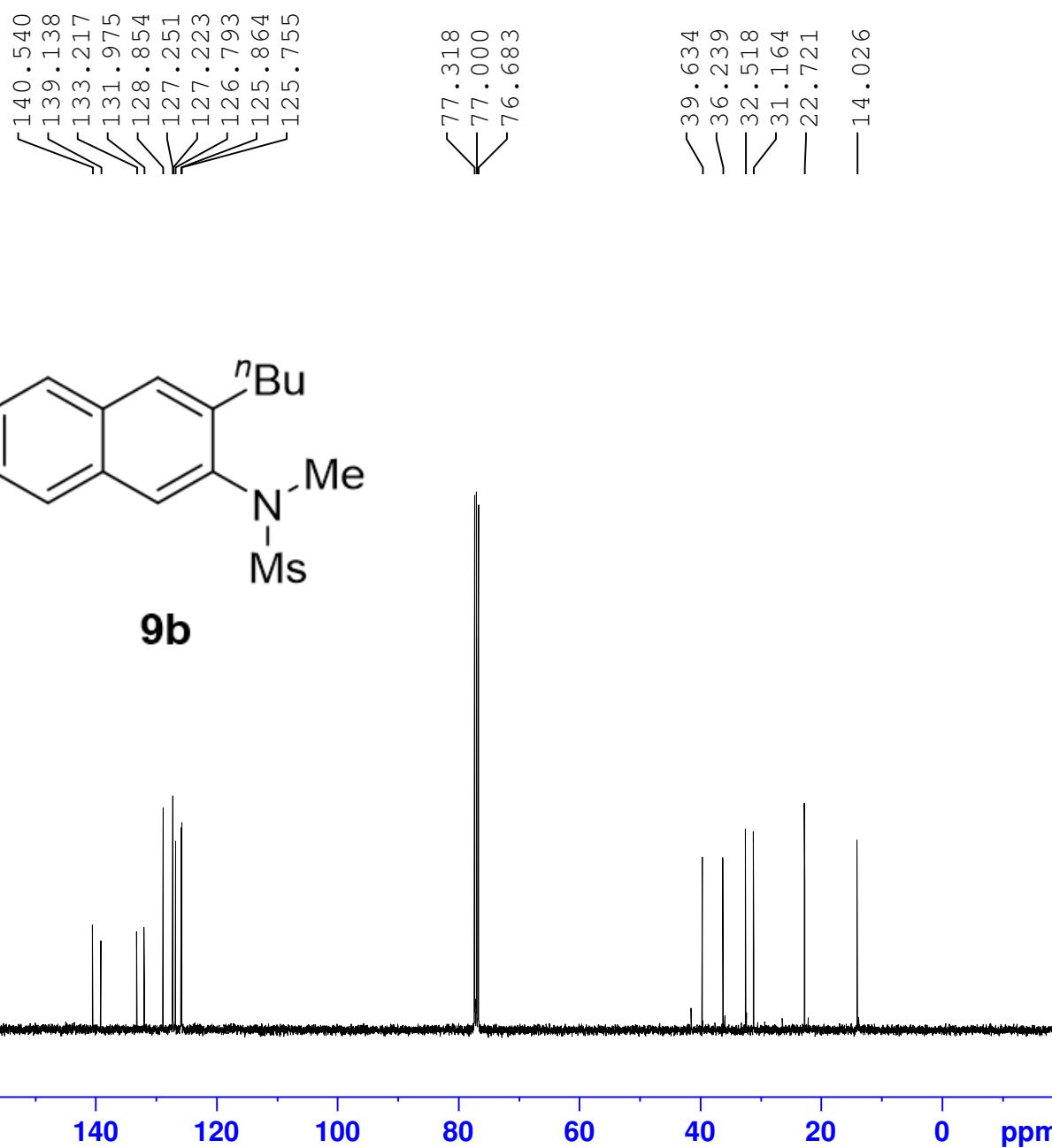
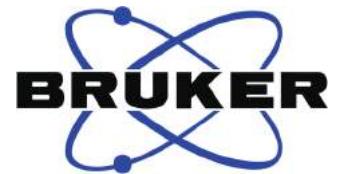


