Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Cu-catalyzed Oxidative Povarov Reactions Between *N*-Alkyl *N*-Methylanilines and Saturated Oxa- and Thiacycles**

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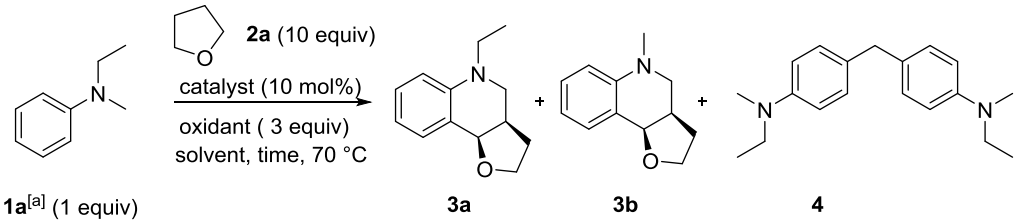
Contents:

1. Table S-1: Reaction optimization for oxidative cycloadditions.

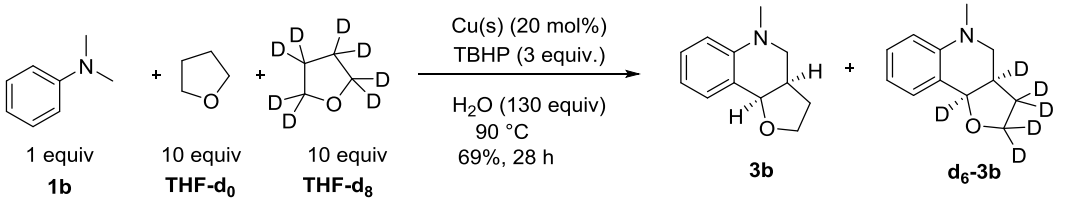


1a[a] (1 equiv) **3a** **3b** **4**

entry	catalyst (10 mol %)	oxidant	solvent	time	yield (%)[b] **1a**	**3a**	**3b**	**4**
1	$FeCl_3$	DTBP	toluene	24 h	36	-	-	-
2	$RuCl_3$	DTBP	toluene	24 h	68	-	-	-
3	CuCl	DTBP	toluene	24 h	61	-	-	-
4	$Cu(OAc)_2$	DTBP	toluene	24 h	60	-	-	-
5	$Cu(CH_3CN)_4$ BF_4	DTBP	toluene	24 h	70	12	-	-
6	CuIPrCl	TBHP	toluene	24 h	28	15	-	-
7	CuBr	TBHP	toluene	24 h	18	-	-	-
8	$Cu(OTf)_2$	O_2 (1 atm)	toluene	24 h	-	-	-	-[c]
9	$Cu(OTf)_2$	DDQ	toluene	24 h	trace	-	-	-[c]

DTBP = Di-*tert*-butyl peroxide in decane. [a] [**1a**] = 0.37 M, [b] isolated yield after silica column purification, [c] complecated mixture of products

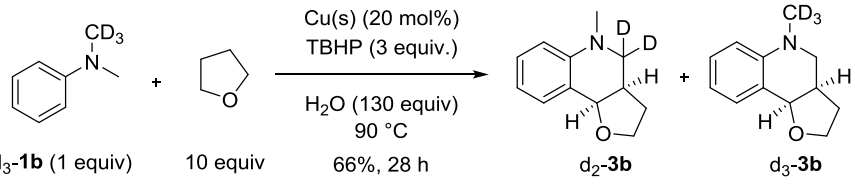
2. Scheme S-1and S-2: kinetic isotope effects



1 equiv **1b** 10 equiv **THF-d_0** 10 equiv **THF-d_8** **3b** **d_6-3b**

3b:**d_6-3b** = 5.7:1 (by ^{1}H NMR)

k_H/K_D = 5.7

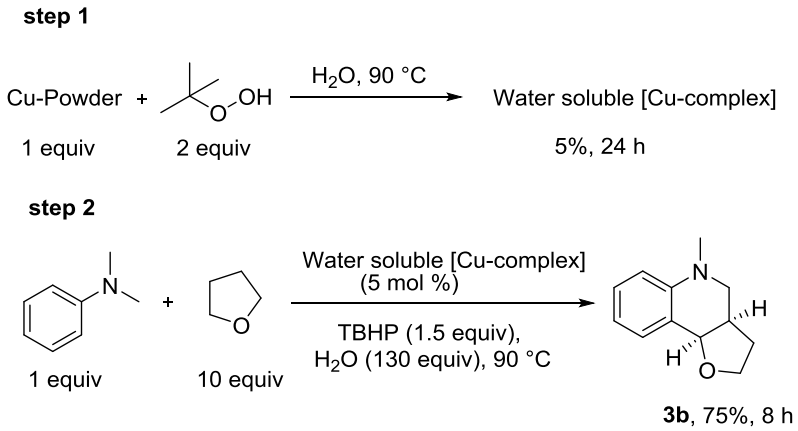


d_3-**1b** (1 equiv) 10 equiv d_2-**3b** d_3-**3b**

d_2-**3b**:d_3-**3b** = 1:2.8 (by ^{1}H NMR)

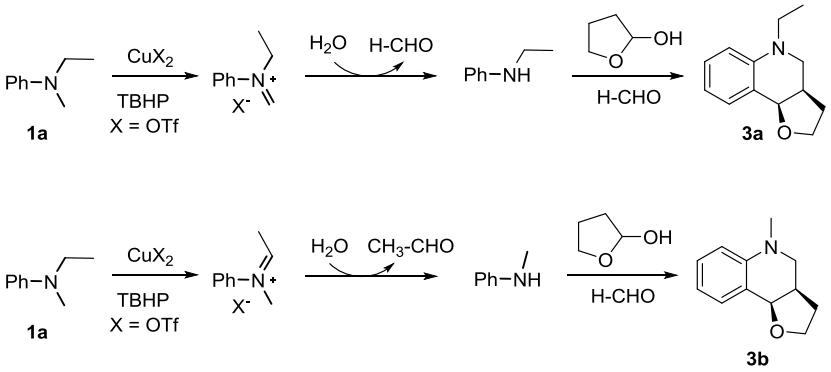
k_H/K_D = 2.8

Scheme S-3:



Step 1: To a water (20 mL) solution of copper powder (0.5 g, 7.94 mmol) was added 70% aqueous solution of *t*-butyl hydroperoxide (2.0 mL, 15.87 mmol) dropwise over 5 minutes. The resulting mixture was stirred at 90 °C for 24 h, and this hot mixture was filtered through a filter paper. The filtrate was concentrated in vacuum to give slightly blue Cu-complex (25 mg) contained 28.03% Cu. **Step 2**: By following the standard procedures for catalytic operation using this copper complex (2.59 mg, 5 mol%), *N*, *N*-dimethyl aniline **1b** (100 mg, 0.83 mmol), THF (0.65 mL, 8.25 mmol) and TBHP (0.16 mL, 1.24 mmol) in hot water (90 ^{0}C, 8 h) afforded compound **3b** (117 mg, 75%, 0.62 mmol) as yellow oil.

Scheme S-4:

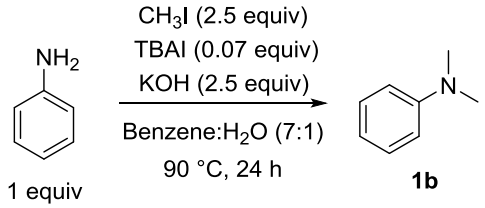


3. Representative Synthetic procedures:

Unless otherwise noted, all reactions were carried out under nitrogen atmosphere in oven-dried glassware using standard syringe, cannula and septa apparatus. Tetrahydrofuran and hexane were dried with sodium metal, benzophenone and distilled before use. Toluene, dichloromethane and 1, 2-dichloroethane were dried over CaH_2 and distilled before use. Methanol and triethylamine were stored over 4Å molecular sieves prior to use. Reagents were purchased from commercial sources and used without purification, unless otherwise stated. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker 400 MHz, Varian 500 MHz and a Bruker 600 MHz spectrometers using chloroform-d ($CDCl_3$) and DCM-d_2 (CD_2Cl_2) as the internal standard. For compound **2c**[s1], **2d**[s1], *N*, *N*-Dialkylanilines[s2-5], tetrahydrofuran-2-ol (lactol)[s6] and d_6-**1b**[s7] were prepared according to known literature procedures. The spectral data for products **3b**-**c**, **3f**, **3e**, **5a**, **5c**, **and 5d** are in agreement with reported data.[s8-9] Compound **2b, 2e, 2f, 2g**, **1c**, **1f** are commercially available.

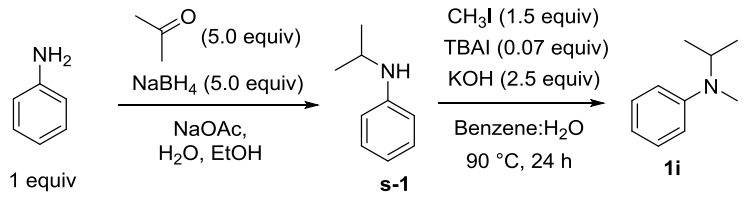
A] General procedure for synthesis of Dialkyl anilines.

a) Synthesis of *N*, *N*-dimethylaniline (1b).[s2]



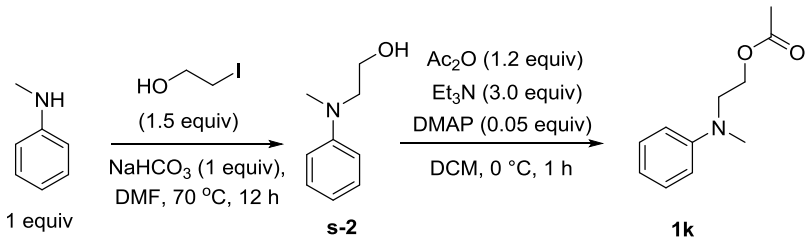
General procedure 1: To a benzene and water (7:1) solution (32 mL) of aniline (2.0 g, 21.48 mmol), tetra-*n*-butylammonium iodide (0.56 g, 1.50 mmol), potassium hydroxide (3.01 g, 53.70 mmol), was added iodomethane (7.68 g, 53.70 mmol) dropwise at room temperature; the reaction mixture was heated at 90 °C for 24 h before it was cooled to RT. The organic layer was extracted with diethyl ether and washed successively with water, saturated sodium carbonate, and dried over magnesium sulfate. The solution was concentrated and eluted through a silica gel column with hexane to give compound **1b** as a light yellow oil (1.8 g, 69.2%, 14.85 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26 ~ 7.22 (m, 2 H), 6.76 ~ 6.70 (m, 3 H), 2.94 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.6, 129.0, 116.5, 112.6, 40.5; HRMS calcd for $C_8H_{11}N$: 121.0891, found: 121.0891.

b) Synthesis of *N*-isopropyl-*N*-methylaniline (1i).[s 4]



To a water and ethanol (1:1) solution (50 mL) of aniline (2 g, 22.0 mmol), sodium acetate trihydrate (8.7 g, 64.5 mmol), acetic acid (20 mL), and acetone (8 mL, 107.5 mmol), at 0 °C was added sodium borohydride (4.1 g, 107.5 mmol) slowly; the reaction mixture was stirred at RT for 30 min before basified with 10% aqueous solution of NaOH. The organic layer was extracted with diethyl ether, concentrated and eluted through a silica gel column with hexane to give compound **s-1** as light yellow oil (2.1 g, 70.6%, 15.53 mmol). With the preceding procedure using iodomethane (3.33 g, 23.29 mmol) and **s-1** (2.1 g, 15.53 mmol), desired **1i** (1.6 g, 10.72 mmol, 69.6%) was obtained as a colourless oil. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.25 ~ 7.23 (m, 2 H), 6.80 (d, *J* = 7.8 Hz, 2 H), 6.72 ~ 6.69 (m, 1 H), 4.13 ~ 4.08 (m, 1 H), 2.74 (s, 3 H), 1.17 (d, *J* = 6.6 Hz, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 129.1, 116.4, 113.3, 48.9, 29.7, 19.3.

c) Synthesis of 2-(methyl(phenyl)amino)ethyl acetate (1k).



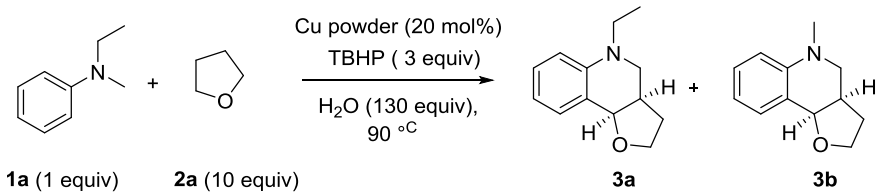
To a DMF (15 mL) solution of *N*-methyl aniline (1.0 g, 9.33 mmol), and sodium hydrogencarbonate (0.78 g, 9.33 mmol) was added 2-iodoethanol (2.4 g, 14.0 mmol) dropwise over 5 min; the mixture was heated to 70 °C for 12 h before it was cooled to RT. The organic layer was extracted with ether and washed successively with water, saturated sodium carbonate, and brine then dried over anhydrous magnesium sulfate. The solution was concentrated and eluted through a silica gel column to give compound **s-2** as yellow oil (1.3 g, 92%, 8.59 mmol).

To a DCM (25 mL) solution of **s-2** (1.3 g, 8.59 mmol) was added 4-Dimethylaminopyridine (52 mg, 0.42 mmol), acetic anhydride (10.3 mmol, 1.1 g) and Et_3N (25.79 mmol, 3.6 mL) at 0 °C and the resulting mixture was stirred for 1 h at room temperature. The reaction mixture was quenched with water and extracted with dichloromethane, concentrated under reduced pressure. The residue was eluted through a silica gel column using hexane and ethyl acetate (9:1) as

eluents to afford acetate **1k** as a yellow liquid (1.1 g, 5.69 mmol, 66.2%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 ~ 7.20 (m, 2 H), 6.74 ~ 6.69 (m, 3 H), 4.24 (t, *J* = 6.2 Hz, 2 H), 3.58 (t, *J* = 6.1 Hz, 2 H), 2.96 (s, 3 H), 2.00 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.0, 149.0, 129.2, 125.0, 116.7, 112.3, 61.5, 51.2, 38.6, 20.9; HRMS calcd for: $C_{11}H_{15}NO_2$: 193.1103, found: 193.1111.

4] Standard procedures for catalytic operations:

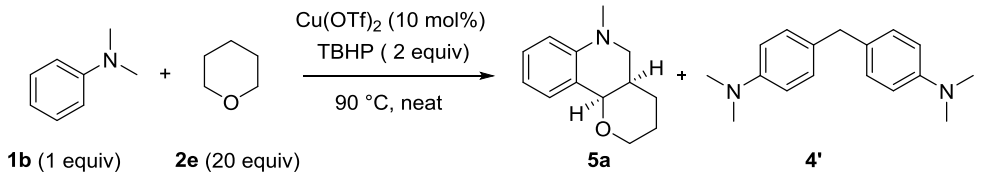
a) Typical procedure for the synthesis of 5-ethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3a).



Condition A : Under nitrogen atmosphere, a reaction flask was charged with copper powder (9.32 mg, 0.15 mmol), tetrahydrofuran (0.6 mL, 7.40 mmol), *N*-ethyl-*N*-methylaniline (100 mg, 0.74 mmol), water (2 mL, 130 equiv), and to this mixture was added 70% aqueous solution of *tert*-butyl hydroperoxide (0.28 mL, 2.22 mmol) dropwise over 5 minutes. The resulting mixture was stirred at 90 °C for 28 h under air. After completion, this reaction mixture was cooled to room temperature, extracted with ethyl acetate, and concentrated in vacuum, and the crude residue was purified by a silica column using hexane and ethyl acetate (9:1) as eluents to afford **3a** (93 mg, 0.45 mmol, 62%) and **3b** (15 mg, 0.07 mmol, 10%) as yellow oil. Spectral data for **3a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (dd, *J* = 7.4, 1.8 Hz, 1 H), 7.17 ~ 7.13 (m, 1 H), 6.71 ~ 6.66 (m, 2 H), 4.53 (d, *J* = 5.2 Hz, 1 H), 3.96 ~ 3.90 (m, 1 H), 3.83 ~ 3.77 (m, 1 H), 3.54 ~ 3.45 (m, 1 H), 3.30 ~ 3.21 (m, 1 H), 2.98 (dd, *J* = 11.4, 5.2 Hz, 1 H), 2.84 (t, *J* = 11.2 Hz, 1 H), 2.44 ~ 2.39 (m, 1 H), 2.27 ~ 2.19 (m, 1 H), 1.77 ~ 1.70 (m, 1 H), 1.11 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.4, 131.7, 129.0, 121.2, 116.5, 111.6, 76.1, 65.0, 49.2, 45.3, 35.6, 30.0, 10.6; HRMS calcd for $C_{13}H_{17}NO$: 203.1310, found: 203.1309. Spectral data for **3b**[s9]**:** ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (dd, *J* = 7.5, 1.6 Hz, 1 H), 7.22 ~ 7.18 (m, 1 H), 6.78 ~ 6.74 (m, 1 H), 6.70 (d, *J* = 8.3 Hz, 1 H), 4.58 (d, *J* = 5.4 Hz, 1 H), 3.97 ~ 3.91 (m, 1 H), 3.83 ~ 3.77 (m, 1 H), 3.02 ~ 2.98 (m, 1 H), 2.88 (s, 3 H), 2.78 (t, *J* = 11.1 Hz, 1 H), 2.56 ~ 2.49 (m, 1 H), 2.28 ~ 2.19 (m, 1 H), 1.78 ~ 1.71 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 147.1, 131.1, 129.0, 121.7, 117.4, 111.8, 75.8, 65.1, 52.5, 39.3, 35.9, 30.0; HRMS calcd for $C_{12}H_{15}NO$: 189.1154, found: 189.1145.

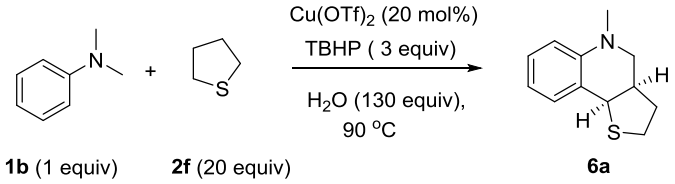
Condition B: similar to **condition A** except cyclic ether **2b-2d** (20.0 equiv) were used as neat.

b) Typical procedure for the synthesis of 6-methyl-3,4,4a,5,6,10b-hexahydro-2*H*-pyrano[3,2-*c*]quinolone (5a).



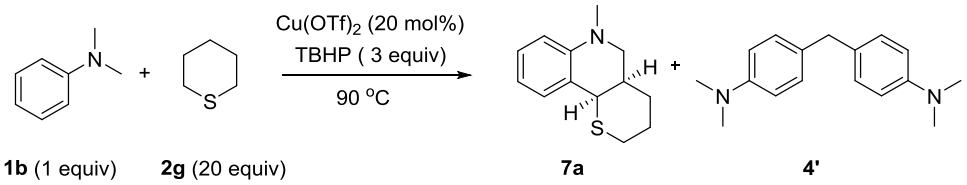
Under argon atmosphere, a reaction flask was charged with $Cu(OTf)_2$ (29.8 mg, 0.08 mmol), pyran **2e** (1.42 g, 16.51 mmol), *N*, *N* dimethyl aniline **1b** (100 mg, 0.82 mmol), and to this mixture was added 70% aqueous solution of *tert*-Butyl hydroperoxide (0.21 mL, 1.65 mmol) dropwise over 5 min at room temperature. The resulting mixture was stirred at 90 °C for 24 h under air tight condition. After completion, reaction mixture was cooled to room temperature, concentrated in vacuum and the crude residue was purified by a silica column using hexane and ethyl acetate (9:1) as eluents to afford **5a** (75.5 mg, 0.37 mmol, 45%) and **4'** (25.2 mg, 0.1 mmol, 12%) both as Yellow oil; spectral data for **5a**[s9]: ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.18 (dd, *J* = 7.5, 1.5 Hz, 1 H) 7.16 ~ 7.13 (m, 1 H), 6.65 (t, *J* = 7.2 Hz, 1 H), 6.60 (d, *J* = 8.4 Hz, 1 H), 4.40 (d, *J* = 3.0 Hz, 1 H), 3.95 ~ 3.93 (m, 1 H), 3.65 (td, *J* = 2.6, 10.9 Hz, 1 H), 3.51 (t, *J* = 11.1 Hz, 1 H), 2.95 ~ 2.92 (m, 1 H), 2.88 (s, 3 H), 2.15 ~ 2.10 (m, 1 H), 1.91 ~ 1.85 (m, 1 H), 1.79 ~ 1.72 (m, 2 H), 1.46 ~ 1.41 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 146.5, 130.7, 129.4, 121.7, 116.4, 111.4, 74.2, 67.4, 51.1, 39.0, 32.4, 25.5, 22.6; HRMS calcd for $C_{13}H_{17}NO$: 203.1310, found: 203.1313. Spectral data for **4'**: ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.03 (d, *J* = 8.4 Hz, 4 H), 6.67 (d, *J* = 8.4 Hz, 4 H), 3.76 (s, 2 H), 2.88 (s, 12 H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 149.6, 130.8, 129.5, 113.3, 41.0, 40.2; HRMS calcd for $C_{17}H_{22}N_2$: 254.1783, found: 254.1780.

c) Typical procedure for the synthesis of 5-methyl-2,3,3a,4,5,9b-hexahydrothieno[3,2-c]quinolone (6a).



Under argon atmosphere, a reaction flask was charged with $Cu(OTf)_2$ (59.7 mg, 0.16 mmol), water (2 mL, 130.0 equiv), tetrahydro thiophene **2f** (16.5 mmol, 1.45 g), *N*, *N* dimethylaniline **1b** (100 mg, 0.82 mmol), and to this mixture was added 70% aqueous solution of *tert*-butyl hydroperoxide (0.318 mL, 2.47 mmol) dropwise over 5 min at room temperature. The resulting mixture was stirred at 90 °C for 24 h under air tight condition. After completion, reaction mixture was cooled to room temperature, extracted with ethyl acetate, concentrated in vacuum and the crude residue was purified by a silica column using hexane and ethyl acetate (19:1) as eluents to afford **6a** (84.7 mg, 0.41 mmol, 50%) as Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.11 ~ 7.07 (m, 2 H), 6.71 ~ 6.62 (m, 2 H), 4.62 (d, *J* = 5.8 Hz, 1 H), 3.22 ~ 3.16 (m, 1 H), 2.97 ~ 2.94 (m, 3 H), 2.89 (s, 3 H), 2.73 ~ 2.66 (m, 1 H), 2.26 ~ 2.15 (m, 1 H), 2.12 ~ 2.05 (m, 1 H); ^{13}C NMR (100 MHz, CDCl3): δ 145.4, 130.4, 128.0, 123.2, 117.1, 111.8, 51.2, 48.7, 40.1, 39.3, 34.4, 30.1; HRMS calcd for $C_{12}H_{15}NS$: 205.0925; found: 205.0918.

d) Typical procedure for the synthesis of 6-methyl-3,4,4a,5,6,10b-hexahydro-2H-thiopyrano[3,2-c]quinoline (7a).



Under argon atmosphere, a reaction flask was charged with $Cu(OTf)_2$ (59.7 mg, 0.16 mmol), tetrahydro thiopyran **2g** (16.50 mmol, 1.68 g), *N*, *N* dimethylaniline **1b** (100 mg, 0.82 mmol), and to this mixture was added 70% aqueous solution of *tert*-Butyl hydroperoxide (0.318 mL, 2.47 mmol) dropwise over 5 min at room temperature. The resulting mixture was stirred at 90 °C for 24 h under air tight condition. After completion, reaction mixture was cooled to room temperature, concentrated in vacuum and the crude residue was purified by a silica column using hexane and ethyl acetate (19:1) as eluents to afford **7a** (83.2 mg, 0.38 mmol, 46%) and **4'** (33.4 mg, 0.13 mmol, 16%) both as yellow oil; spectral data for **7a**: ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.22 ~ 7.21 (m, 1 H), 7.09 ~ 7.06 (m, 1 H), 6.60 (t, *J* = 7.3 Hz, 1 H), 6.54 (d, *J* = 8.2 Hz, 1 H), 4.14 (d, *J* = 3.0 Hz, 1 H), 3.89 (t, *J* = 10.2 Hz, 1 H), 3.04 ~ 3.01 (m, 1 H), 2.89 (s, 3 H), 2.74 ~ 2.70 (m, 1 H), 2.53 ~ 2.50 (m, 1 H), 2.45 ~ 2.43 (m, 1 H), 1.81 ~ 1.75 (m, 4 H); ^{13}C NMR (150

MHz, $CDCl_3$): δ 146.0, 129.6, 128.5, 121.6, 115.9, 110.9, 51.9, 43.1, 38.9, 32.4, 29.4, 28.4, 22.9; HRMS calcd for $C_{13}H_{17}NS$: 219.1082, found: 219.1081.

5. References:

s1. P. Canonne, G. B. Foscolos, D. Bélanger, *J. Org. Chem.*, 1980, **45**, 1828-1835.

s2. M. O. Ratnikov, M. P. Doyle, *J. Am. Chem. Soc*., 2013, **135**, 1549-1557.

s3. J. Barluenga, A. M. Bayon, G. Asensio, *Chem. Commun.*, 1984, 1334-1335.

s4. E. C. Ashby, G. Fred Willard, *J. Org. Chem.*, 1978, **43**, 4750-4758.

s5. G. Cerichelli, G. Mancini, *Tetrahydron*, 1994, **50**, 3797-3802.

s6. A. G. M. Barrett, H. B. Broughton, *J. Org. Chem.*, 1986**, 51**, 495-503**.**

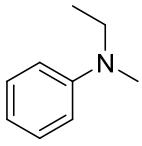
s7. W. Yang, Y. Luo, W. Liu, X. Deng, X. Du, M. Li, *J. Label Compd. Radiopharm*, 2011, **54**, 211–213.

s8. M. Satoru, T. Koji, M. Masahiro, N. Masakatsu, *Heterocycles* 1992, **34**, 1177-1182.

s9. B. V. Subba Reddy, Harish Grewal, *Tetrahedron Lett*. 2011, **52**, 761-763.

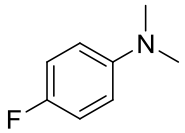
6. Spectral data.

Spectral data for *N*-ethyl-*N*-methylaniline (1a).[s5]



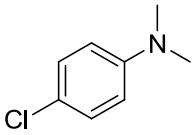
Purified by silica column using hexane as eluent to afford **1a** as yellow oil; ^1H NMR (400 MHz, $CDCl_3$): δ 7.28 ~ 7.24 (m, 2 H), 6.76 ~ 6.70 (m, 3 H), 3.43 (q, *J* = 7.1 Hz, 2 H), 2.93 (s, 3 H), 1.15 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.1, 129.1, 116.0, 112.4, 46.8, 37.4, 11.1; HRMS calcd for $C_9H_{13}N$: 135.1048, found: 135.1051.

Spectral data for 4-fluoro-*N*,*N*-dimethylaniline (1d).[s2]



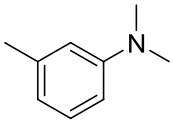
Purified by silica column using hexane as eluent to afford **1d** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.96 ~ 6.92 (m, 2 H), 6.69 ~ 6.67 (m, 2 H), 2.89 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6 (d, *J* = 233.7 Hz), 147.5, 115.3 (d, *J* = 21.8 Hz), 113.9 (d, *J* = 7.2 Hz), 41.4; HRMS calcd for $C_8H_{10}FN$: 139.0797, found: 139.0800.

Spectral data for 4-chloro-*N*,*N*-dimethylaniline (1e).[s2]



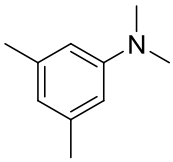
Purified by silica column using hexane as eluent to afford **1e** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15 (d, *J* = 9.0 Hz, 2 H), 6.62 (d, *J* = 9.0 Hz, 2 H), 2.90 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.1, 128.7, 121.3, 113.6, 40.5; HRMS calcd for $C_8H_{10}ClN$: 155.0502, found: 155.0497.

Spectral data for *N*,*N*,3-trimethylaniline (1g).



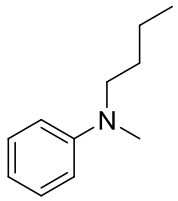
Purified by silica column using hexane as eluent to afford **1g** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 ~ 7.13 (m, 1 H), 6.59 ~ 6.57 (m, 3 H), 2.94 (s, 6 H), 2.34 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.7, 138.7, 128.9, 117.6, 113.4, 109.9, 40.7, 21.9.

Spectral data for *N*,*N*,3,5-tetramethylaniline (1h).



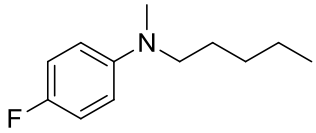
Purified by silica column using hexane as eluent to afford **1h** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.49 ~ 6.48 (m, 3H), 2.99 (s, 6 H), 2.37 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.8, 138.5, 118.7, 110.7, 40.7, 21.7; HRMS calcd for $C_{10}H_{15}N$: 149.1204, found: 149.1205.

Spectral data for *N*-butyl-*N*-methylaniline (1j).[s5]



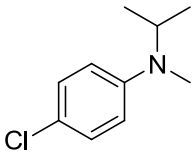
Purified by silica column using hexane as eluent to afford **1j** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 ~ 7.25 (m, 2 H), 6.76 ~ 6.69 (m, 3 H), 3.34 (t, *J* = 7.4 Hz, 2 H), 2.95 (s, 3 H), 1.63 ~ 1.55 (m, 2 H), 1.41 ~ 1.36 (m, 2 H), 0.99 (t, *J* = 7.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.3, 129.1, 115.7, 112.0, 52.5, 38.2, 28.8, 20.3, 14.0; HRMS calcd for: $C_{11}H_{17}N$: 163.1361, found: 163.1364.

Spectral data for 4-fluoro-*N*-methyl-*N*-pentylaniline (1o).



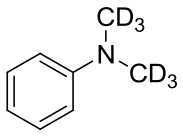
Purified by silica column using hexane as eluent to afford **1o** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.93 ~ 6.87 (m, 2 H), 6.63 ~ 6.59 (m, 2 H), 3.22 (t, *J* = 7.4 Hz, 2 H), 2.85 (s, 3 H), 1.56 ~1.49 (m, 2 H), 1.36 ~ 1.25 (m, 4 H), 0.89 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.1 (d, *J* = 234.0 Hz), 146.3, 115.4 (d, *J* = 22.0 Hz), 113.4 (d, *J* = 7.0 Hz), 53.6, 38.7, 29.3, 26.2, 22.6 14.1; HRMS calcd for $C_{12}H_{18}FN$: 195.1423, found: 195.1423

Spectral data for 4-chloro-N-isopropyl-N-methylaniline (1p).



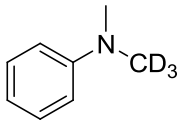
Purified by silica column using hexane as eluent to afford **1p** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15 (d, *J* = 8.1 Hz, 2 H), 6.69 (d, *J* = 8.1 Hz, 2 H), 4.05 ~ 3.99 (m, 1 H), 2.69 (s, 3 H), 1.15 (d, *J* = 6.6 Hz, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.8, 128.8, 121.0, 114.4, 49.2, 29.8, 19.2; HRMS calcd for $C_{10}H_{14}ClN$: 183.0815, found: 183.0819.

Spectral data for d_6 -*N*,*N*-dimethylaniline (d_6-1b).[s7]



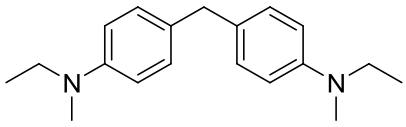
Purified by silica column using hexane as eluent to afford d_6-**1b** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 ~ 7.29 (m, 2 H), 6.82 ~ 6.79 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.7, 129.0, 116.5, 112.5, 39.6 (m); HRMS calcd for: $C_8H_5D_6N$: 127.1268, found: 127.1265.

Spectral data for d_3 -*N*,*N*-dimethylaniline (d_3-1b).



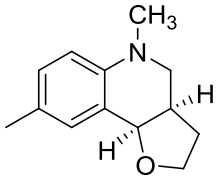
Purified by silica column using hexane as eluent to afford d_3-**1b** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 ~ 7.24 (m, 2 H), 6.77 ~ 6.72 (m, 3 H), 2.95 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.7, 129.0, 116.6, 112.6, 40.5; HRMS calcd for: $C_8H_8D_3N$: 124.1080, found: 124.1074.

Spectral data for 4,4'-methylenebis(N-ethyl-N-methylaniline) (4).



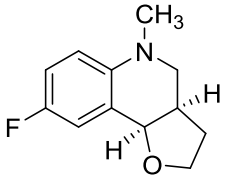
Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.07 (d, *J* = 8.5 Hz, 4 H), 6.68 (d, *J* = 8.6 Hz, 4 H), 3.82 (s, 2 H), 3.37 (q, *J* = 7.0 Hz, 4 H), 2.88 (s, 6 H), 1.12 (t, *J* = 7.0 Hz, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.5, 129.8, 129.5, 112.8, 47.0, 39.8, 37.6, 11.2; HRMS calcd for $C_{19}H_{26}N_2$: 282.2096, found: 282.2091.

Spectral data for 5,8-dimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3c).[s8]



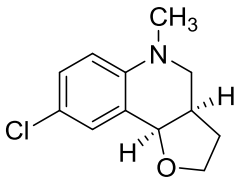
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3c** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 (s, 1 H), 7.00 (dd, *J* = 8.4, 1.8 Hz, 1 H), 6.62 (d, *J* = 8.3 Hz, 1 H), 4.56 (d, *J* = 5.6 Hz, 1 H), 3.96 ~ 3.90 (m, 1 H), 3.82 ~ 3.76 (m, 1 H), 2.96 (dd, *J* = 11.1, 5.2 Hz, 1 H), 2.84 (s, 3 H), 2.71 (t, *J* = 10.9 Hz, 1 H), 2.57 ~ 2.49 (m, 1 H), 2.27 ~ 2.18 (m, 4 H), 1.78 ~ 1.71 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.0, 131.4, 129.5, 126.6, 121.8, 112.0, 75.8, 65.2, 52.9, 39.4, 36.2, 30.1, 20.1; HRMS calcd for $C_{13}H_{17}NO$: 203.1310, found: 203.1308.

Spectral data for 8-fluoro-5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3d).



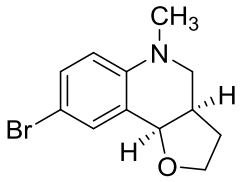
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3d** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06 (dd, *J* = 9.2, 3.2 Hz, 1 H), 6.91 ~ 6.86 (m, 1 H), 6.60 (dd, *J* = 9.2, 4.8 Hz, 1 H), 4.54 (d, *J* = 5.8 Hz, 1 H), 3.94 ~ 3.89 (m, 1 H), 3.81 ~ 3.75 (m, 1 H), 2.98 (dd, *J* = 11.1, 5.0 Hz, 1 H), 2.83 (s, 3 H), 2.70 (t, *J* = 10.8 Hz, 1 H), 2.56 ~ 2.52 (m, 1 H), 2.27 ~ 2.18 (m, 1 H), 1.79 ~ 1.71 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6 (d, *J* = 235 Hz), 143.8, 123.3 (d, *J* = 28 Hz), 117.1 (d, *J* = 21 Hz), 115.4 (d, *J* = 22 Hz), 112.8 (d, *J* = 7.0 Hz), 75.5, 65.4, 52.9, 39.7, 36.3, 30.0; HRMS calcd for $C_{12}H_{14}FNO$: 207.1059, found: 207.1056.

Spectral data for 8-chloro-5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3e).[s9]



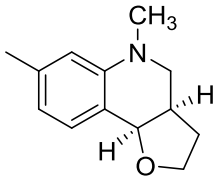
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3e** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 2.6 Hz, 1 H), 7.11 (dd, *J* = 8.8, 2.6 Hz, 1 H), 6.58 (d, *J* = 8.8 Hz, 1 H), 4.51 (d, *J* = 5.6 Hz, 1 H), 3.94 ~ 3.88 (m, 1 H), 3.81 ~ 3.75 (m, 1 H), 2.99 (dd, *J* = 11.3, 5.2 Hz, 1 H), 2.84 (s, 3 H), 2.73 (t, *J* = 11.0 Hz, 1 H), 2.52 ~ 2.47 (m, 1 H), 2.25 ~ 2.20 (m, 1 H), 1.77 ~ 1.70 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 130.6, 128.7, 123.2, 122.0, 113.0, 75.3, 65.2, 52.4, 39.3, 35.9, 29.9; HRMS calcd for $C_{12}H_{14}ClNO$: 223.0764, found: 223.0766.

Spectral data for 8-bromo-5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3f).[s9]



Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3f** as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 2.3 Hz, 1 H), 7.24 (dd, *J* = 8.8, 2.4 Hz, 1 H), 6.53 (d, *J* = 8.8 Hz, 1 H), 4.50 (d, *J* = 5.5 Hz, 1 H), 3.94 ~ 3.88 (m, 1 H), 3.81 ~ 3.75 (m, 1 H), 2.99 (dd, *J* = 11.3, 5.2 Hz, 1 H), 2.84 (s, 3 H), 2.74 (t, *J* = 11.0 Hz, 1 H), 2.52 ~ 2.46 (m, 1 H), 2.26 ~ 2.17 (m, 1 H), 1.77 ~ 1.69 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 133.5, 131.6, 123.7, 113.5, 109.2, 75.3, 65.2, 52.3, 39.3, 35.8, 29.8; HRMS calcd for $C_{12}H_{14}BrNO$: 267.0259, found: 267.0260 and 269.0240.

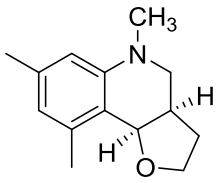
Spectral data for 5,7-dimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3g).



Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3g** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 7.6 Hz, 1 H), 6.60 ~ 6.58 (m, 1 H), 6.52 (s, 1 H), 4.57 (d, *J* = 5.5 Hz, 1 H), 3.96 ~ 3.90 (m, 1 H), 3.82 ~ 3.76 (m, 1 H), 2.98 (dd, *J* = 11.2, 5.2 Hz, 1 H), 2.88 (s, 3 H), 2.77 (t, *J* = 11.0 Hz, 1 H), 2.54 ~ 2.46 (m, 1 H), 2.31 (s, 3 H), 2.27 ~ 2.18 (m, 1 H), 1.78 ~ 1.70 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.0, 138.8, 131.0, 118.9, 118.4,

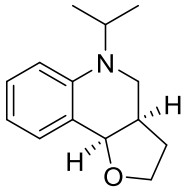
112.4, 75.7, 65.1, 52.6, 39.3, 36.0, 30.0, 21.7; HRMS calcd for $C_{13}H_{17}NO$: 203.1310, found: 203.1308.

Spectral data for 5,7,9-trimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3h).



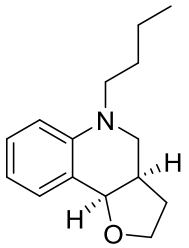
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3h** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.44 (s, 1 H), 6.38 (s, 1 H), 4.56 (d, *J* = 5.0 Hz, 1 H), 3.95 ~ 3.90 (m, 1 H), 3.80 ~ 3.74 (m, 1 H), 2.91 (dd, *J* = 11.1, 5.2 Hz, 1 H), 2.87 (s, 3 H), 2.74 (t, *J* = 11.4 Hz, 1 H), 2.46 ~ 2.38 (m, 1 H), 2.35 (s, 3 H), 2.28 ~ 2.18 (m, 4 H), 1.75 ~ 1.68 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.4, 139.7, 138.3, 120.6, 117.1, 110.4, 73.7, 64.5, 52.3, 39.8, 35.8, 29.8, 21.6, 19.1; HRMS calcd for $C_{14}H_{19}NO$: 217.1467, found: 217.1469.

Spectral data for 5-isopropyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3i).



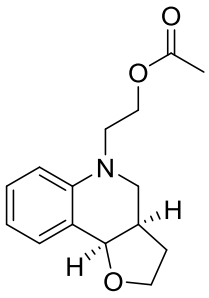
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3i** as yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.33 (dd, *J* = 7.5, 1.6 Hz, 1 H), 7.17 ~ 7.14 (m, 1 H), 6.77 (d, *J* = 8.4 Hz, 1 H), 6.70 ~ 6.67 (m, 1 H), 4.53 (d, *J* = 5.6 Hz, 1 H), 4.11 ~ 4.07 (m, 1 H), 3.93 ~ 3.89 (m, 1 H), 3.79 ~ 3.75 (m, 1 H), 3.10 (dd, *J* = 11.6, 4.8 Hz, 1 H), 2.50 (t, *J* = 11.1 Hz, 1 H), 2.36 ~ 2.32 (m, 1 H), 2.26 ~ 2.20 (m, 1 H), 1.78 ~ 1.73 (m, 1 H), 1.22 (d, *J* = 6.7 Hz, 3 H), 1.10 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 146.1, 131.8, 128.9, 122.1, 116.6, 111.6, 76.1, 65.1, 46.3, 41.2, 36.3, 30.2, 20.3, 17.6; HRMS calcd for $C_{14}H_{19}NO$: 217.1467, found: 217.1464.

Spectral data for 5-butyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3j).



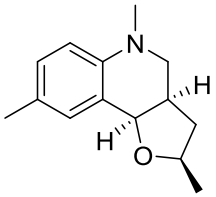
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3j** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (dd, *J* = 7.9, 1.7 Hz, 1 H), 7.17 ~ 7.12 (m, 1 H), 6.69 ~ 6.51 (m, 2 H), 4.52 (d, *J* = 5.2 Hz, 1 H), 3.95 ~ 3.90 (m, 1 H), 3.82 ~ 3.75 (m, 1 H), 3.35 ~ 3.28 (m, 1 H), 3.23 ~ 3.16 (m, 1 H), 3.00 (dd, *J* = 11.5, 5.2 Hz, 1 H), 2.85 (t, *J* = 11.2 Hz, 1 H), 2.42 ~ 2.39 (m, 1 H), 2.27 ~ 2.20 (m, 1 H), 1.74 ~ 1.71 (m, 1 H), 1.59 ~ 1.51 (m, 2 H), 1.39 ~ 1.33 (m, 2 H), 0.94 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 131.7, 129.0, 120.8, 116.3, 111.4, 76.0, 64.9, 51.1, 50.1, 35.5, 29.9, 28.1, 20.5, 14.0; HRMS calcd for $C_{15}H_{21}NO$: 231.1623, found: 231.1599.

Spectral data for 2-(2,3,3a,4-tetrahydrofuro[3,2-c]quinolin-5(9bH)-yl)ethyl acetate (3k).



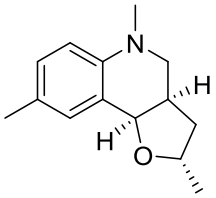
Purified by silica column using hexane and ethyl acetate (4:1) as eluent to afford **3k** as yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 7.8, 1.8 Hz, 1 H), 7.17 ~ 7.14 (m, 1 H), 6.72 ~ 6.70 (m, 2 H), 4.52 (d, *J* = 5.4 Hz, 1 H), 4.24 (t, *J* = 6.6 Hz, 2 H), 3.94 ~ 3.90 (m, 1 H), 3.82 ~ 3.78 (m, 1 H), 3.61 ~ 3.56 (m, 1 H), 3.53 ~ 3.48 (m, 1 H), 3.09 (dd, *J* = 12.0, 5.4 Hz, 1 H), 2.94 (t, *J* = 11.4 Hz, 1 H), 2.42 ~ 2.38 (m, 1 H), 2.25 ~ 2.19 (m, 1 H), 2.02 (s, 3 H), 1.75 ~ 1.70 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.9, 145.1, 131.8, 129.1, 121.0, 117.1, 111.3, 75.9, 65.0, 61.1, 50.7, 49.6, 35.5, 29.7, 20.9; HRMS calcd for : $C_{15}H_{19}NO_3$: 261.1365, found: 261.1370.

Spectral data for (trans)-2,5,8-trimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3l):



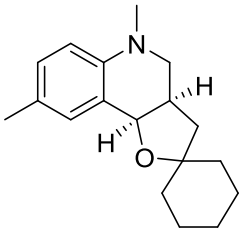
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3l** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.15 (s, 1 H), 6.98 (dd, *J* = 8.4, 2.0 Hz, 1 H), 6.61 (d, *J* = 8.4 Hz, 1 H), 4.51(d, *J* = 5.6 Hz, 1 H), 4.06 ~ 4.00 (m, 1 H), 2.91 (dd, *J* = 11.0, 5.0 Hz, 1 H), 2.83 (s, 3 H), 2.71 (t, *J* = 11.0 Hz, 1 H), 2.51 ~ 2.43 (m, 1 H), 2.40 ~ 2.33 (m, 1 H), 2.22 (s, 3 H), 1.29 (d, *J* = 6.0 Hz, 3 H), 1.22 ~ 1.18 (m, 1 H); ^{13}C NMR (150 MHz, CD_3COCD_3) : δ 146.5, 132.5, 129.9, 126.4, 123.5, 112.6, 76.7, 73.8, 54.7, 39.5, 38.6, 38.0, 21.9, 20.3; HRMS calcd for $C_{14}H_{19}NO$: 217.1467, found: 217.1468.

Spectral data for (cis)-2,5,8-trimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3l):



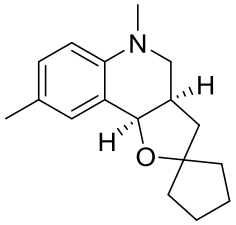
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3l** as yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.15 (s, 1 H), 6.97 (dd, *J* = 8.4, 1.2 Hz, 1 H), 6.59 (d, *J* = 8.4 Hz, 1 H), 4.82 (d, *J* = 6.0 Hz, 1 H), 4.21 ~ 4.16 (m, 1 H), 2.98 (dd, *J* = 11.4, 4.8 Hz, 1 H), 2.82 (s, 3 H), 2.77 (t, *J* =10.8 Hz, 1 H), 2.65 ~ 2.62 (m, 1 H), 2.23 (s, 3 H), 1.99 ~ 1.95 (m, 1 H), 1.83 ~ 1.79 (m, 1 H), 1.27 (d, *J* = 6.0 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 145.2, 131.3, 129.4, 126.9, 122.9, 112.0, 74.9, 72.3, 53.0, 39.6, 38.3, 37.8, 21.8, 20.2; HRMS calcd for $C_{14}H_{19}NO$: 217.1467, found: 217.1462.

Spectral data of 5',8'-dimethyl-3a',4',5',9b'-tetrahydro-3'H-spiro[cyclohexane-1,2'-furo[3,2-c]quinoline] (3m).



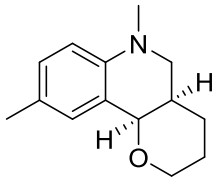
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3m** as colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.13 (d, *J* = 1.8 Hz, 1 H), 6.96 (dd, *J* = 8.3, 2.0 Hz, 1 H), 6.60 (d, *J* = 8.3 Hz, 1 H), 4.70 (d, *J* = 5.4 Hz, 1 H), 2.89 (dd, *J* = 11.0, 5.3 Hz, 1 H), 2.85 ~ 2.78 (m, 4 H), 2.52 ~ 2.44 (m, 1 H), 2.22 (s, 3 H), 2.09 (dd, *J* = 12.8, 8.3 Hz, 1 H), 1.76 ~ 1.44 (m, 11 H); ^{13}C NMR (100 MHz, CDCl3): δ 145.2, 131.7, 129.4, 126.5, 122.5, 112.0, 81.0, 73.5, 53.4, 40.6, 39.8, 39.5, 37.2, 37.1, 25.7, 24.3, 23.5, 20.2; HRMS calcd for $C_{18}H_{26}NO$[M + H]: 272.1936; found: [M + H] :272.2009.

Spectral data of 5',8'-dimethyl-3a',4',5',9b'-tetrahydro-3'H-spiro[cyclopentane-1,2'-furo[3,2-c]quinoline] (3n).



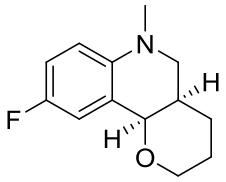
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **3n** as colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.10 (s, 1 H), 6.95 (d, *J* = 8.2 Hz, 1 H), 6.60 (d, *J* = 8.3 Hz, 1 H), 4.58 (d, *J* = 5.6 Hz, 1 H), 2.93 ~ 2.78 (m, 5 H), 2.51 ~ 2.43 (m, 1 H), 2.30 ~ 2.17 (m, 5 H), 2.03 ~ 1.51 (m, 8 H); ^{13}C NMR (100 MHz, CDCl3): δ 145.3, 131.6, 129.5, 126.5, 122.3, 112.0, 89.0, 74.5, 53.4, 41.4, 39.9, 39.5 (one qt. carbon merged), 37.0, 24.1, 24.0, 20.2; HRMS calcd for $C_{17}H_{23}NO$: 257.1780; found: 257.1852.

Spectral data for 6,9-dimethyl-3,4,4a,5,6,10b-hexahydro-2*H*-pyrano[3,2-*c*]quinolone (5d).[s8]



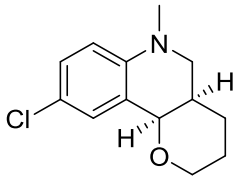
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **5d** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.01 (s, 1 H), 6.96 (dd, *J* = 8.4, 2.0 Hz, 1 H), 6.54 (d, *J* = 8.4 Hz, 1 H), 4.36 (d, *J* =2.8 Hz, 1 H), 3.96 ~ 3.93 (m, 1 H), 3.64 (td, *J* = 10.9, 2.5 Hz, 1 H), 3.46 (t, *J* = 11.0 Hz, 1 H), 2.90 ~ 2.87 (m, 1 H), 2.85 (s, 3 H), 2.20 (s, 3 H), 2.16 ~ 2.08 (m, 1 H), 1.92 ~ 1.84 (m, 1 H), 1.80 ~ 1.69 (m, 2 H), 1.45 ~ 1.39 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.5, 131.1, 130.0, 125.7, 121.8, 111.7, 74.2, 67.5, 51.2, 39.3, 32.6, 25.7, 22.5, 20.1; HRMS calcd for $C_{14}H_{19}NO$: 217.1467, found: 217.1472.

Spectral data for 9-fluoro-6-methyl-3,4,4a,5,6,10b-hexahydro-2*H*-pyrano[3,2-*c*]quinolone (5b).



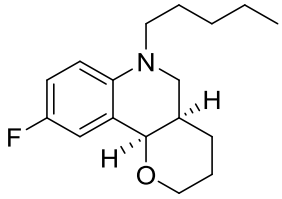
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **5b** as yellow oil; ^{1}H NMR (600 MHz, CD_2Cl_2): δ 6.91 (dd, *J* = 9.0, 3.0 Hz, 1 H), 6.86 (dt, *J* = 8.7, 3.1 Hz, 1 H), 6.53 (dd, *J* = 4.6 Hz, 9.0 Hz, 1 H), 4.39 (d, *J* = 3.2 Hz, 1 H), 3.86 ~ 3.82 (m, 1 H), 3.63 ~ 3.60 (m, 1 H), 3.39 (t, *J* = 10.8 Hz, 1 H), 2.96 ~ 2.94 (m, 1 H), 2.85 (s, 3 H), 2.11 ~ 2.08 (m, 1 H), 1.89 ~ 1.84 (m, 1 H), 1.78 ~ 1.75 (m, 2 H), 1.48 ~ 1.43 (m, 1 H); ^{13}C NMR (150 MHz, CD_2Cl_2) : δ 156.1, 143.8, 123.6 (d, *J* = 6.0 Hz), 116.7 (d, *J* = 22.5 Hz), 115.7 (d, *J* = 22.5 Hz), 112.4 (d, *J* = 7.5 Hz), 74.0, 67.2, 51.9, 39.7, 33.0, 25.6, 23.2 ; HRMS calcd for $C_{13}H_{16}FNO$: 221.1216, found: 221.1211.

9-chloro-6-methyl-3,4,4a,5,6,10b-hexahydro-2*H*-pyrano[3,2-*c*]quinolone (5c).[s9]



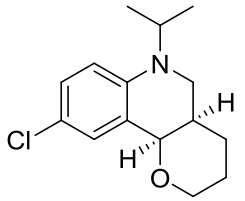
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **5c** as yellow oil; ^{1}H NMR (600 MHz, CD_2Cl_2): δ 7.12 (d, *J* = 2.4 Hz, 1 H), 7.08 (dd, *J* = 9.0, 3.0 Hz, 1 H), 6.53 (d, *J* =8.4 Hz, 1 H), 4.36 (d, *J* = 3.0 Hz, 1 H), 3.88 ~ 3.85 (m, 1 H), 3.64 ~ 3.60 (m, 1 H), 3.45 (t, *J* =10.8 Hz, 1 H), 2.98 ~ 2.96 (m, 1 H), 2.87 (s, 3 H), 2.10 ~ 2.07 (m, 1 H), 1.88 ~ 1.84 (m, 1 H), 1.77 ~ 1.70 (m, 2 H), 1.46 ~ 1.43 (m, 1 H)**;** ^{13}C NMR (150 MHz, CD_2Cl_2): δ 145.7, 130.3, 129.1, 123.8, 120.7, 112.7, 74.0, 67.4, 51.5, 39.2, 32.7, 25.6, 23.1; HRMS calcd for $C_{13}H_{16}ClNO$: 237.0920, found: 237.0918.

Spectral data for 9-fluoro-6-pentyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (5h).



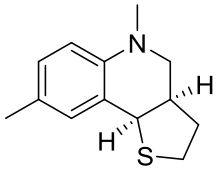
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **5h** as yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 6.92 (dd, *J* = 9.0, 3.6 Hz, 1 H), 6.82 (dt, *J* = 8.7, 3.0 Hz, 1 H), 6.46 (dd, *J* = 4.2, 9.0 Hz, 1 H), 4.39 (d, *J* = 3.0 Hz, 1 H), 3.89 ~ 3.86 (m, 1 H), 3.64 ~ 3.60 (m, 1 H), 3.45 (t, *J* = 10.8 Hz, 1 H), 3.24 ~ 3.19 (m, 1 H), 3.15 ~ 3.12 (m, 1 H), 2.98 ~ 2.96 (m, 1 H), 2.07 ~ 2.05 (m, 1 H), 1.86 ~ 1.81 (m, 1H), 1.76 ~ 1.69 (m, 2 H), 1.54 ~ 1.51 (m, 1 H), 1.48 ~ 1.44 (m, 1 H), 1.37 ~ 1.26 (m, 5 H), 0.89 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.6 (d, *J* = 234.0 Hz), 141.9, 122.0 (d, *J* = 6.0 Hz), 116.6 (d, *J* = 21.0 Hz), 115.7 (d, *J* = 22.5 Hz), 111.6 (d, *J* = 7.5 Hz), 73.7, 66.7, 51.9, 49.5, 32.3, 29.4, 25.8, 25.1, 22.8, 22.6, 14.0; HRMS calcd for $C_{17}H_{24}FNO$: 277.1842, found: 277.1840.

Spectral data for 9-chloro-6-isopropyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (5i).



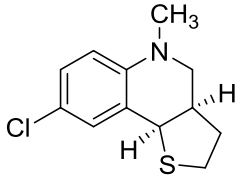
Purified by silica column using hexane and ethyl acetate (9:1) as eluent to afford **5i** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 (d, *J* = 2.5 Hz, 1 H), 7.04 (dd, *J* = 8.9, 2.5 Hz, 1 H), 6.61 (d, *J* = 9.0 Hz, 1 H), 4.36 (d, *J* = 3.0 Hz, 1 H), 4.08 ~ 4.01 (m, 1 H), 3.89 ~ 3.86 (m, 1 H), 3.65 ~ 3.59 (m, 1 H), 3.24 (t, *J* = 10.9 Hz, 1 H), 2.97 (dd, *J* = 11.3, 3.4 Hz, 1 H), 1.98 ~ 1.94 (m, 1 H), 1.84 ~ 1.71 (m, 3 H), 1.48 ~ 1.44 (m, 1 H), 1.18 (d, *J* = 6.6 Hz, 3 H), 1.11 (d, *J* = 6.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9, 130.4, 128.9, 123.1, 120.0, 112.1, 73.7, 66.7, 46.7, 39.9, 31.9, 25.2, 22.7, 19.8, 18.2; HRMS calcd for : $C_{15}H_{20}ClNO$: 265.1233, found: 265.1230.

Spectral data of 5,8-dimethyl-2,3,3a,4,5,9b-hexahydrothieno[3,2-c]quinolone (6b).



Purified by silica column using hexane and ethyl acetate (19:1) as eluent to afford **6b** as pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.90 ~ 6.89 (m, 2 H), 6.55 (d, *J* = 8.8 Hz, 1 H), 4.59 (d, *J* = 6.0 Hz, 1 H), 3.11 (dd, *J* = 11.0, 9.8 Hz, 1 H), 2.96 ~ 2.90 (m, 3 H), 2.84 (s, 3 H), 2.72 ~ 2.65 (m, 1 H), 2.25 ~ 2.05 (m, 5 H); ^{13}C NMR (100 MHz, CDCl3): δ 143.3, 130.9, 128.6, 126.4, 123.5, 112.0, 51.6, 48.6, 40.4, 39.6, 34.5, 30.2, 20.2; EI-MS calcd for $C_{13}H_{17}NS$: 219.1082; found: 219.1084.

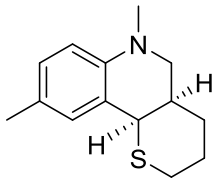
Spectral data for 8-chloro-5-methyl-2,3,3a,4,5,9b-hexahydrothieno[3,2-c]quinoline (6c).



Purified by silica column using hexane and ethyl acetate (19:1) as eluent to afford **6c** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.04 ~ 7.00 (m, 2 H), 6.52 (d, *J* = 8.4 Hz, 1 H), 4.53 (d, *J* =

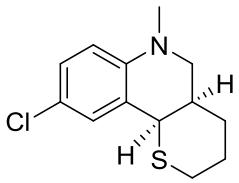
5.9 Hz, 1 H), 3.18 ~ 3.12 (m, 1 H), 2.97 ~ 2.92 (m, 3 H), 2.85 (s, 3 H), 2.70 ~ 2.62 (m, 1 H), 2.24 ~ 2.15 (m, 1 H), 2.10~ 2.00 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 129.8, 127.7, 124.8, 121.5, 112.9, 51.0, 48.3, 40.0, 39.5, 34.3, 30.1; HRMS calcd for $C_{12}H_{14}ClNS$: 239.0535, found: 239.0537.

Spectral data for 6,9-dimethyl-3,4,4a,5,6,10b-hexahydro-2H-thiopyrano[3,2-c]quinoline (7b).



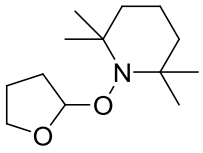
Purified by silica column using hexane and ethyl acetate (19:1) as eluent to afford **7b** as yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.03 (d, *J* = 1.8 Hz, 1 H), 6.90 ~ 6.88 (m, 1 H), 6.48 (d, *J* = 8.4 Hz, 1 H), 4.11 (d, *J* = 3.0 Hz, 1 H), 3.84 (t, *J* = 10.8 Hz, 1 H), 2.99 ~ 2.96 (m, 1 H), 2.86 (s, 3 H), 2.74 ~ 2.70 (m, 1 H), 2.52 ~ 2.50 (m, 1 H), 2.44 ~ 2.42 (m, 1 H), 2.19 (s, 3 H), 1.79 ~ 1.73 (m, 4 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 144.0, 130.2, 129.1, 125.1, 121.8, 111.3, 52.0, 43.1, 39.2, 32.7, 29.5, 28.5, 23.0, 20.2; HRMS calcd for $C_{14}H_{19}NS$: 233.1238, found: 233.1230.

Spectral data for 9-chloro-6-methyl-3,4,4a,5,6,10b-hexahydro-2H-thiopyrano[3,2-c]quinoline (7c).



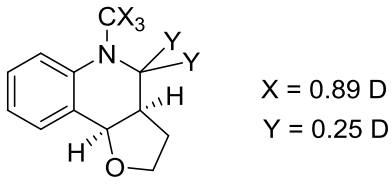
Purified by silica column using hexane and ethyl acetate (19:1) as eluent to afford **7c** as yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 2.32 Hz, 1 H), 7.00 (dd, *J* = 8.8, 2.5 Hz, 1 H), 6.43 (d, *J* = 8.7 Hz, 1 H), 4.05 (d, *J* = 3.4 Hz, 1 H), 3.78 (t, *J* = 10.6 Hz, 1 H), 3.05 (dd, *J* = 11.1, 3.7 Hz, 1 H), 2.86 (s, 3 H), 2.70 ~ 2.64 (m, 1 H), 2.53 ~ 2.48 (m, 1 H), 2.41 ~ 2.36 (m, 1 H), 1.81 ~ 1.71 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.5, 129.1, 128.1, 123.0, 120.3, 111.8, 52.2, 42.5, 39.0, 32.4, 28.9, 28.0, 23.2; HRMS calcd for : $C_{13}H_{16}ClNS$: 253.0692, found: 253.0698.

Spectral data for 2,2,6,6-tetramethyl-1-(tetrahydrofuran-2-yloxy)piperidine (8).



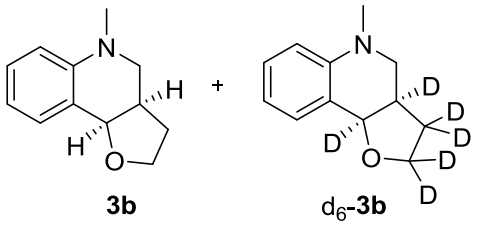
Purified by silica column using hexane and ethyl acetate (4:1) as eluent to afford Colourless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.34 ~ 5.32 (m, 1 H), 3.87 ~ 3.76 (m, 2 H), 1.99 ~ 1.84 (m, 3 H), 1.78 ~ 1.72 (m, 1 H), 1.47 ~ 1.40 (m, 5 H), 1.30 ~ 1.27 (m, 1 H), 1.22 ~ 1.01 (m, 12 H); ^{13}C NMR (100 MHz, $CDCl_3$): 109.6, 66.6, 60.1, 58.6, 40.1, 39.7, 33.9, 33.3, 31.2, 23.9, 20.4, 20.0, 17.2; HRMS calcd $C_{13}H_{25}NO_2$: 227.1885, found: 227.1858.

Spectral data for d_5-5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (d_5-3b).



Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (dd, *J* = 7.4, 1.4 Hz, 1 H), 7.21 ~ 7.16 (m, 1 H), 6.77 ~ 6.73 (m, 1 H), 6.68 (d, *J* = 8.2 Hz, 1 H), 4.58 (d, *J* = 5.5 Hz, 1 H), 3.96 ~ 3.90 (m, 1 H), 3.83 ~ 3.77 (m, 1 H), 2.99 (dd, *J* = 11.2, 5.2 Hz, 0.76 H), 2.88 (s, 0.38 H), 2.77 (t, *J* = 11.0 Hz, 0.76 H), 2.54 ~ 2.49 (m, 1 H), 2.28 ~ 2.19 (m, 1 H), 1.78 ~ 1.73 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.2, 131.2, 129.1, 121.7, 117.4, 111.8, 75.8, 65.2, 52.5, 36.0, 30.1; HRMS calcd for $C_{12}H_{12}D_3NO$: 192.1342, found: 192.1339; HRMS calcd for $C_{12}H_{10}D_5NO$: 194.1467, found: 194.1463.

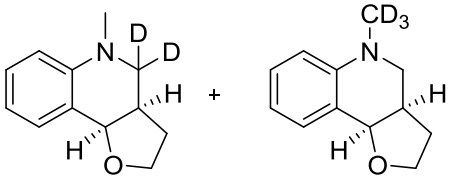
Spectral data for 5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (**3b**) and d_6-**5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline** (d_6-**3b**).



3b d_6-**3b**

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (dd, *J* = 7.4, 1.4 Hz, 1 H), 7.21 ~ 7.17 (m, 1 H), 6.77 ~ 6.73 (m, 1 H), 6.69 (d, *J* = 8.3 Hz, 1 H), 4.58 (d, *J* = 5.5 Hz, 0.84 H), 3.96 ~ 3.90 (m, 0.85 H), 3.83 ~ 3.77 (m, 0.86 H), 3.01 (dd, *J* = 11.2, 5.2 Hz, 1 H), 2.88 (s, 3 H), 2.77 (t, *J* = 11.1 Hz, 1 H), 2.56 ~ 2.48 (m, 0.85 H), 2.29 ~ 2.18 (m, 0.85 H), 1.83 ~ 1.71 (m, 0.86 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.2, 131.2, 129.1, 121.7, 117.4, 111.8, 75.9, 65.2, 52.6, 39.3, 36.0, 30.1; HRMS calcd for **3b** $C_{12}H_{15}NO$: 189.1154, found: 189.1161; HRMS calcd for d6-**3b** $C_{12}H_9D_6NO$: 195.1530, found: 195.1536.

Spectral data for d_2-5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (d_2-**3b**) **and d_3-5-methyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline** (d_3-**3b**).

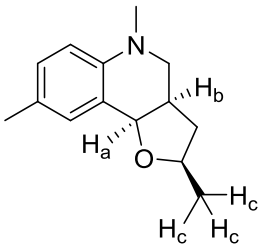


d_2-**3b** d_3-**3b**

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 7.4, 1 H), 7.22 ~ 7.18 (m, 1 H), 6.78 ~ 6.74 (m, 1 H), 6.73 ~ 6.68 (m, 1 H), 4.59 (d, *J* = 5.4 Hz, 1 H), 3.97 ~ 3.91 (m, 1 H), 3.84 ~ 3.78 (m, 1 H), 3.01 ~ 2.98 (m, 0.70 H), 2.88 (s, 0.76 H), 2.80 ~ 2.75 (m, 0.70 H), 2.55 ~ 2.49 (m, 1 H), 2.28 ~ 2.20 (m, 1 H), 1.79 ~ 1.72 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.2, 131.2, 129.0, 121.7, 117.4, 111.8, 75.8, 65.1, 52.6, 52.5, 39.5, 39.3, 36.0, 35.8, 30.1; HRMS calcd for **d3-3b:** $C_{12}H_{12}D_3NO$: 192.1342, found: 192.1345; HRMS calcd for **d2-3b**: $C_{12}H_{13}D_2NO$: 191.1279, found: 191.1275.

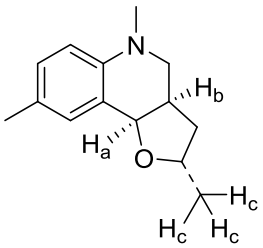
NOE of compounds.

NOE spectra for (trans)-2,5,8-trimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3l).



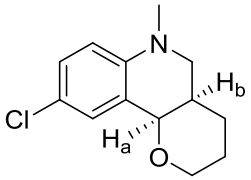
Entry	Irradiation	Intensity increase %
1	H_a (δ 4.51)	H_b (δ 2.51 ~ 2.43, 0.65%)
2	H_b (δ 2.51 ~ 2.43)	H_a (δ 4.51, 1.26%)

NOE spectra for (cis)-2,5,8-trimethyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-c]quinoline (3l).



Entry	Irradiation	Intensity increase %
1	H_a (δ 4.82)	H_b (δ 2.65 ~ 2.62, 0.75%)
2	H_b (δ 2.65 ~ 2.62)	H_a (δ 4.82, 0.89%)
3	H_c (δ 1.27)	H_a (δ 4.82, 0.09%)

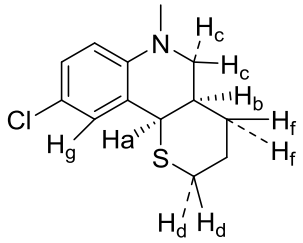
NOE spectra for 9-chloro-6-methyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (5c).



Entry	Irradiation	Intensity increase %
1	H_a (δ 4.36)	H_b (δ 2.10 ~ 2.07, 1.08%)

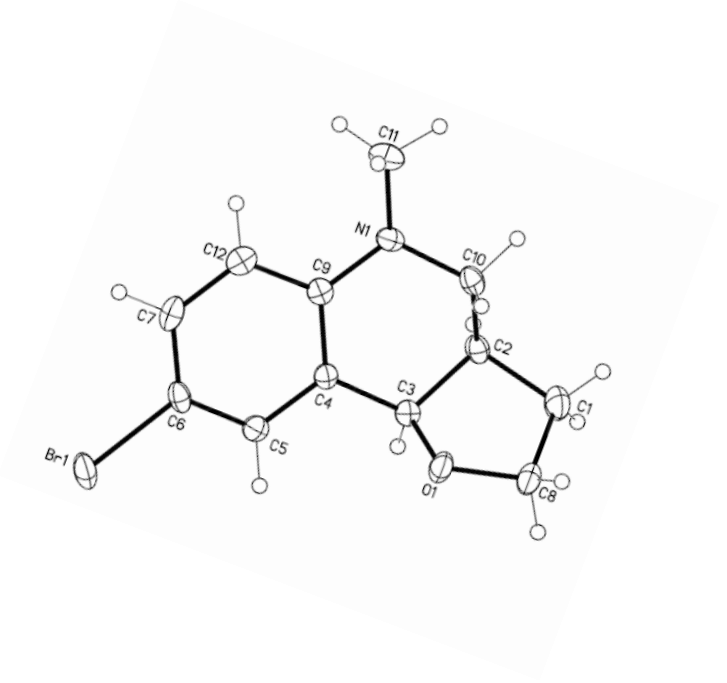
2	H_b (δ 2.10 ~ 2.07)	H_a (δ 4.36, 0.90%)

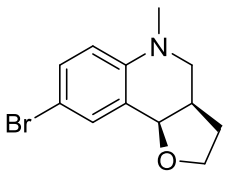
NOE spectra for 9-chloro-6-methyl-3,4,4a,5,6,10b-hexahydro-2H-thiopyrano[3,2-c]quinoline (7c).



Entry	Irradiation	Intensity increase %
1	H_a (δ 4.05)	H_b (δ 2.70 ~ 2.64, 0.47%), H_d (δ 2.41 ~ 2.36, 1.24%), H_g (δ 7.23, 1.14%)
2	H_b (δ 2.70 ~ 2.64)	H_a (δ 4.05, 0.63%)

7. X-ray crystallographic data of compound 3f.





3f

Table 1. Crystal data and structure refinement for 140825LT_0m.

Identification code	140825lt_0m	
Empirical formula	C12 H14 Br N O	
Formula weight	268.15	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 8.8904(6) Å	α= 90°.
	b = 13.0832(9) Å	β= 90°.
	c = 19.2524(11) Å	γ = 90°.
Volume	2239.3(3) $Å^3$	
Z	8	
Density (calculated)	1.591 Mg/m^3	
Absorption coefficient	3.644 mm^{-1}	
F(000)	1088	
Crystal size	0.30 x 0.10 x 0.10 mm^3	
Theta range for data collection	2.116 to 26.411°.	
Index ranges	-11<=h<=10, -16<=k<=15, -23<=l<=24	
Reflections collected	15606	
Independent reflections	2295 [R(int) = 0.0350]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9485 and 0.6826	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2295 / 0 / 137	
Goodness-of-fit on F^2	1.068	
Final R indices [I>2sigma(I)]	R1 = 0.0257, wR2 = 0.0518	
R indices (all data)	R1 = 0.0385, wR2 = 0.0581	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.352 and -0.380 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 140825LT_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	2042(1)	3791(1)	438(1)	27(1)
O(1)	1391(2)	3059(1)	3310(1)	29(1)

N(1)	739(2)	5854(2)	3203(1)	18(1)
C(1)	2068(3)	3736(2)	4418(1)	28(1)
C(2)	2144(2)	4564(2)	3856(1)	18(1)
C(3)	2331(2)	3938(2)	3198(1)	20(1)
C(4)	1822(2)	4464(2)	2546(1)	15(1)
C(5)	2146(2)	4007(2)	1911(1)	17(1)
C(6)	1673(2)	4461(2)	1301(1)	18(1)
C(7)	934(2)	5383(2)	1311(1)	21(1)
C(8)	1556(3)	2776(2)	4027(1)	29(1)
C(9)	1040(2)	5392(2)	2570(1)	16(1)
C(10)	706(2)	5183(2)	3807(1)	19(1)
C(11)	-310(3)	6709(2)	3220(1)	26(1)
C(12)	609(2)	5849(2)	1939(1)	20(1)

Table 3. Bond lengths [Å] and angles [°] for 140825LT_0m.

Br(1)-C(6)	1.907(2)
O(1)-C(8)	1.436(3)
O(1)-C(3)	1.438(3)
N(1)-C(9)	1.387(3)
N(1)-C(10)	1.457(3)
N(1)-C(11)	1.457(3)
C(1)-C(2)	1.532(3)
C(1)-C(8)	1.533(3)
C(1)-H(5)	0.9900
C(1)-H(1)	0.9900
C(2)-C(10)	1.516(3)
C(2)-C(3)	1.518(3)
C(2)-H(4)	1.0000
C(3)-C(4)	1.501(3)
C(3)-H(13)	1.0000
C(4)-C(5)	1.391(3)
C(4)-C(9)	1.400(3)
C(5)-C(6)	1.382(3)
C(5)-H(11)	0.9500
C(6)-C(7)	1.374(3)
C(7)-C(12)	1.385(3)
C(7)-H(2)	0.9500

C(8)-H(14)	0.9900
C(8)-H(3)	0.9900
C(9)-C(12)	1.407(3)
C(10)-H(7)	0.9900
C(10)-H(6)	0.9900
C(11)-H(8)	0.9800
C(11)-H(10)	0.9800
C(11)-H(9)	0.9800
C(12)-H(12)	0.9500
C(8)-O(1)-C(3)	106.99(17)
C(9)-N(1)-C(10)	116.30(19)
C(9)-N(1)-C(11)	118.55(18)
C(10)-N(1)-C(11)	115.58(17)
C(2)-C(1)-C(8)	104.23(17)
C(2)-C(1)-H(5)	110.9
C(8)-C(1)-H(5)	110.9
C(2)-C(1)-H(1)	110.9
C(8)-C(1)-H(1)	110.9
H(5)-C(1)-H(1)	108.9
C(10)-C(2)-C(3)	109.18(17)
C(10)-C(2)-C(1)	112.61(18)
C(3)-C(2)-C(1)	102.29(19)
C(10)-C(2)-H(4)	110.8
C(3)-C(2)-H(4)	110.8
C(1)-C(2)-H(4)	110.8
O(1)-C(3)-C(4)	108.51(17)
O(1)-C(3)-C(2)	103.97(17)
C(4)-C(3)-C(2)	114.72(19)
O(1)-C(3)-H(13)	109.8
C(4)-C(3)-H(13)	109.8
C(2)-C(3)-H(13)	109.8
C(5)-C(4)-C(9)	120.36(19)
C(5)-C(4)-C(3)	118.37(19)
C(9)-C(4)-C(3)	121.27(19)
C(6)-C(5)-C(4)	119.9(2)
C(6)-C(5)-H(11)	120.0
C(4)-C(5)-H(11)	120.0

C(7)-C(6)-C(5)	120.7(2)
C(7)-C(6)-Br(1)	119.93(16)
C(5)-C(6)-Br(1)	119.35(18)
C(6)-C(7)-C(12)	119.9(2)
C(6)-C(7)-H(2)	120.0
C(12)-C(7)-H(2)	120.0
O(1)-C(8)-C(1)	106.88(19)
O(1)-C(8)-H(14)	110.3
C(1)-C(8)-H(14)	110.3
O(1)-C(8)-H(3)	110.3
C(1)-C(8)-H(3)	110.3
H(14)-C(8)-H(3)	108.6
N(1)-C(9)-C(4)	120.20(19)
N(1)-C(9)-C(12)	121.4(2)
C(4)-C(9)-C(12)	118.3(2)
N(1)-C(10)-C(2)	110.75(17)
N(1)-C(10)-H(7)	109.5
C(2)-C(10)-H(7)	109.5
N(1)-C(10)-H(6)	109.5
C(2)-C(10)-H(6)	109.5
H(7)-C(10)-H(6)	108.1
N(1)-C(11)-H(8)	109.5
N(1)-C(11)-H(10)	109.5
H(8)-C(11)-H(10)	109.5
N(1)-C(11)-H(9)	109.5
H(8)-C(11)-H(9)	109.5
H(10)-C(11)-H(9)	109.5
C(7)-C(12)-C(9)	120.7(2)
C(7)-C(12)-H(12)	119.7
C(9)-C(12)-H(12)	119.7

Symmetry transformations used to generate equivalent atoms:

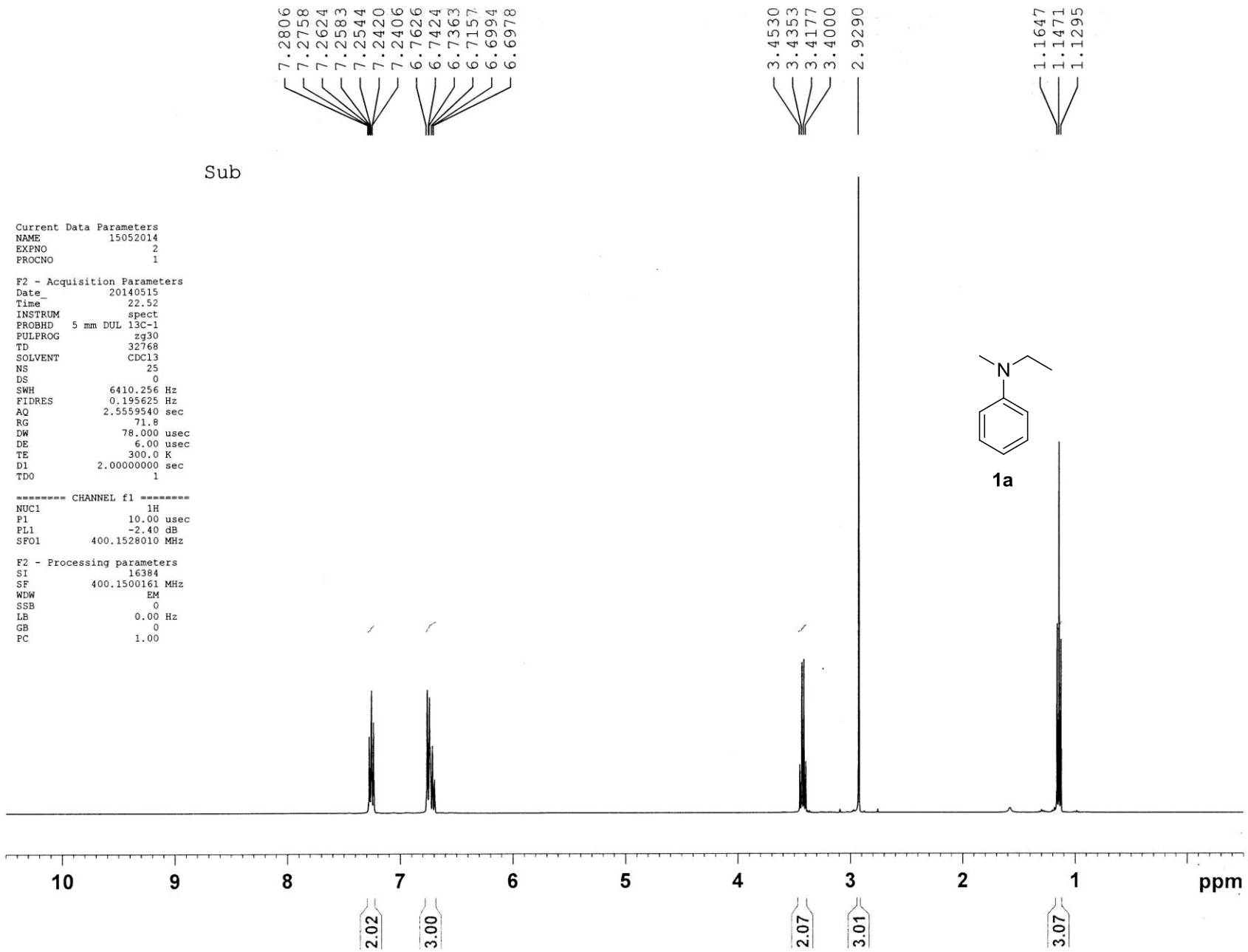
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 140825LT_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	34(1)	32(1)	13(1)	-3(1)	3(1)	-9(1)

O(1)	54(1)	17(1)	14(1)	3(1)	-6(1)	-9(1)
N(1)	19(1)	16(1)	21(1)	-3(1)	2(1)	1(1)
C(1)	37(1)	30(2)	16(1)	1(1)	-3(1)	-1(1)
C(2)	19(1)	21(1)	14(1)	-2(1)	-2(1)	-3(1)
C(3)	24(1)	18(1)	16(1)	0(1)	0(1)	2(1)
C(4)	15(1)	16(1)	15(1)	0(1)	0(1)	-3(1)
C(5)	17(1)	16(1)	19(1)	0(1)	2(1)	-2(1)
C(6)	19(1)	23(1)	13(1)	-3(1)	2(1)	-9(1)
C(7)	18(1)	26(2)	18(1)	7(1)	-4(1)	-7(1)
C(8)	48(2)	23(2)	16(1)	5(1)	-1(1)	-2(1)
C(9)	13(1)	17(1)	18(1)	-1(1)	0(1)	-5(1)
C(10)	18(1)	22(1)	16(1)	-3(1)	3(1)	-5(1)
C(11)	23(1)	22(2)	31(1)	-6(1)	1(1)	4(1)
C(12)	16(1)	20(1)	25(1)	2(1)	-2(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 140825LT_0m.