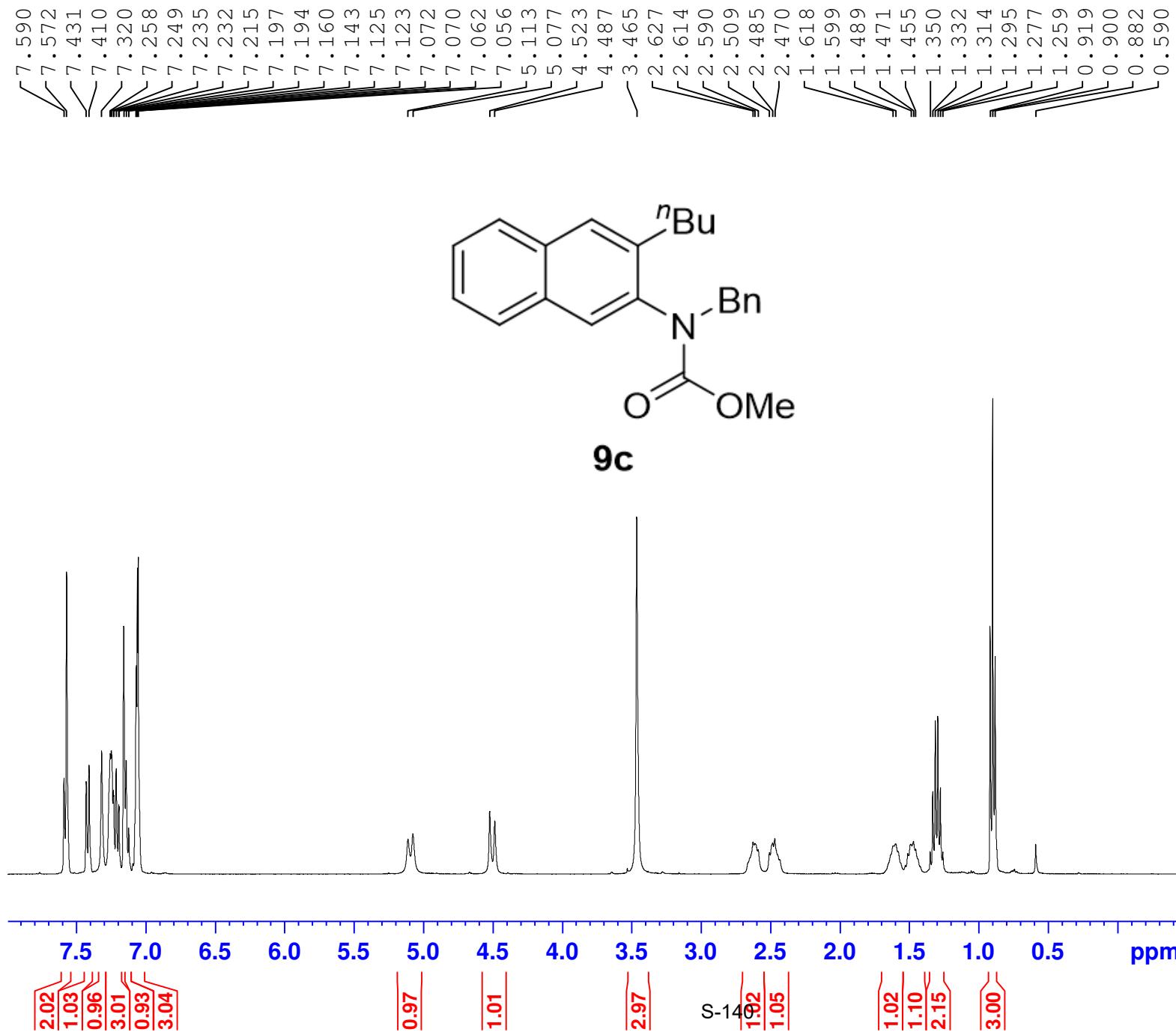
Current Data Parameters
 NAME wuan-200-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160203
 Time 1.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 49.32
 DW 62.400 usec
 DE 6.50 usec
 TE 296.8 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 ¹H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME wuan-209-1
 EXPNO 4
 PROCNO 1

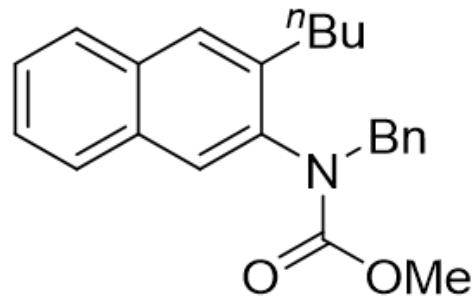
F2 - Acquisition Parameters
 Date_ 20160605
 Time 23.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 88.84
 DW 62.400 usec
 DE 6.50 usec
 TE 330.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