	x	y	z	U(eq)
H(5)	1337	3923	4784	33
H(1)	3067	3629	4634	33
H(4)	3032	5020	3931	22
H(13)	3403	3719	3148	23
H(11)	2691	3383	1897	21
H(2)	646	5701	888	25
H(14)	2314	2226	4075	35
H(3)	587	2527	4215	35
H(7)	-165	4714	3772	22
H(6)	584	5597	4234	22
H(8)	56	7255	2915	38
H(10)	-390	6968	3697	38
H(9)	-1301	6480	3061	38
H(12)	90	6483	1944	24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DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
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F2 - Processing parameters
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WDW EM
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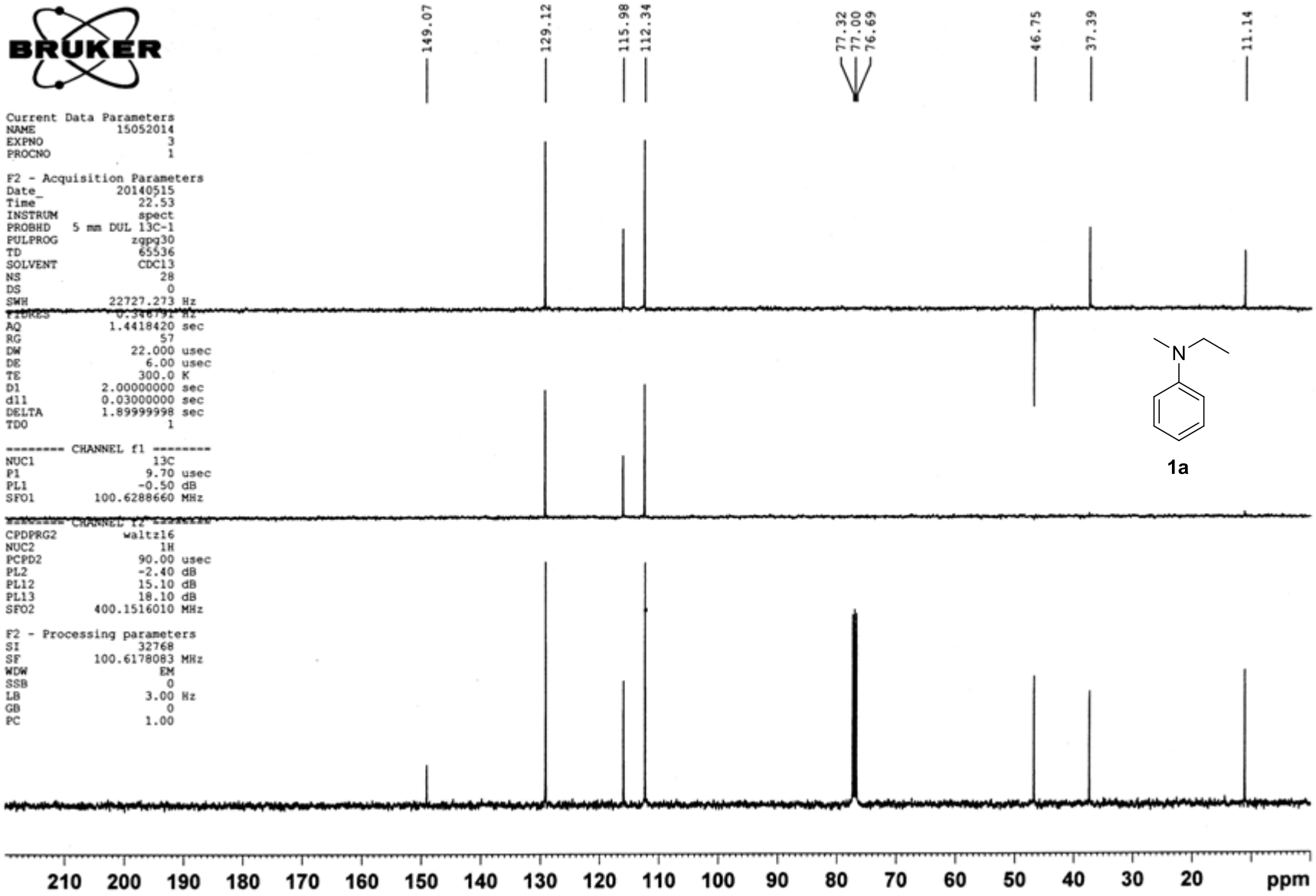
3.07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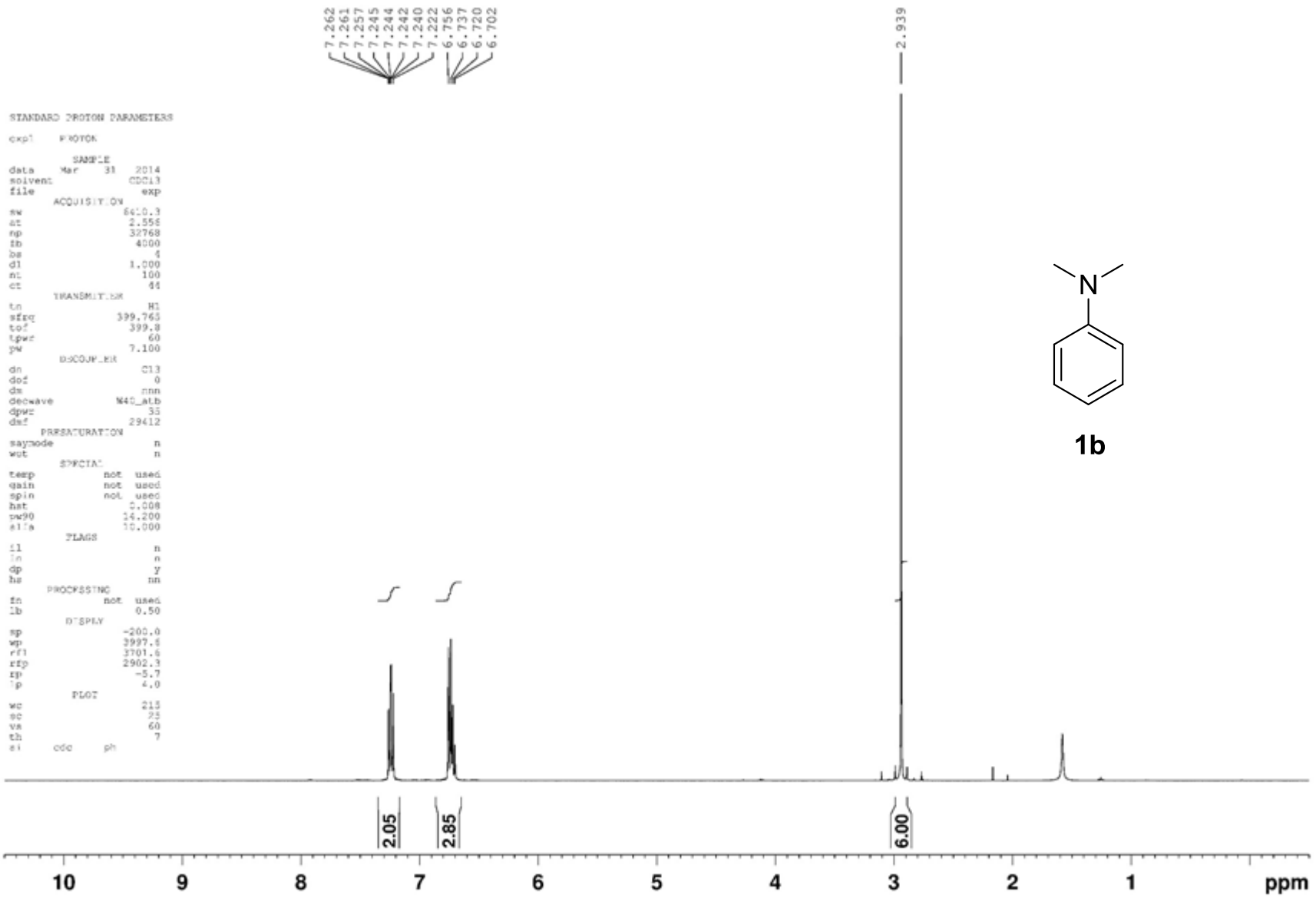
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vs 60
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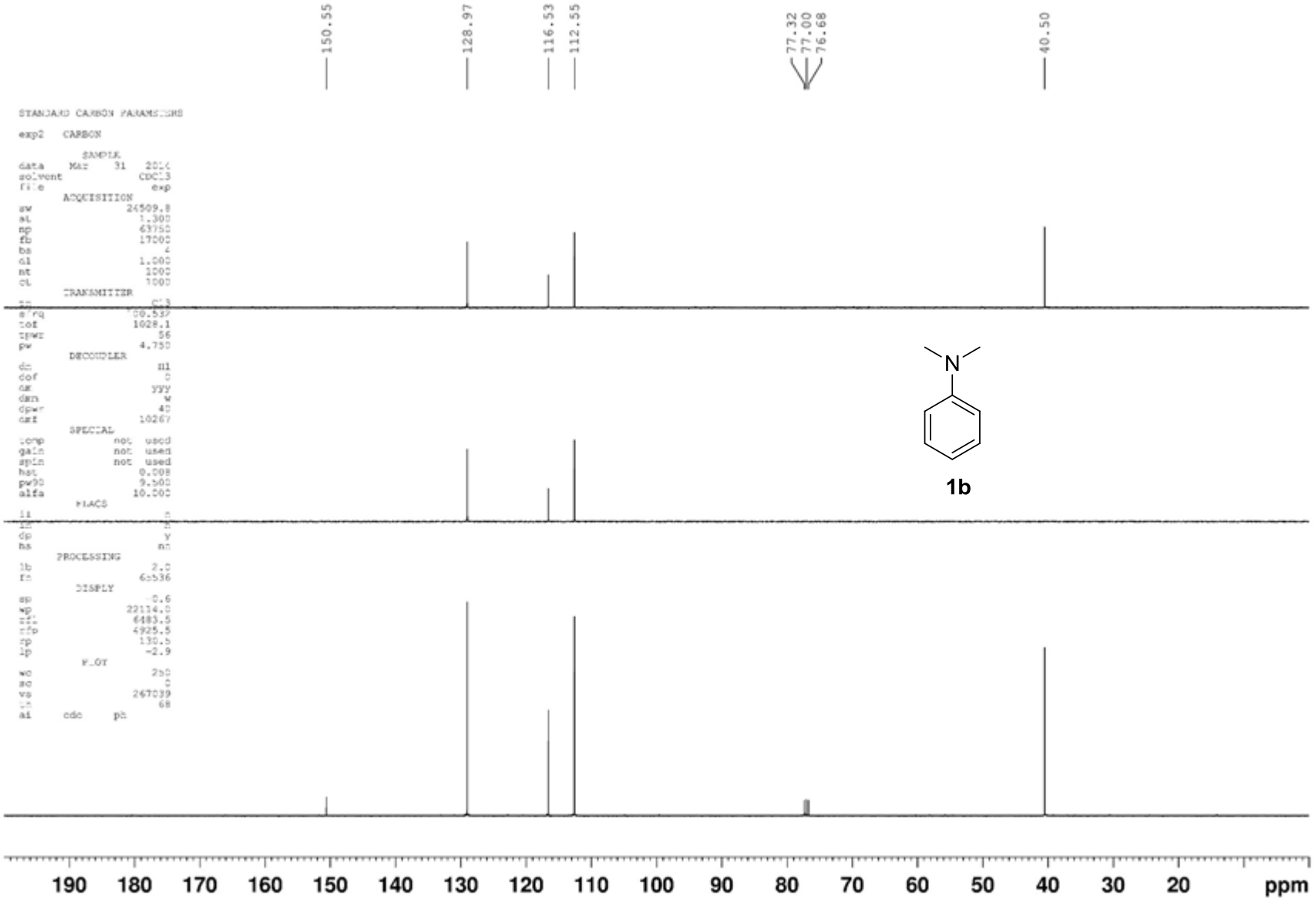
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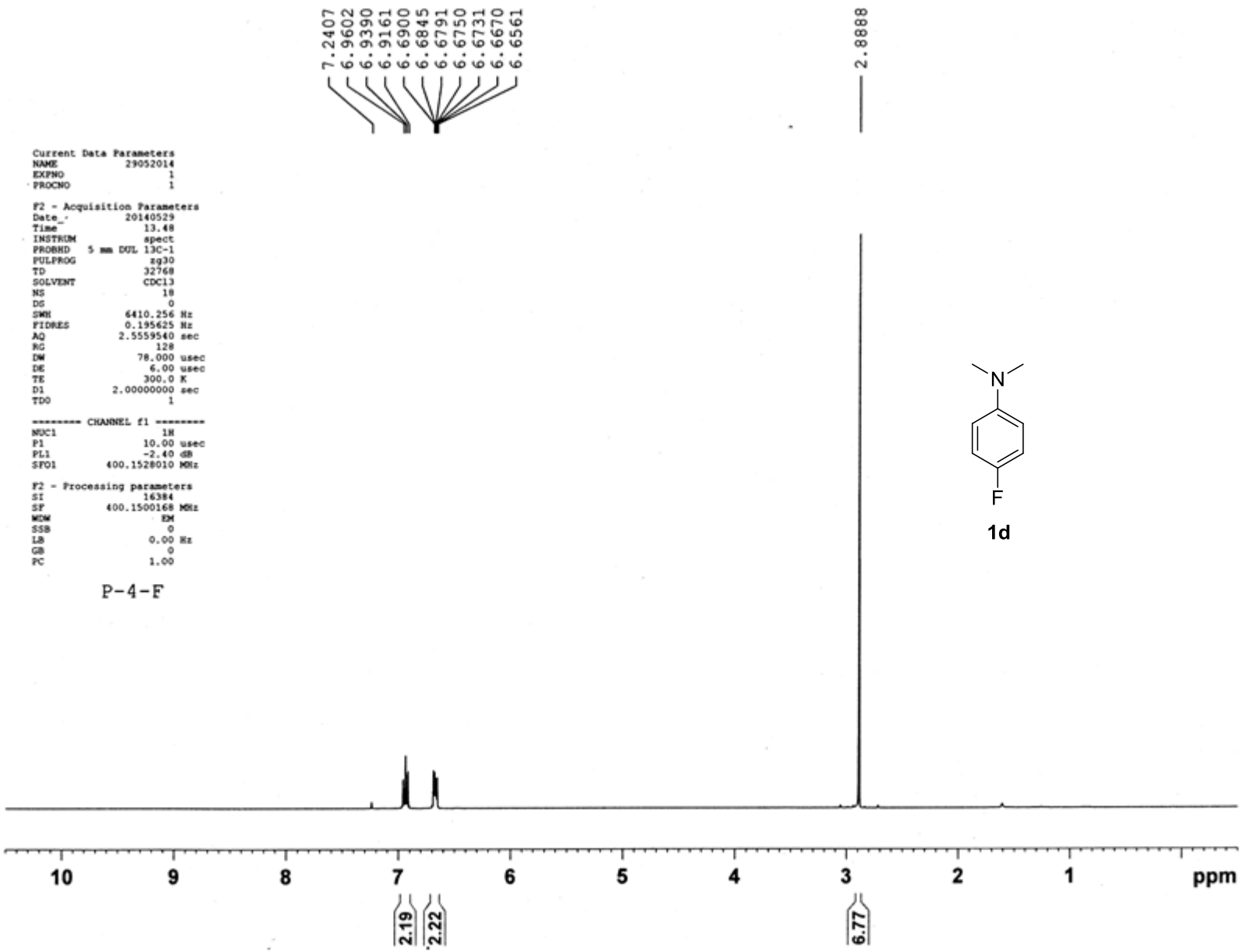
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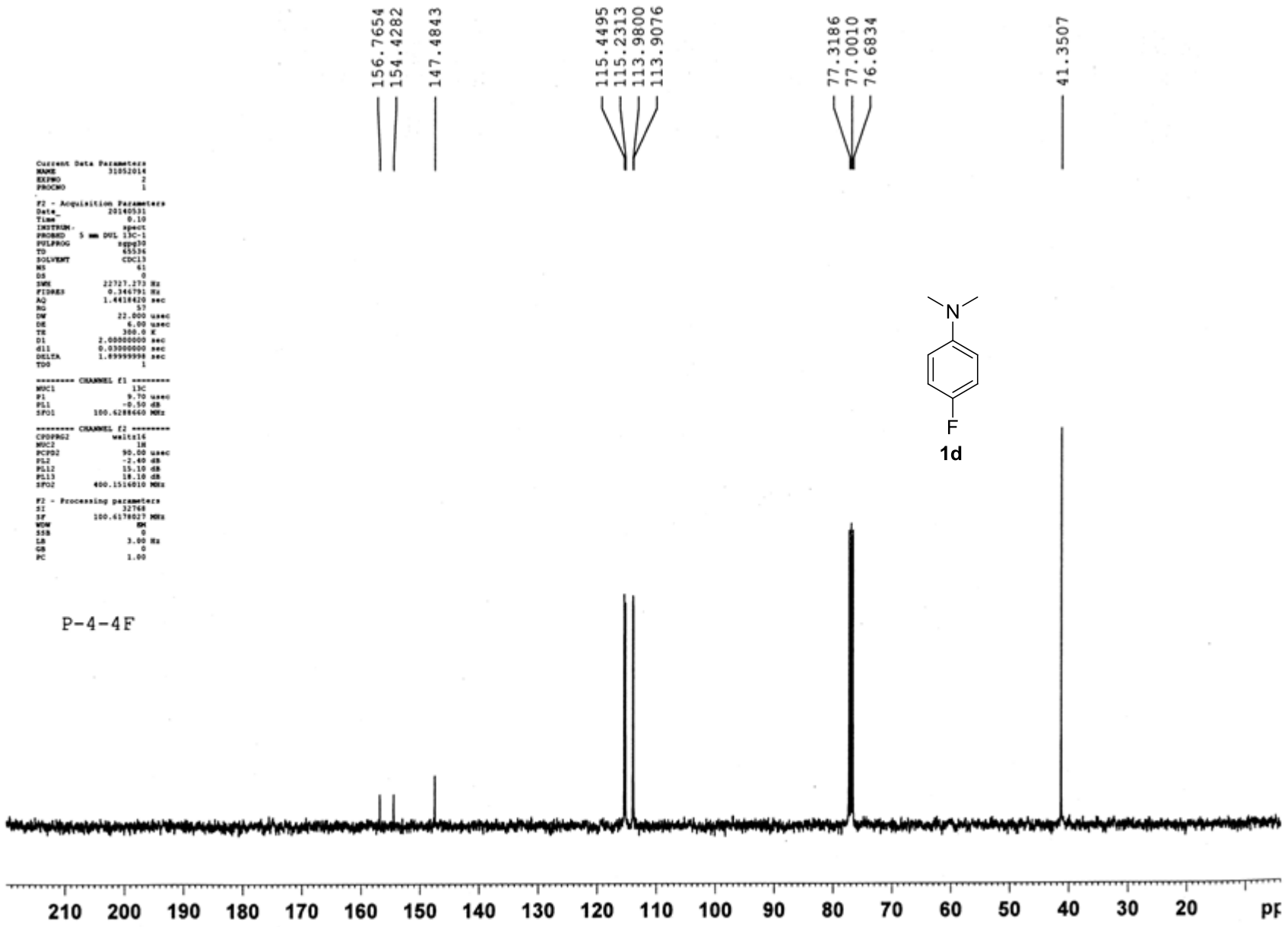
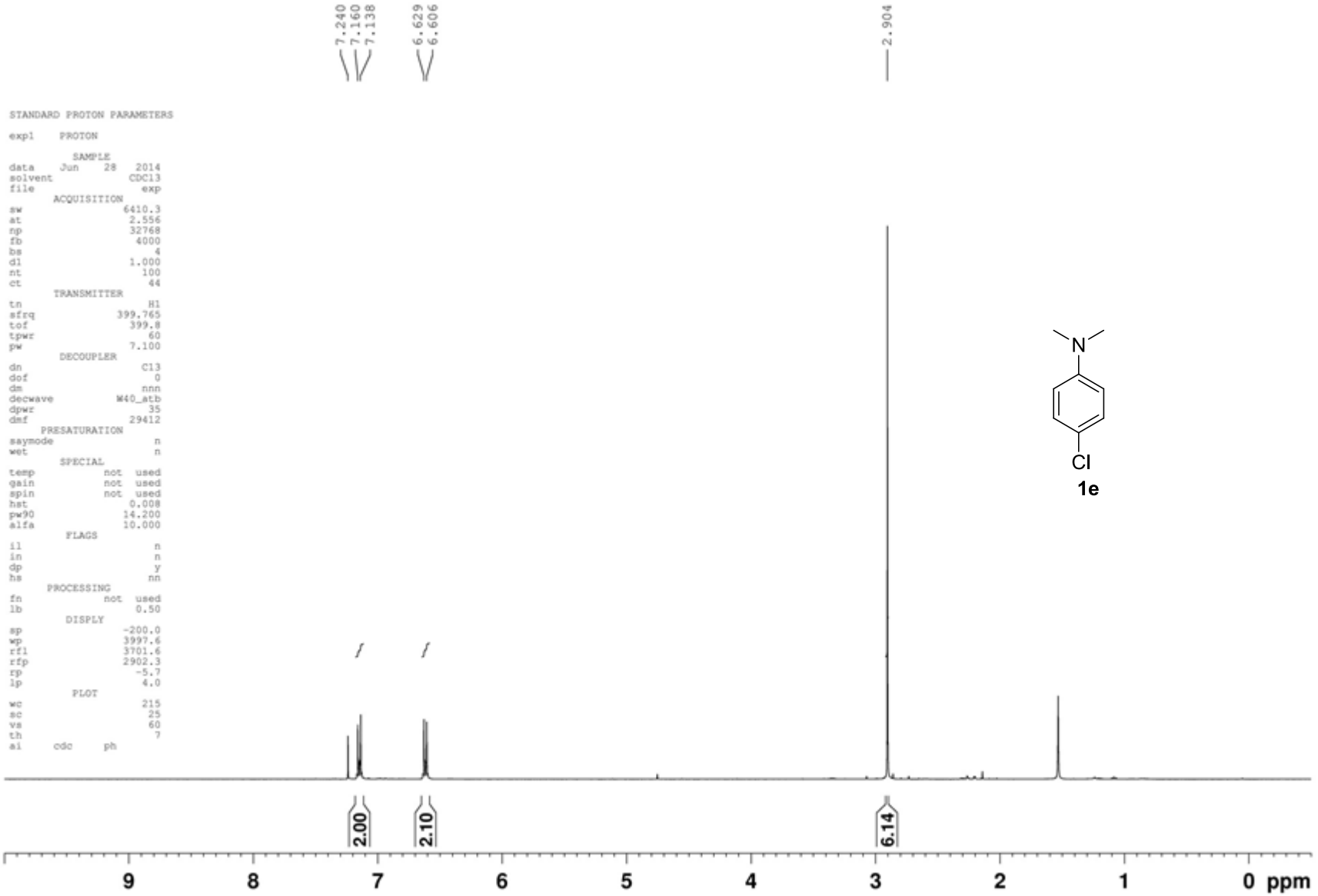
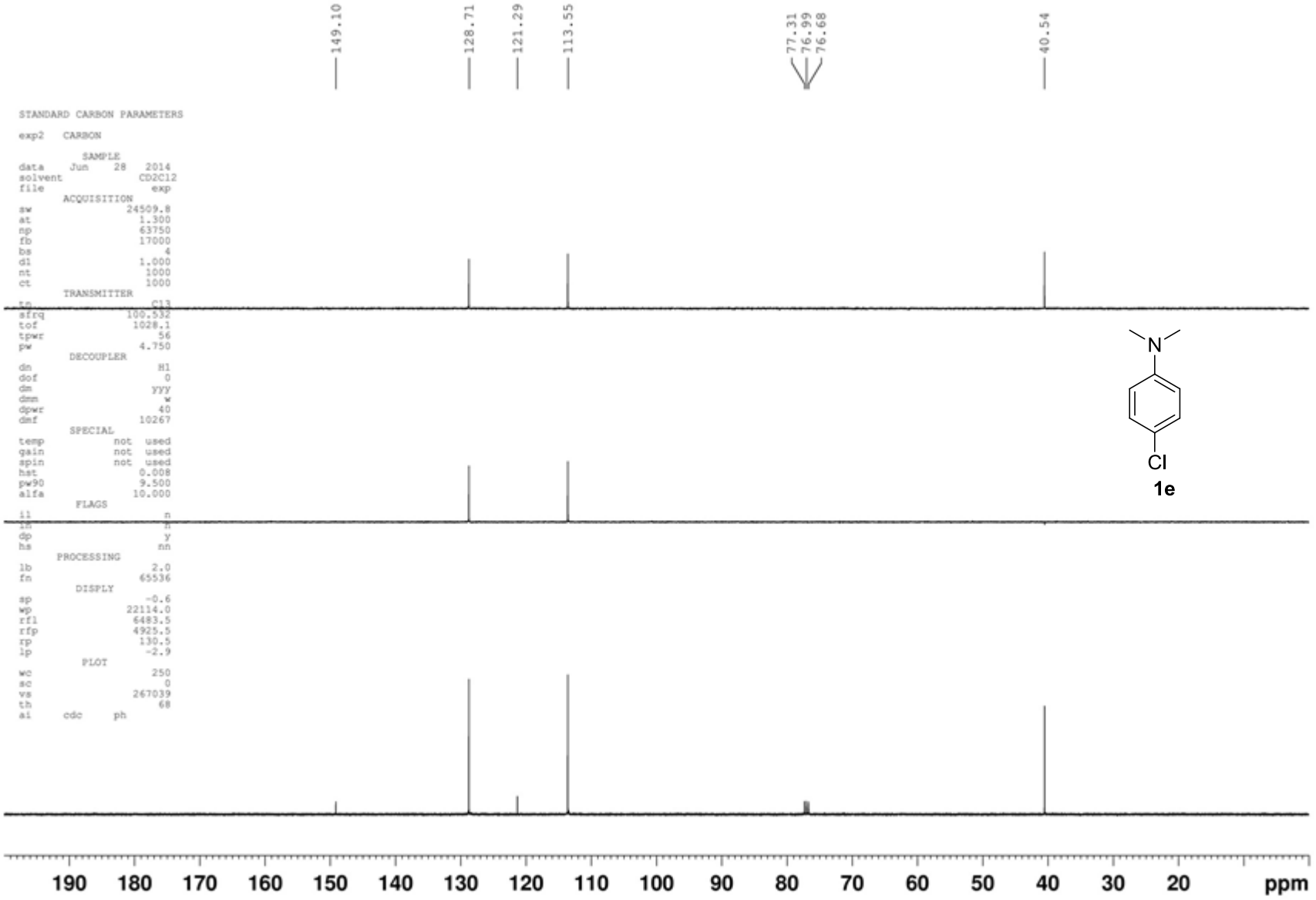
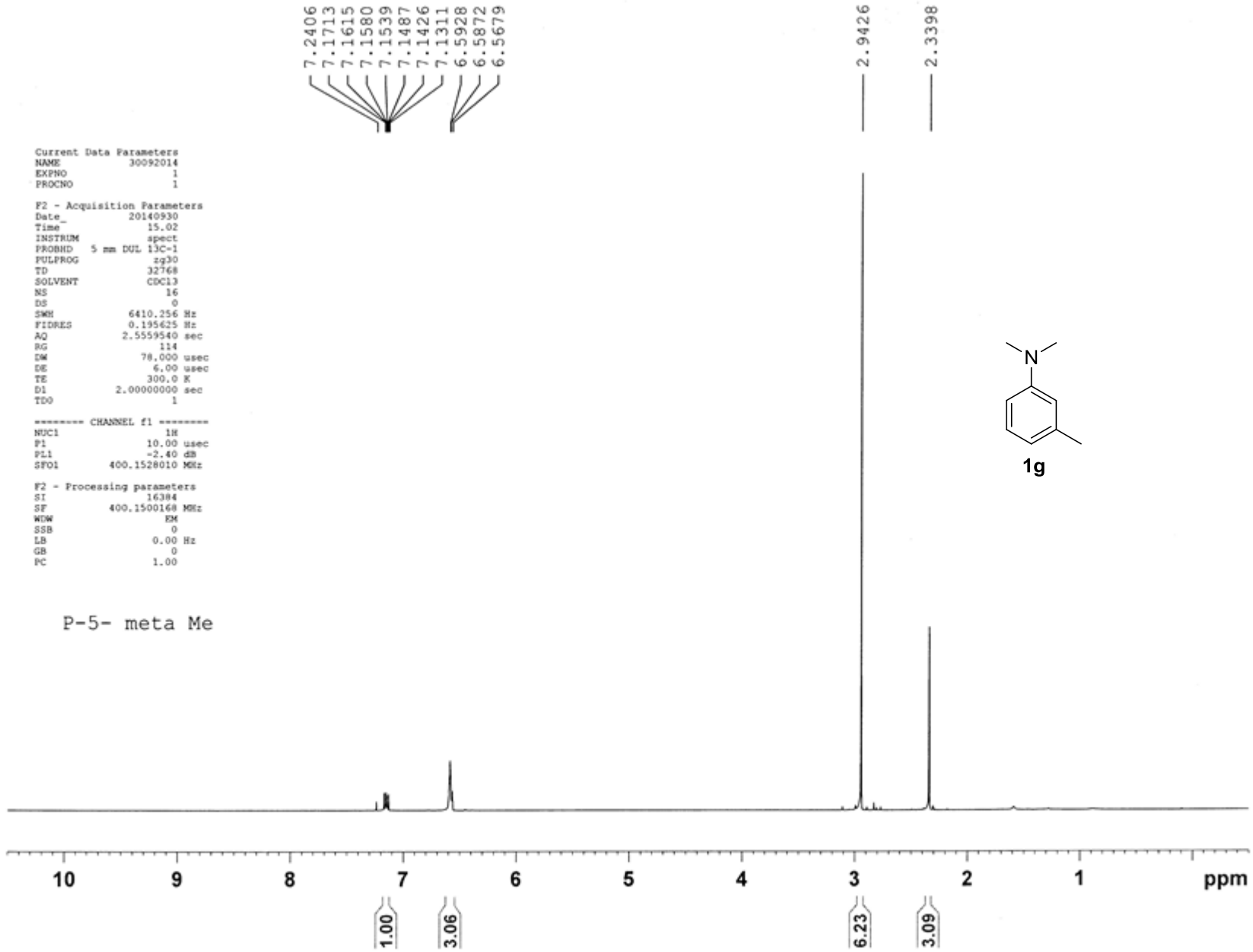
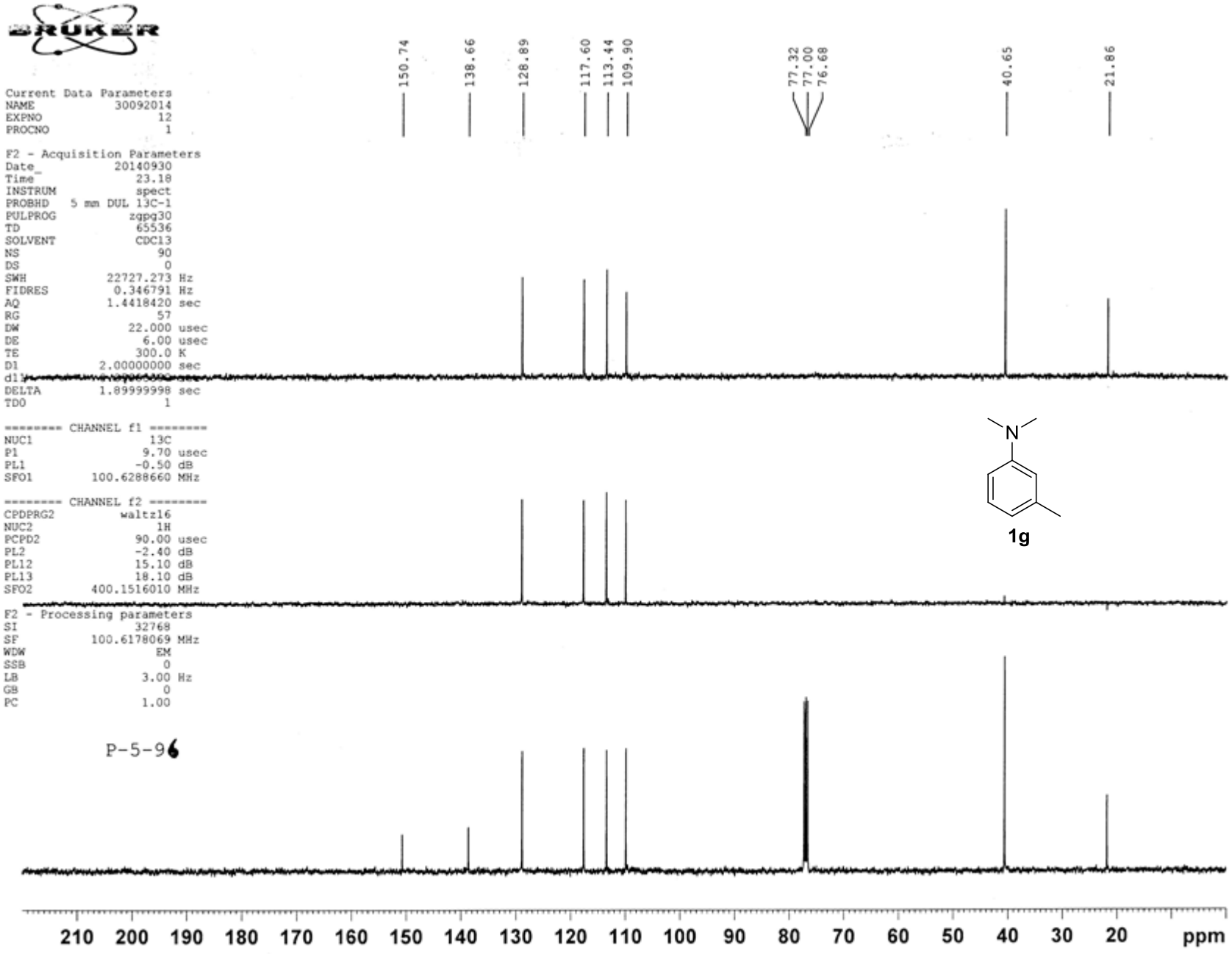
6.6731
6.6670
6.6561
2.8888
Current Data Parameters
NAME 29052014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140529
Time 13.48
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 18
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-4-F
N
F
1d
10
9
8
7
6
5
4
3
2
1
ppm
2.19
2.22
6.77

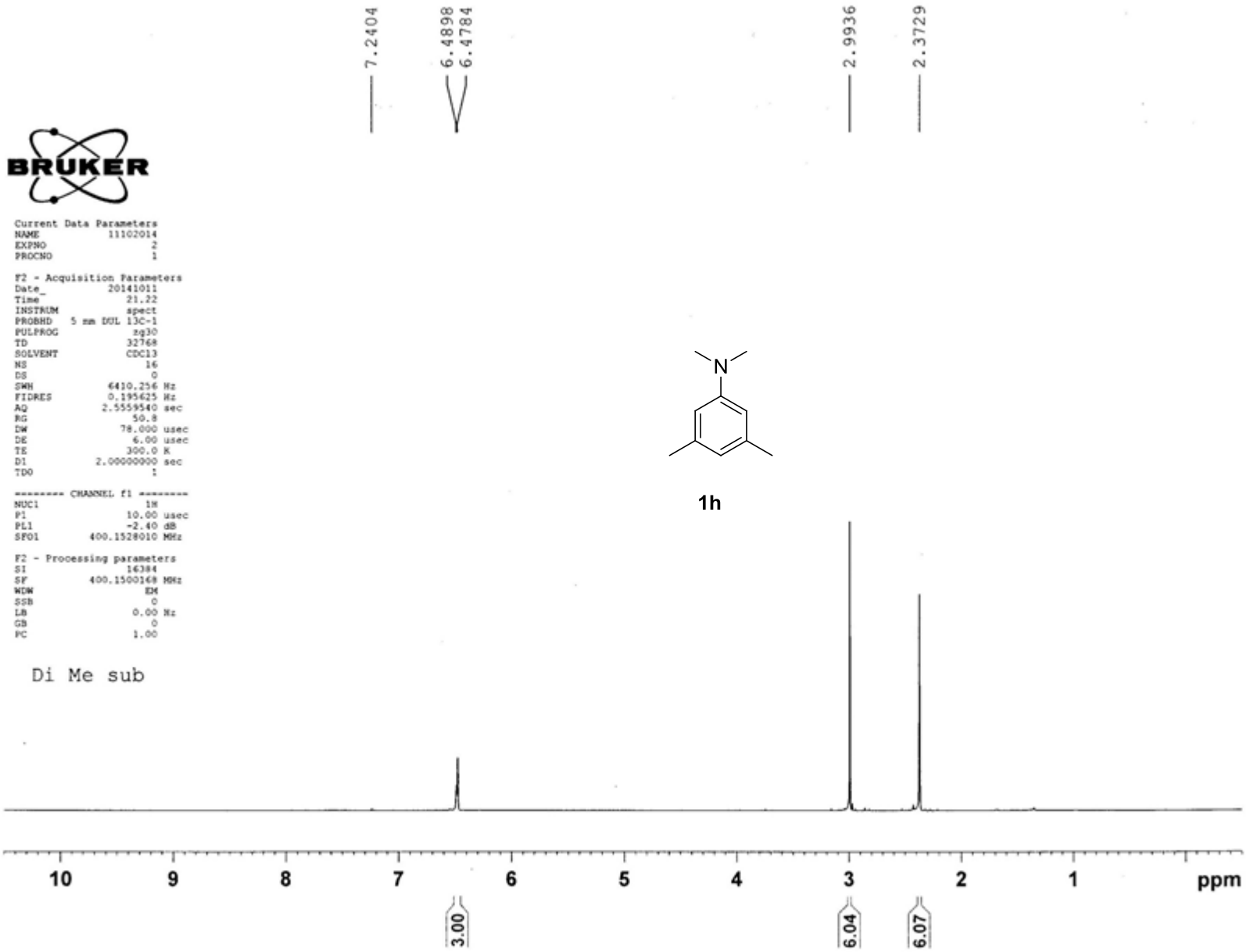

156.7654
154.4282
147.4843
115.4495
115.2313
113.9800
113.9076
77.3186
77.0010
76.6834
41.3507
Current Data Parameters
NAME 31052014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140531
Time 0.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 61
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288460 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1316010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178027 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-4-4F
N
F
1d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 pp

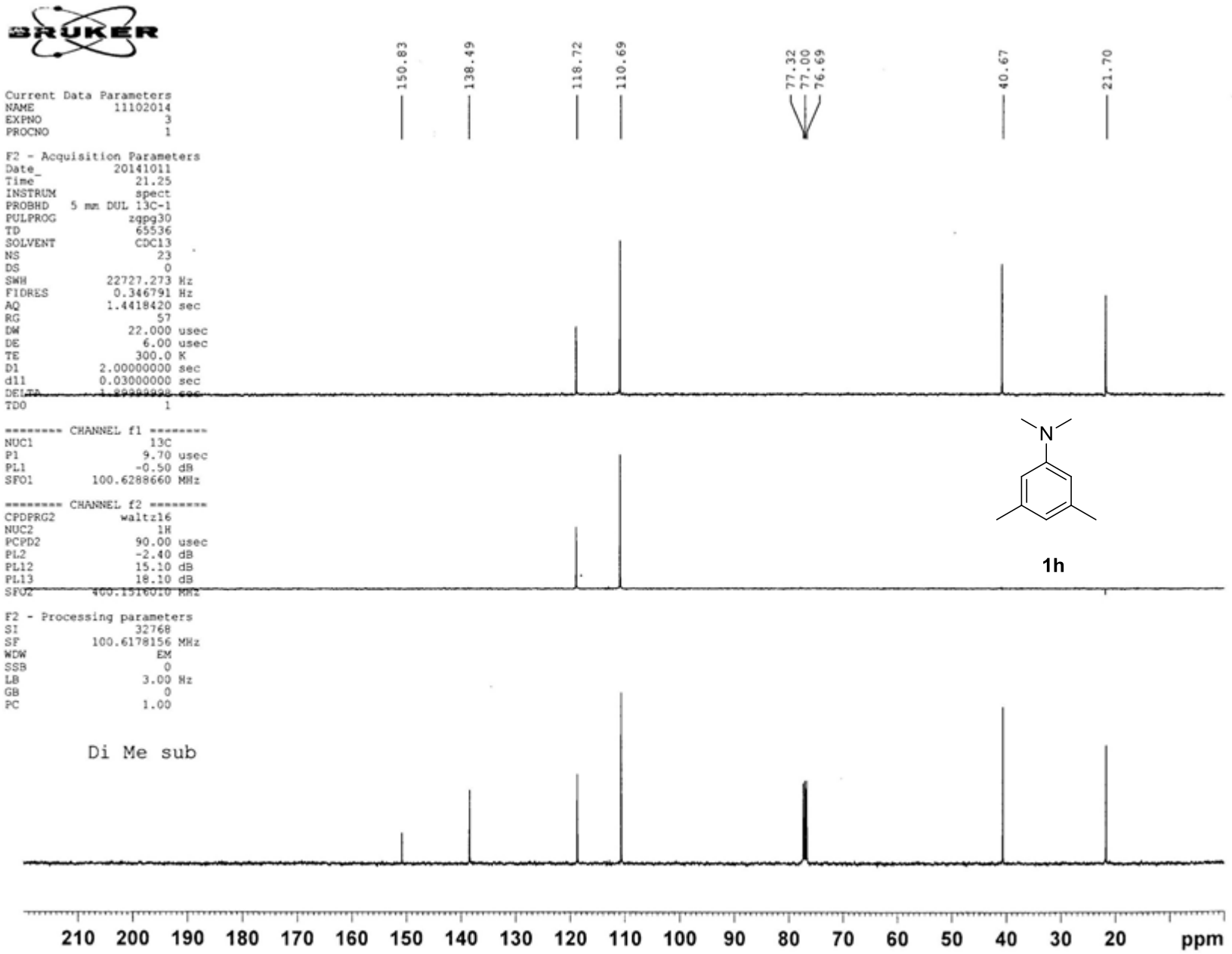

7.240
7.160
7.138
6.629
6.606
2.904
STANDARD PROTON PARAMETERS
expl PROTON
SAMPLE
data Jun 28 2014
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 2.556
np 32768
fb 4000
bs 4
d1 1.000
nt 100
ct 44
TRANSMITTER
tn H1
sfrq 399.765
tof 399.8
tpwr 60
pw 7.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_atb
dpwr 35
dmf 29412
PRESATURATION
saymode n
wet n
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 14.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
lb 0.50
DISPLY
sp -200.0
wp 3997.6
rf1 3701.6
rfp 2902.3
rp -5.7
lp 4.0
PLOT
wc 215
sc 25
vs 60
th 7
al cdc ph
N
Cl
1e
2.00
2.10
6.14
9
8
7
6
5
4
3
2
1
0 ppm

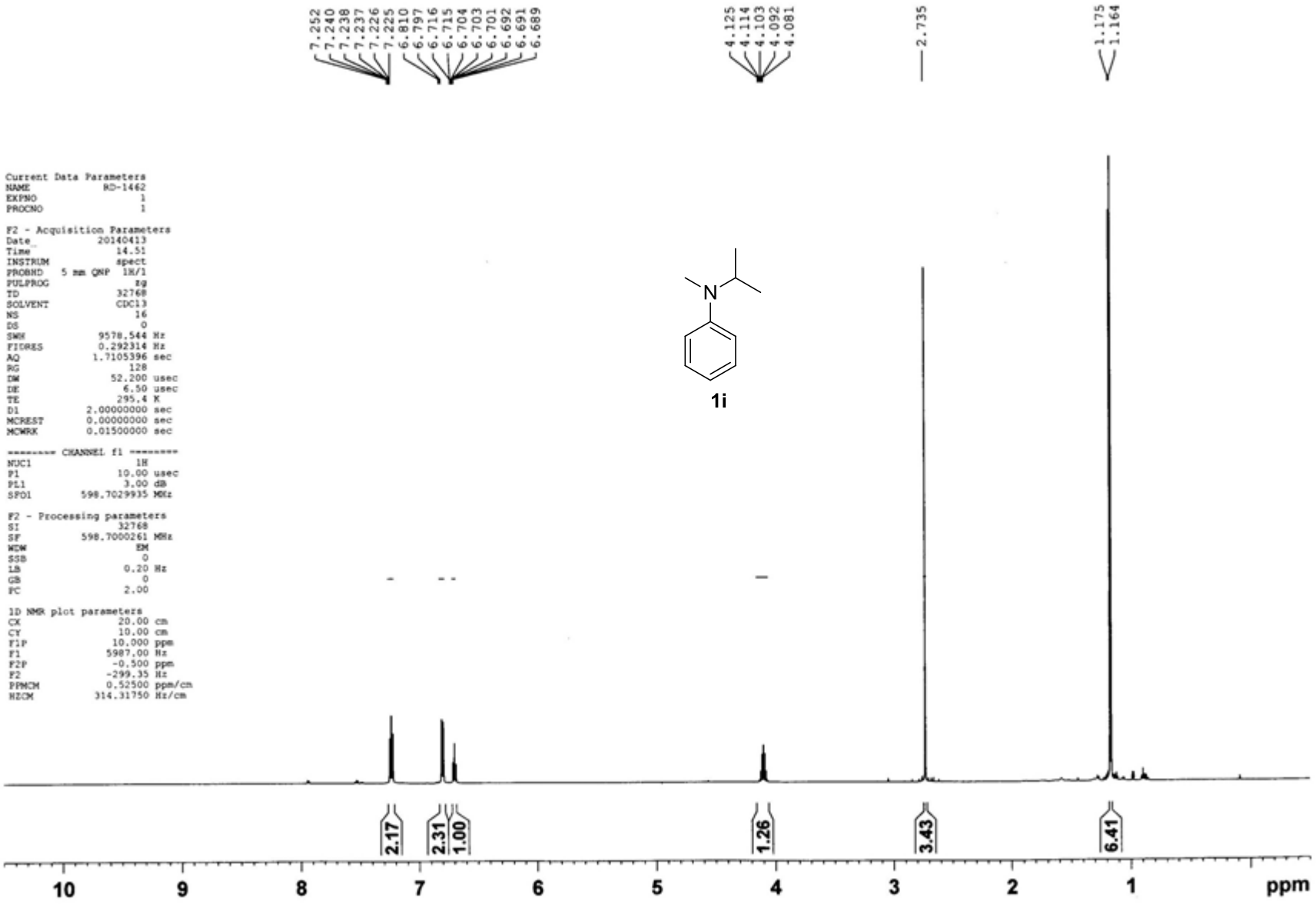

149.10
128.71
121.29
113.55
77.31
76.99
76.68
40.54
STANDARD CARBON PARAMETERS
exp2 CARBON
SAMPLE
data Jun 28 2014
solvent CD2Cl2
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 4
d1 1.000
nt 1000
ct 1000
TRANSMITTER
tn C13
sfrq 100.532
tof 1028.1
tpwr 56
pw 4.750
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 40
dmf 10267
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.0
fn 65536
DISPLY
sp -0.6
wp 22114.0
rfl 6483.5
rfp 4925.5
rp 130.5
lp -2.9
PLOT
wc 250
sc 0
vs 267039
th 68
ai cdc ph
N
Cl
1e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

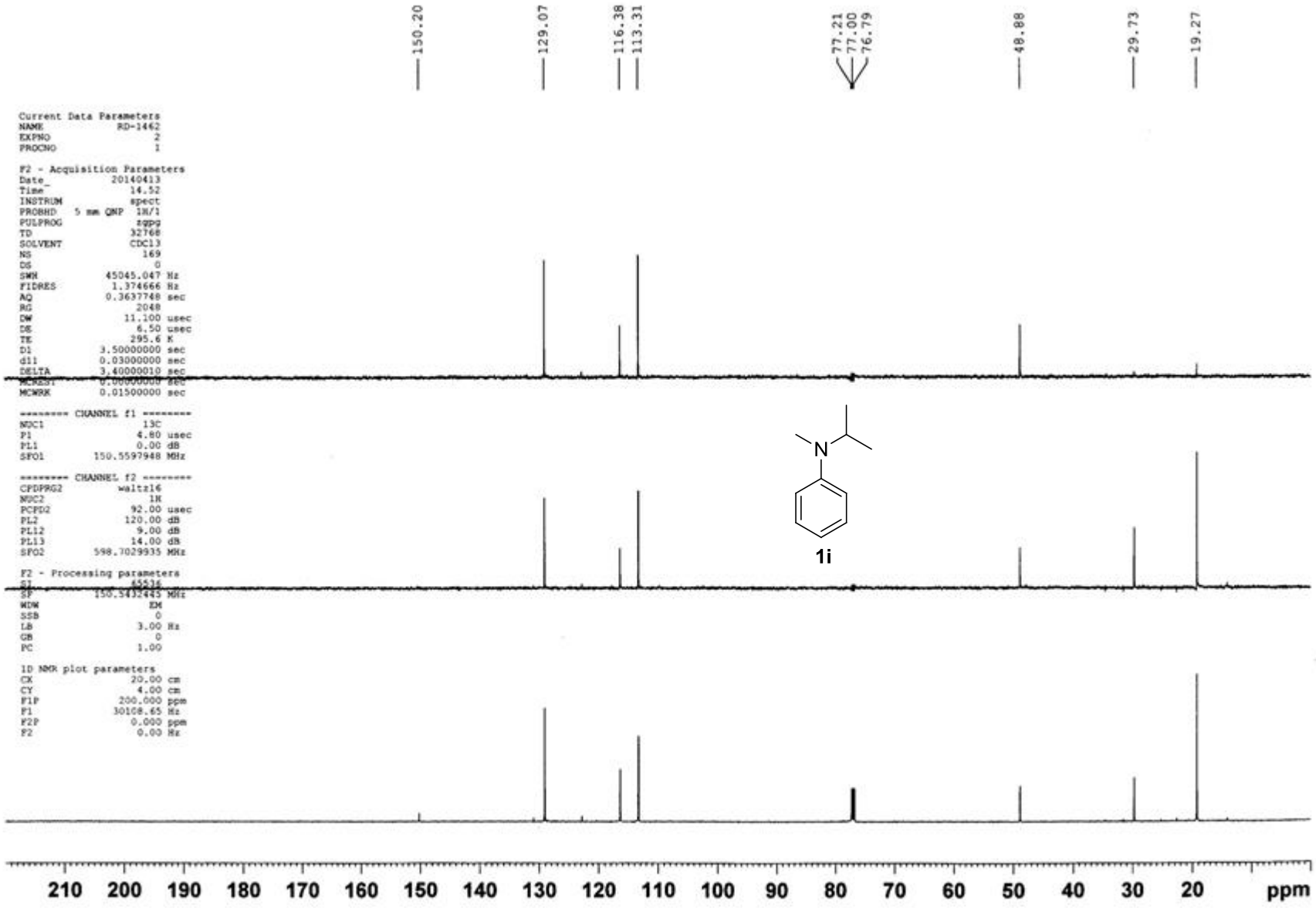

7.2406
7.1713
7.1615
7.1580
7.1539
7.1487
7.1426
7.1311
6.5928
6.5872
6.5679
2.9426
2.3398
Current Data Parameters
NAME 30092014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140930
Time 15.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 114
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-5- meta Me
N
1g
10
9
8
7
6
5
4
3
2
1
ppm
1.00
3.06
6.23
3.09

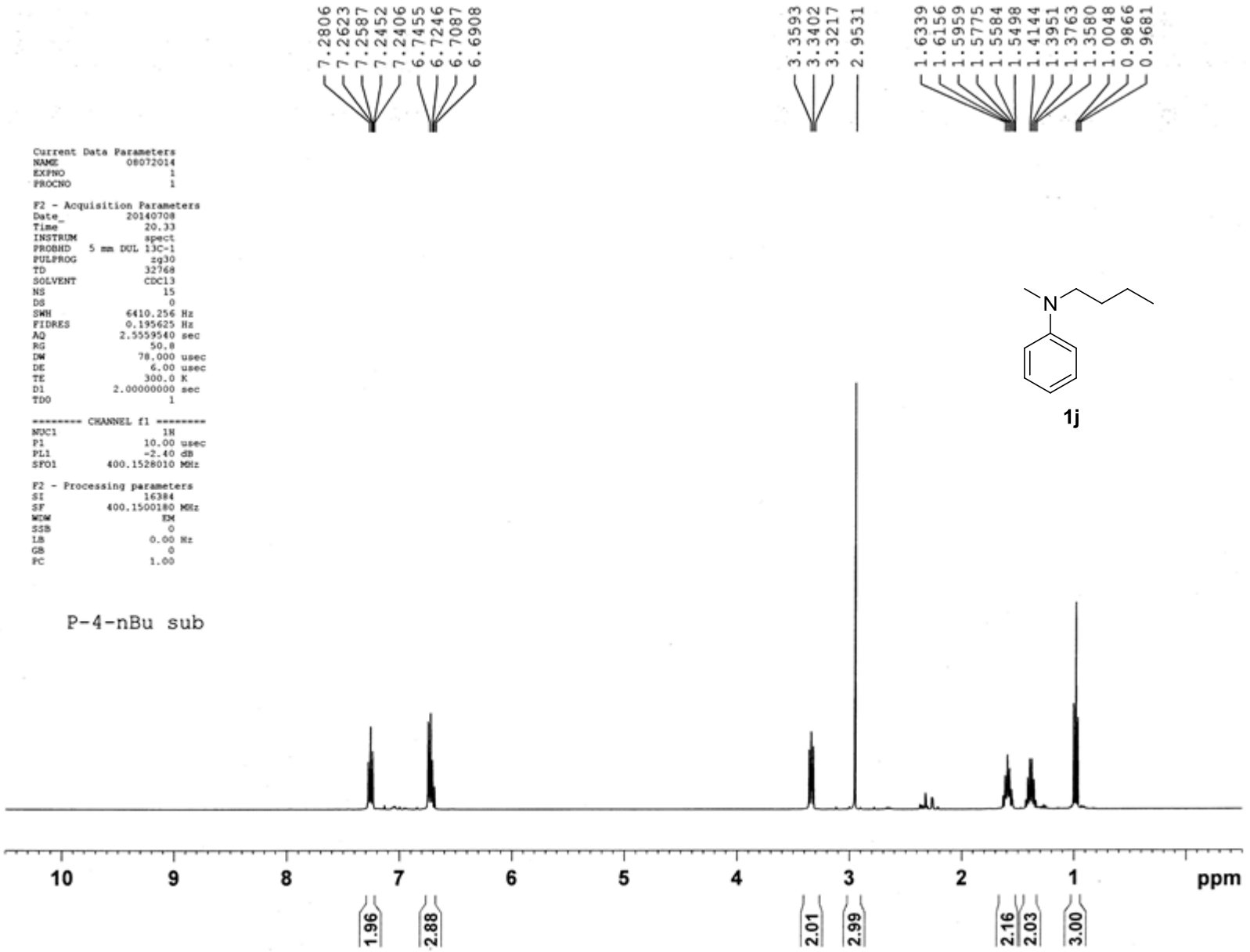

150.74
138.66
128.89
117.60
113.44
109.90
77.32
77.00
76.68
40.65
21.86
Current Data Parameters
NAME 30092014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140930
Time 23.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 90
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178069 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-5-96
N
1g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

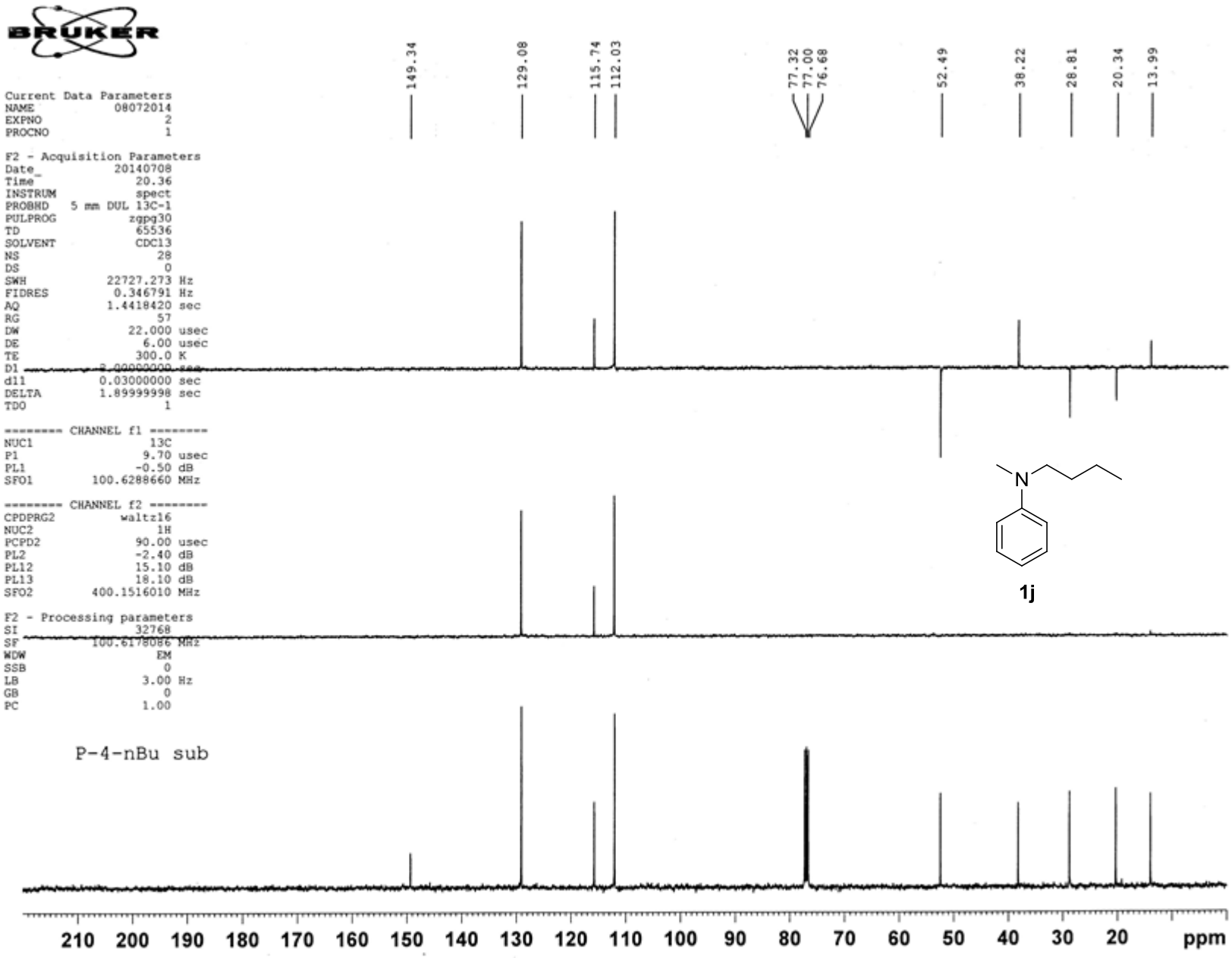

7.2404
6.4898
6.4784
2.9936
2.3729
BRUKER
Current Data Parameters
NAME 11102014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141011
Time 21.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 50.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Di Me sub
N
1h
10
9
8
7
6
5
4
3
2
1
ppm
3.00
6.04
6.07

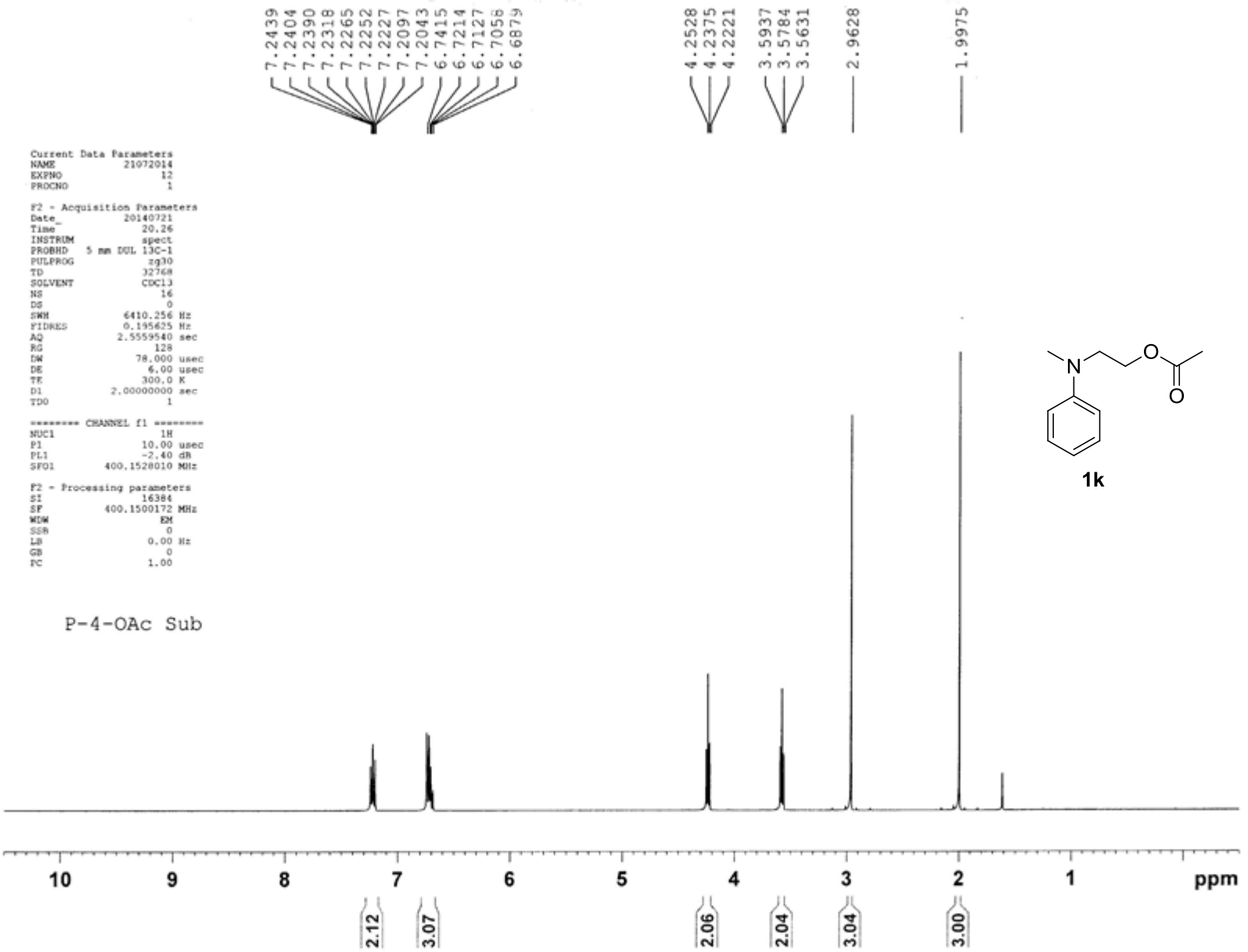

150.83
138.49
118.72
110.69
77.32
77.00
76.69
40.67
21.70
Current Data Parameters
NAME 11102014
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141011
Time 21.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zqpg30
TD 65536
SOLVENT CDCl3
NS 23
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
F2 - Processing parameters
SI 32768
SF 100.6178156 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
1h
Di Me sub
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