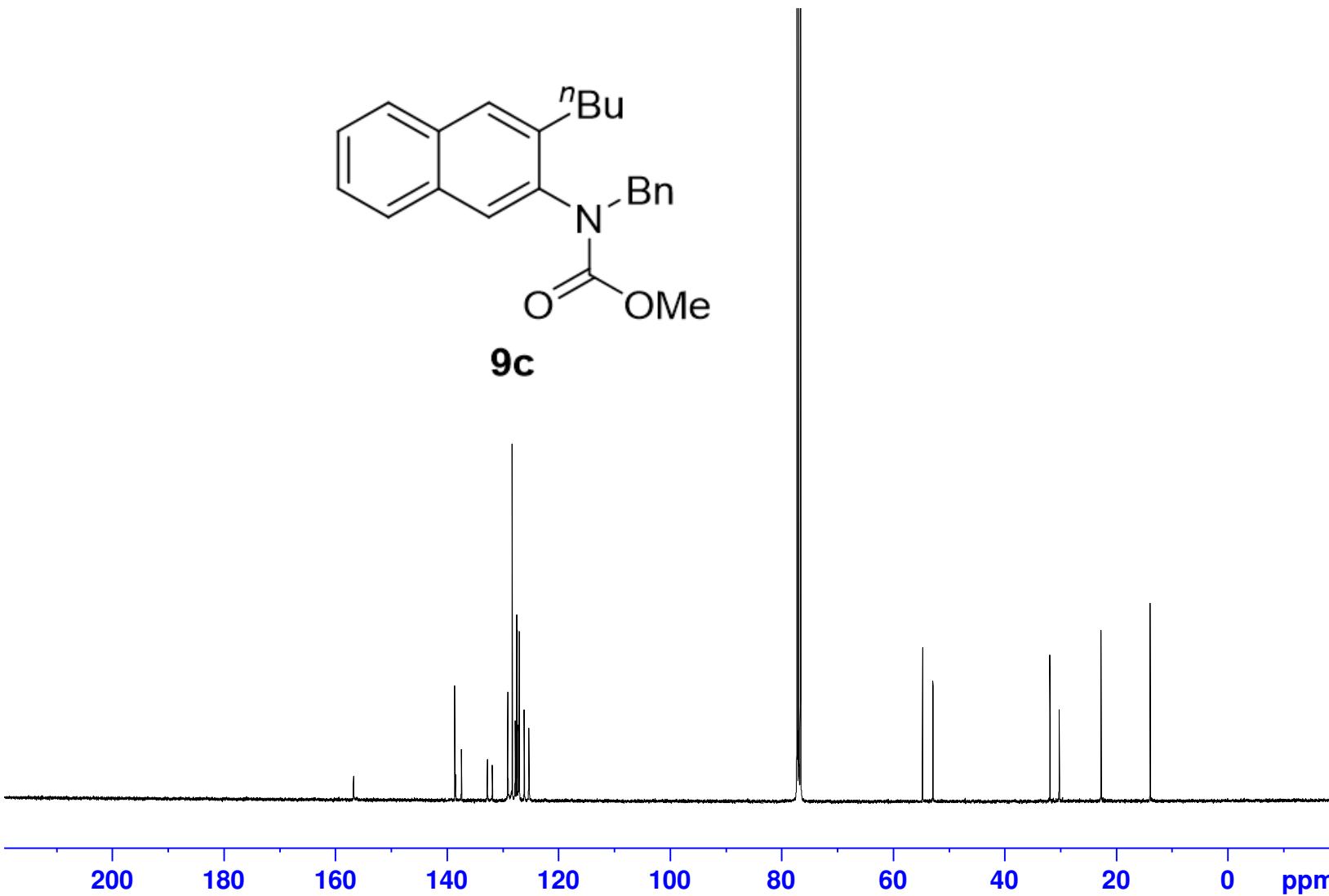
F2 - Processing parameters
 SI 65536
 SF 400.1299957 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



156.828
 138.676
 138.531
 137.480
 132.811
 131.945
 129.161
 128.375
 127.838
 127.593
 127.514
 127.237
 127.104
 126.233
 125.398



77.318
 77.001
 76.683
 54.789
 52.934
 31.957
 30.286
 22.788
 -13.996



Current Data Parameters
 NAME wuan-209
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160203
 Time 1.17
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10330
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.9 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127716 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