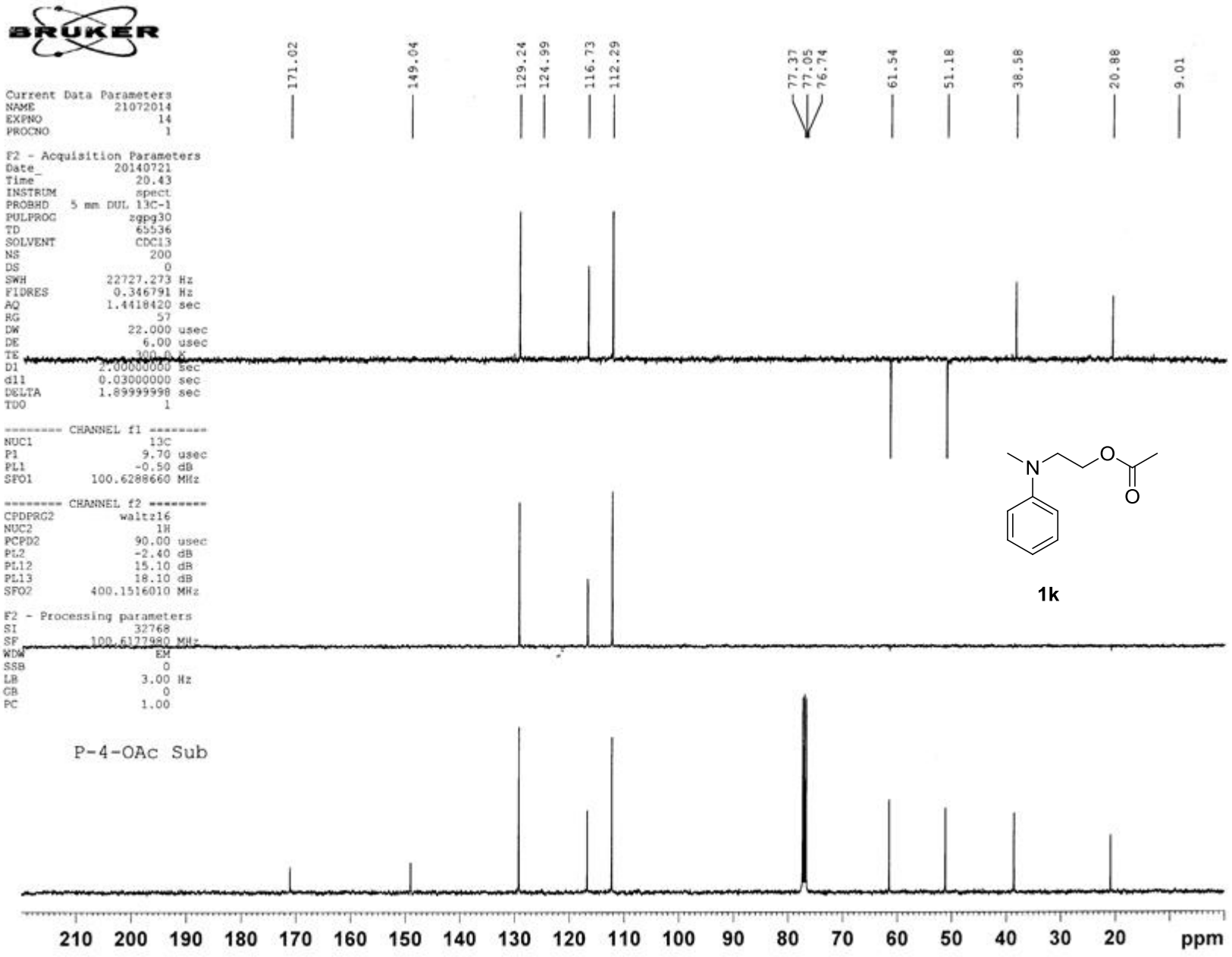

7.252
7.240
7.238
7.237
7.226
7.225
6.810
6.797
6.716
6.715
6.704
6.703
6.701
6.692
6.691
6.689
4.125
4.114
4.103
4.092
4.081
2.735
1.175
1.164
Current Data Parameters
NAME RD-1462
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140413
Time 14.51
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9578.544 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 128
DW 52.200 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 598.7029935 MHz
F2 - Processing parameters
SI 32768
SF 598.7000261 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 5987.00 Hz
F2P -0.500 ppm
F2 -299.35 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.31750 Hz/cm
N
1i
2.17
2.31
1.00
1.26
3.43
6.41
10
9
8
7
6
5
4
3
2
1
ppm

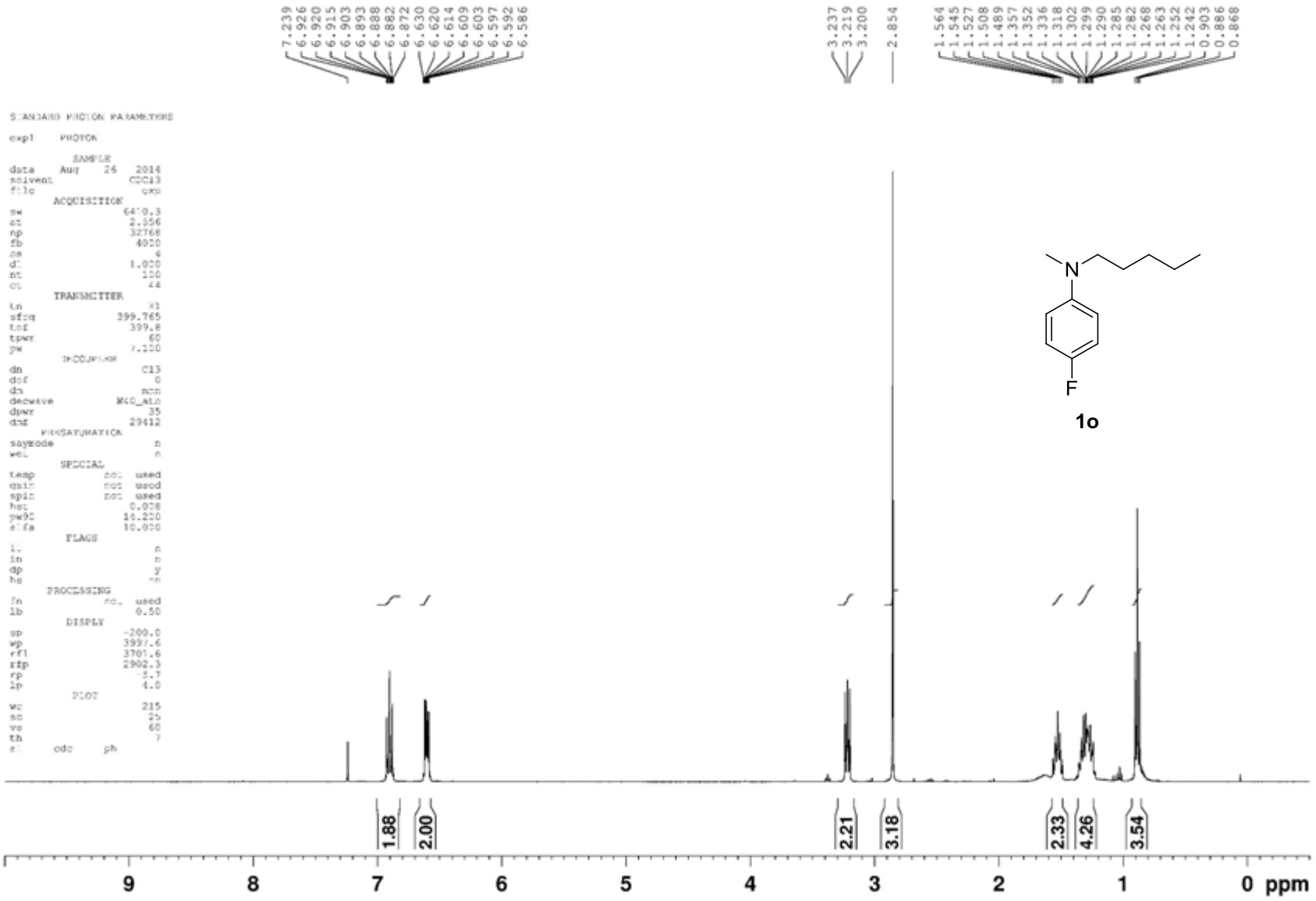

150.20
129.07
116.38
113.31
77.21
77.00
76.79
48.88
29.73
19.27
Current Data Parameters
NAME RD-1462
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140413
Time 14.52
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 169
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 295.6 K
D1 3.50000000 sec
d11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
-------- CHANNEL f1 --------
NUC1 13C
P1 4.80 usec
PL1 0.00 dB
SFO1 150.5597948 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 598.7029935 MHz
F2 - Processing parameters
SI 45516
SF 150.5432445 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.000 ppm
F1 30108.65 Hz
F2P 0.000 ppm
F2 0.00 Hz
N
1i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

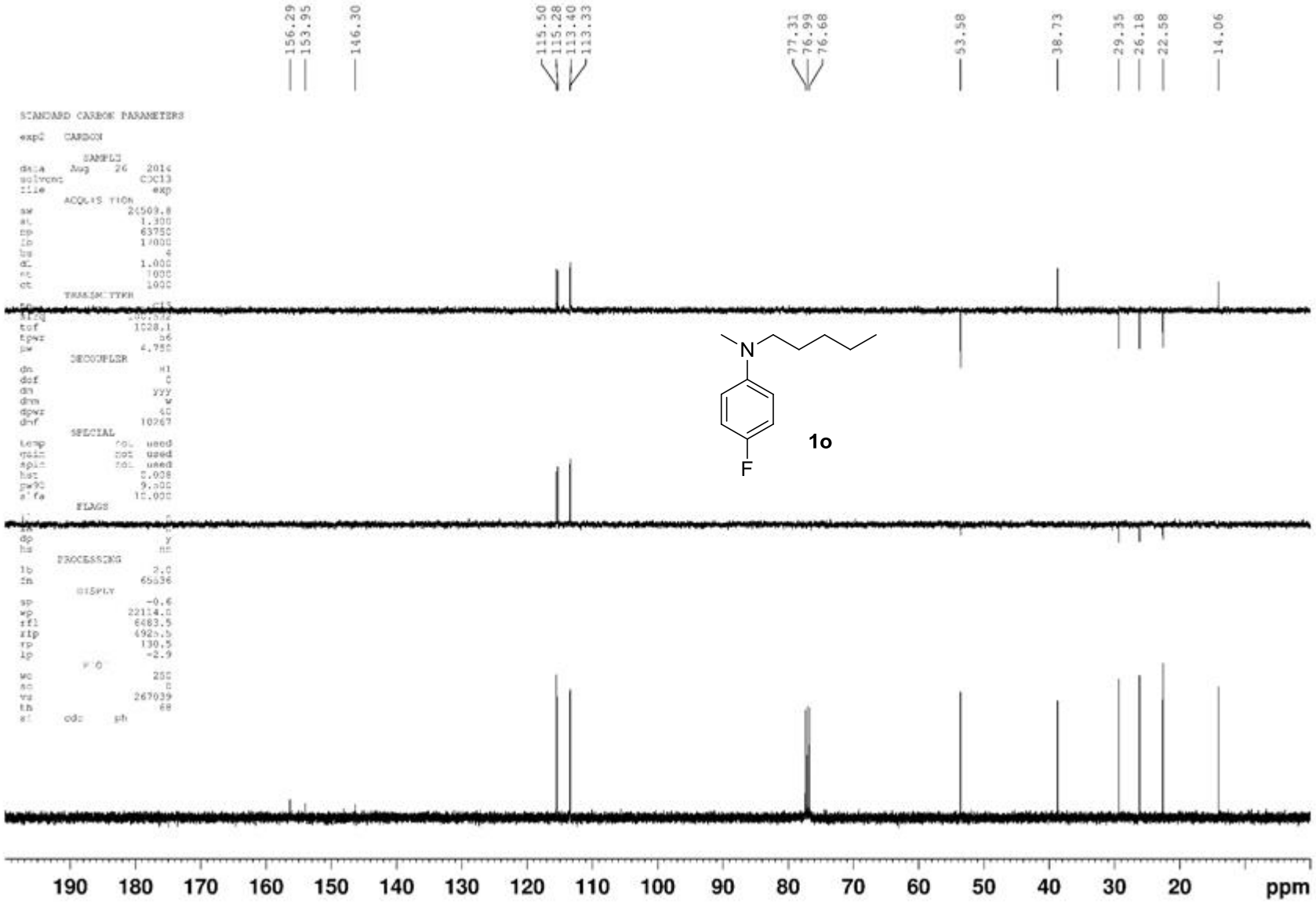

7.2806
7.2623
7.2587
7.2452
7.2406
6.7455
6.7246
6.7087
6.6908
3.3593
3.3402
3.3217
2.9531
1.6339
1.6156
1.5959
1.5775
1.5584
1.5498
1.4144
1.3951
1.3763
1.3580
1.0048
0.9866
0.9681
Current Data Parameters
NAME 08072014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140708
Time_ 20.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 15
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 50.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500180 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-4-nBu sub
N
1j
10
9
8
7
6
5
4
3
2
1
ppm
1.96
2.88
2.01
2.99
2.16
2.03
3.00

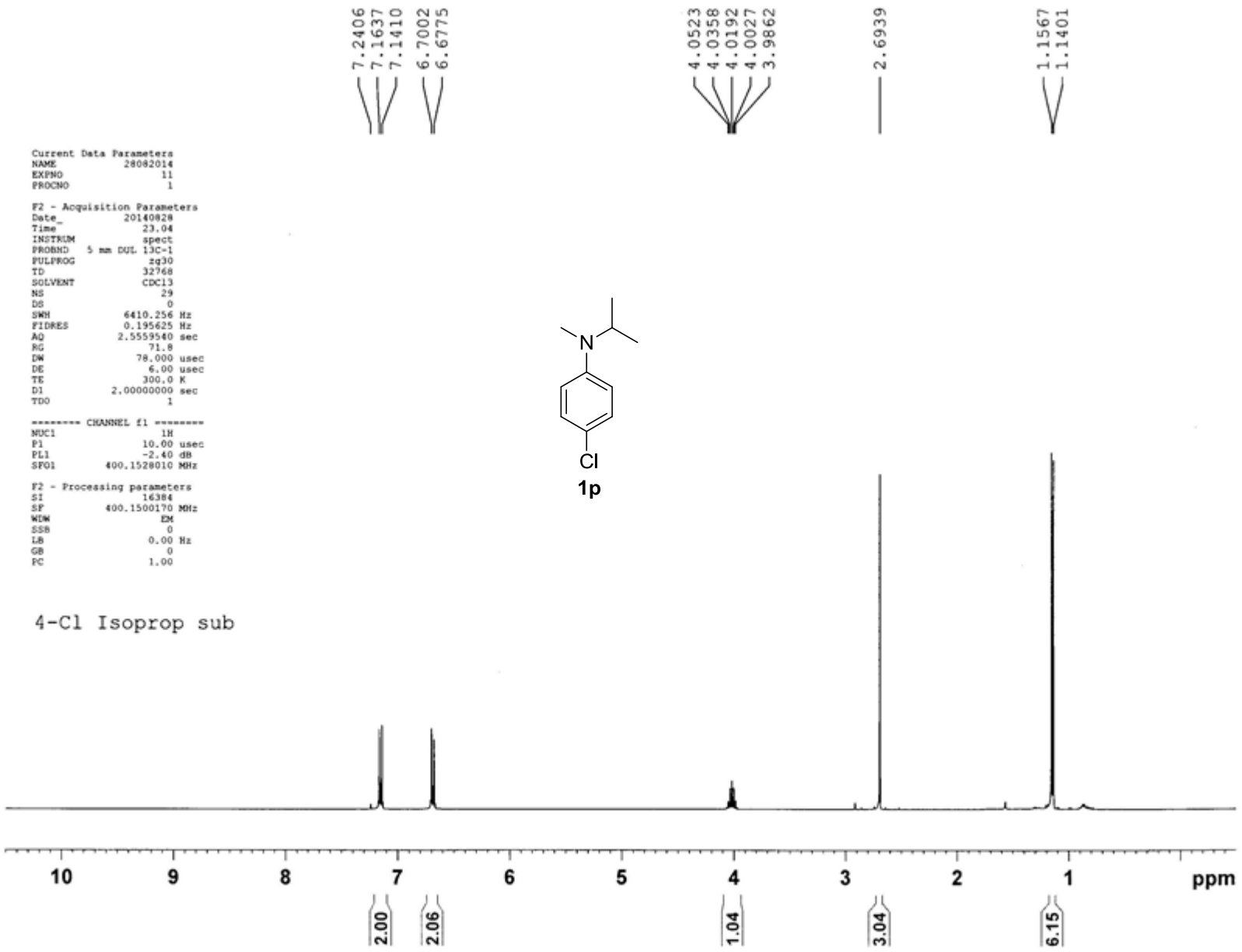

149.34
129.08
115.74
112.03
77.32
77.00
76.68
52.49
38.22
28.81
20.34
13.99
Current Data Parameters
NAME 08072014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140708
Time 20.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 28
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178086 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-4-nBu sub
N
1j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

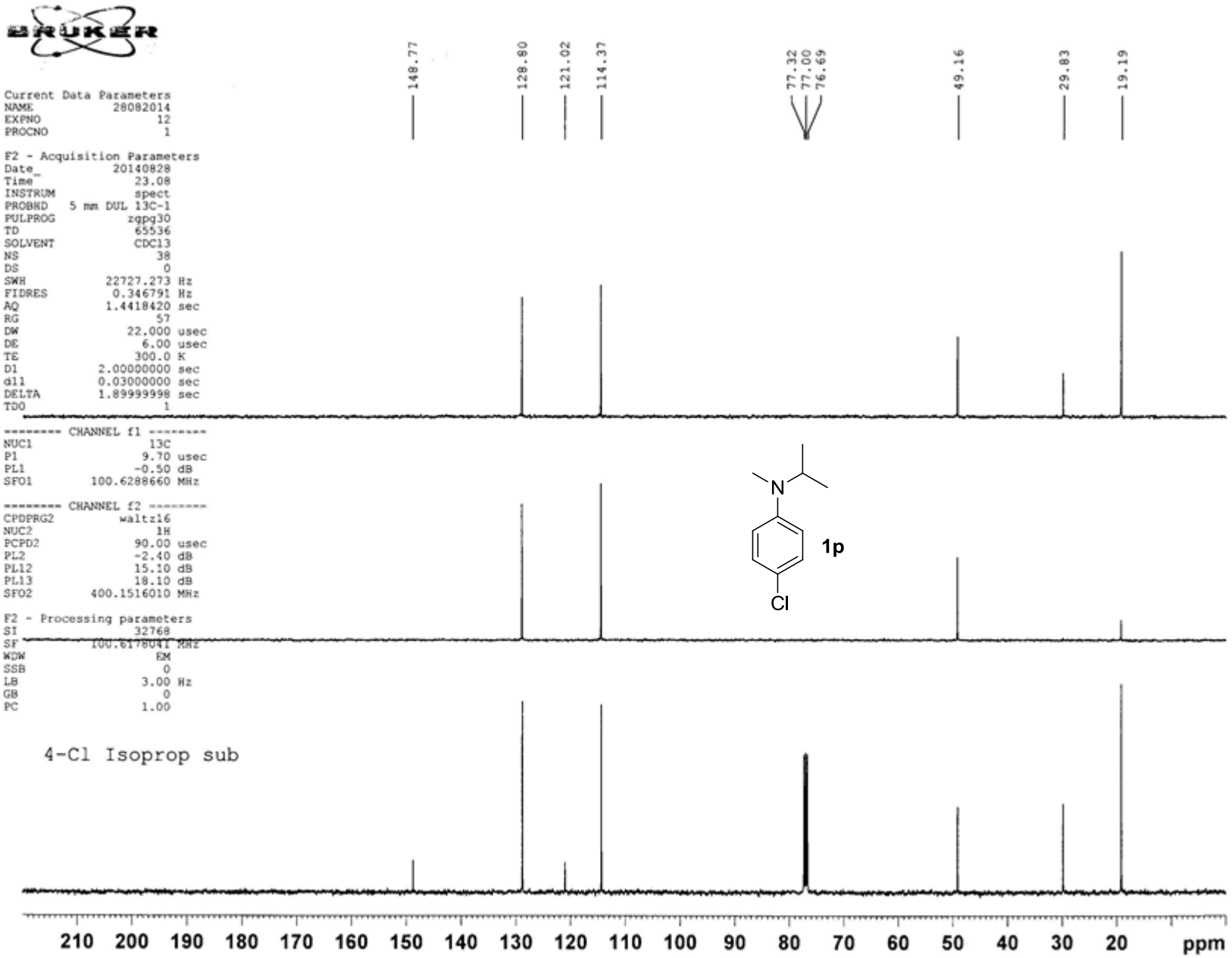

7.2439
7.2404
7.2390
7.2318
7.2265
7.2252
7.2227
7.2097
7.2043
6.7415
6.7214
6.7127
6.7056
6.6879
4.2528
4.2375
4.2221
3.5937
3.5784
3.5631
2.9628
1.9975
Current Data Parameters
NAME 21072014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140721
Time 20.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-4-OAc Sub
N
O
O
1k
10
9
8
7
6
5
4
3
2
1
ppm
2.12
3.07
2.06
2.04
3.04
3.00

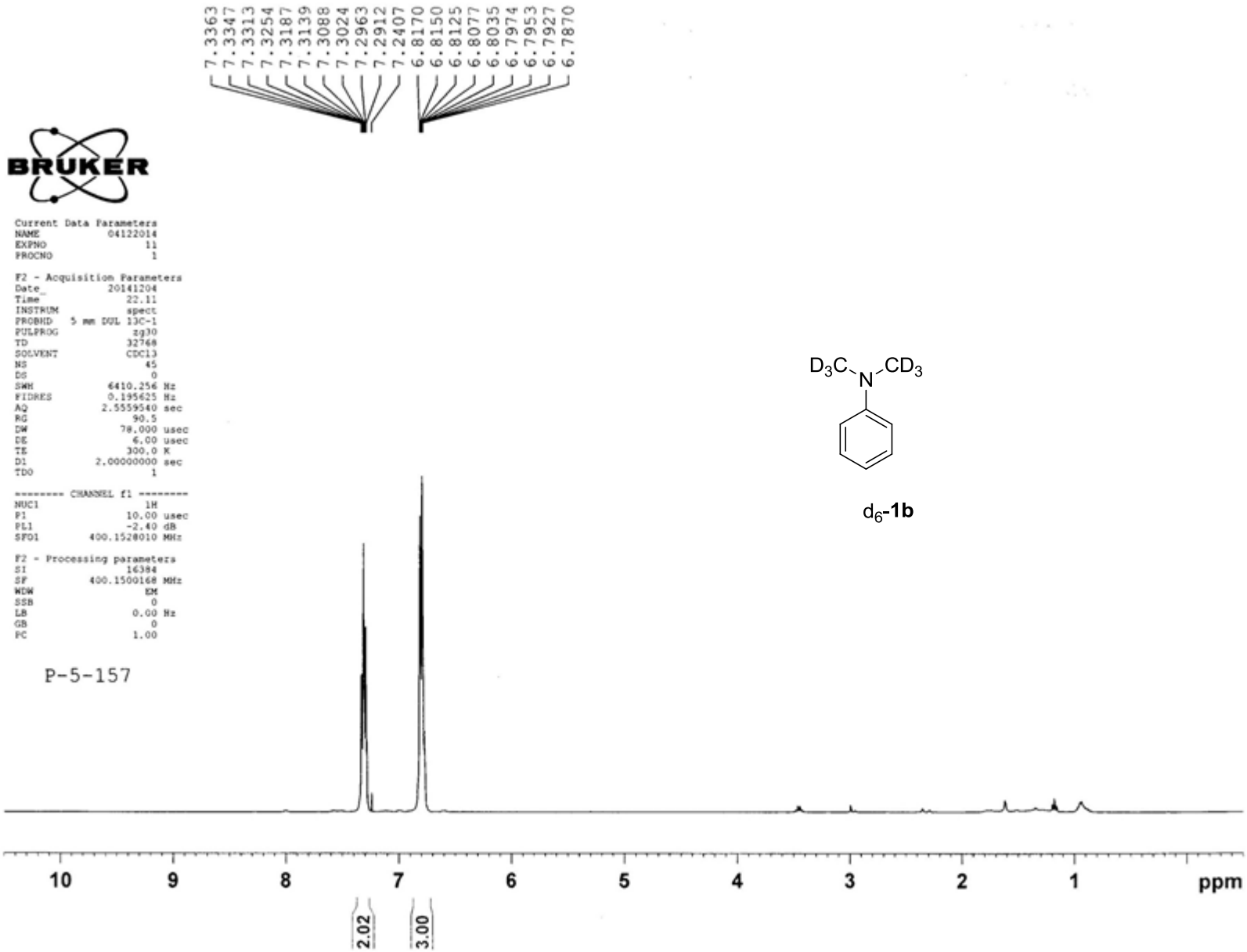

171.02
149.04
129.24
124.99
116.73
112.29
77.37
77.05
76.74
61.54
51.18
38.58
20.88
9.01
Current Data Parameters
NAME 21072014
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140721
Time 20.43
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6127980 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-4-OAc Sub
N
O
O
1k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

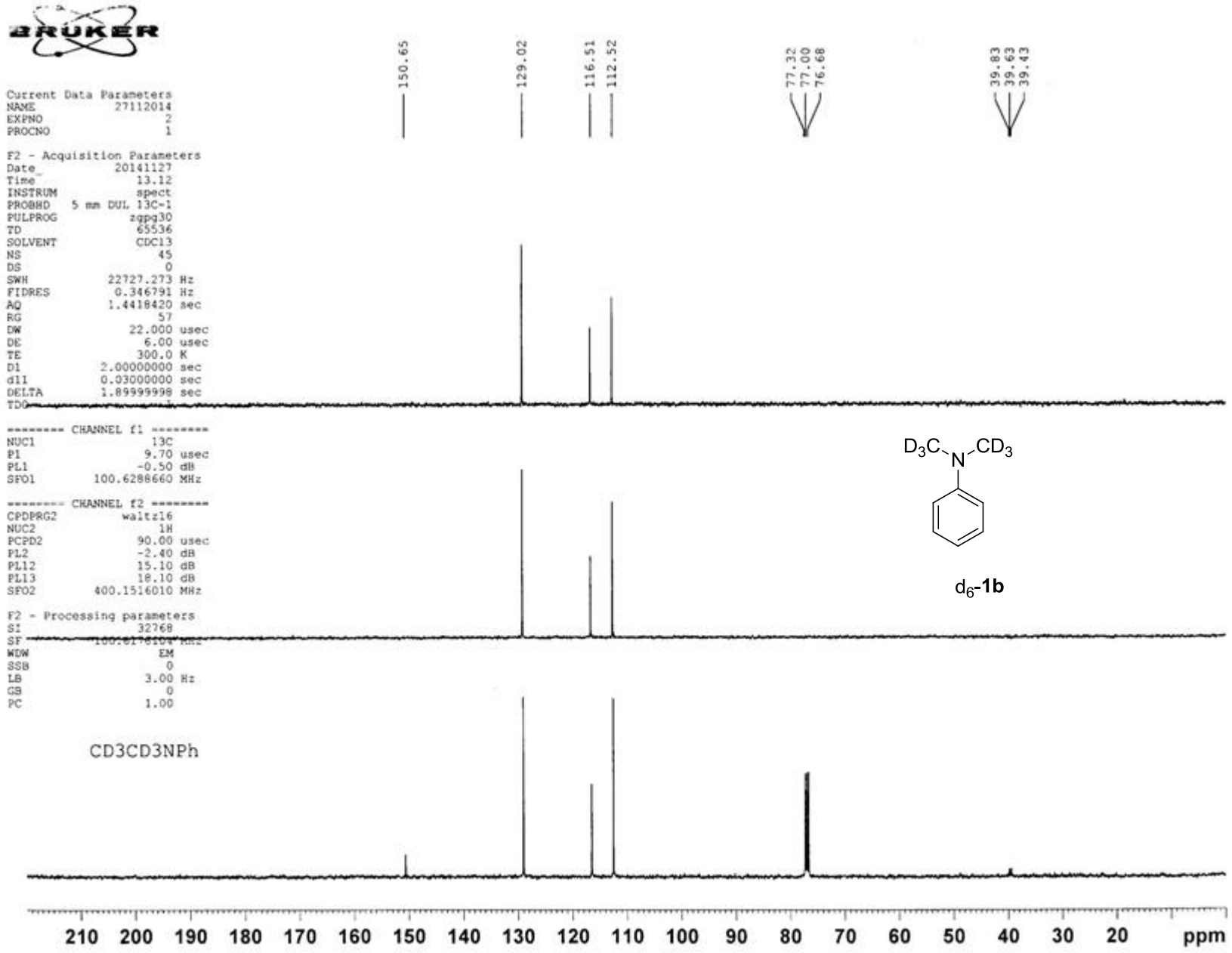

7.239
6.926
6.920
6.915
6.903
6.893
6.888
6.882
6.872
6.630
6.620
6.614
6.609
6.603
6.597
6.592
6.586
3.237
3.219
3.200
2.854
1.564
1.545
1.527
1.508
1.489
1.357
1.352
1.336
1.318
1.302
1.299
1.290
1.285
1.282
1.268
1.263
1.252
1.242
0.903
0.886
0.868
N
F
1o
1.88
2.00
2.21
3.18
2.33
4.26
3.54
9
8
7
6
5
4
3
2
1
0 ppm

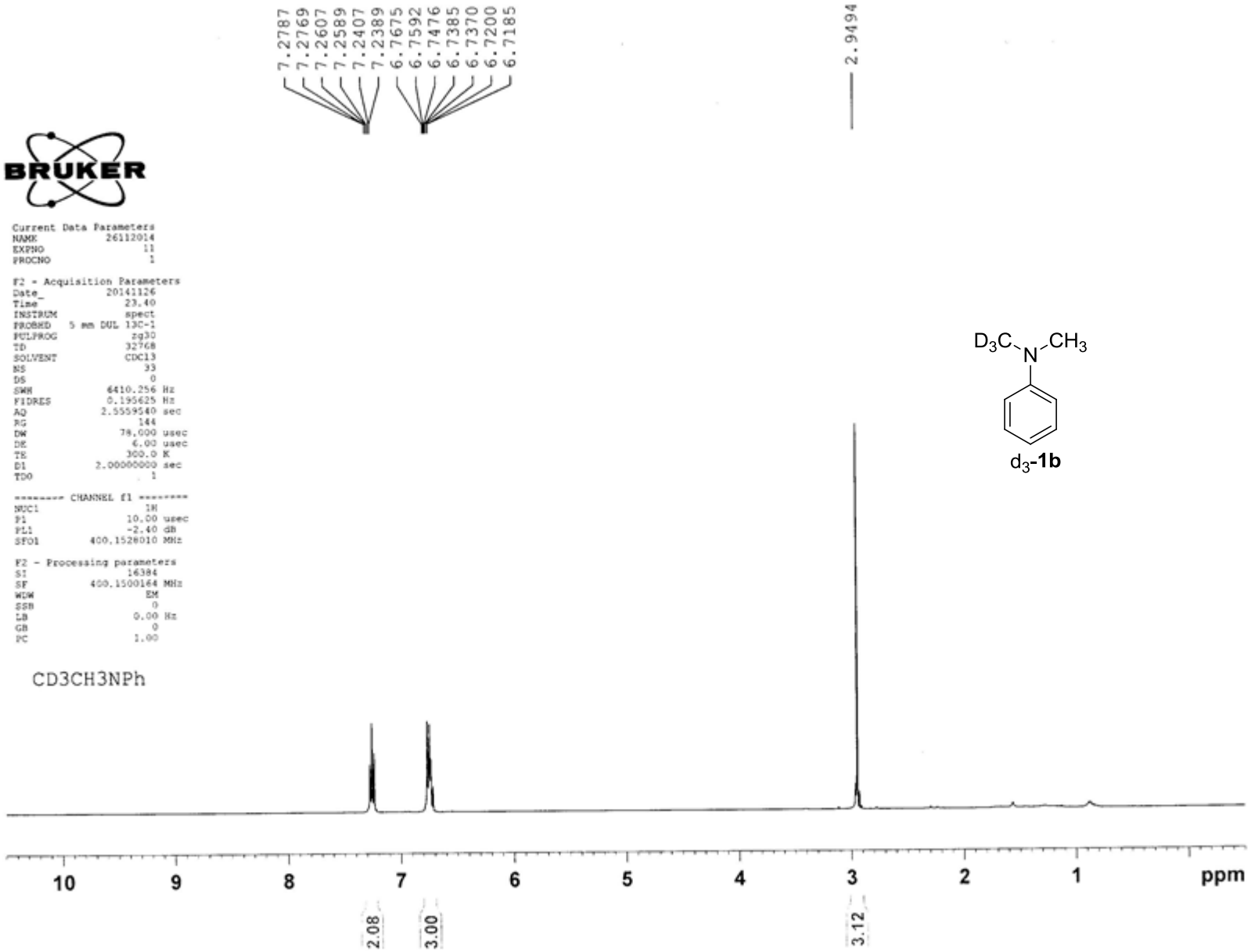

156.29
153.95
146.30
115.50
115.28
113.40
113.33
77.31
76.99
76.68
53.58
38.73
29.35
26.18
22.58
14.06
N
F
1o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

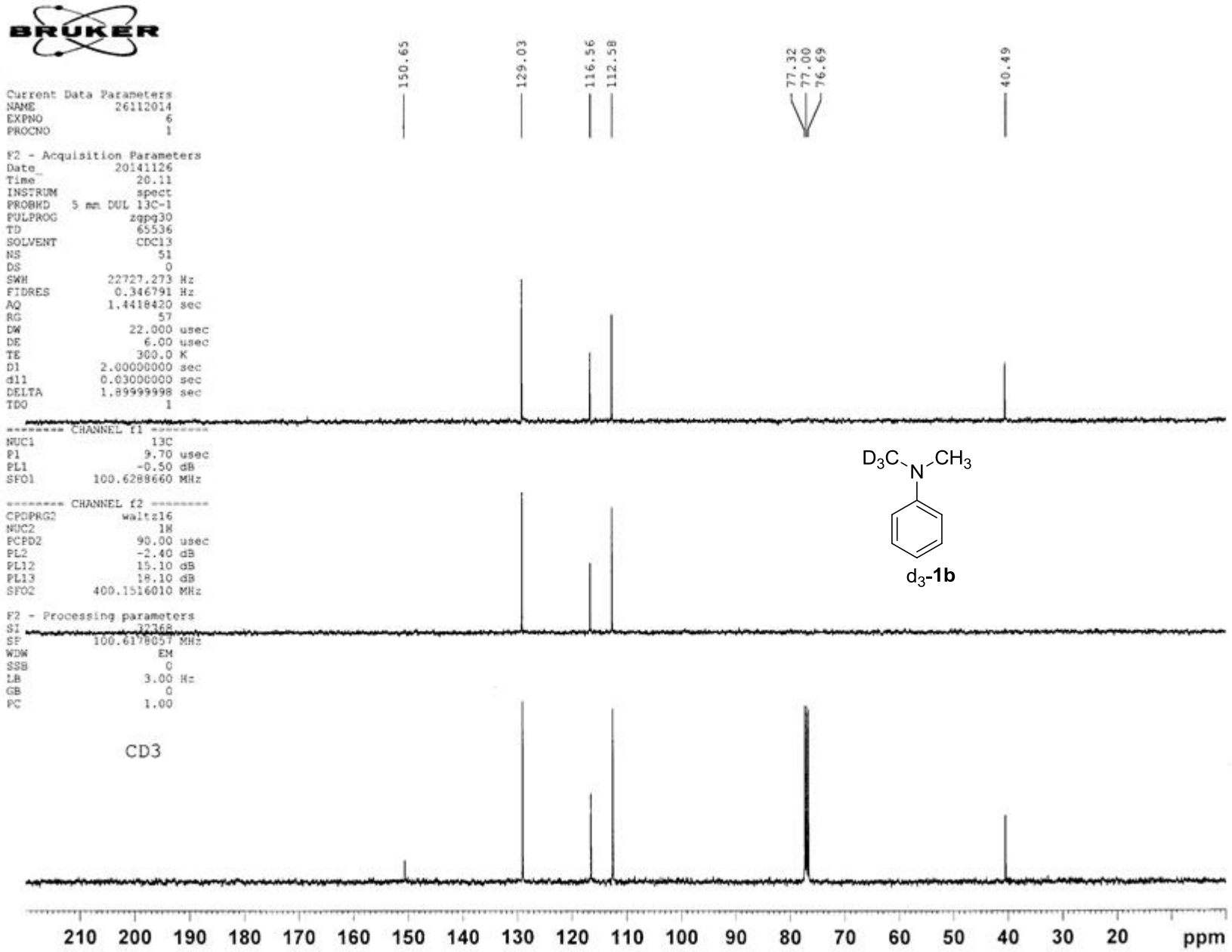

7.2406
7.1637
7.1410
6.7002
6.6775
4.0523
4.0358
4.0192
4.0027
3.9862
2.6939
1.1567
1.1401
Current Data Parameters
NAME 28082014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140828
Time 23.04
INSTRUM spect
PROBND 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 29
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 71.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500170 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
N
Cl
1p
4-Cl Isoprop sub
10
9
8
7
6
5
4
3
2
1
ppm
2.00
2.06
1.04
3.04
6.15

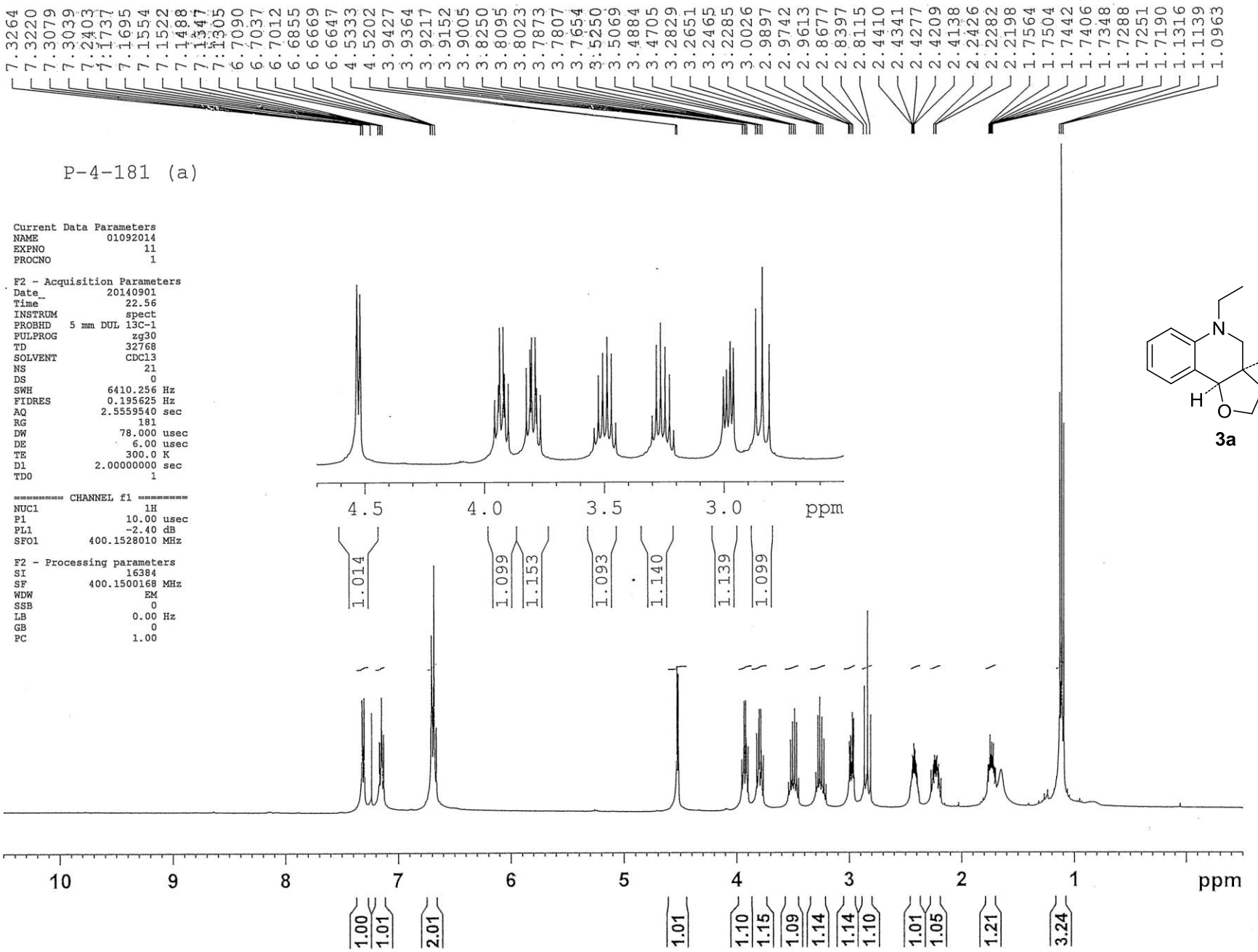

148.77
128.80
121.02
114.37
77.32
77.00
76.69
49.16
29.83
19.19
Current Data Parameters
NAME 28082014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140828
Time 23.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 38
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178041 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
Cl
1p
4-Cl Isoprop sub
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

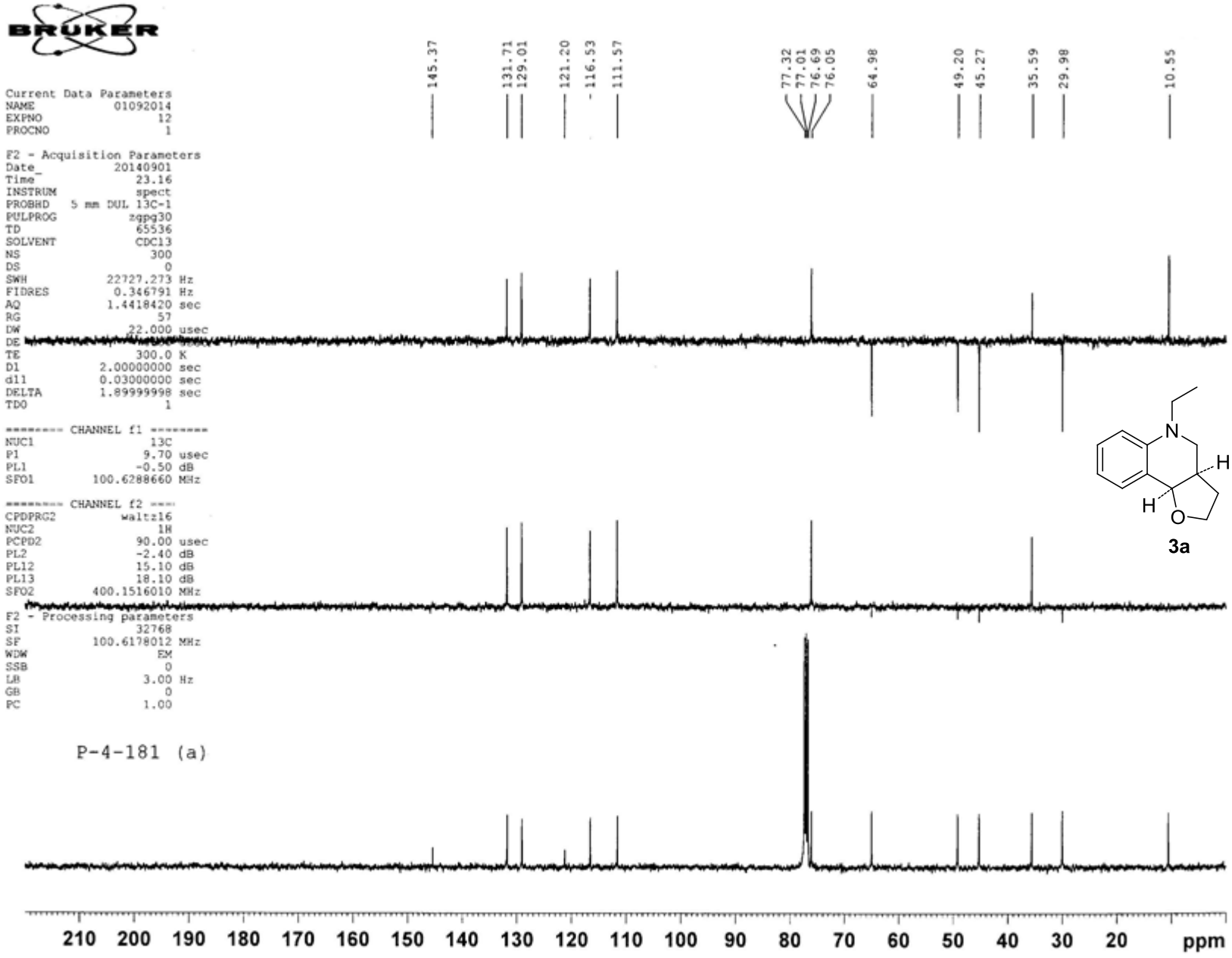

7.3363
7.3347
7.3313
7.3254
7.3187
7.3139
7.3088
7.3024
7.2963
7.2912
7.2407
6.8170
6.8150
6.8125
6.8077
6.8035
6.7974
6.7953
6.7927
6.7870
BRUKER
Current Data Parameters
NAME 04122014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141204
Time 22.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 45
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-5-157
D3C
N
CD3
d6-1b
10
9
8
7
6
5
4
3
2
1
ppm
2.02
3.00

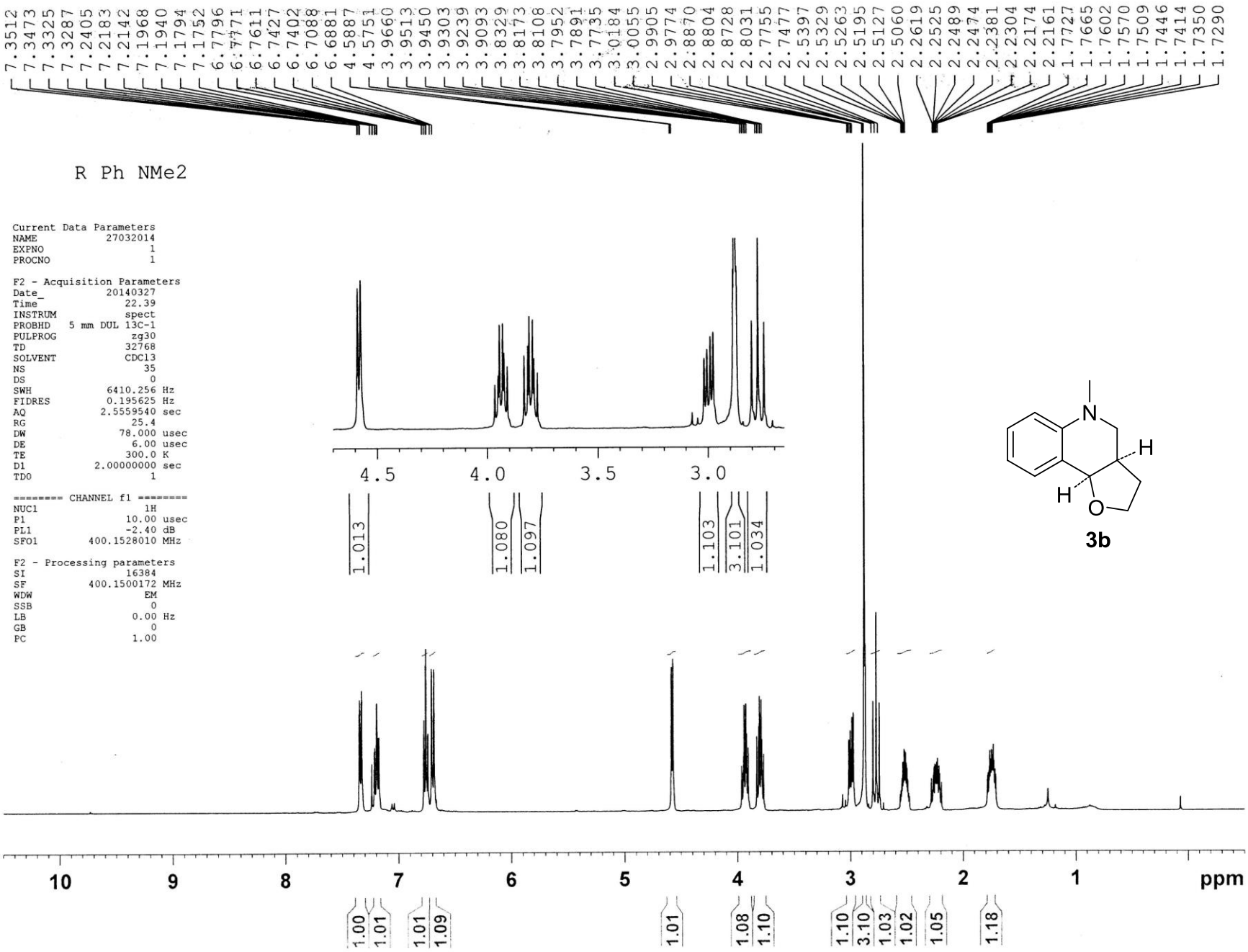

150.65
129.02
116.51
112.52
77.32
77.00
76.68
39.83
39.63
39.43
Current Data Parameters
NAME 27112014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141127
Time 13.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 45
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6278104 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
CD3CD3NPh
D3C N CD3
d6-1b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

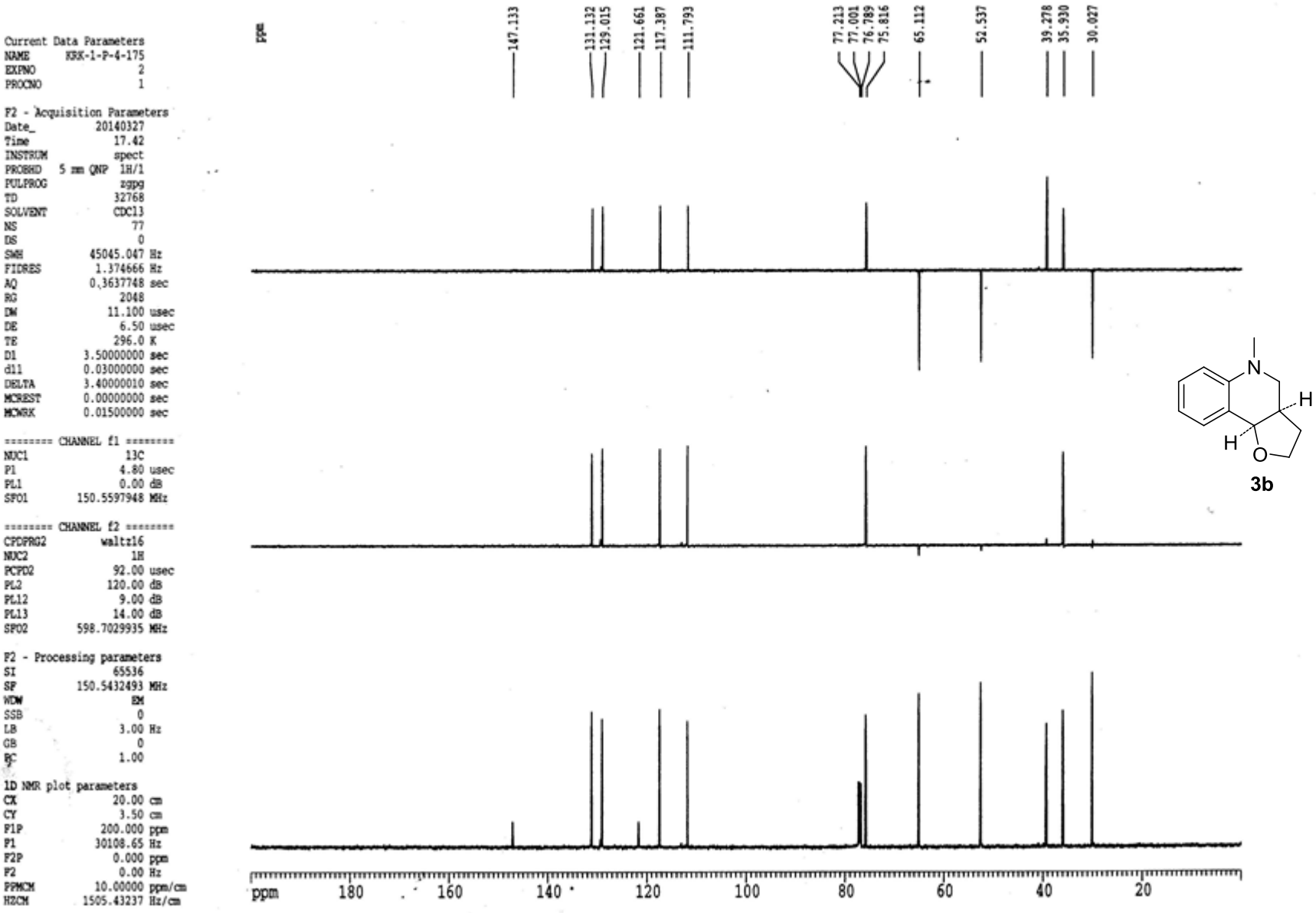

7.2787
7.2769
7.2607
7.2589
7.2407
7.2389
6.7675
6.7592
6.7476
6.7385
6.7370
6.7200
6.7185
2.9494
BRUKER
Current Data Parameters
NAME 26112014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141126
Time 23.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 33
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 144
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500164 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
CD3CH3NPh
D3C N CH3
d3-1b
10
9
8
7
6
5
4
3
2
1
ppm
2.08
3.00
3.12

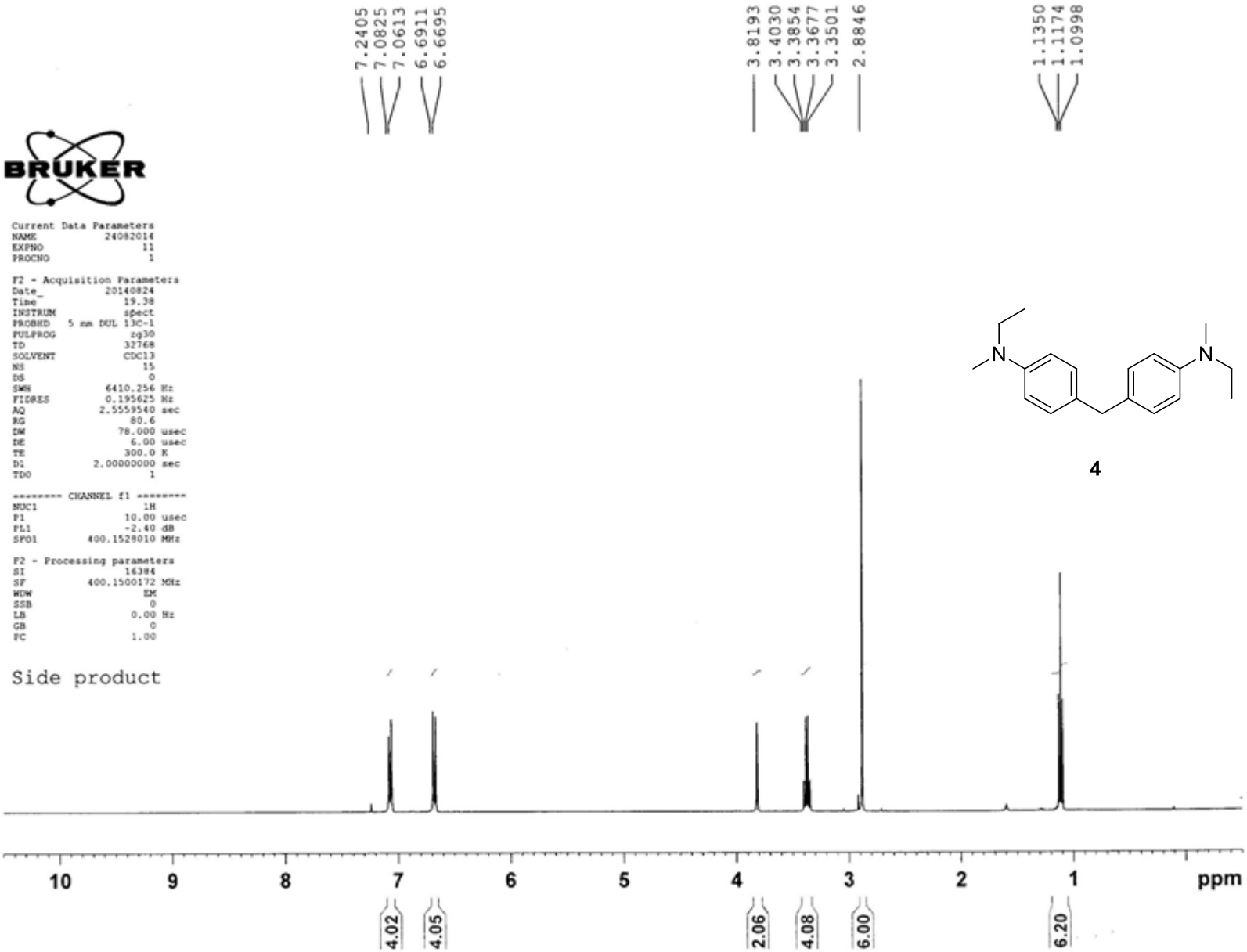

BRUKER
150.65
129.03
116.56
112.58
77.32
77.00
76.69
40.49
Current Data Parameters
NAME 26112014
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141126
Time 20.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178057 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
CD3
D3C N CH3
d3-1b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

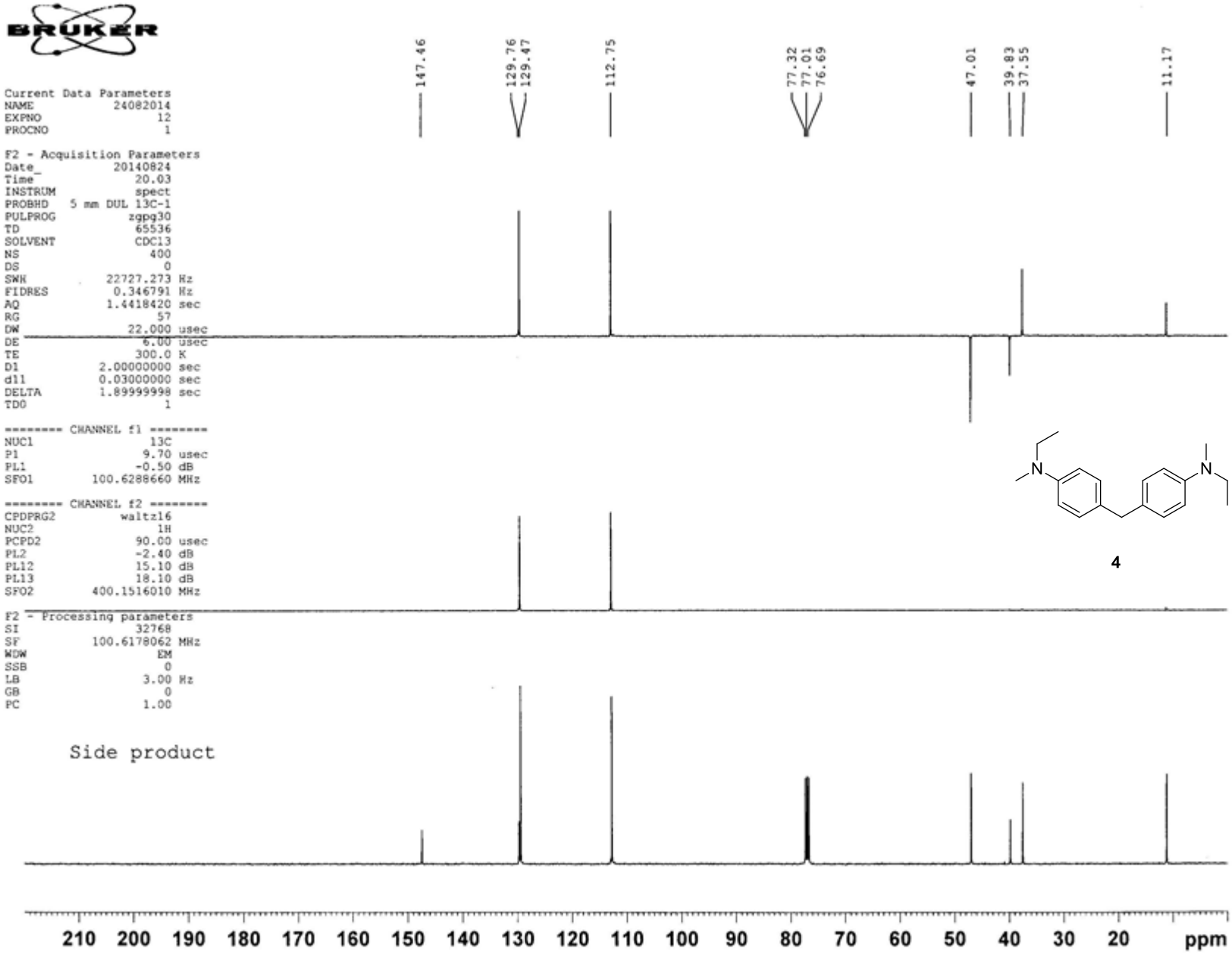

7.3264
7.3220
7.3079
7.3039
7.2403
7.1737
7.1695
7.1554
7.1522
7.1488
7.1347
7.1305
6.7090
6.7037
6.7012
6.6855
6.6669
6.6647
4.5333
4.5202
3.9427
3.9364
3.9217
3.9152
3.9005
3.8250
3.8095
3.8023
3.7873
3.7807
3.7654
3.5250
3.5069
3.4884
3.4705
3.2829
3.2651
3.2465
3.2285
3.0026
2.9897
2.9742
2.9613
2.8677
2.8397
2.8115
2.4410
2.4341
2.4277
2.4209
2.4138
2.2426
2.2282
2.2198
1.7564
1.7504
1.7442
1.7406
1.7348
1.7288
1.7251
1.7190
1.1316
1.1139
1.0963
P-4-181 (a)
Current Data Parameters
NAME 01092014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140901
Time 22.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 21
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 181
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.5
4.0
3.5
3.0
ppm
1.014
1.099
1.153
1.093
1.140
1.139
1.099
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
2.01
1.01
1.10
1.15
1.09
1.14
1.14
1.10
1.01
1.05
1.21
3.24
N
H
H
O
3a

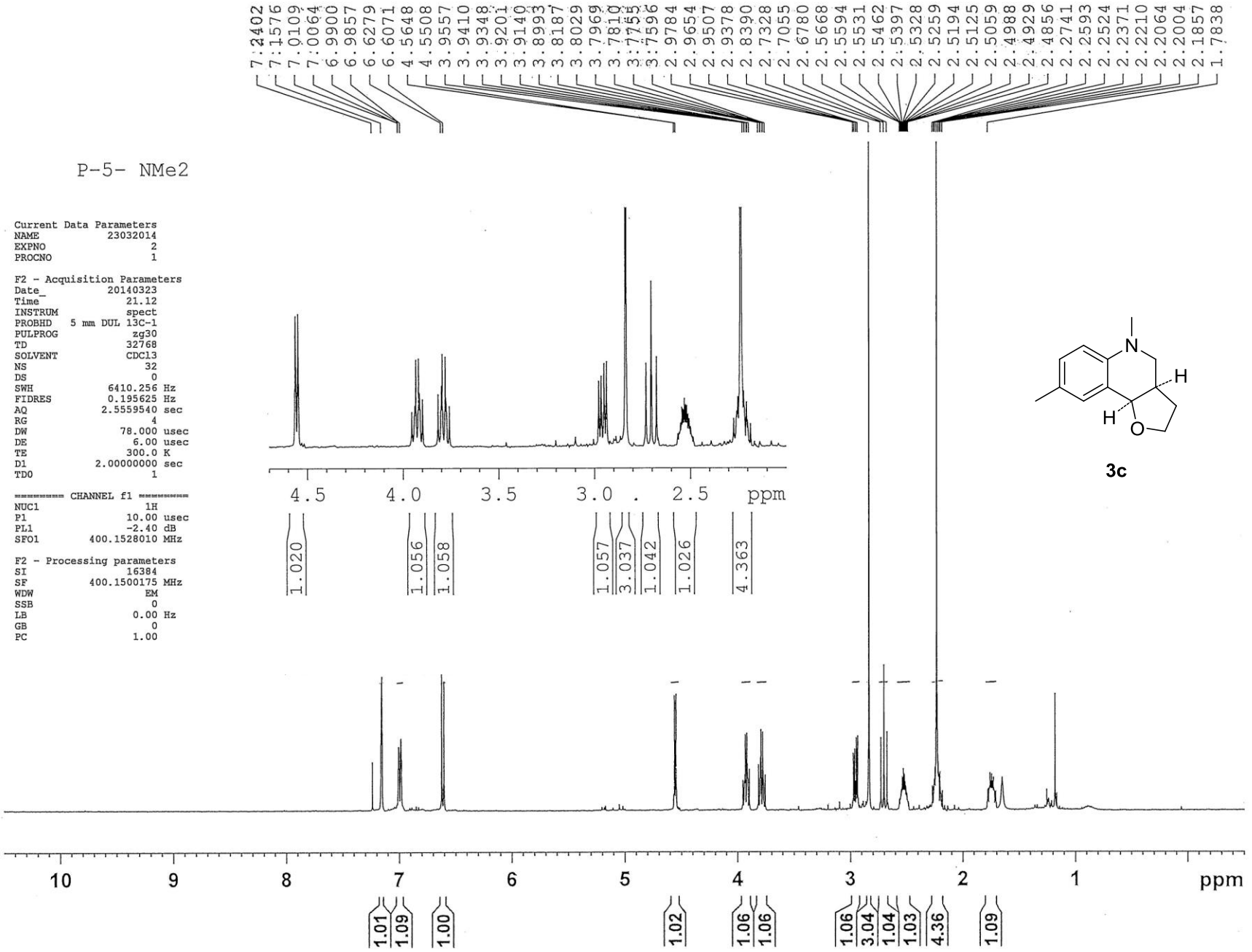

BRUKER
Current Data Parameters
NAME 01092014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140901
Time 23.16
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178012 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-4-181 (a)
145.37
131.71
129.01
121.20
116.53
111.57
77.32
77.01
76.69
76.05
64.98
49.20
45.27
35.59
29.98
10.55
N
H
H
O
3a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

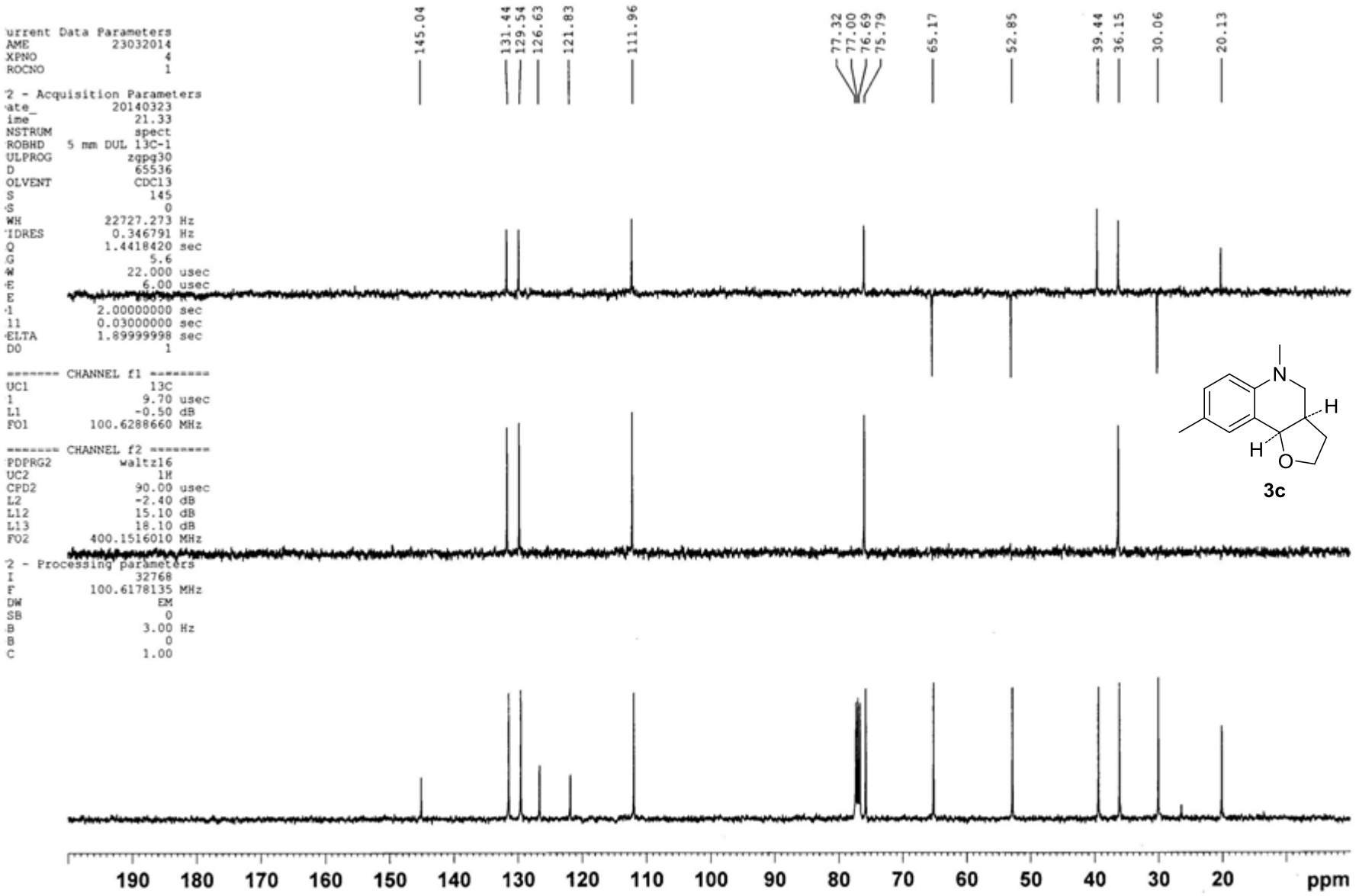

R Ph NMe2
Current Data Parameters
NAME 27032014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140327
Time 22.39
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 35
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 25.4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.3512 7.3473 7.3325 7.3287 7.2405 7.2183 7.2142 7.1968 7.1940 7.1794 7.1752 6.7796 6.7771 6.7611 6.7427 6.7402 6.7088 6.6881 4.5887 4.5751 3.9660 3.9513 3.9450 3.9303 3.9239 3.9093 3.8329 3.8173 3.8108 3.7952 3.7891 3.7735 3.0184 3.0055 2.9905 2.9774 2.8870 2.8804 2.8728 2.8031 2.7755 2.7477 2.5397 2.5329 2.5263 2.5195 2.5127 2.5060 2.2619 2.2525 2.2489 2.2474 2.2381 2.2304 2.2174 2.2161 1.7727 1.7665 1.7602 1.7570 1.7509 1.7446 1.7414 1.7350 1.7290
4.5 4.0 3.5 3.0
1.013 1.080 1.097 1.103 3.101 1.034
10 9 8 7 6 5 4 3 2 1 ppm
1.00 1.01 1.01 1.09 1.01 1.08 1.10 1.10 3.10 1.03 1.02 1.05 1.18
N H H O
3b


Current Data Parameters
NAME KSK-1-P-4-175
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140327
Time 17.42
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 77
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 296.0 K
D1 3.50000000 sec
d11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.80 usec
PL1 0.00 dB
SFO1 150.5597948 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 598.7029935 MHz
F2 - Processing parameters
SI 65536
SF 150.5432493 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 3.50 cm
F1P 200.000 ppm
F1 30108.65 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1505.43237 Hz/cm
ppm
147.133
131.132
129.015
121.661
117.387
111.793
77.213
77.001
76.789
75.816
65.112
52.537
39.278
35.930
30.027
N
H
H
O
3b
ppm
180
160
140
120
100
80
60
40
20


7.2405
7.0825
7.0613
6.6911
6.6695
3.8193
3.4030
3.3854
3.3677
3.3501
2.8846
1.1350
1.1174
1.0998
BRUKER
Current Data Parameters
NAME 24082014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140824
Time 19.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 15
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 80.6
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Side product
N
N
4
10
9
8
7
6
5
4
3
2
1
ppm
4.02
4.05
2.06
4.08
6.00
6.20


147.46
129.76
129.47
112.75
77.32
77.01
76.69
47.01
39.83
37.55
11.17
Current Data Parameters
NAME 24082014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140824
Time 20.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178062 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Side product
4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


P-5- NMe2
Current Data Parameters
NAME 23032014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140323
Time 21.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500175 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.2402
7.1576
7.0109
7.0064
6.9900
6.9857
6.6279
6.6071
4.5648
4.5508
3.9557
3.9410
3.9348
3.9201
3.9140
3.8993
3.8187
3.8029
3.7969
3.7810
3.7755
3.7596
2.9784
2.9654
2.9507
2.9378
2.8390
2.7328
2.7055
2.6780
2.5668
2.5594
2.5531
2.5462
2.5397
2.5328
2.5259
2.5194
2.5125
2.5059
2.4988
2.4929
2.4856
2.2741
2.2593
2.2524
2.2371
2.2210
2.2064
2.2004
2.1857
1.7838
4.5
4.0
3.5
3.0
2.5
ppm
1.020
1.056
1.058
1.057
3.037
1.042
1.026
4.363
10
9
8
7
6
5
4
3
2
1
ppm
1.01
1.09
1.00
1.02
1.06
1.06
1.06
3.04
1.04
1.03
4.36
1.09
N
H
H
O
3c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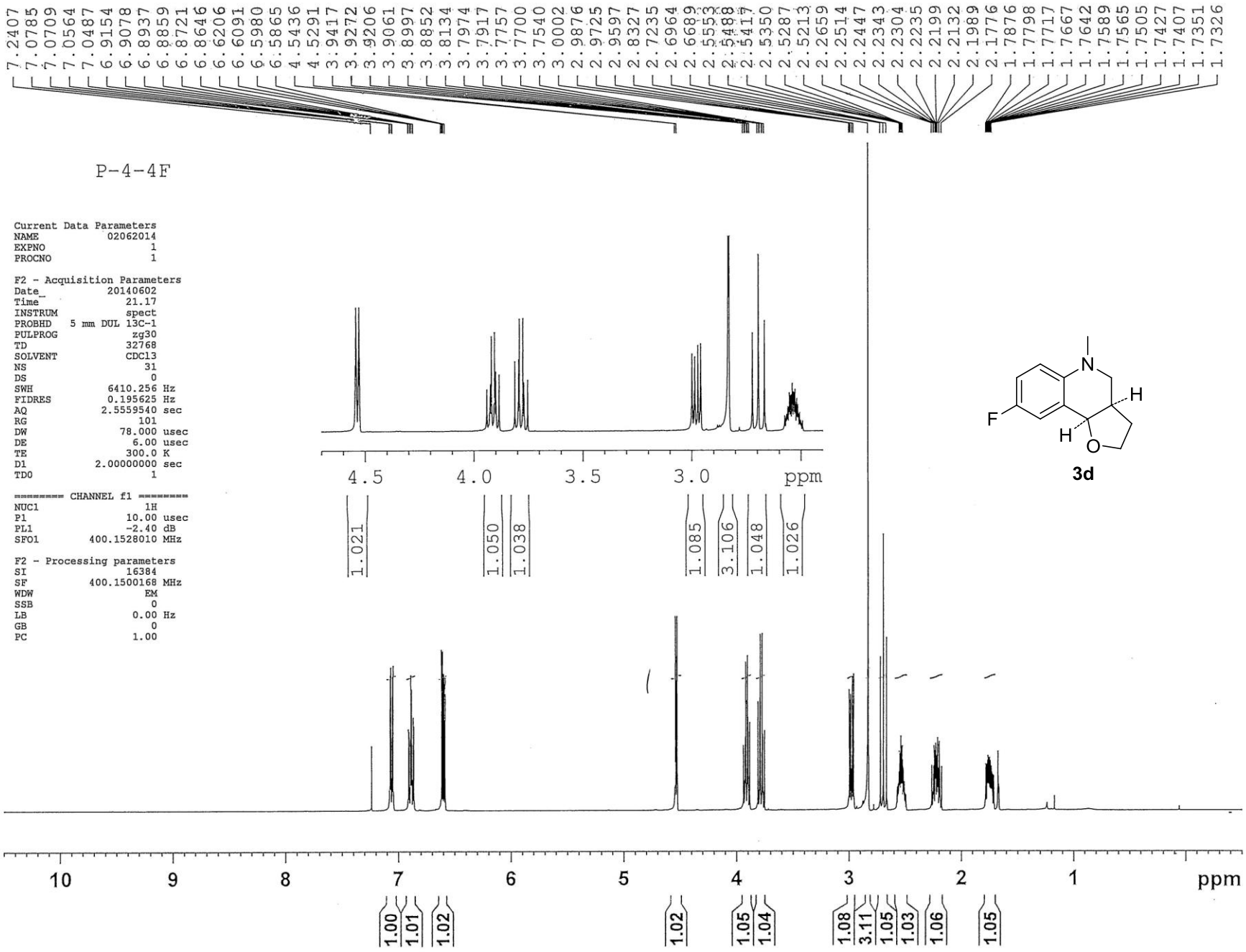
129.54
126.63
121.83
111.96
77.32
77.00
76.69
75.79
65.17
52.85
39.44
36.15
30.06
20.13
urrent Data Parameters
AME 23032014
XPNO 4
ROCNO 1
2 - Acquisition Parameters
ate_ 20140323
ime 21.33
NSTRUM spect
ROBHD 5 mm DUL 13C-1
ULPROG zgpg30
D 65536
OLVENT CDCl3
S 145
S 0
WH 22727.273 Hz
IDRES 0.346791 Hz
Q 1.4418420 sec
G 5.6
W 22.000 usec
E 6.00 usec
E
1 2.00000000 sec
11 0.03000000 sec
ELTA 1.89999998 sec
D0 1
======= CHANNEL f1 ========
UC1 13C
1 9.70 usec
L1 -0.50 dB
FO1 100.6288660 MHz
======= CHANNEL f2 ========
PDPRG2 waltz16
UC2 1H
CPD2 90.00 usec
L2 -2.40 dB
L12 15.10 dB
L13 18.10 dB
FO2 400.1516010 MHz
2 - Processing parameters
I 32768
F 100.6178135 MHz
DW EM
SB 0
B 3.00 Hz
B 0
C 1.00
N
H
H
O
3c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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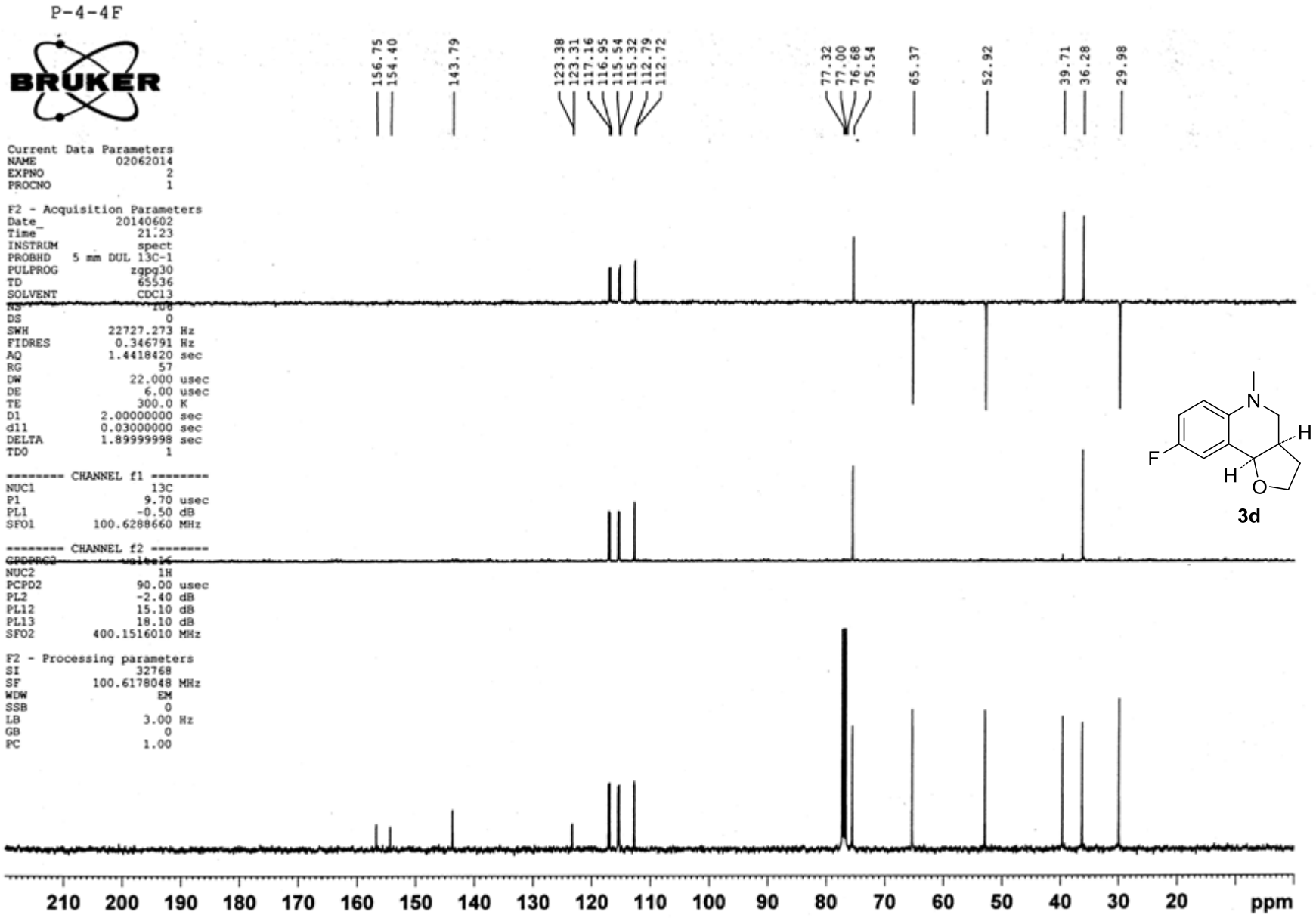
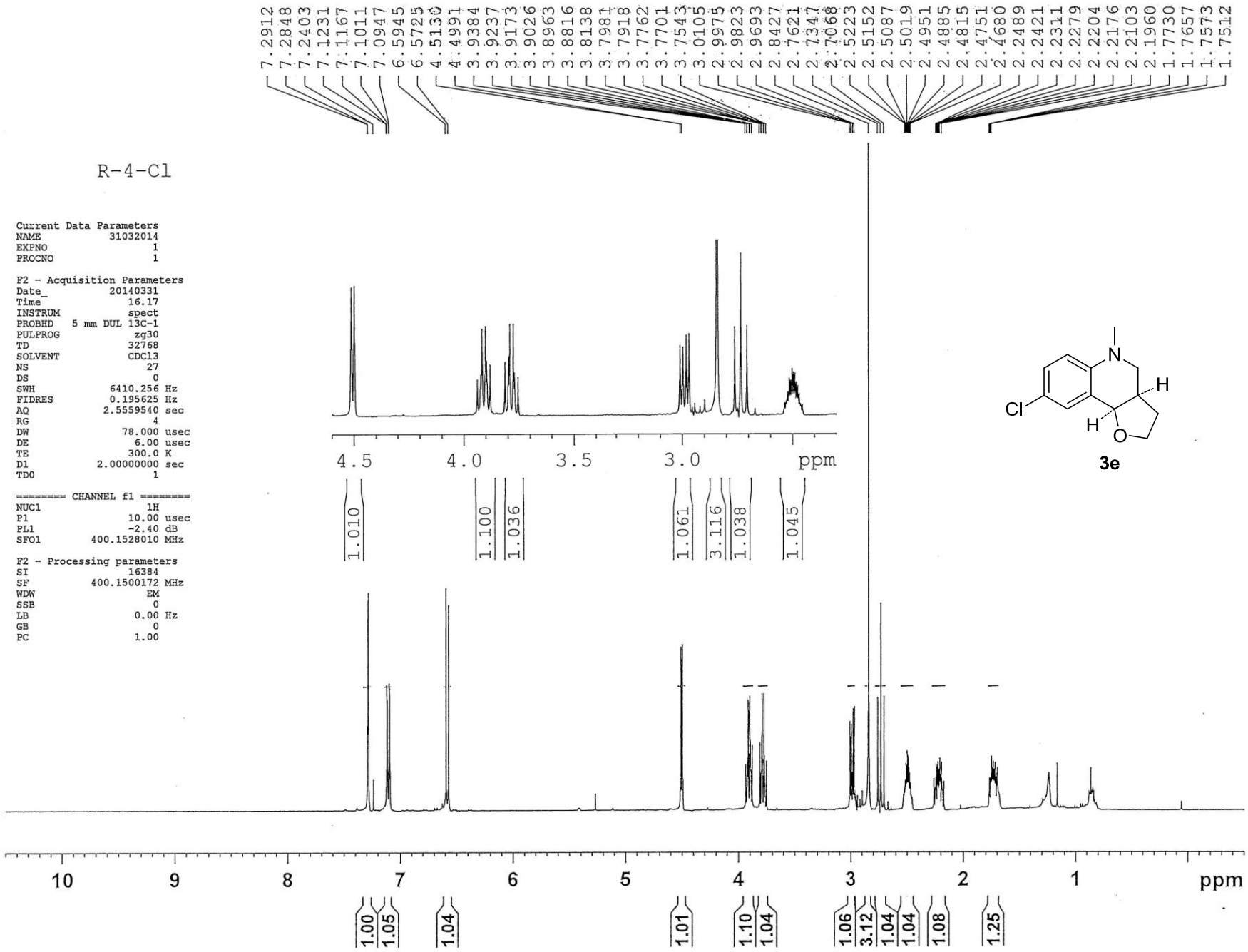
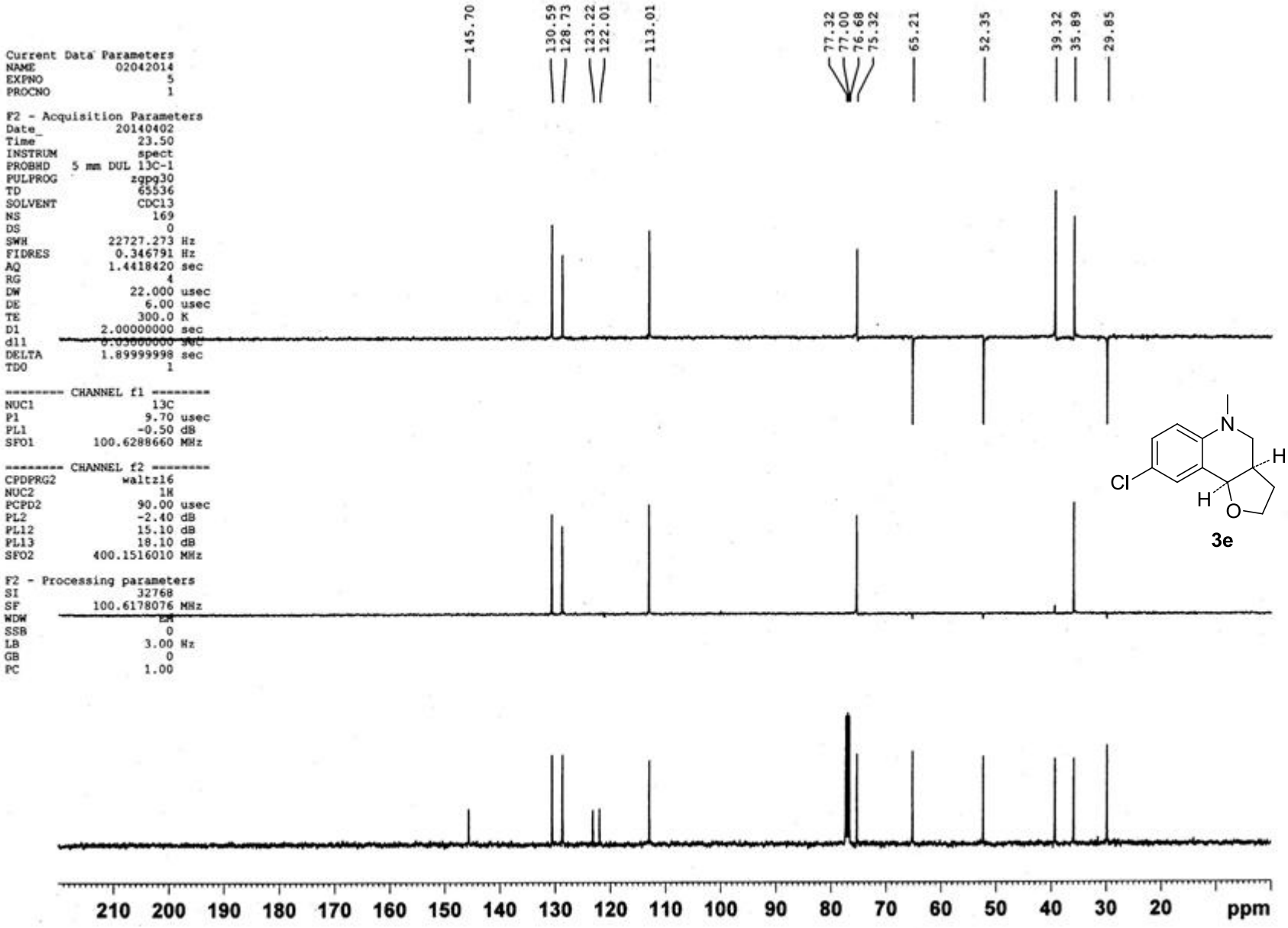
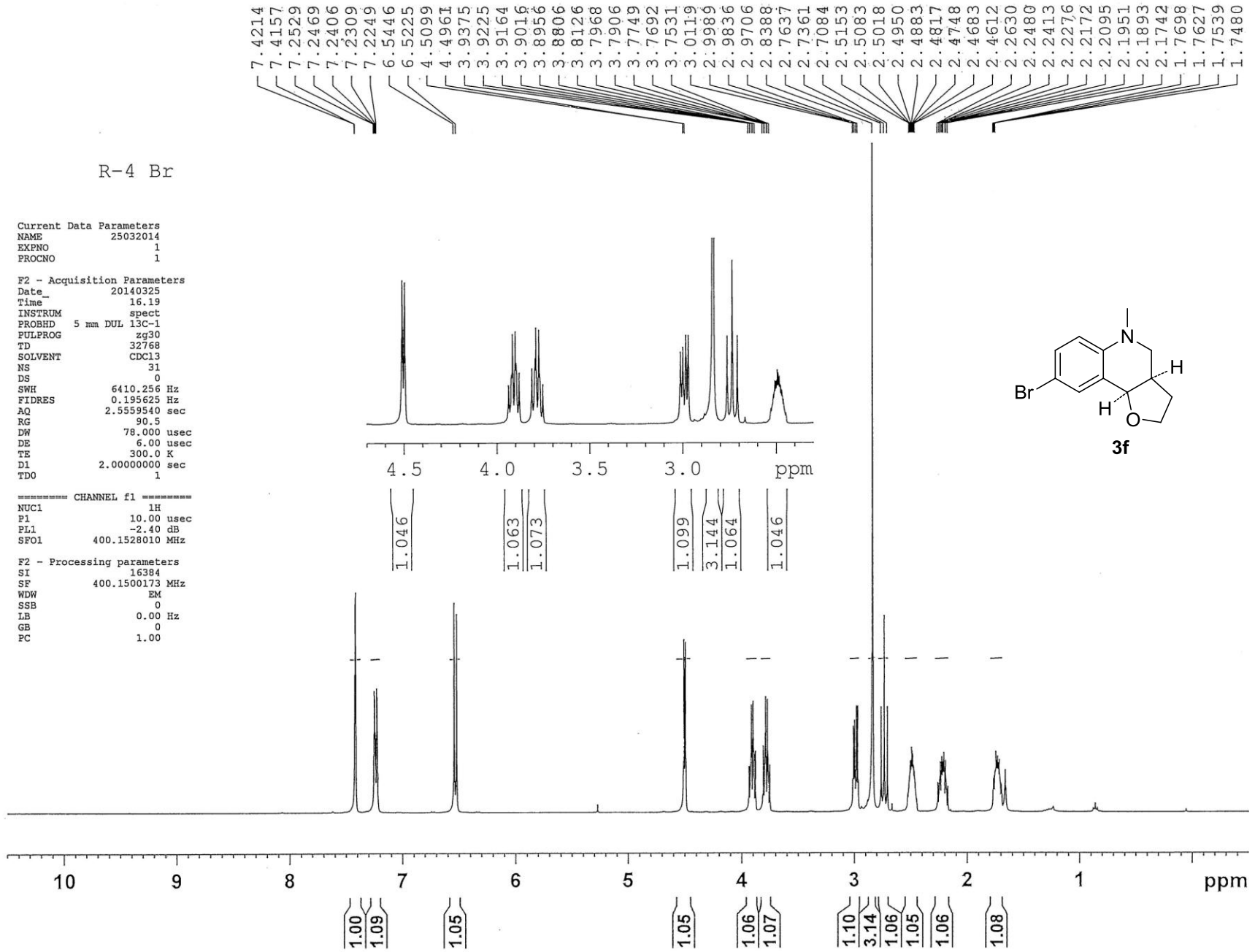
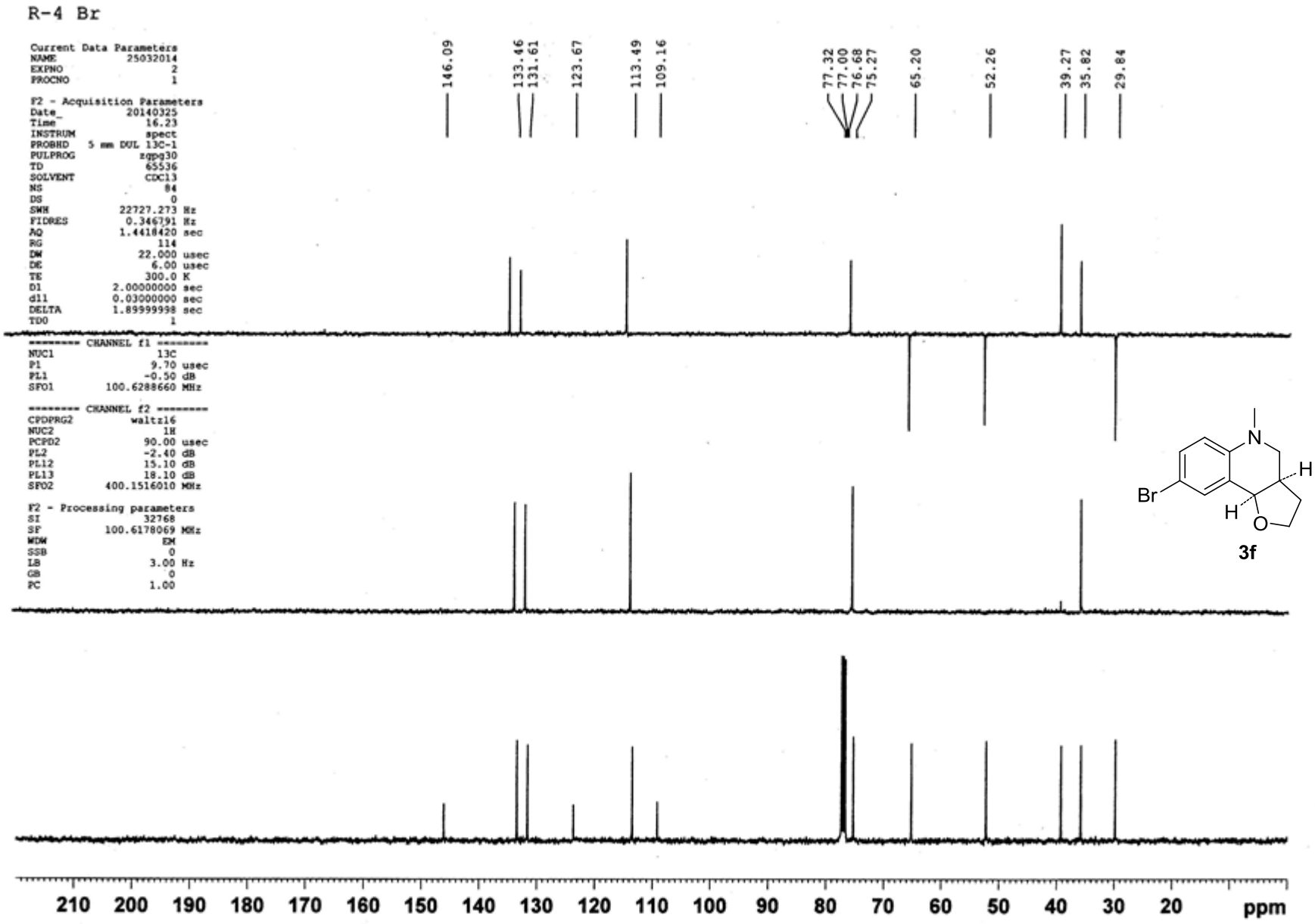
7.0709
7.0564
7.0487
6.9154
6.9078
6.8937
6.8859
6.8721
6.8646
6.6206
6.6091
6.5980
6.5865
4.5436
4.5291
3.9417
3.9272
3.9206
3.9061
3.8997
3.8852
3.8134
3.7974
3.7917
3.7757
3.7700
3.7540
3.0002
2.9876
2.9725
2.9597
2.8327
2.7235
2.6964
2.6689
2.5553
2.5488
2.5417
2.5350
2.5287
2.5213
2.2659
2.2514
2.2447
2.2343
2.2304
2.2235
2.2199
2.2132
2.1989
2.1776
1.7876
1.7798
1.7717
1.7667
1.7642
1.7589
1.7565
1.7505
1.7427
1.7407
1.7351
1.7326
P-4-4F
Current Data Parameters
NAME 02062014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140602
Time 21.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 31
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 101
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.5
4.0
3.5
3.0
ppm
1.021
1.050
1.038
1.085
3.106
1.048
1.026
N
H
F
H
O
3d
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.02
1.02
1.05
1.04
1.08
3.11
1.05
1.03
1.06
1.05