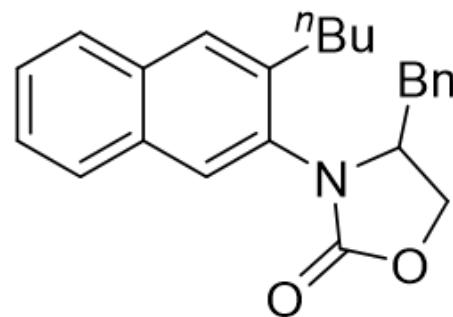


Current Data Parameters
 NAME wuan-212-3
 EXPNO 3
 PROCNO 1

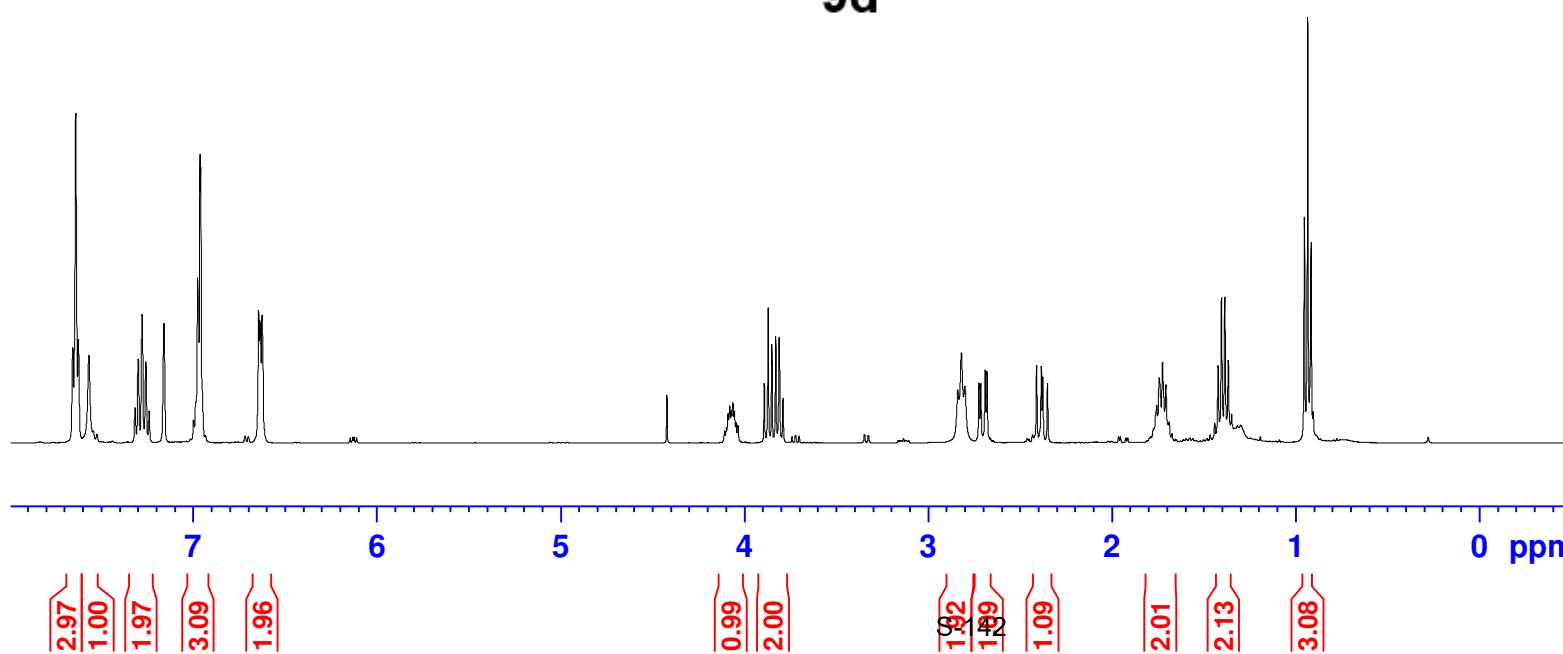
F2 - Acquisition Parameters
 Date_ 20160605
 Time 23.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 4
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 70.97
 DW 62.400 usec
 DE 6.50 usec
 TE 330.1 K
 D1 1.0000000 sec
 TDO 1