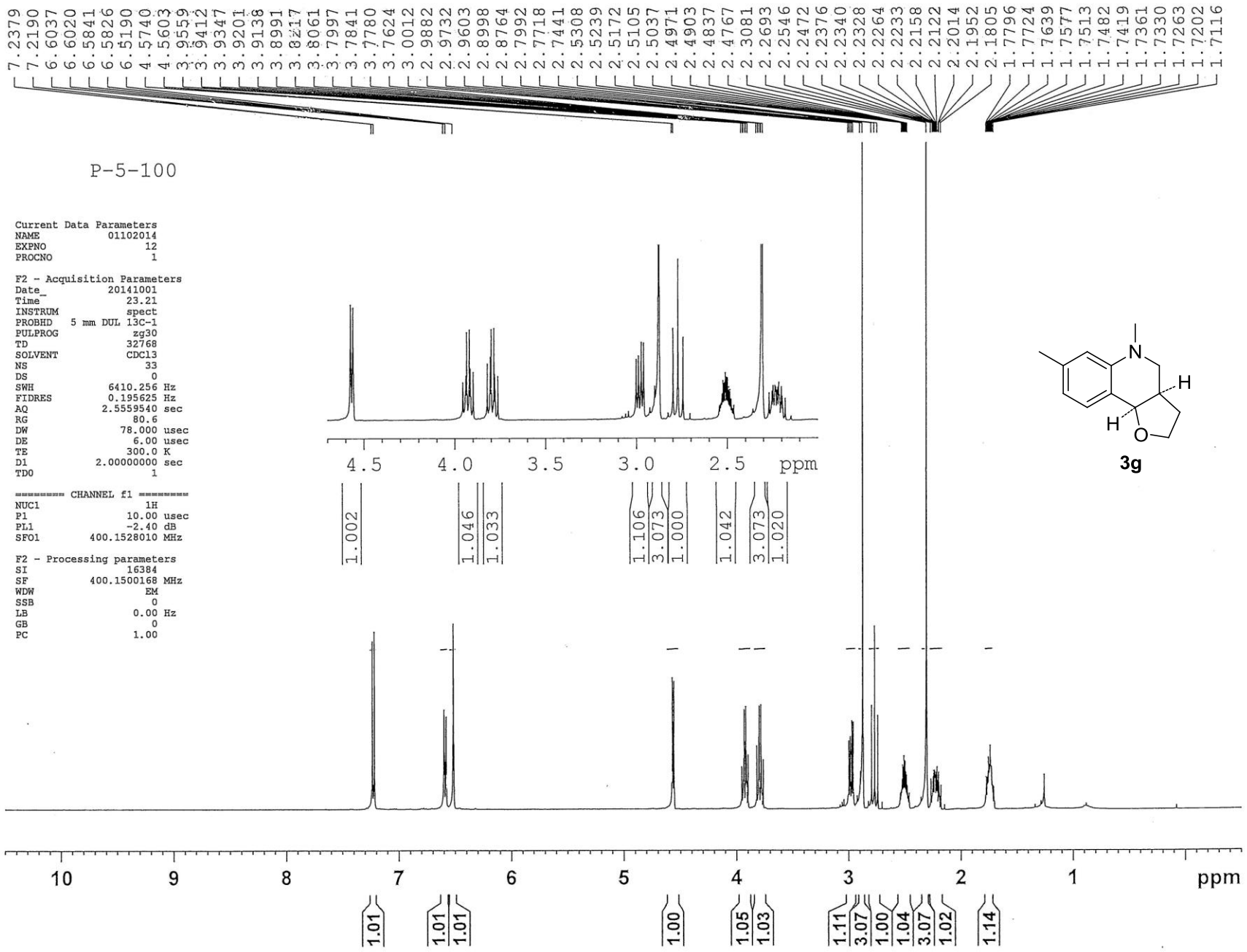

P-4-4F
BRUKER
156.75
154.40
143.79
123.38
123.31
117.16
116.95
115.54
115.32
112.79
112.72
77.32
77.00
76.68
75.54
65.37
52.92
39.71
36.28
29.98
Current Data Parameters
NAME 02062014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140602
Time 21.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 108
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178048 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
H
F
H
O
3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

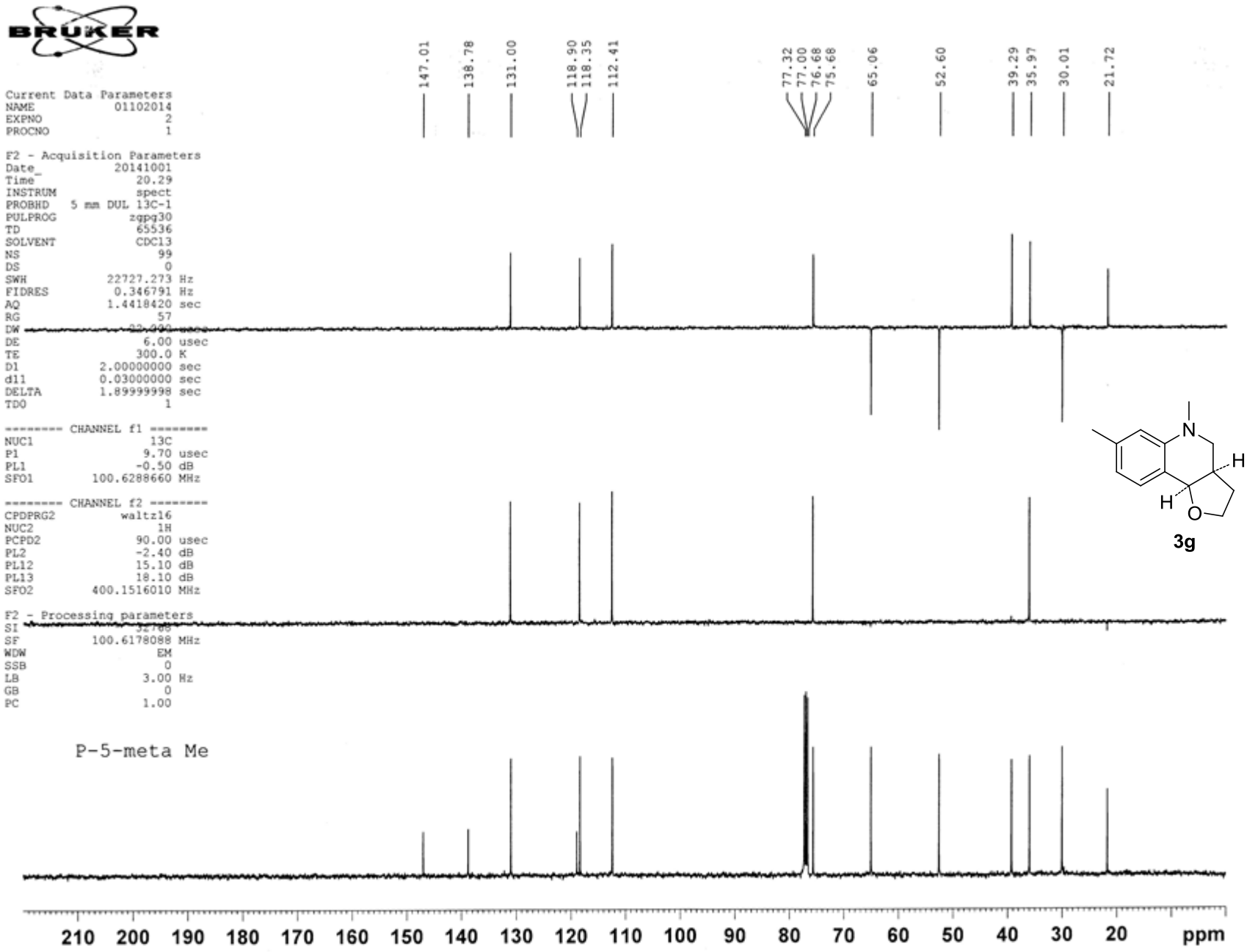

R-4-Cl
Current Data Parameters
NAME 31032014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140331
Time 16.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 27
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 4
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.2912 7.2848 7.2403 7.1231 7.1167 7.1011 7.0947 6.5945 6.5725 4.5130 4.4991 3.9384 3.9237 3.9173 3.9026 3.8963 3.8816 3.8138 3.7981 3.7918 3.7762 3.7701 3.7543 3.0105 2.9975 2.9823 2.9693 2.8427 2.7621 2.7347 2.7068 2.5223 2.5152 2.5087 2.5019 2.4951 2.4885 2.4815 2.4751 2.4680 2.2489 2.2421 2.2311 2.2279 2.2204 2.2176 2.2103 2.1960 1.7730 1.7657 1.7573 1.7512
1.010 1.100 1.036 1.061 3.116 1.038 1.045
1.00 1.05 1.04 1.01 1.10 1.04 1.06 3.12 1.04 1.04 1.08 1.25
ppm
Cl
N
H
H
O
3e

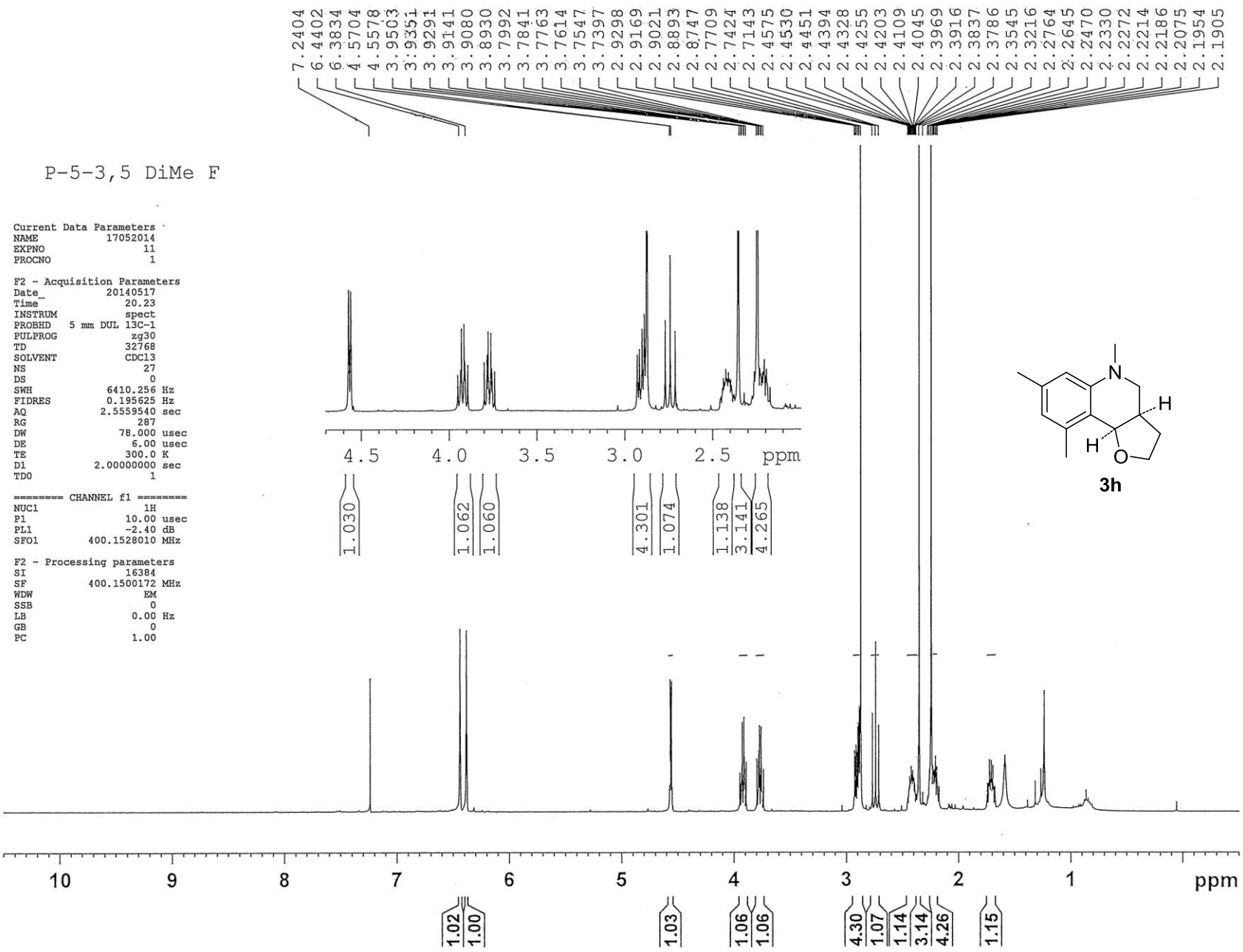

145.70
130.59
128.73
123.22
122.01
113.01
77.32
77.00
76.68
75.32
65.21
52.35
39.32
35.89
29.85
Current Data Parameters
NAME 02042014
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140402
Time 23.50
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 169
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 4
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178076 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
H
Cl
H
O
3e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


R-4 Br
Current Data Parameters
NAME 25032014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140325
Time 16.19
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 31
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500173 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.4214 7.4157 7.2529 7.2469 7.2406 7.2309 7.2249 6.5446 6.5225 4.5099 4.4961 3.9375 3.9225 3.9164 3.9016 3.8956 3.8806 3.8126 3.7968 3.7906 3.7749 3.7692 3.7531 3.0119 2.9989 2.9836 2.9706 2.8388 2.7637 2.7361 2.7084 2.5153 2.5083 2.5018 2.4950 2.4883 2.4817 2.4748 2.4683 2.4612 2.2630 2.2480 2.2413 2.2276 2.2172 2.2095 2.1951 2.1893 2.1742 1.7698 1.7627 1.7539 1.7480
1.046 1.063 1.073 1.099 3.144 1.064 1.046
4.5 4.0 3.5 3.0 ppm
10 9 8 7 6 5 4 3 2 1 ppm
1.00 1.09 1.05 1.05 1.06 1.07 1.10 3.14 1.06 1.05 1.06 1.08
Br
N
H
H
O
3f


R-4 Br
Current Data Parameters
NAME 25032014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140325
Time 16.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 114
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178069 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
146.09
133.46
131.61
123.67
113.49
109.16
77.32
77.00
76.68
75.27
65.20
52.26
39.27
35.82
29.84
N
H
Br
H
O
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


P-5-100
Current Data Parameters
NAME 01102014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141001
Time 23.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 33
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 80.6
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
3g


147.01
138.78
131.00
118.90
118.35
112.41
77.32
77.00
76.68
75.68
65.06
52.60
39.29
35.97
30.01
21.72
Current Data Parameters
NAME 01102014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141001
Time 20.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 99
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178088 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-5-meta Me
N
H
H
O
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


P-5-3,5 DiMe F
Current Data Parameters
NAME 17052014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140517
Time 20.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 27
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 287
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.2404
6.4402
6.3834
4.5704
4.5578
3.9503
3.9351
3.9291
3.9141
3.9080
3.8930
3.7992
3.7841
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3.7614
3.7547
3.7397
2.9298
2.9169
2.9021
2.8893
2.8747
2.7709
2.7424
2.7143
2.4575
2.4530
2.4451
2.4394
2.4328
2.4255
2.4203
2.4109
2.4045
2.3969
2.3916
2.3837
2.3786
2.3545
2.3216
2.2764
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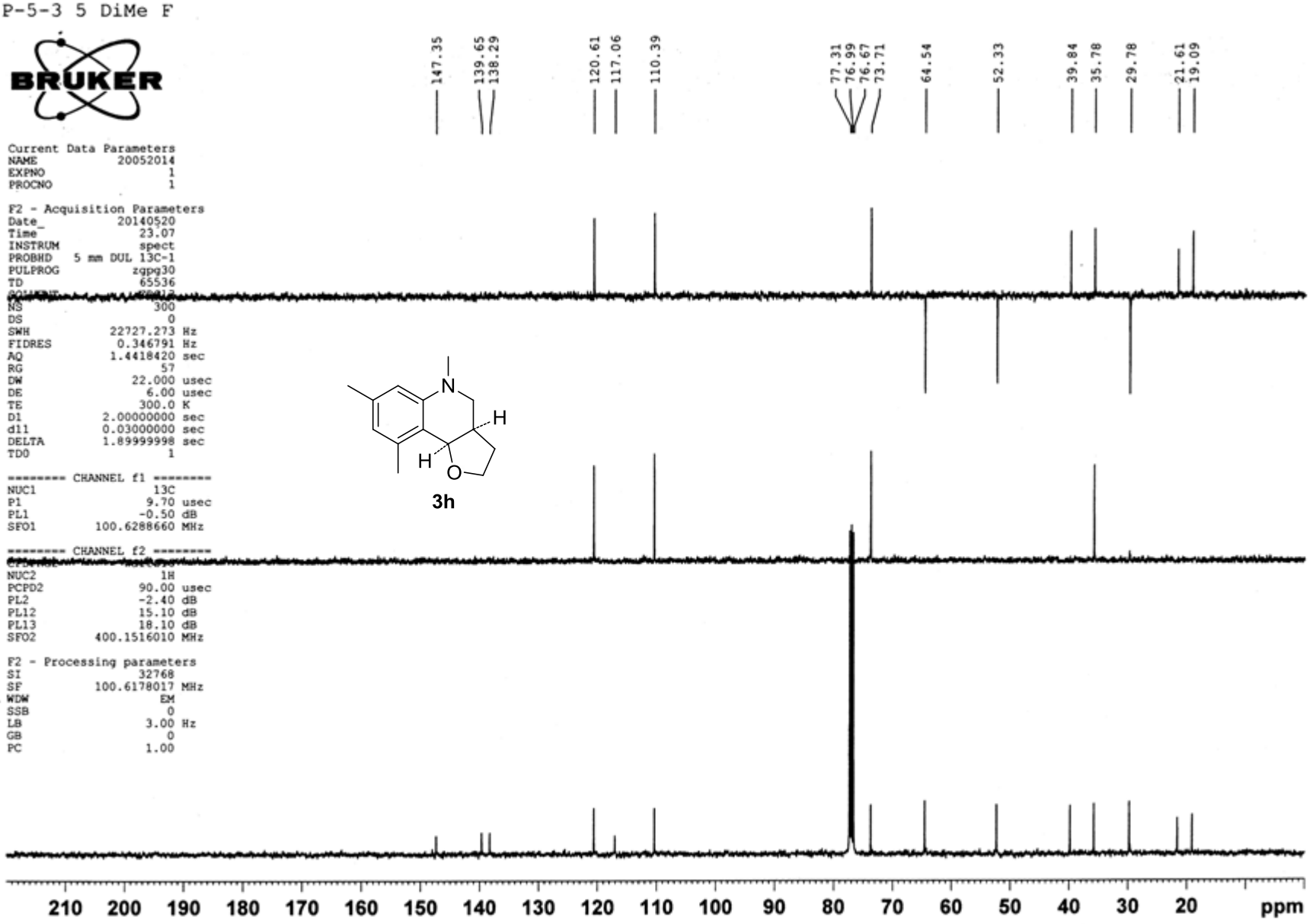
2.2470
2.2330
2.2272
2.2214
2.2186
2.2075
2.1954
2.1905
4.5
4.0
3.5
3.0
2.5
ppm
1.030
1.062
1.060
4.301
1.074
1.138
3.141
4.265
N
H
H
O
3h
10
9
8
7
6
5
4
3
2
1
ppm
1.02
1.00
1.03
1.06
1.06
4.30
1.07
1.14
3.14
4.26
1.15


P-5-3 5 DiMe F
147.35
139.65
138.29
120.61
117.06
110.39
77.31
76.99
76.67
73.71
64.54
52.33
39.84
35.78
29.78
21.61
19.09
Current Data Parameters
NAME 20052014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140520
Time 23.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
NS 300
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178017 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
H
H
O
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

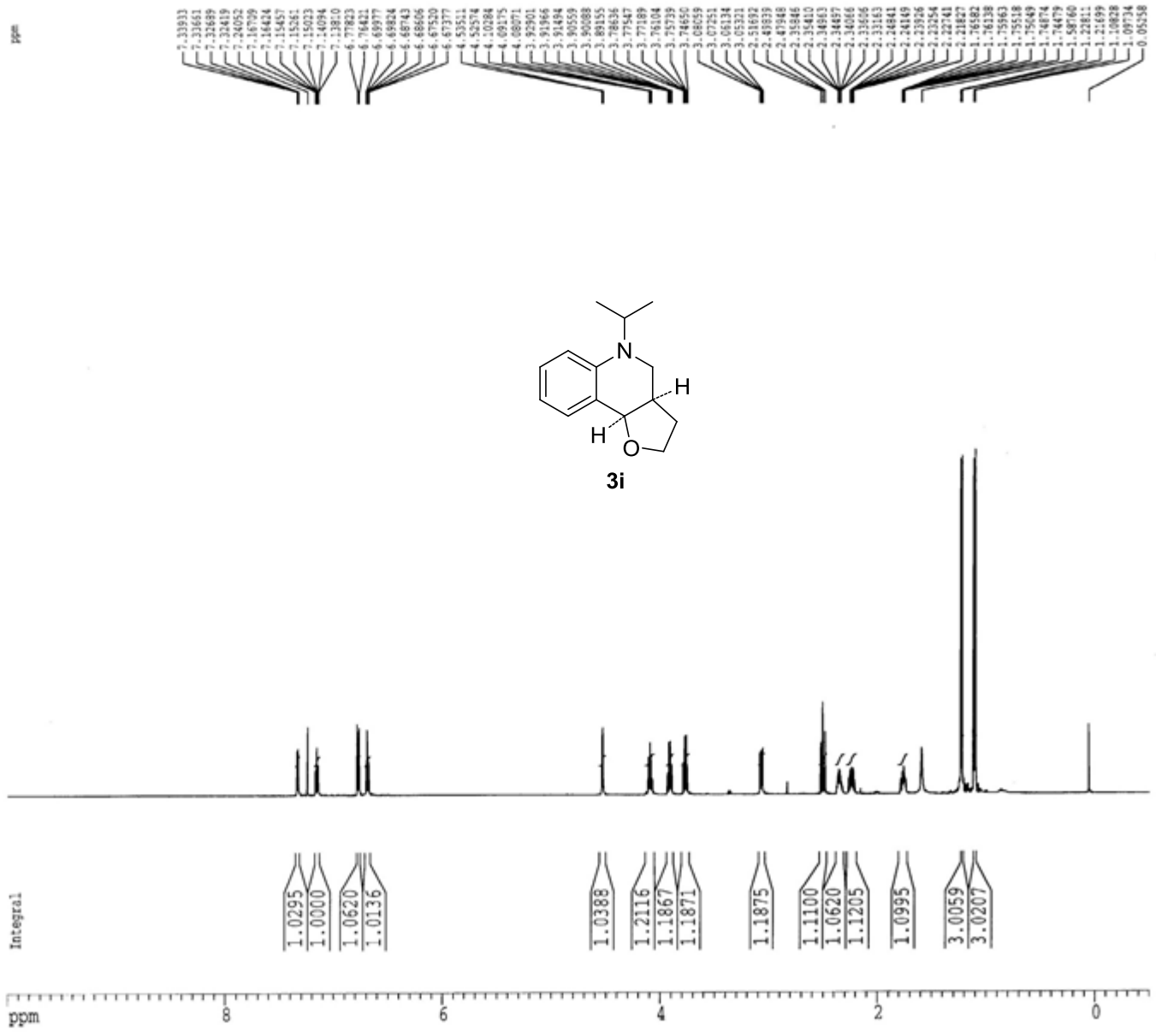
```
Current Data Parameters
NAME            RD-1463P
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140417
Time               17.46
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            9578.544 Hz
FIDRES         0.292314 Hz
AQ             1.7105396 sec
RG                  128
DW                52.200 usec
DE                 6.50 usec
TE                297.5 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1                3.00 dB
SFO1         598.7029935 MHz

F2 - Processing parameters
SI                32768
SF           598.7000261 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 6.00 cm
F1P               10.000 ppm
F1              5987.00 Hz
F2P               -0.500 ppm
F2              -299.35 Hz
PPMCM             0.52500 ppm/cm
HZCM            314.31750 Hz/cm
```



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Current Data Parameters
NAME              RD-1463P
EXPNO                    2
PROCNO                   1

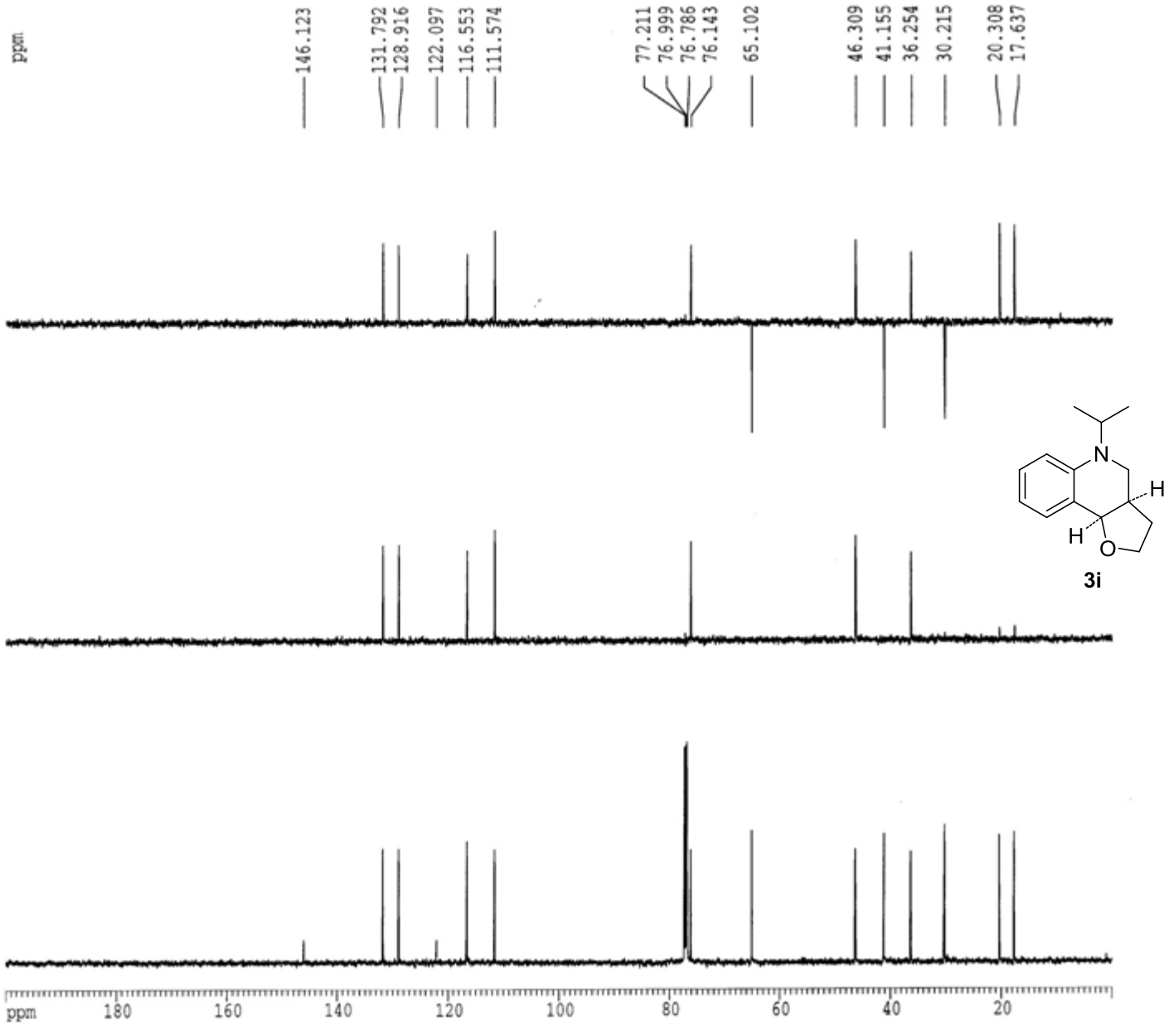
F2 - Acquisition Parameters
Date_              20140417
Time                 18.04
INSTRUM              spect
PROBHD   5 mm QNP  1H/1
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                      270
DS                        0
SWH               45045.047 Hz
FIDRES             1.374666 Hz
AQ                 0.3637748 sec
RG                     2048
DW                   11.100 usec
DE                    6.50 usec
TE                  298.8 K
D1                3.50000000 sec
d11               0.03000000 sec
DELTA             3.40000010 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

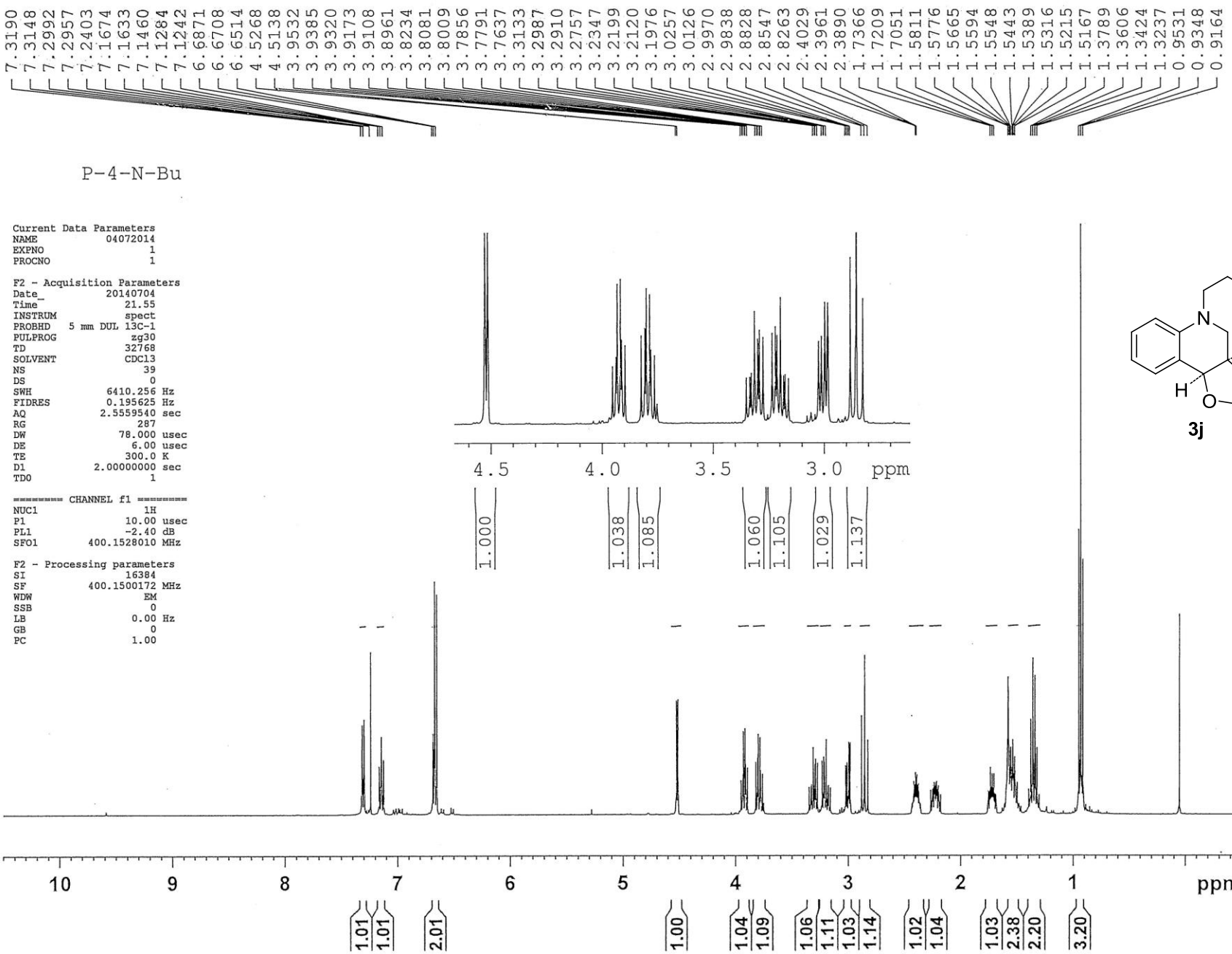
======== CHANNEL f1 ========
NUC1                    13C
P1                     4.80 usec
PL1                    0.00 dB
SFO1           150.5597948 MHz

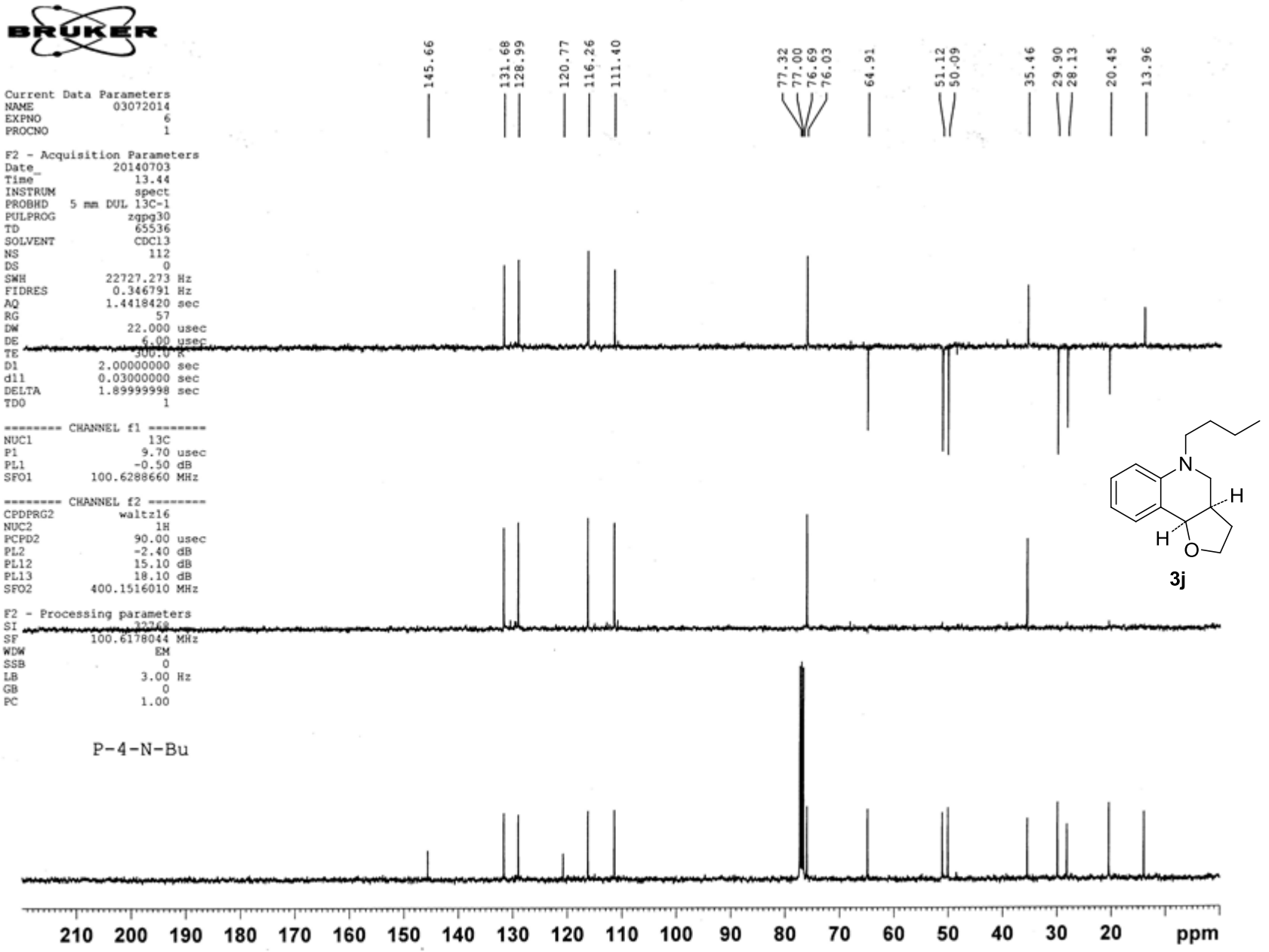
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 92.00 usec
PL2                  120.00 dB
PL12                   9.00 dB
PL13                  14.00 dB
SFO2           598.7029935 MHz

F2 - Processing parameters
SI                     65536
SF             150.5632390 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                      4.00 cm
F1P                  200.000 ppm
F1                 30108.65 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1505.43237 Hz/cm
```




7.3190 7.3148 7.2992 7.2957 7.2403 7.1674 7.1633 7.1460 7.1284 7.1242 6.6871 6.6708 6.6514 4.5268 4.5138 3.9532 3.9385 3.9320 3.9173 3.9108 3.8961 3.8234 3.8081 3.8009 3.7856 3.7791 3.7637 3.3133 3.2987 3.2910 3.2757 3.2347 3.2199 3.2120 3.1976 3.0257 3.0126 2.9970 2.9838 2.8828 2.8547 2.8263 2.4029 2.3961 2.3890 1.7366 1.7209 1.7051 1.5811 1.5776 1.5665 1.5594 1.5548 1.5443 1.5389 1.5316 1.5215 1.5167 1.3789 1.3606 1.3424 1.3237 0.9531 0.9348 0.9164
P-4-N-Bu
Current Data Parameters
NAME 04072014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140704
Time 21.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 39
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 287
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.5 4.0 3.5 3.0 ppm
1.000 1.038 1.085 1.060 1.105 1.029 1.137
10 9 8 7 6 5 4 3 2 1 ppm
1.01 1.01 2.01 1.00 1.04 1.09 1.06 1.11 1.03 1.14 1.02 1.04 1.03 2.38 2.20 3.20
N H H O
3j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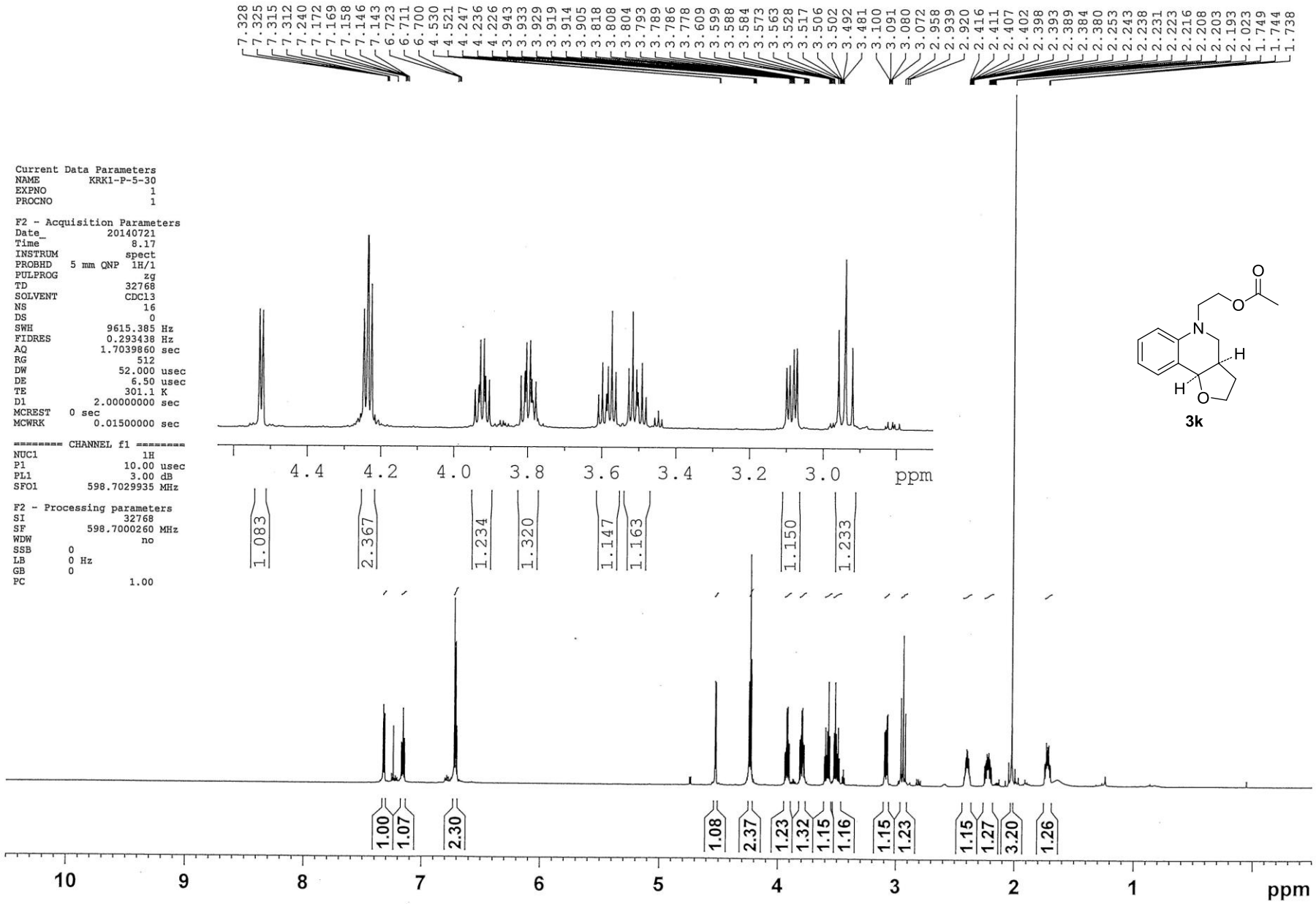
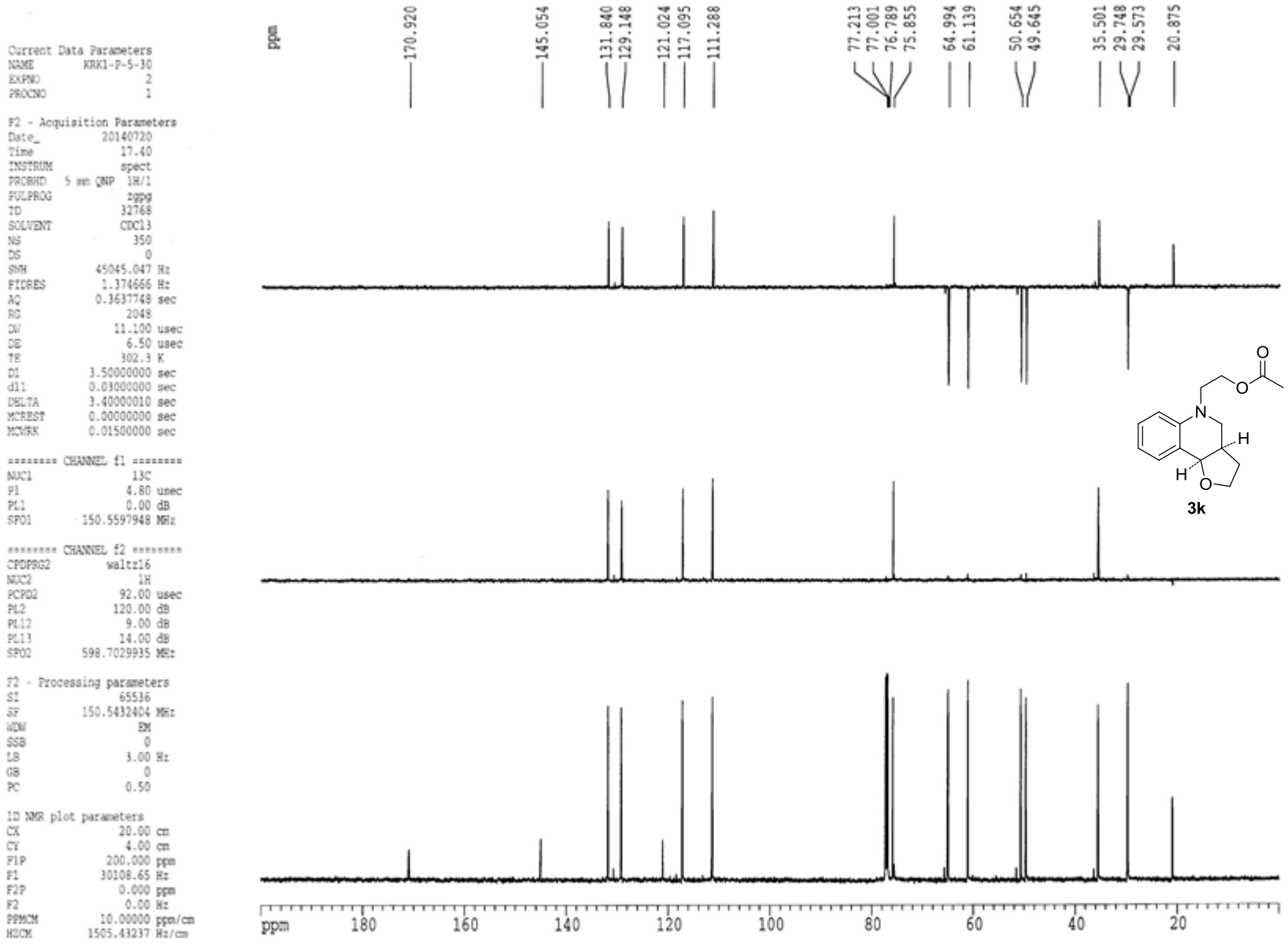
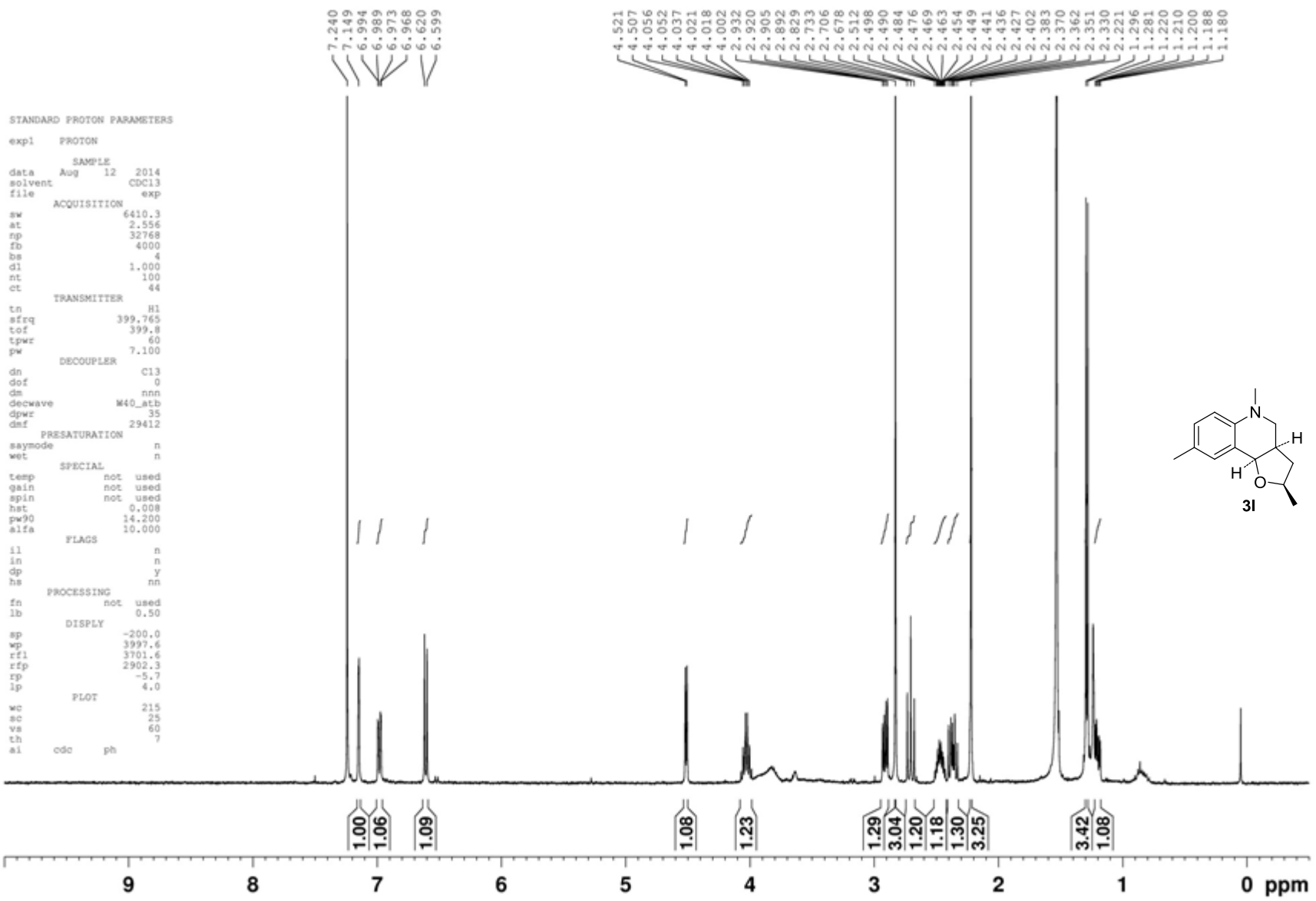
NAME 03072014
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140703
Time 13.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 112
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178044 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-4-N-Bu
145.66
131.68
128.99
120.77
116.26
111.40
77.32
77.00
76.69
76.03
64.91
51.12
50.09
35.46
29.90
28.13
20.45
13.96
N
H
H
O
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.328 7.325 7.315 7.312 7.240 7.172 7.169 7.158 7.146 7.143 6.723 6.711 6.700 4.530 4.521 4.247 4.236 4.226 3.943 3.933 3.929 3.919 3.914 3.905 3.818 3.808 3.804 3.793 3.789 3.786 3.778 3.609 3.599 3.588 3.584 3.573 3.563 3.528 3.517 3.506 3.502 3.492 3.481 3.100 3.091 3.080 3.072 2.958 2.939 2.920 2.416 2.411 2.407 2.402 2.398 2.393 2.389 2.384 2.380 2.253 2.243 2.238 2.231 2.223 2.216 2.208 2.203 2.193 2.023 1.749 1.744 1.738
Current Data Parameters
NAME KRK1-P-5-30
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140721
Time 8.17
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 512
DW 52.000 usec
DE 6.50 usec
TE 301.1 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 598.7029935 MHz
F2 - Processing parameters
SI 32768
SF 598.7000260 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 ppm
1.083 2.367 1.234 1.320 1.147 1.163 1.150 1.233
1.00 1.07 2.30 1.08 2.37 1.23 1.32 1.15 1.16 1.15 1.23 1.15 1.27 3.20 1.26
10 9 8 7 6 5 4 3 2 1 ppm
O
O
N
H
H
O
3k


Current Data Parameters
NAME KRK1-P-5-30
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140720
Time 17.40
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 350
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 302.3 K
D1 3.50000000 sec
d11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.80 usec
PL1 0.00 dB
SFO1 150.5597948 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 598.7029935 MHz
F2 - Processing parameters
SI 65536
SF 150.5432404 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 0.50
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.000 ppm
F1 30108.65 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1505.43237 Hz/cm
ppm
170.920
145.054
131.840
129.148
121.024
117.095
111.288
77.213
77.001
76.789
75.855
64.994
61.139
50.654
49.645
35.501
29.748
29.573
20.875
O
O
N
H
H
O
3k
ppm
180
160
140
120
100
80
60
40
20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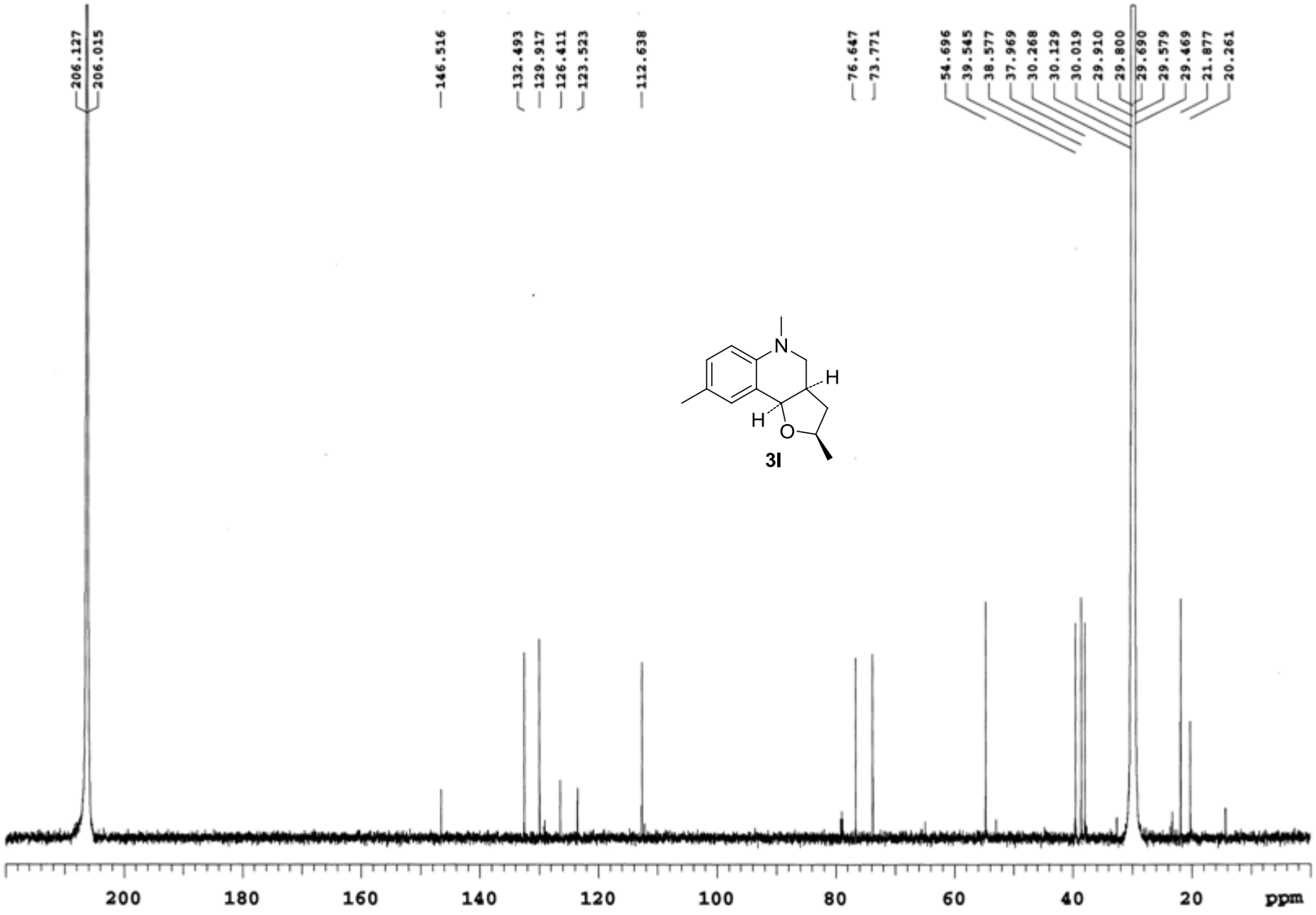
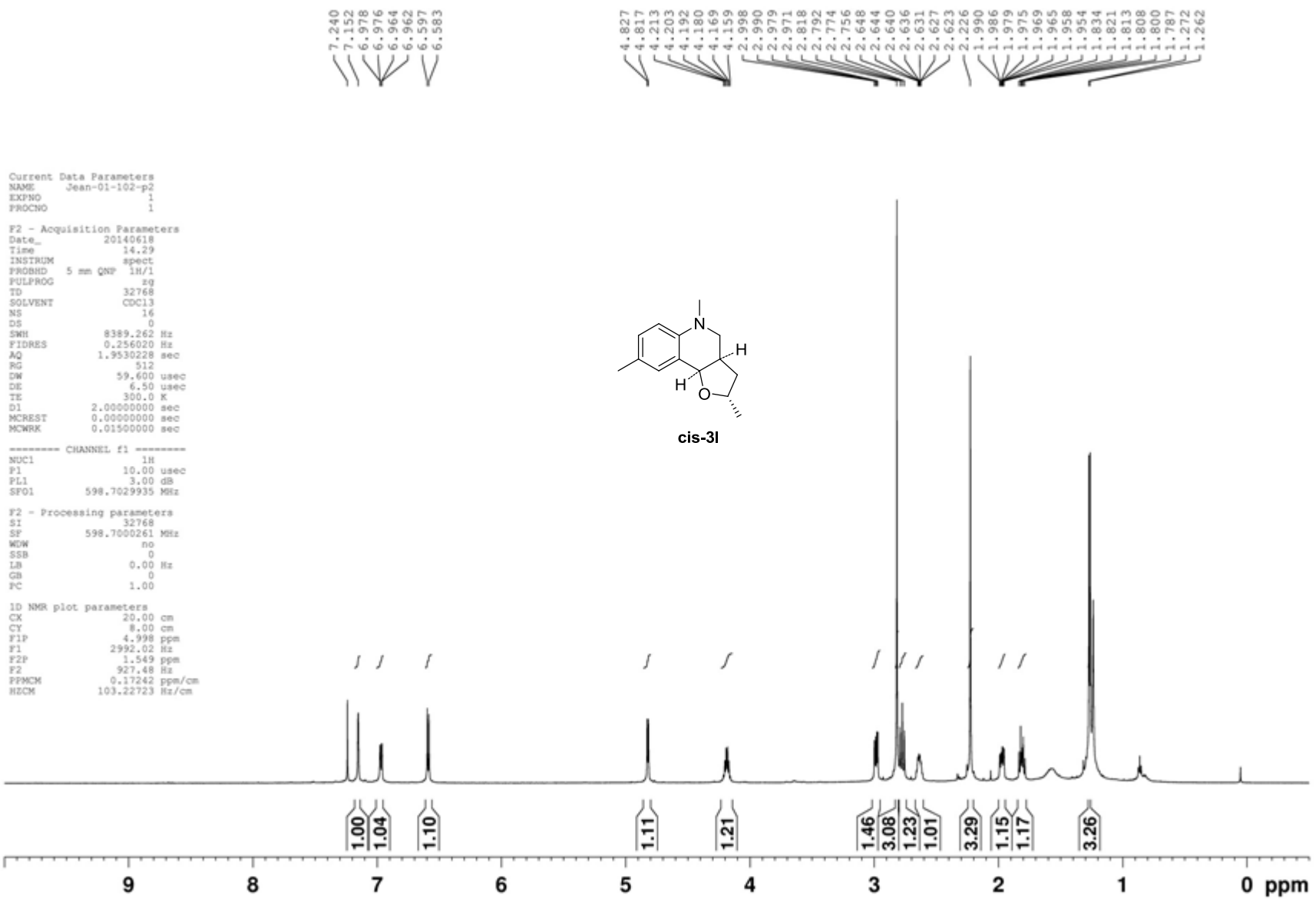
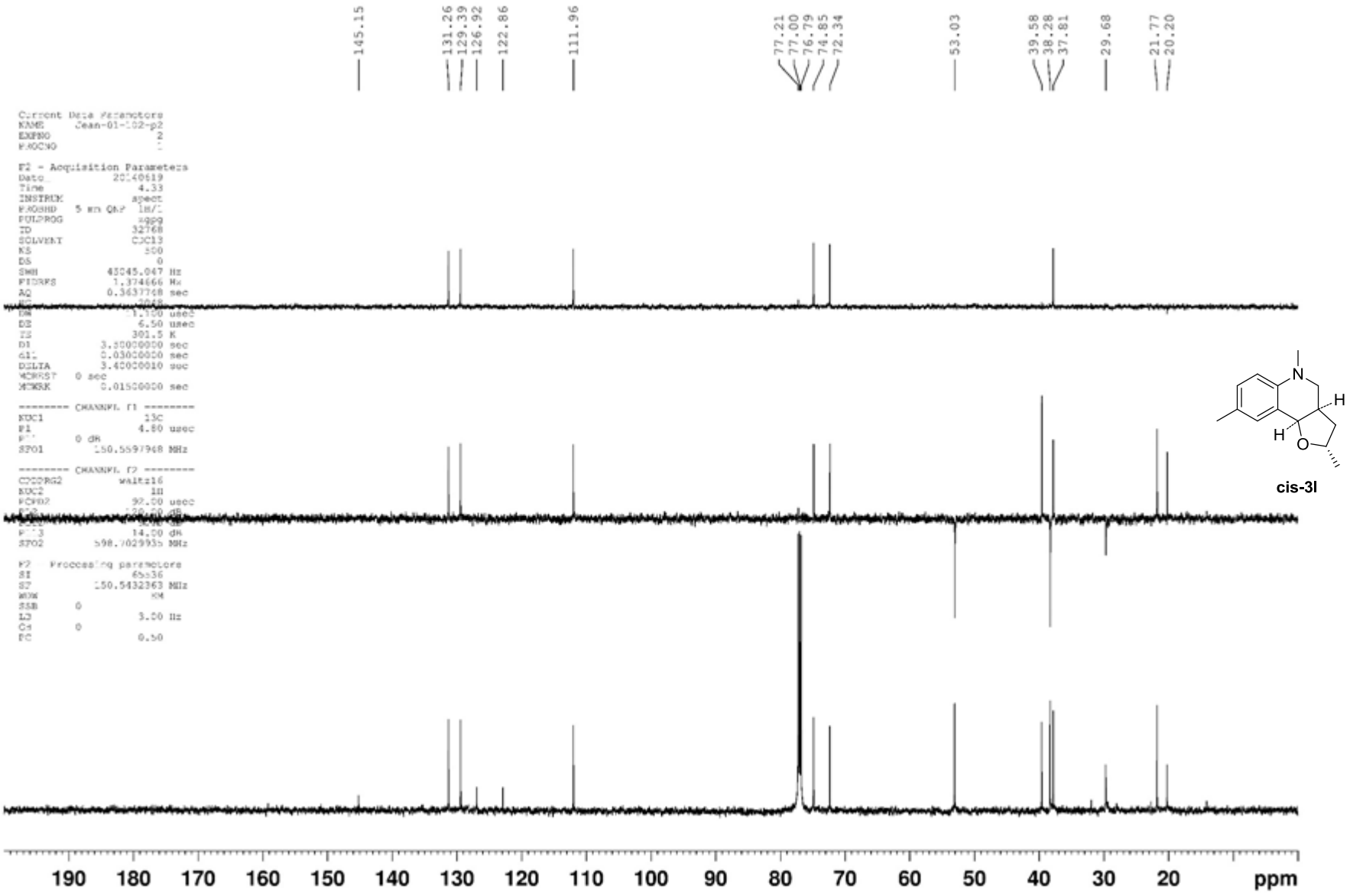
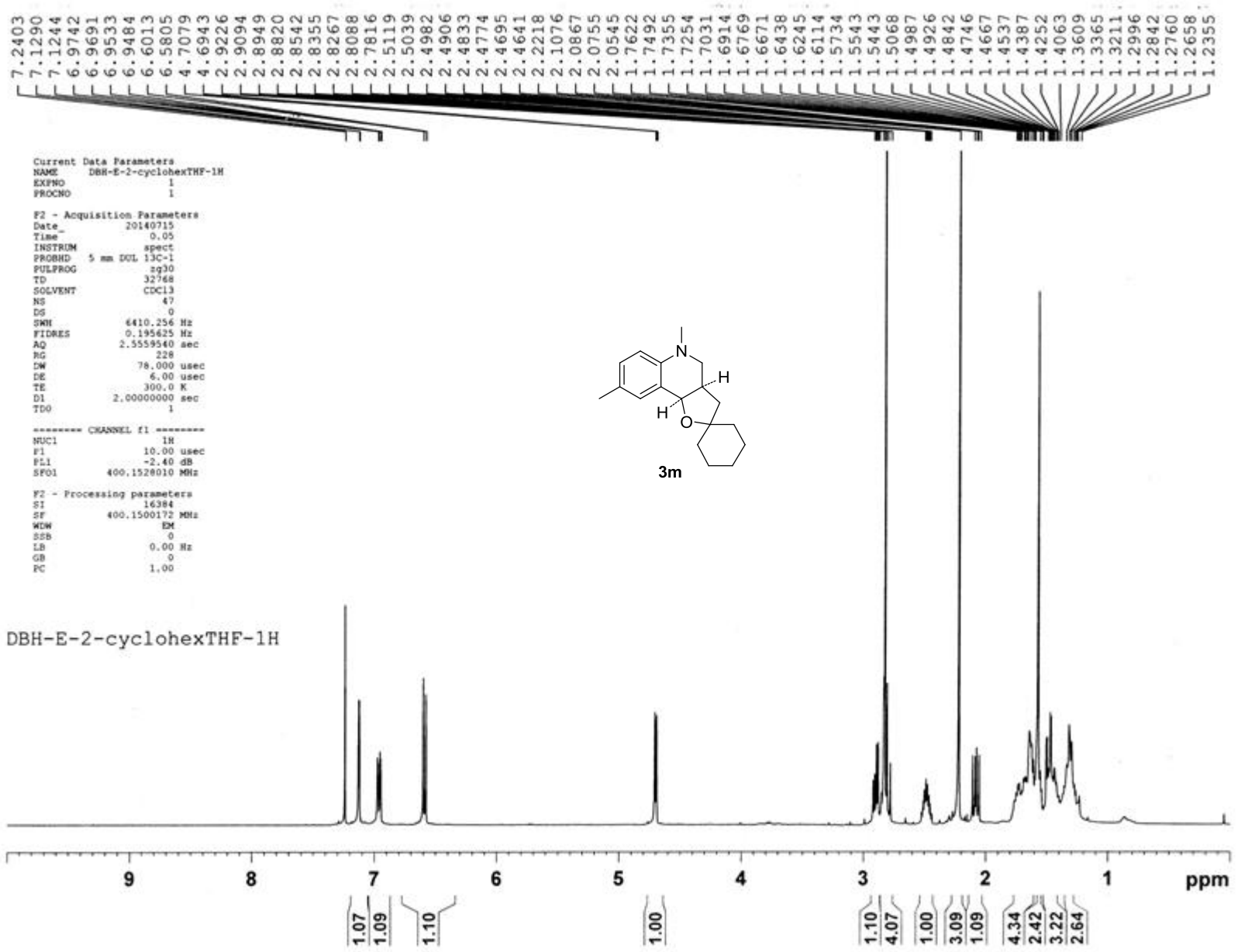
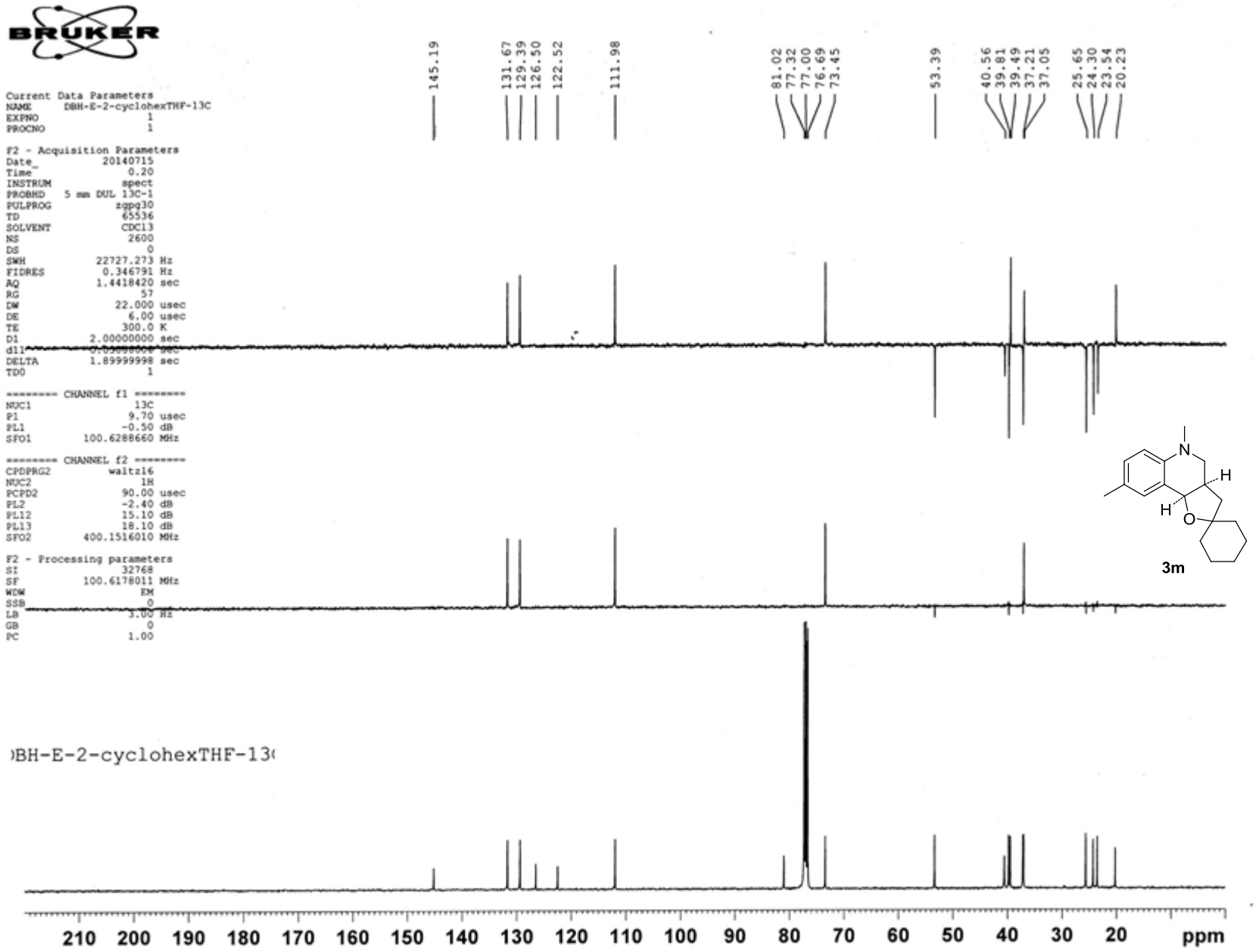
expl PROTON
SAMPLE
data Aug 12 2014
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 2.556
np 32768
fb 4000
bs 4
d1 1.000
nt 100
ct 44
TRANSMITTER
tn H1
sfrq 399.765
tof 399.8
tpwr 60
pw 7.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_atb
dpwr 35
dmf 29412
PRESATURATION
saymode n
wet n
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 14.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
lb 0.50
DISPLY
sp -200.0
wp 3997.6
rf1 3701.6
rfp 2902.3
rp -5.7
lp 4.0
PLOT
wc 215
sc 25
vs 60
th 7
ai cdc ph
7.240
7.149
6.994
6.989
6.973
6.968
6.620
6.599
4.521
4.507
4.056
4.052
4.037
4.021
4.018
4.002
2.932
2.920
2.905
2.892
2.829
2.733
2.706
2.678
2.512
2.498
2.490
2.484
2.476
2.469
2.463
2.454
2.449
2.441
2.436
2.427
2.402
2.383
2.370
2.362
2.351
2.330
2.221
1.296
1.281
1.220
1.210
1.200
1.188
1.180
1.00
1.06
1.09
1.08
1.23
1.29
3.04
1.20
1.18
1.30
3.25
3.42
1.08
9
8
7
6
5
4
3
2
1
0 ppm
N
H
H
O
3l

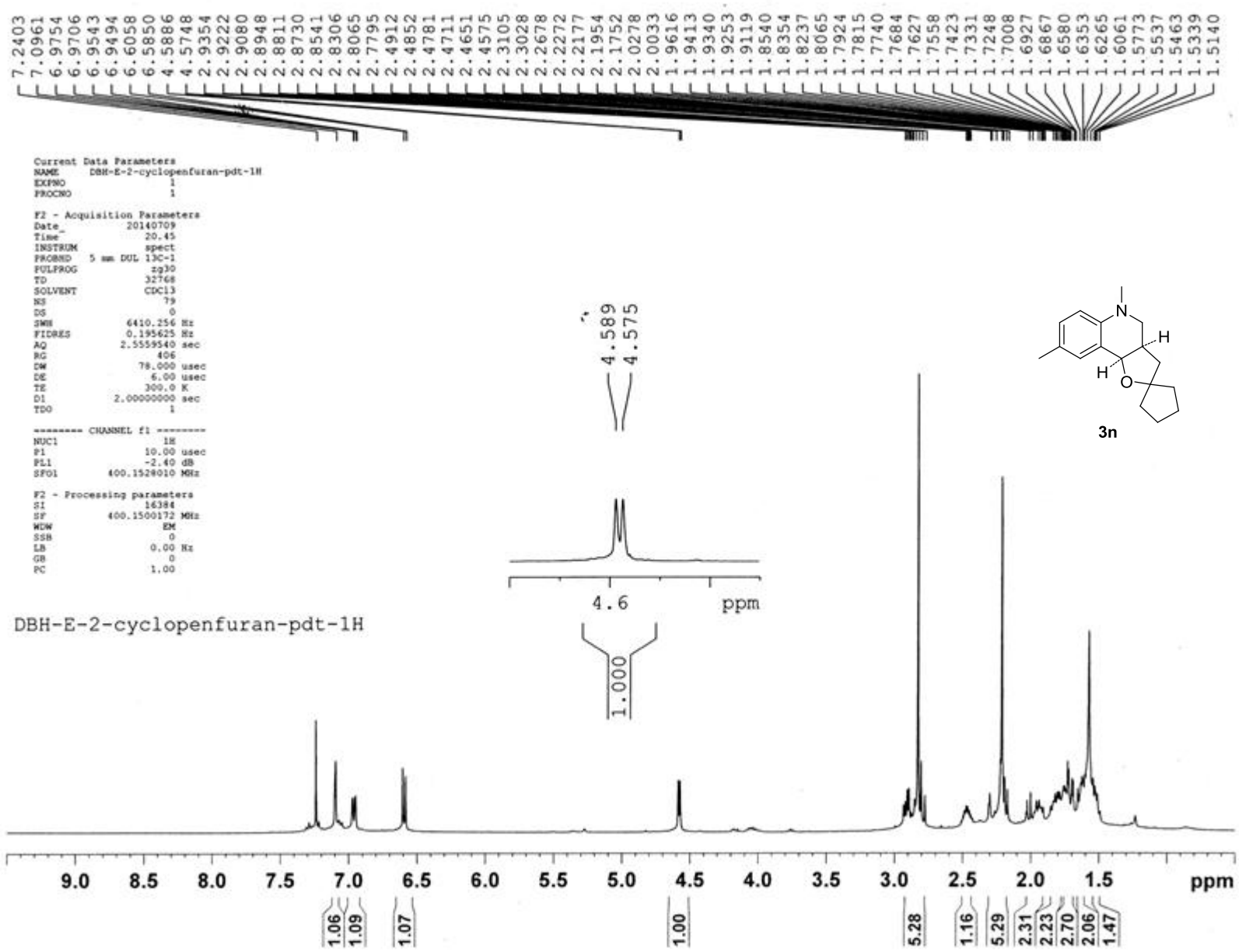

206.127
206.015
146.516
132.493
129.917
126.411
123.523
112.638
76.647
73.771
54.696
39.545
38.577
37.969
30.268
30.129
30.019
29.910
29.800
29.690
29.579
29.469
21.877
20.261
N
H
H
O
3l
200
180
160
140
120
100
80
60
40
20
ppm

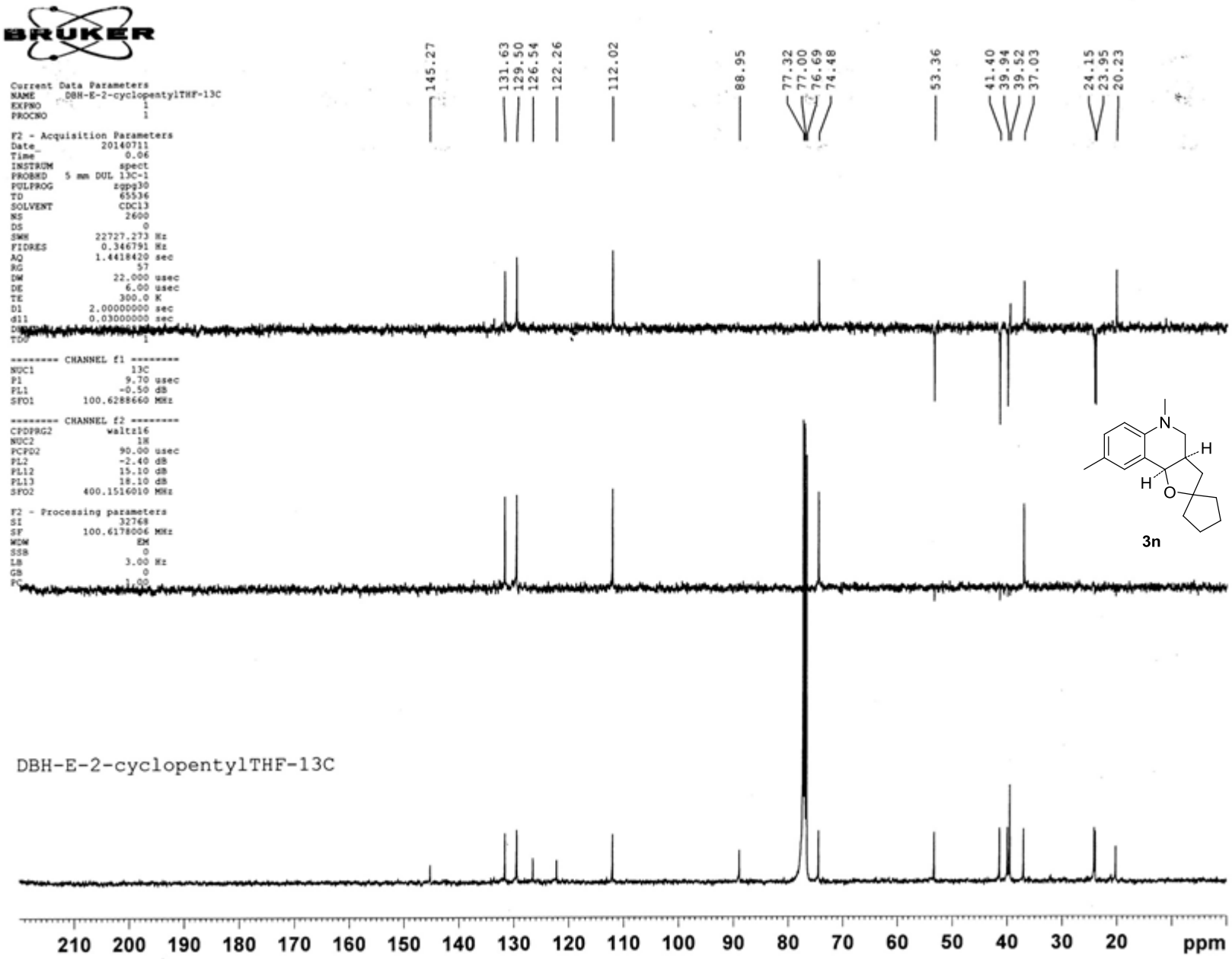

7.240
7.152
6.978
6.976
6.964
6.962
6.597
6.583
4.827
4.817
4.213
4.203
4.192
4.180
4.169
4.159
2.998
2.990
2.979
2.971
2.818
2.792
2.774
2.756
2.648
2.644
2.640
2.636
2.631
2.627
2.623
2.226
1.990
1.986
1.979
1.975
1.969
1.965
1.958
1.954
1.834
1.821
1.813
1.808
1.800
1.787
1.272
1.262
Current Data Parameters
NAME Jean-01-102-p2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140618
Time 14.29
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 512
DW 59.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 598.7029935 MHz
F2 - Processing parameters
SI 32768
SF 598.7000261 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 4.998 ppm
F1 2992.02 Hz
F2P 1.549 ppm
F2 927.48 Hz
PPMCM 0.17242 ppm/cm
HZCM 103.22723 Hz/cm
N
H
H
O
cis-3l
1.00
1.04
1.10
1.11
1.21
1.46
3.08
1.23
1.01
3.29
1.15
1.17
3.26
9
8
7
6
5
4
3
2
1
0 ppm

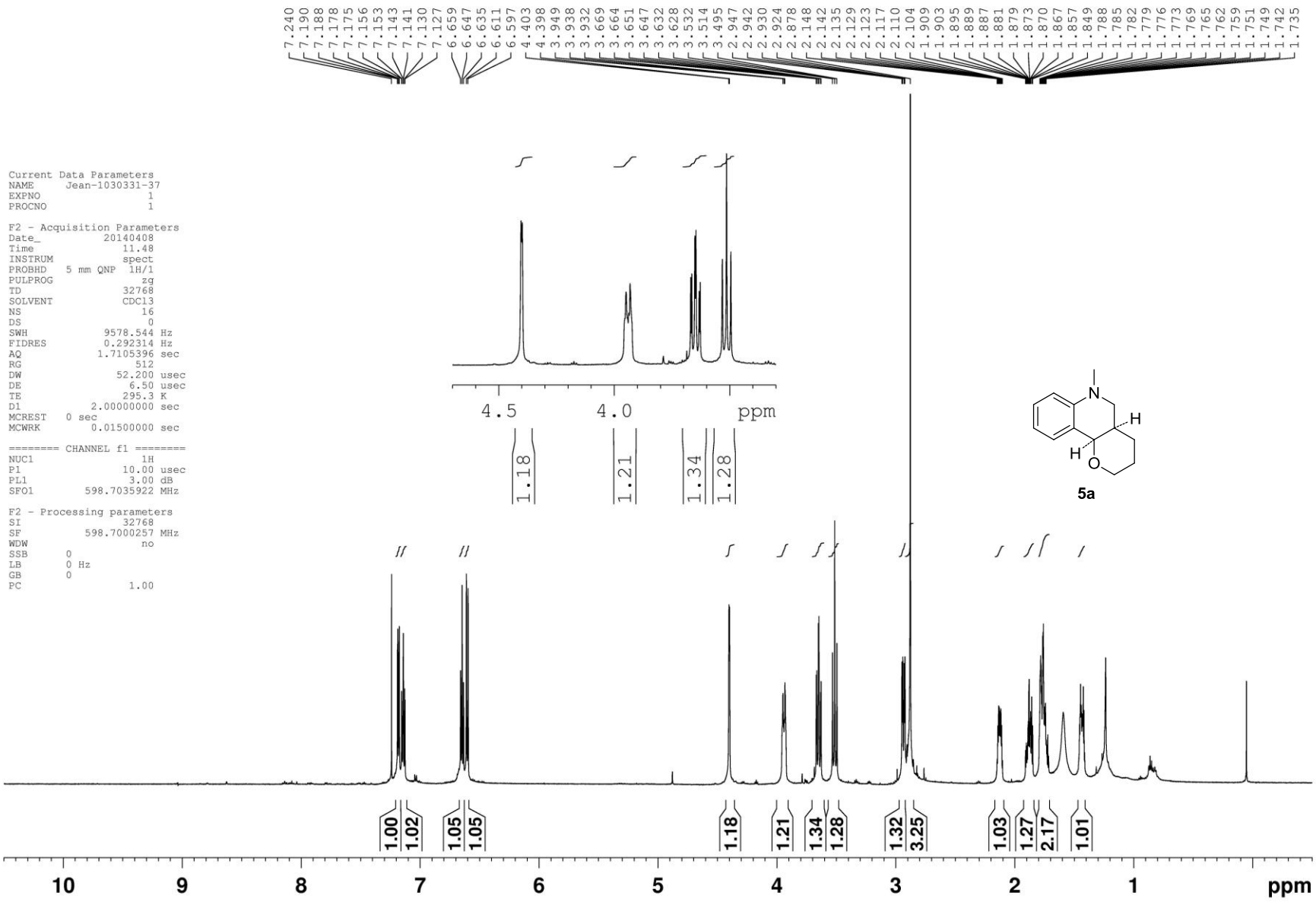

145.15
131.26
129.39
126.92
122.86
111.96
77.21
77.00
76.79
74.85
72.34
53.03
39.58
38.28
37.81
29.68
21.77
20.20
Current Data Parameters
NAME Jean-01-102-p2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date 20140619
Time 4.33
INSTRUM spect
PROBHD 5 mm QNP 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 500
DS 0
SWH 43545.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637768 sec
RG 1.100 usec
DW 6.50 usec
DE 301.5 K
TE 3.30000000 sec
D1 0.03000020 sec
D11 3.40000010 sec
DELTA 0 sec
MCREST 0.01500000 sec
MCWSK
CHANNEL f1
NUC1 13C
P1 4.80 usec
PL1 0 dB
SFO1 150.5597968 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 14.00 dB
SFO2 599.7029935 MHz
F2 Processing parameters
SI 65536
SF 150.5432363 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 0.50
N
H
H
O
cis-3l
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

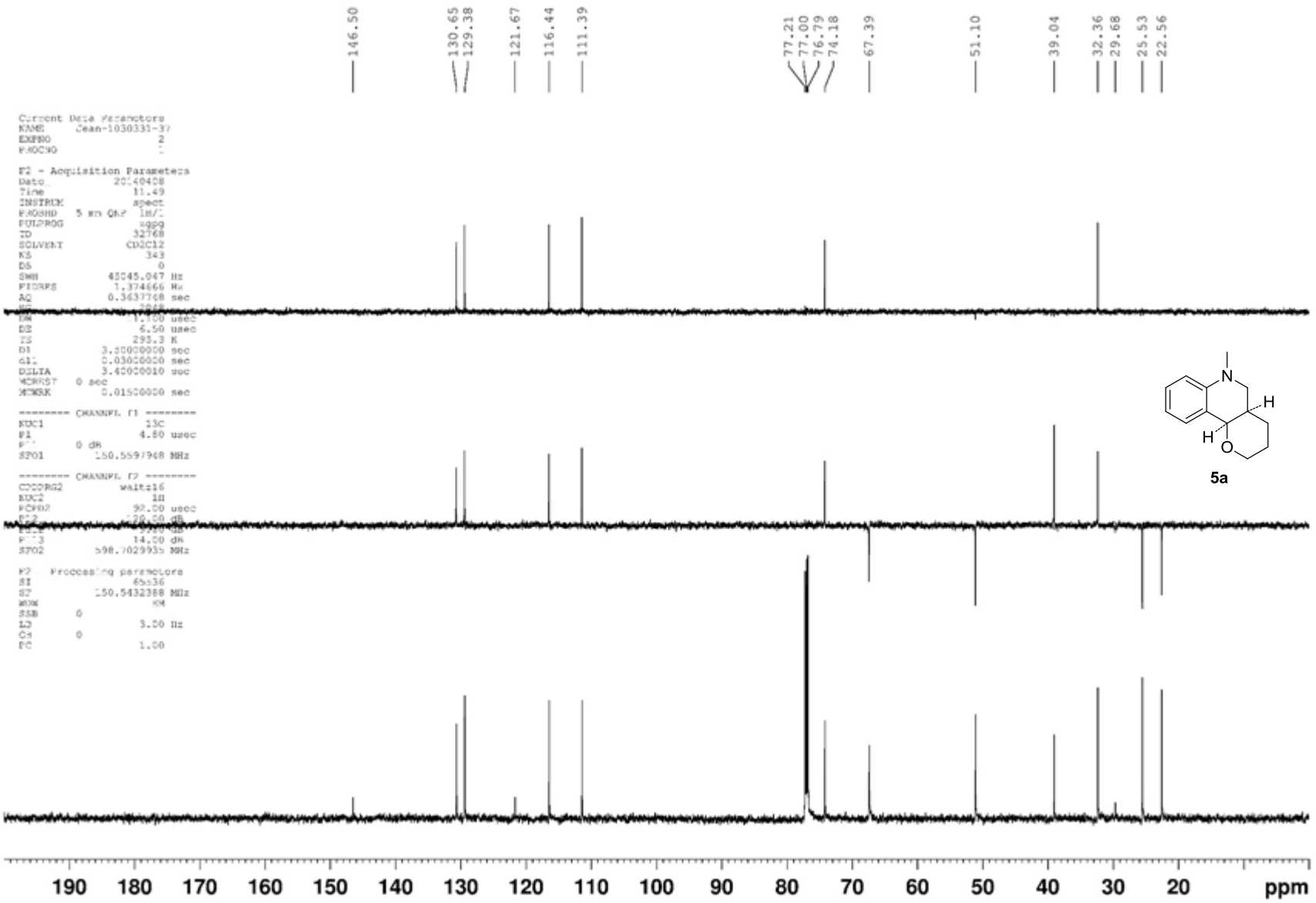

7.2403
7.1290
7.1244
6.9742
6.9691
6.9533
6.9484
6.6013
6.5805
4.7079
4.6943
2.9226
2.9094
2.8949
2.8820
2.8542
2.8355
2.8267
2.8088
2.7816
2.5119
2.5039
2.4982
2.4906
2.4833
2.4774
2.4695
2.4641
2.2218
2.1076
2.0867
2.0755
2.0545
1.7622
1.7492
1.7355
1.7254
1.7031
1.6914
1.6769
1.6671
1.6438
1.6245
1.6114
1.5734
1.5543
1.5443
1.5068
1.4987
1.4926
1.4842
1.4746
1.4667
1.4537
1.4387
1.4252
1.4063
1.3609
1.3365
1.3211
1.2996
1.2842
1.2760
1.2658
1.2355
Current Data Parameters
NAME DBH-E-2-cyclohexTHF-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140715
Time 0.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 47
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 228
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
N
H
H
O
3m
DBH-E-2-cyclohexTHF-1H
9
8
7
6
5
4
3
2
1
ppm
1.07
1.09
1.10
1.00
1.10
4.07
1.00
3.09
1.09
4.34
2.42
3.22
2.64

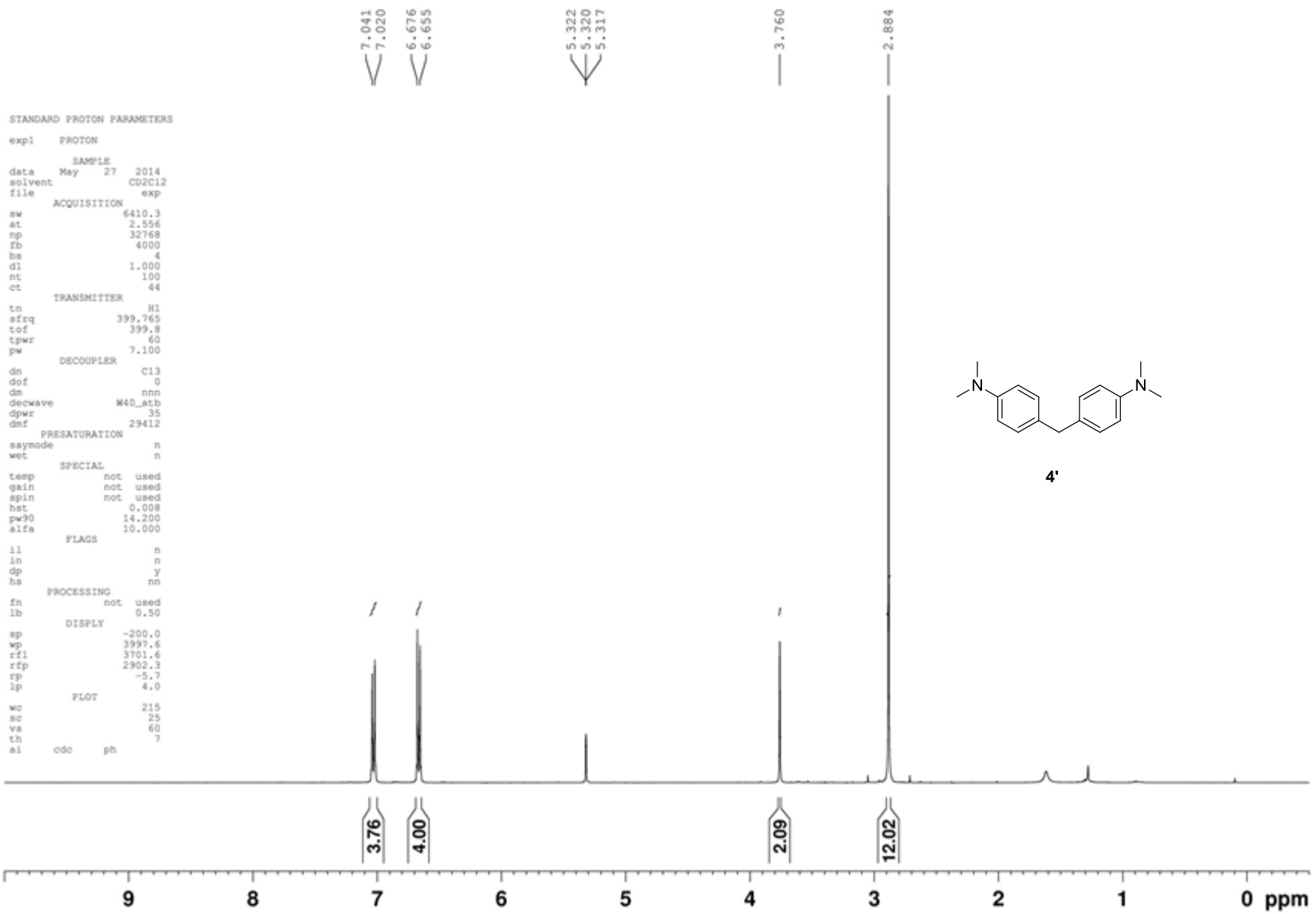

Current Data Parameters
NAME DBH-E-2-cyclohexTHF-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140715
Time 0.20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2600
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178011 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
145.19
131.67
129.39
126.50
122.52
111.98
81.02
77.32
77.00
76.69
73.45
53.39
40.56
39.81
39.49
37.21
37.05
25.65
24.30
23.54
20.23
N
H
H
O
3m
DBH-E-2-cyclohexTHF-13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

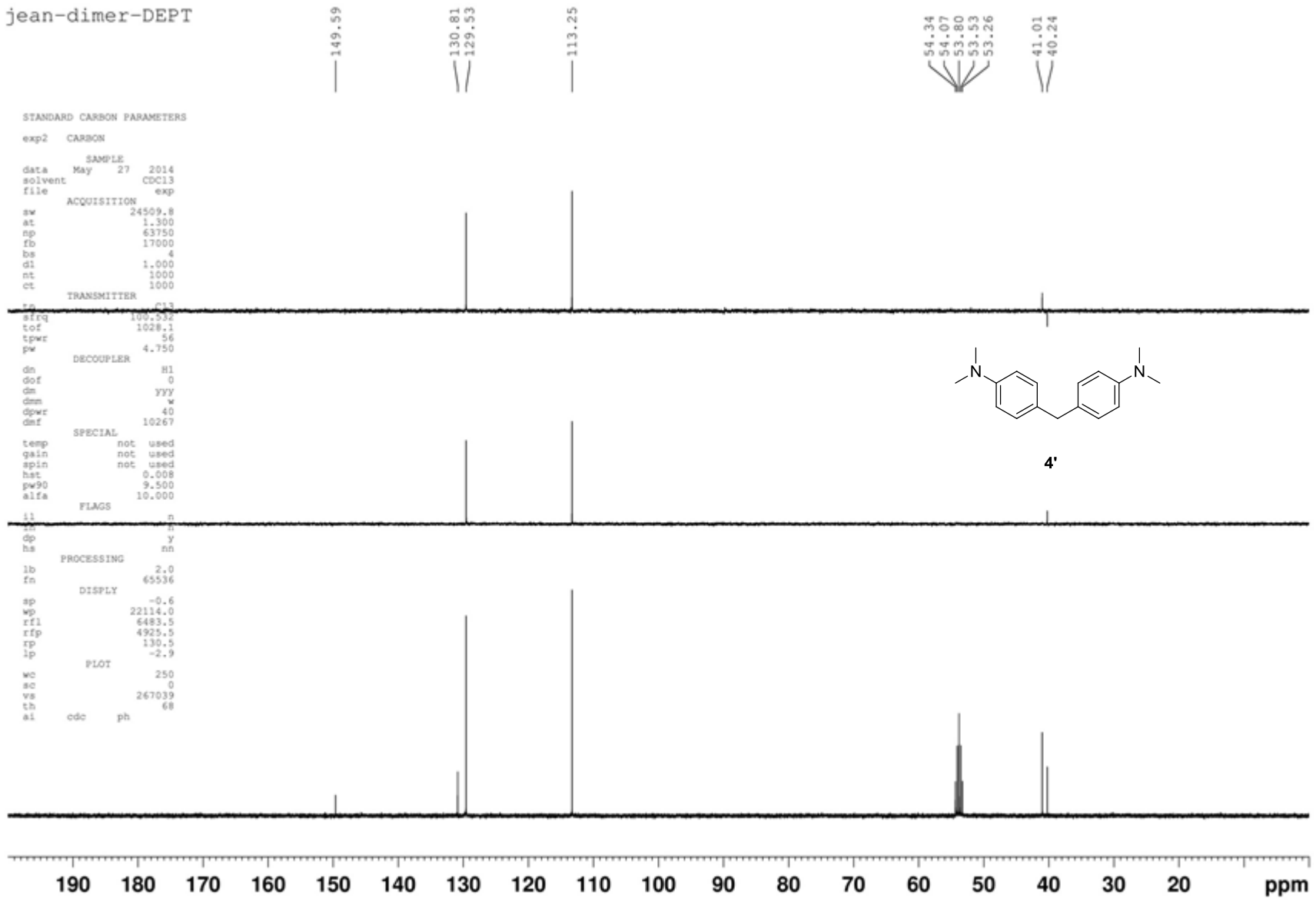

7.2403 7.0961 6.9754 6.9706 6.9543 6.9494 6.6058 6.5850 4.5886 4.5748 2.9354 2.9222 2.9080 2.8948 2.8811 2.8730 2.8541 2.8306 2.8065 2.7795 2.4912 2.4852 2.4781 2.4711 2.4651 2.4575 2.3105 2.3028 2.2678 2.2272 2.2177 2.1954 2.1752 2.0278 2.0033 1.9616 1.9413 1.9340 1.9253 1.9119 1.8540 1.8354 1.8237 1.8065 1.7924 1.7815 1.7740 1.7684 1.7627 1.7558 1.7423 1.7331 1.7248 1.7008 1.6927 1.6867 1.6580 1.6353 1.6265 1.6061 1.5773 1.5537 1.5463 1.5339 1.5140