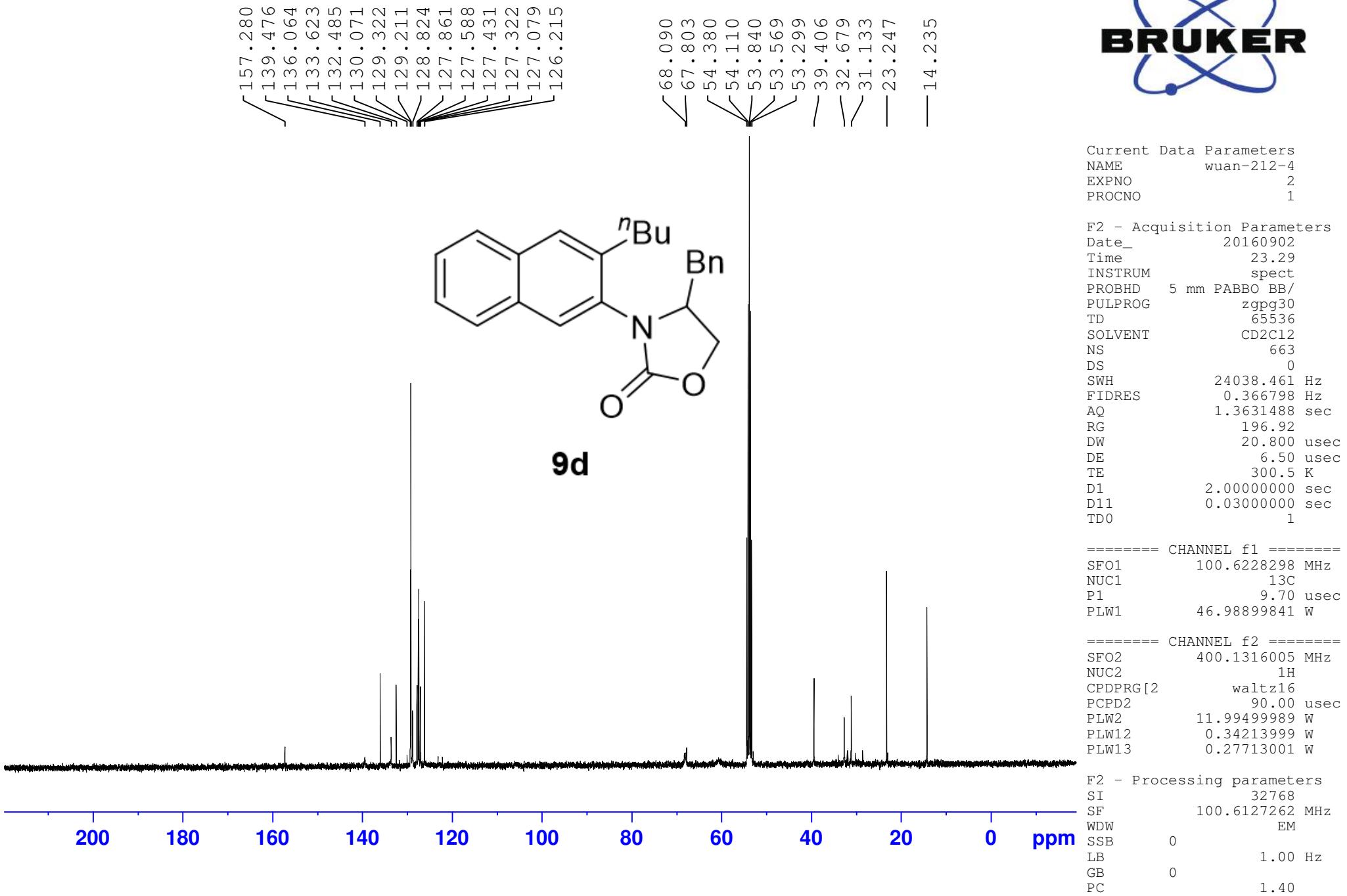
===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1299957 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

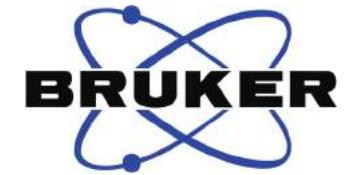
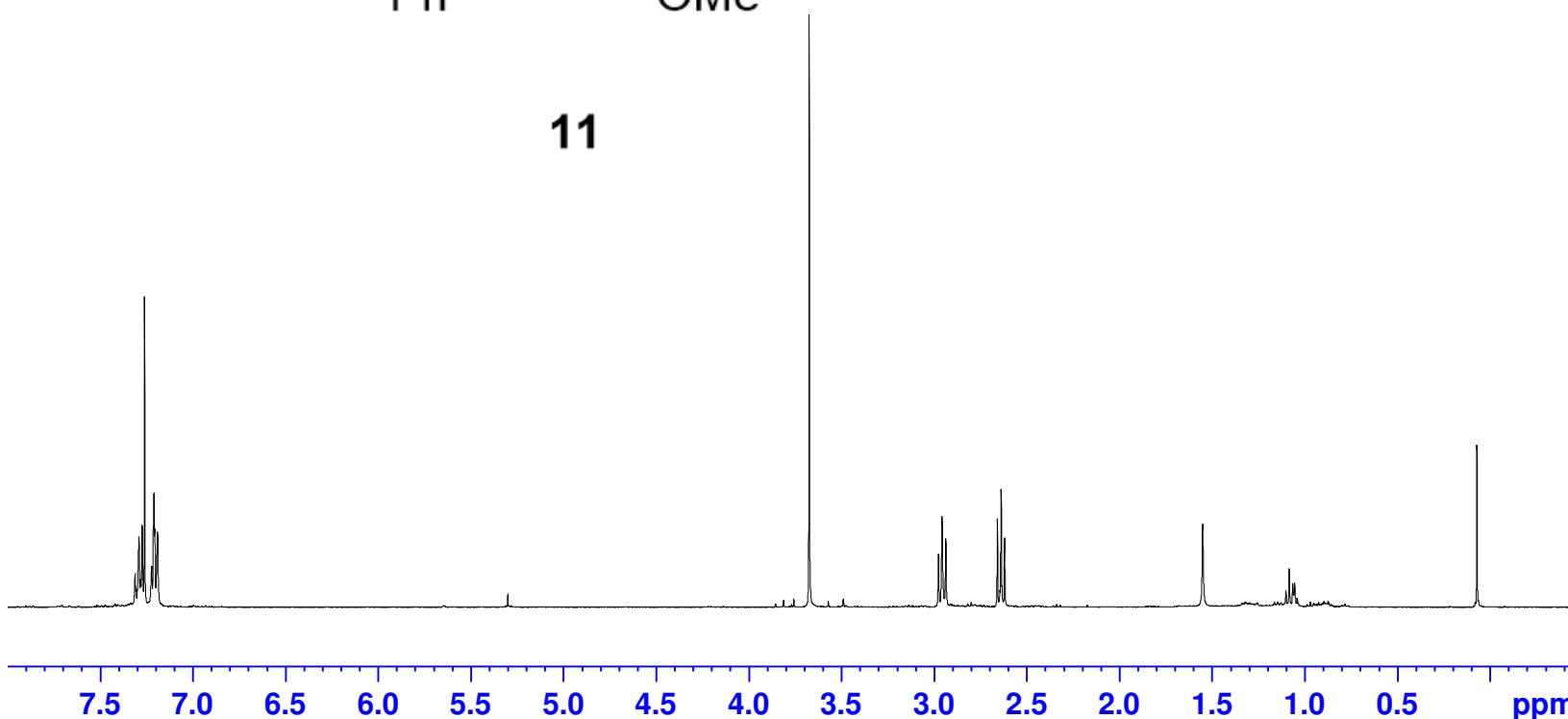
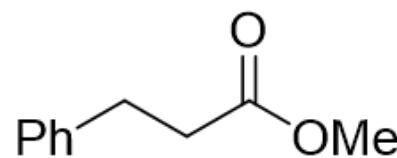


9d





7.306
 7.291
 7.282
 7.273
 7.221
 7.260
 7.209
 7.205
 7.190



Current Data Parameters
 NAME wuan-162-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151221
 Time 13.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.12226 Hz
 AQ 4.0894465 sec
 RG 187.77
 DW 62.400 usec
 DE 6.50 usec
 TE 296.8 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 14.50 usec
 PLW1 11.99499989 W

F2 - Processing parameters
 SI 65536
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME wuan-162-3
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151221
 Time 14.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 574
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 196.92
 DW 20.800 usec
 DE 6.50 usec
 TE 297.7 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 9.70 usec
 PLW1 46.98899841 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 11.99499989 W
 PLW12 0.34213999 W
 PLW13 0.27713001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127701 MHz
 WDW 0 EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