Current Data Parameters
NAME DBH-E-2-cyclopenfuran-pdt-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140709
Time 20.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 79
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 406
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.589
4.575
4.6
ppm
1.000
DBH-E-2-cyclopenfuran-pdt-1H
N
H
H
O
3n
9.0 8.5 8.0 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 ppm
1.06 1.09 1.07 1.00 5.28 1.16 5.29 2.31 2.23 2.70 2.06 1.47

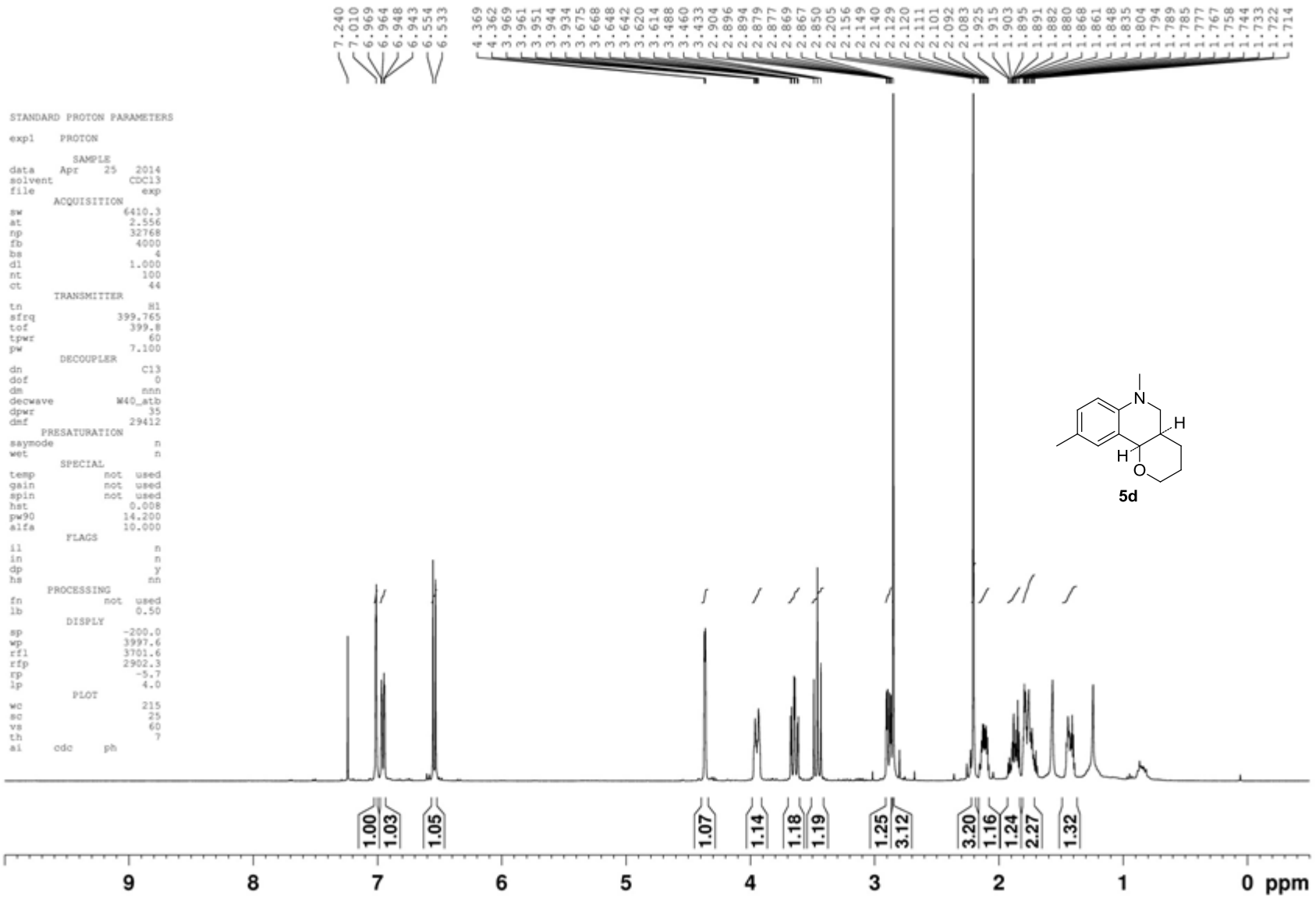

BRUKER
Current Data Parameters
NAME DBH-E-2-cyclopentylTHF-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140711
Time 0.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2600
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178006 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
145.27
131.63
129.50
126.54
122.26
112.02
88.95
77.32
77.00
76.69
74.48
53.36
41.40
39.94
39.52
37.03
24.15
23.95
20.23
N
H
H
O
3n
DBH-E-2-cyclopentylTHF-13C
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

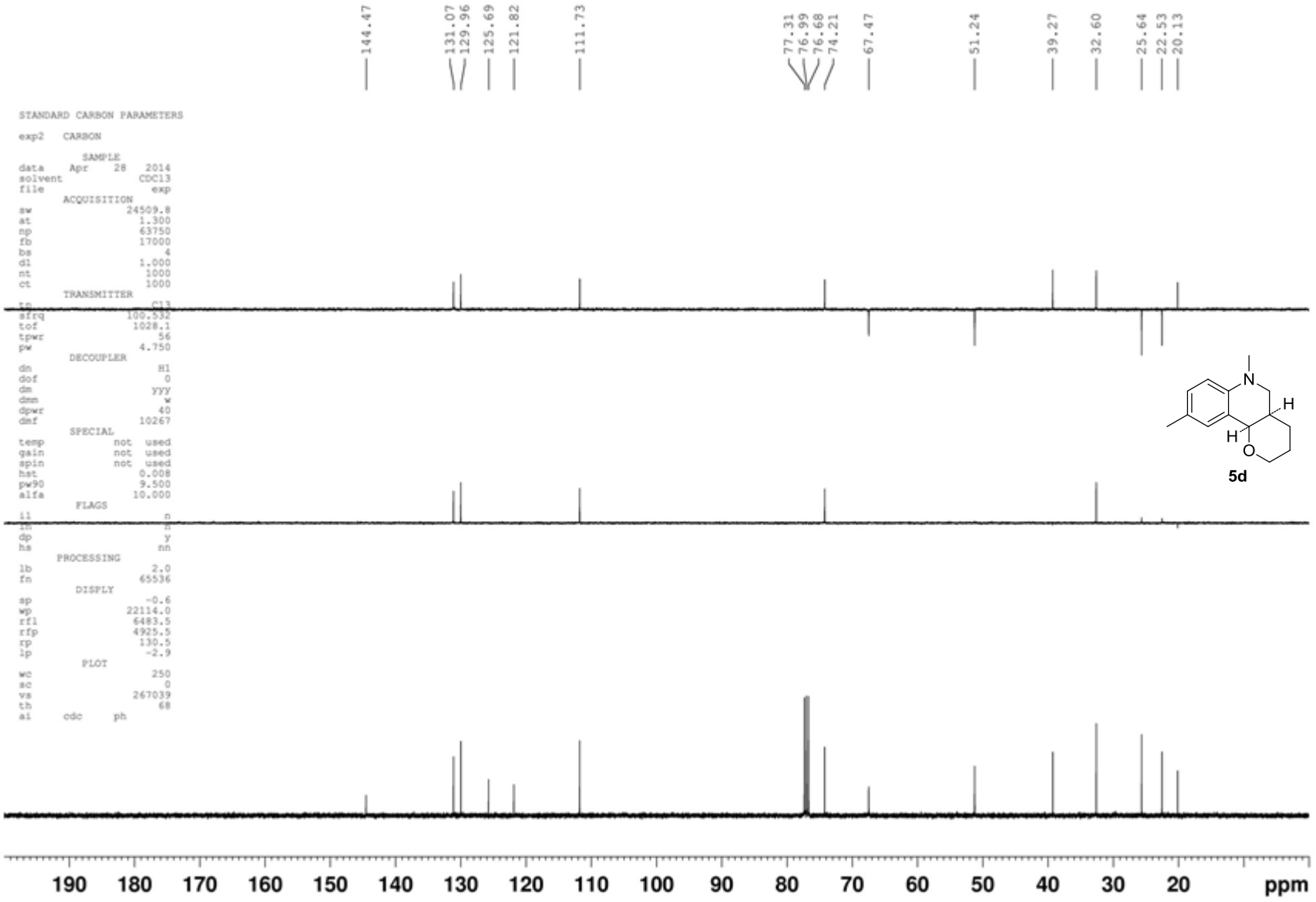

Current Data Parameters
NAME Jean-1030331-37
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140408
Time 11.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9578.544 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 512
DW 52.200 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 598.7035922 MHz
F2 - Processing parameters
SI 32768
SF 598.7000257 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.240 7.190 7.188 7.178 7.175 7.156 7.153 7.143 7.141 7.130 7.127 6.659 6.647 6.635 6.611 6.597 4.403 4.398 3.949 3.938 3.932 3.669 3.664 3.651 3.647 3.632 3.628 3.532 3.514 3.495 2.947 2.942 2.930 2.924 2.878 2.148 2.142 2.135 2.129 2.123 2.117 2.110 2.104 1.909 1.903 1.895 1.889 1.887 1.881 1.879 1.873 1.870 1.867 1.857 1.849 1.788 1.785 1.782 1.779 1.776 1.773 1.769 1.765 1.762 1.759 1.751 1.749 1.742 1.735
4.5
4.0
ppm
1.18
1.21
1.34
1.28
N
H
H
O
5a
1.00
1.02
1.05
1.05
1.18
1.21
1.34
1.28
1.32
3.25
1.03
1.27
2.17
1.01
10
9
8
7
6
5
4
3
2
1
ppm

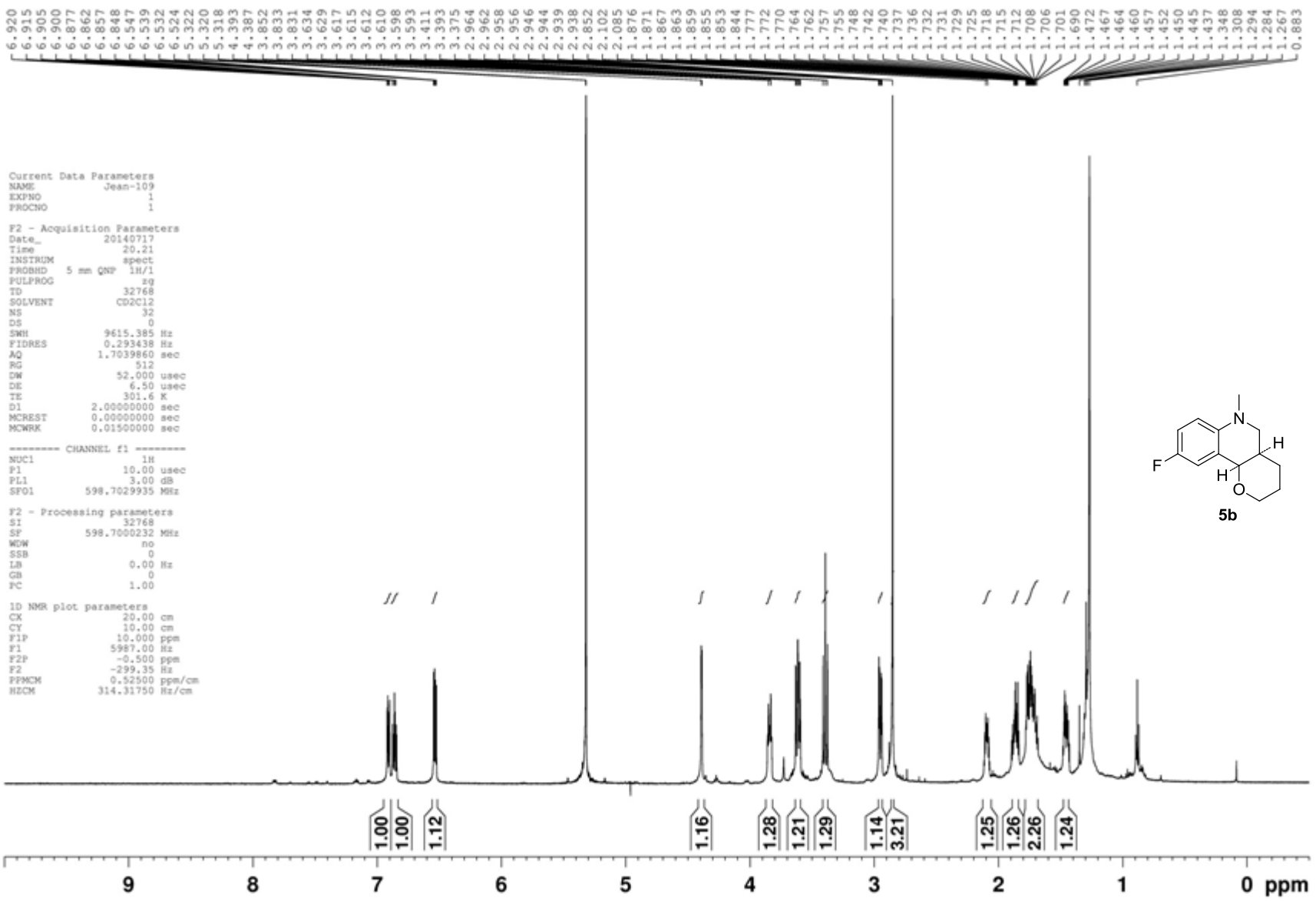

146.50
130.65
129.38
121.67
116.44
111.39
77.21
77.00
76.79
74.18
67.39
51.10
39.04
32.36
29.68
25.53
22.56
N
H
H
O
5a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

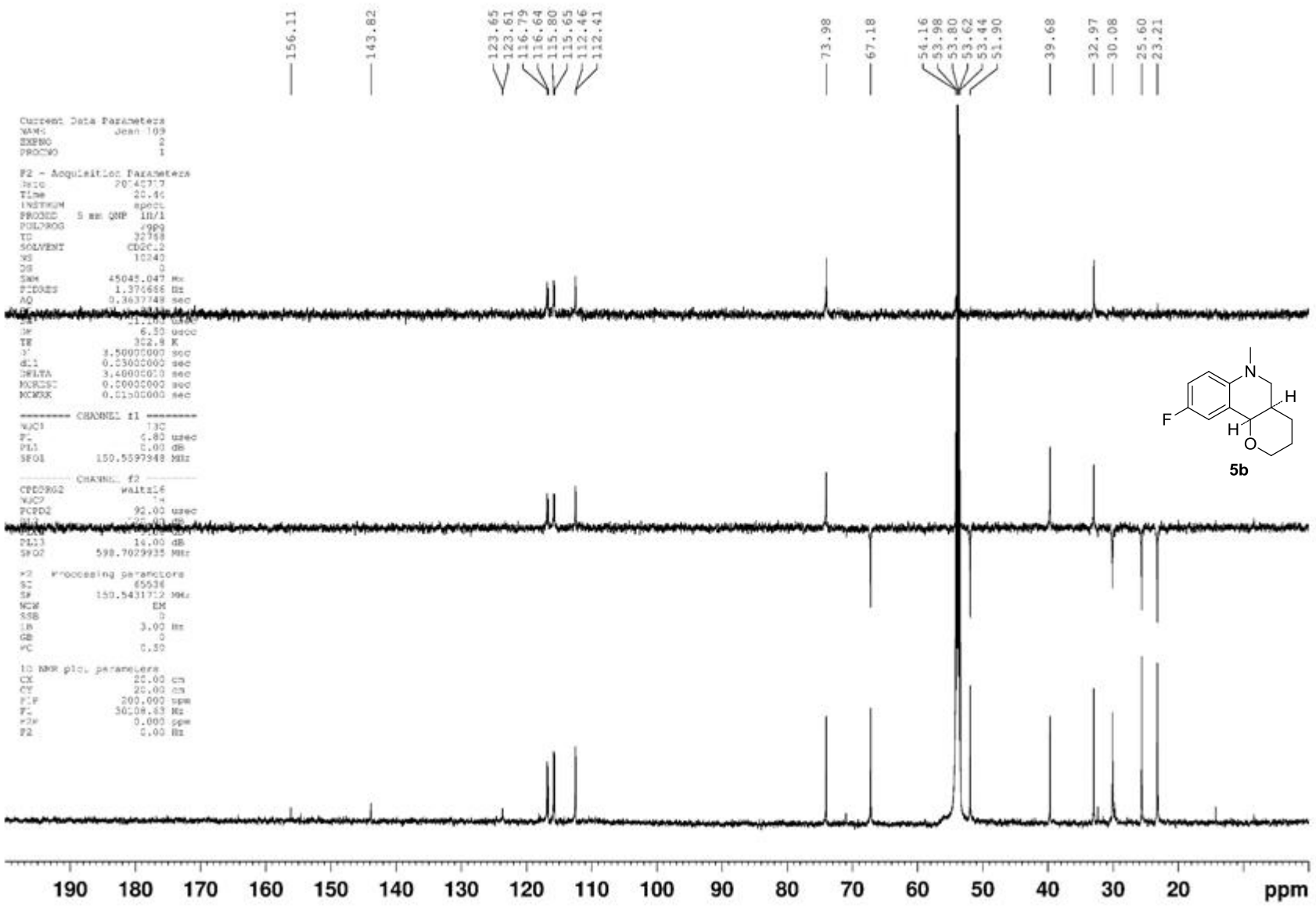

7.041
7.020
6.676
6.655
5.322
5.320
5.317
3.760
2.884
STANDARD PROTON PARAMETERS
expl PROTON
SAMPLE
data May 27 2014
solvent CD2Cl2
file exp
ACQUISITION
sw 6410.3
at 2.556
np 32768
fb 4000
bs 4
d1 1.000
nt 100
ct 44
TRANSMITTER
tn H1
sfrq 399.765
tof 399.8
tpwr 60
pw 7.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_atb
dpwr 35
dmf 29412
PRESATURATION
saymode n
wet n
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 14.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
lb 0.50
DISPLY
sp -200.0
wp 3997.6
rf1 3701.6
rfp 2902.3
rp -5.7
lp 4.0
PLOT
wc 215
sc 25
vs 60
th 7
ai cdc ph
N
N
4'
3.76
4.00
2.09
12.02
9
8
7
6
5
4
3
2
1
0 ppm

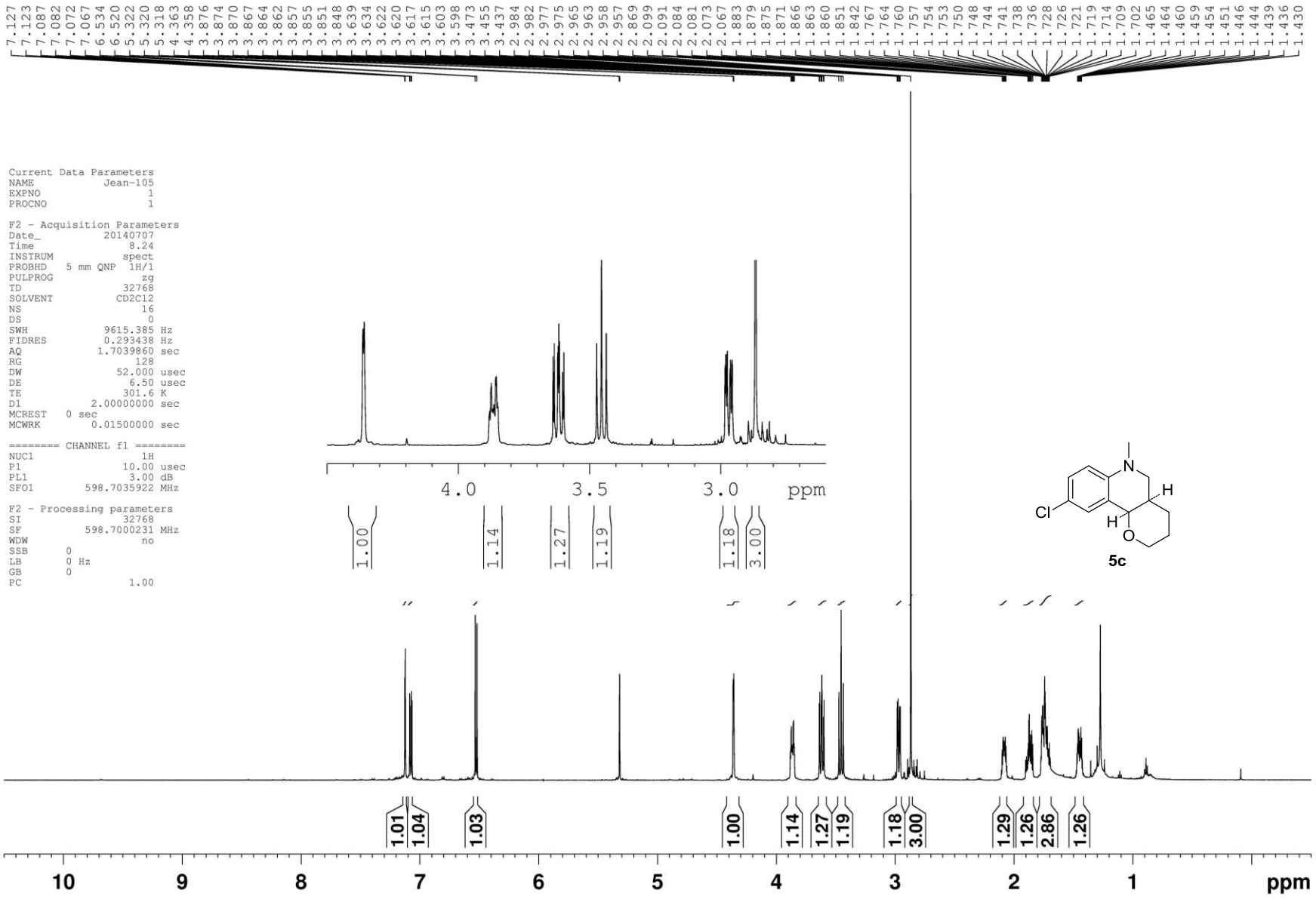

jean-dimer-DEPT
149.59
130.81
129.53
113.25
54.34
54.07
53.80
53.53
53.26
41.01
40.24
STANDARD CARBON PARAMETERS
exp2 CARBON
SAMPLE
data May 27 2014
solvent CDCl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 4
d1 1.000
nt 1000
ct 1000
TRANSMITTER
tof 1028.1
tpwr 56
pw 4.750
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 40
dmf 10267
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
dp y
hs nn
PROCESSING
lb 2.0
fn 65536
DISPLY
sp -0.6
wp 22114.0
rfl 6483.5
rfp 4925.5
rp 130.5
lp -2.9
PLOT
wc 250
sc 0
vs 267039
th 68
ai cdc ph
N
N
4'
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

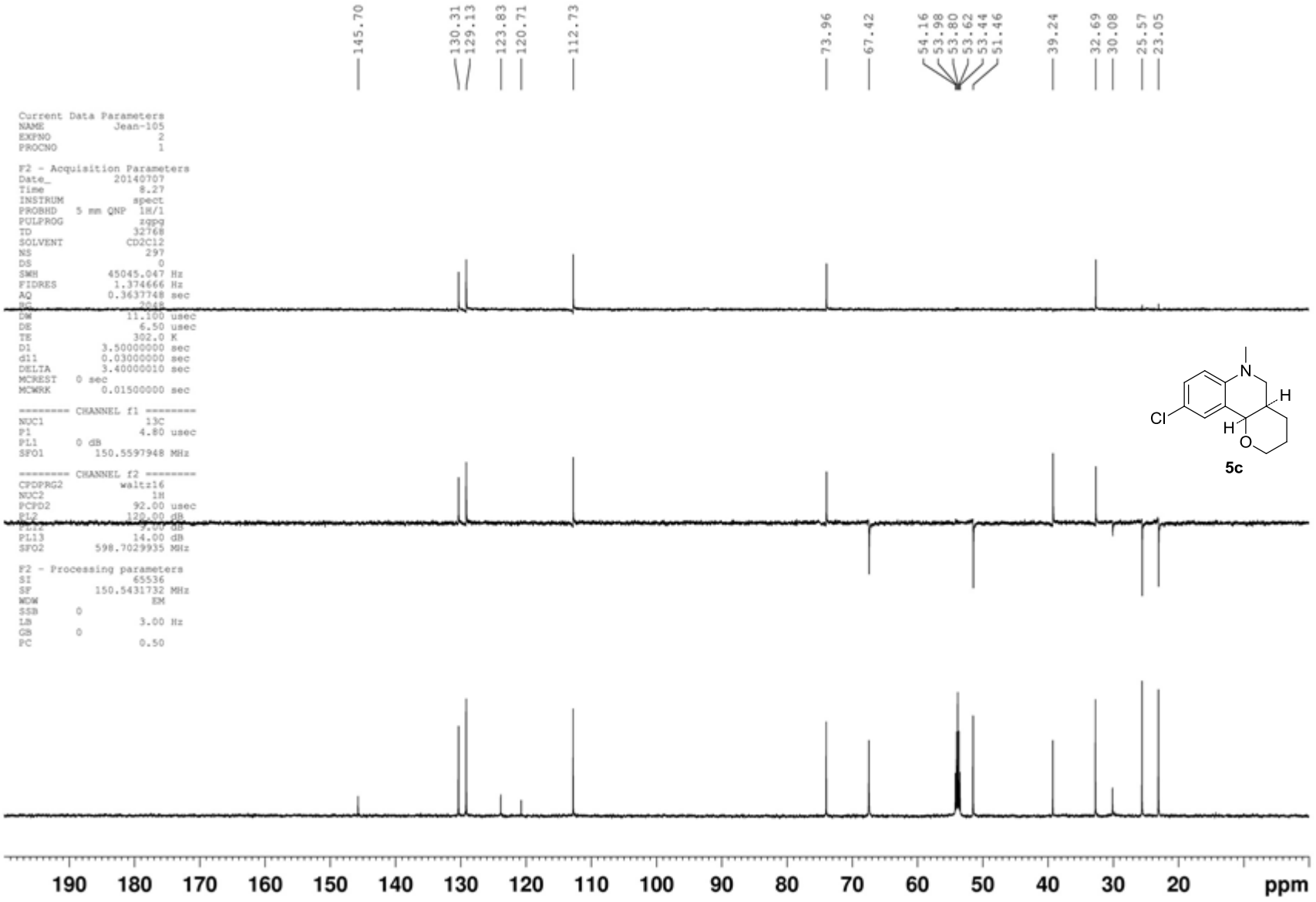

7.240
7.010
6.969
6.964
6.948
6.943
6.554
6.533
4.369
4.362
3.969
3.961
3.951
3.944
3.934
3.675
3.668
3.648
3.642
3.620
3.614
3.488
3.460
3.433
2.904
2.896
2.894
2.879
2.877
2.869
2.867
2.850
2.205
2.156
2.149
2.140
2.129
2.120
2.111
2.101
2.092
2.083
1.925
1.915
1.903
1.895
1.891
1.882
1.880
1.868
1.861
1.848
1.835
1.804
1.794
1.789
1.785
1.777
1.767
1.758
1.744
1.733
1.722
1.714
STANDARD PROTON PARAMETERS
expl PROTON
SAMPLE
data Apr 25 2014
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 2.556
np 32768
fb 4000
bs 4
d1 1.000
nt 100
ct 44
TRANSMITTER
tn H1
sfrq 399.765
tof 399.8
tpwr 60
pw 7.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_atb
dpwr 35
dmf 29412
PRESATURATION
saymode n
wet n
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 14.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
lb 0.50
DISPLY
sp -200.0
wp 3997.6
rfl 3701.6
rfp 2902.3
rp -5.7
lp 4.0
PLOT
wc 215
sc 25
vs 60
th 7
ai cdc ph
N
H
H
O
5d
1.00
1.03
1.05
1.07
1.14
1.18
1.19
1.25
3.12
3.20
1.16
1.24
2.27
1.32
9
8
7
6
5
4
3
2
1
0 ppm

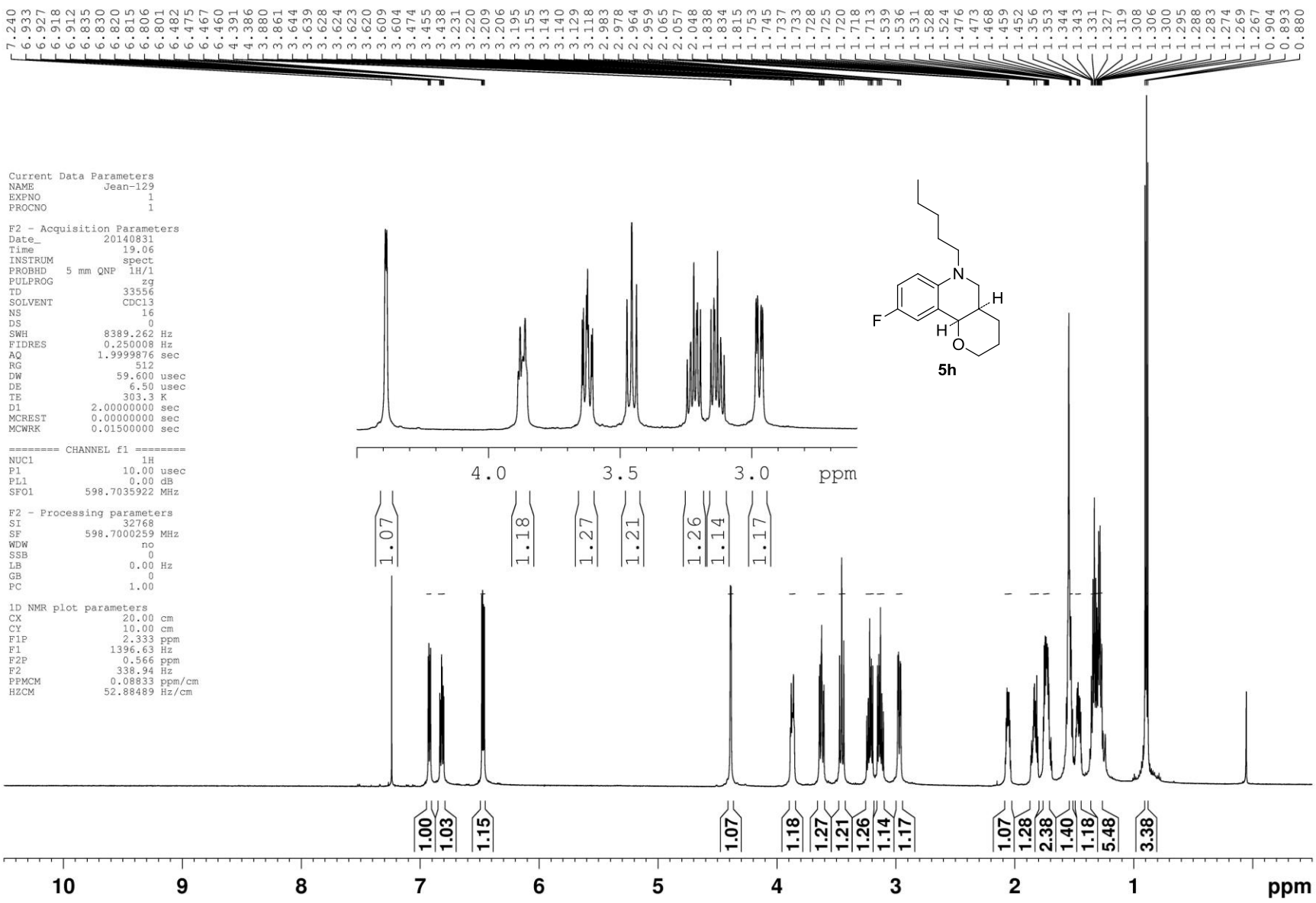

144.47
131.07
129.96
125.69
121.82
111.73
77.31
76.99
76.68
74.21
67.47
51.24
39.27
32.60
25.64
22.53
20.13
STANDARD CARBON PARAMETERS
exp2 CARBON
SAMPLE
data Apr 28 2014
solvent CDCl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 4
d1 1.000
nt 1000
ct 1000
TRANSMITTER
tn C13
sfrq 100.532
tof 1028.1
tpwr 56
pw 4.750
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 40
dmf 10267
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
il n
dp y
hs nn
PROCESSING
lb 2.0
fn 65536
DISPLY
sp -0.6
wp 22114.0
rfl 6483.5
rfp 4925.5
rp 130.5
lp -2.9
PLOT
wc 250
sc 0
vs 267039
th 68
ai cdc ph
N
H
H
O
5d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

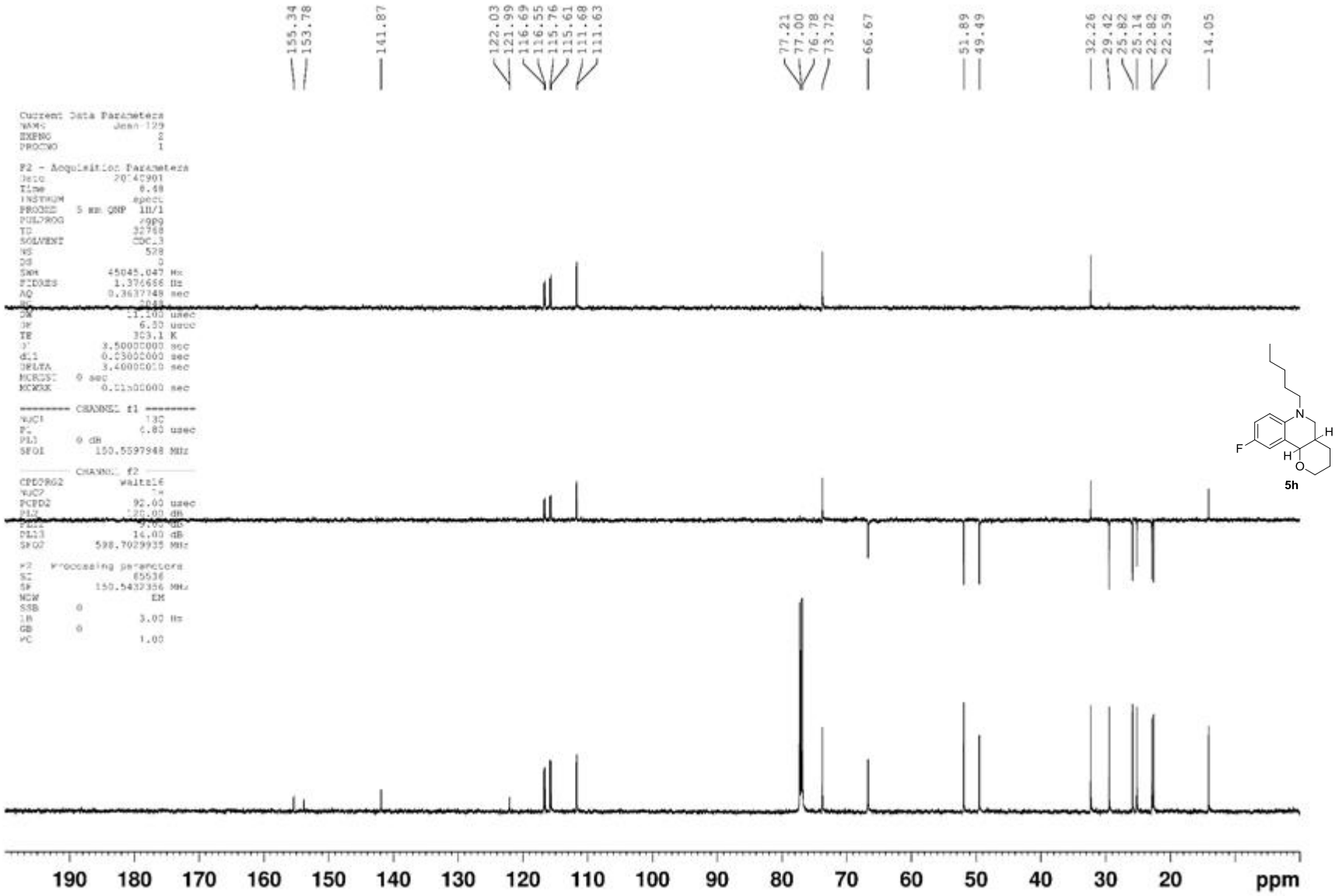

6.920 6.915 6.905 6.900 6.877 6.862 6.857 6.848 6.547 6.539 6.532 6.524 5.322 5.320 5.318 4.393 4.387 3.852 3.833 3.831 3.634 3.629 3.617 3.615 3.612 3.610 3.598 3.593 3.411 3.393 3.375 2.964 2.962 2.958 2.956 2.946 2.944 2.939 2.938 2.852 2.102 2.085 1.876 1.871 1.867 1.863 1.859 1.855 1.853 1.844 1.777 1.772 1.770 1.764 1.762 1.757 1.755 1.748 1.742 1.740 1.737 1.736 1.732 1.731 1.729 1.725 1.718 1.715 1.712 1.708 1.706 1.701 1.690 1.472 1.467 1.464 1.460 1.457 1.452 1.450 1.445 1.437 1.348 1.308 1.294 1.284 1.267 0.883
Current Data Parameters
NAME Jean-109
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140717
Time 20.21
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CD2C12
NS 32
DS 0
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 512
DW 52.000 usec
DE 6.50 usec
TE 301.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 598.7029935 MHz
F2 - Processing parameters
SI 32768
SF 598.7000232 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 5987.00 Hz
F2P -0.500 ppm
F2 -299.35 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.31750 Hz/cm
1.00 1.00 1.12 1.16 1.28 1.21 1.29 1.14 3.21 1.25 1.26 2.26 1.24
9 8 7 6 5 4 3 2 1 0 ppm
F
N
H
H
O
5b

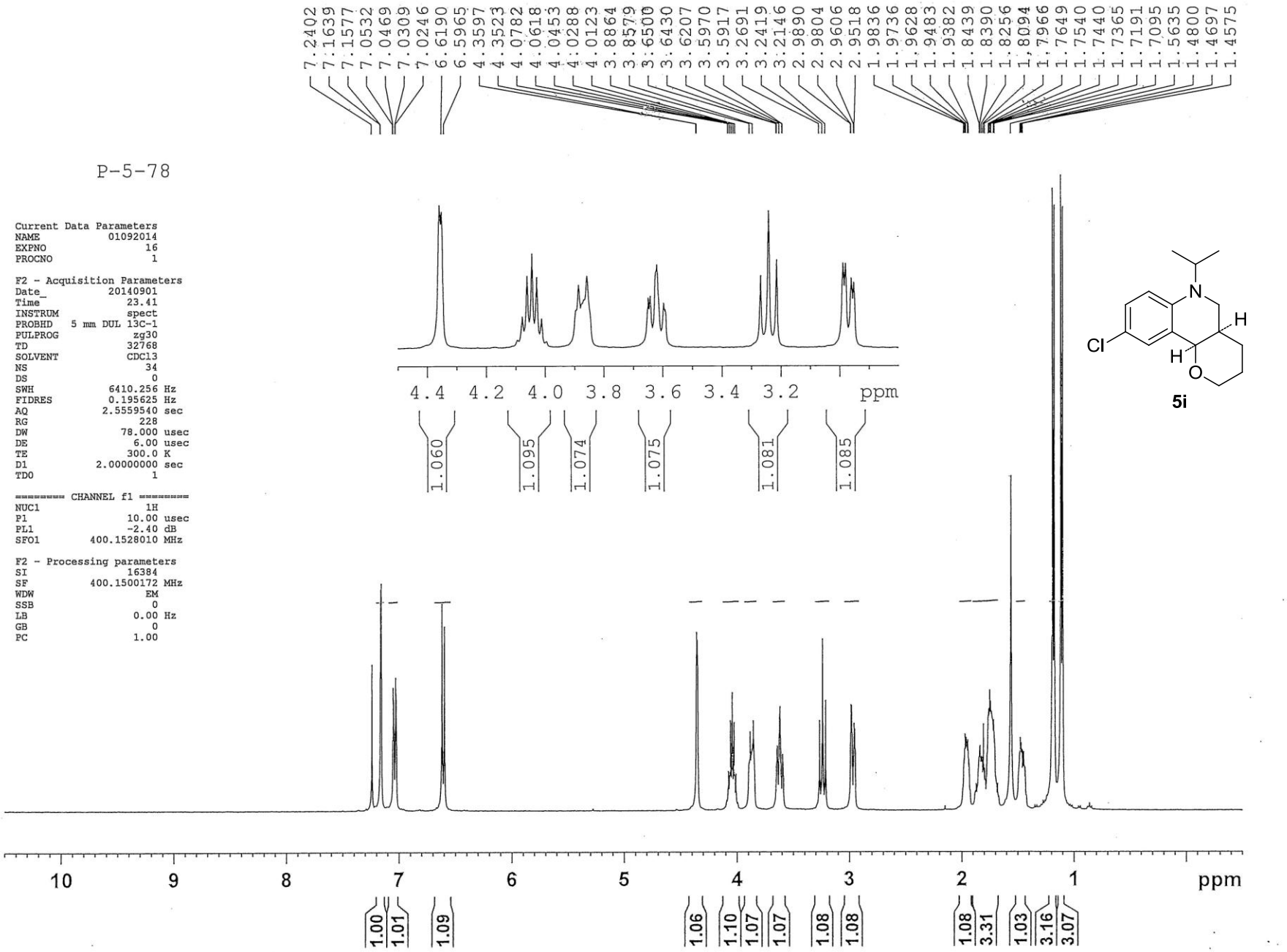

156.11
143.82
123.65
123.61
116.79
116.64
115.80
115.65
112.46
112.41
73.98
67.18
54.16
53.98
53.80
53.62
53.44
51.90
39.68
32.97
30.08
25.60
23.21
F
N
H
H
O
5b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

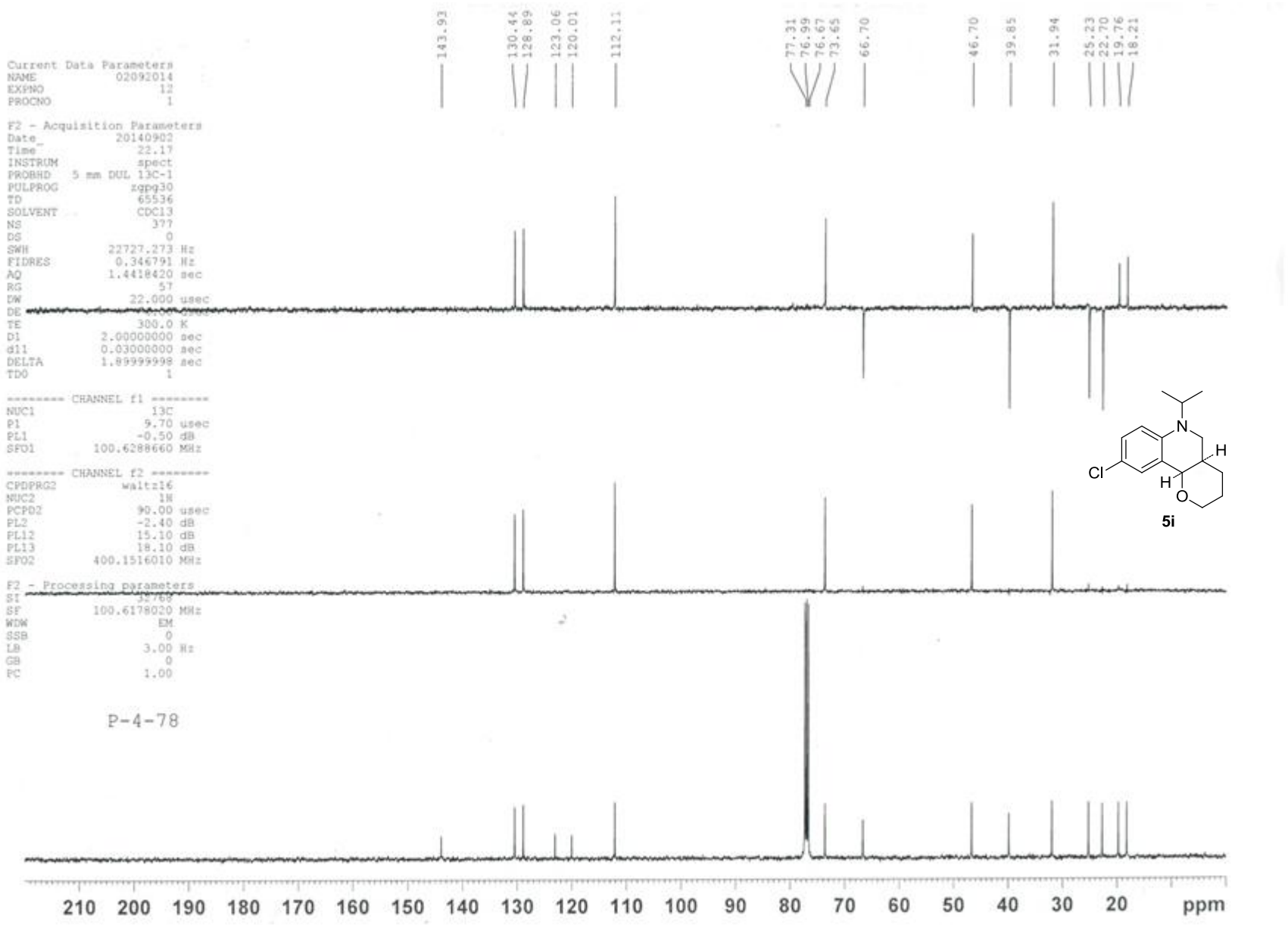

7.127 7.123 7.087 7.082 7.072 7.067 6.534 6.520 5.322 5.320 5.318 4.363 4.358 3.876 3.874 3.870 3.867 3.864 3.862 3.857 3.855 3.851 3.848 3.639 3.634 3.622 3.620 3.617 3.615 3.603 3.598 3.473 3.455 3.437 2.984 2.982 2.977 2.975 2.965 2.963 2.958 2.957 2.869 2.099 2.091 2.084 2.081 2.073 2.067 1.883 1.879 1.875 1.871 1.866 1.863 1.860 1.851 1.842 1.767 1.764 1.760 1.757 1.754 1.753 1.750 1.748 1.744 1.741 1.738 1.736 1.728 1.726 1.721 1.719 1.714 1.709 1.702 1.465 1.464 1.460 1.459 1.454 1.451 1.446 1.444 1.439 1.436 1.430
Current Data Parameters
NAME Jean-105
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140707
Time 8.24
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CD2C12
NS 16
DS 0
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 128
DW 52.000 usec
DE 6.50 usec
TE 301.6 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 598.7035922 MHz
F2 - Processing parameters
SI 32768
SF 598.7000231 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
4.0
3.5
3.0
ppm
1.00
1.14
1.27
1.19
1.18
3.00
N
H
Cl
H
O
5c
1.01
1.04
1.03
1.00
1.14
1.27
1.19
1.18
3.00
1.29
1.26
2.86
1.26
10
9
8
7
6
5
4
3
2
1
ppm

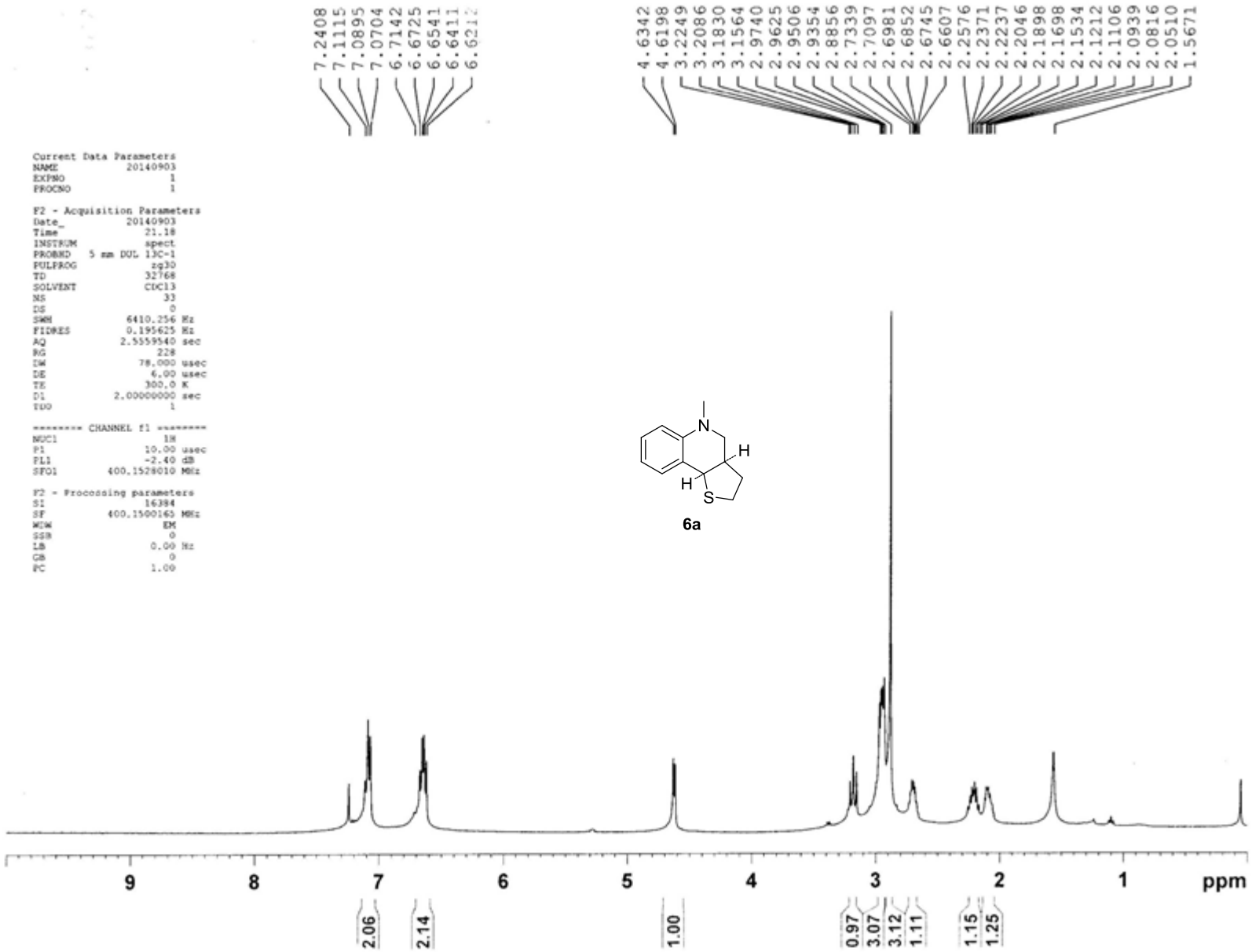

145.70
130.31
129.13
123.83
120.71
112.73
73.96
67.42
54.16
53.98
53.80
53.62
53.44
51.46
39.24
32.69
30.08
25.57
23.05
Current Data Parameters
NAME Jean-105
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140707
Time 8.27
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CD2Cl2
NS 297
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 302.0 K
D1 3.50000000 sec
d11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.80 usec
PL1 0 dB
SFO1 150.5597948 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 598.7029935 MHz
F2 - Processing parameters
SI 65536
SF 150.5431732 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 0.50
Cl
N
H
H
O
5c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

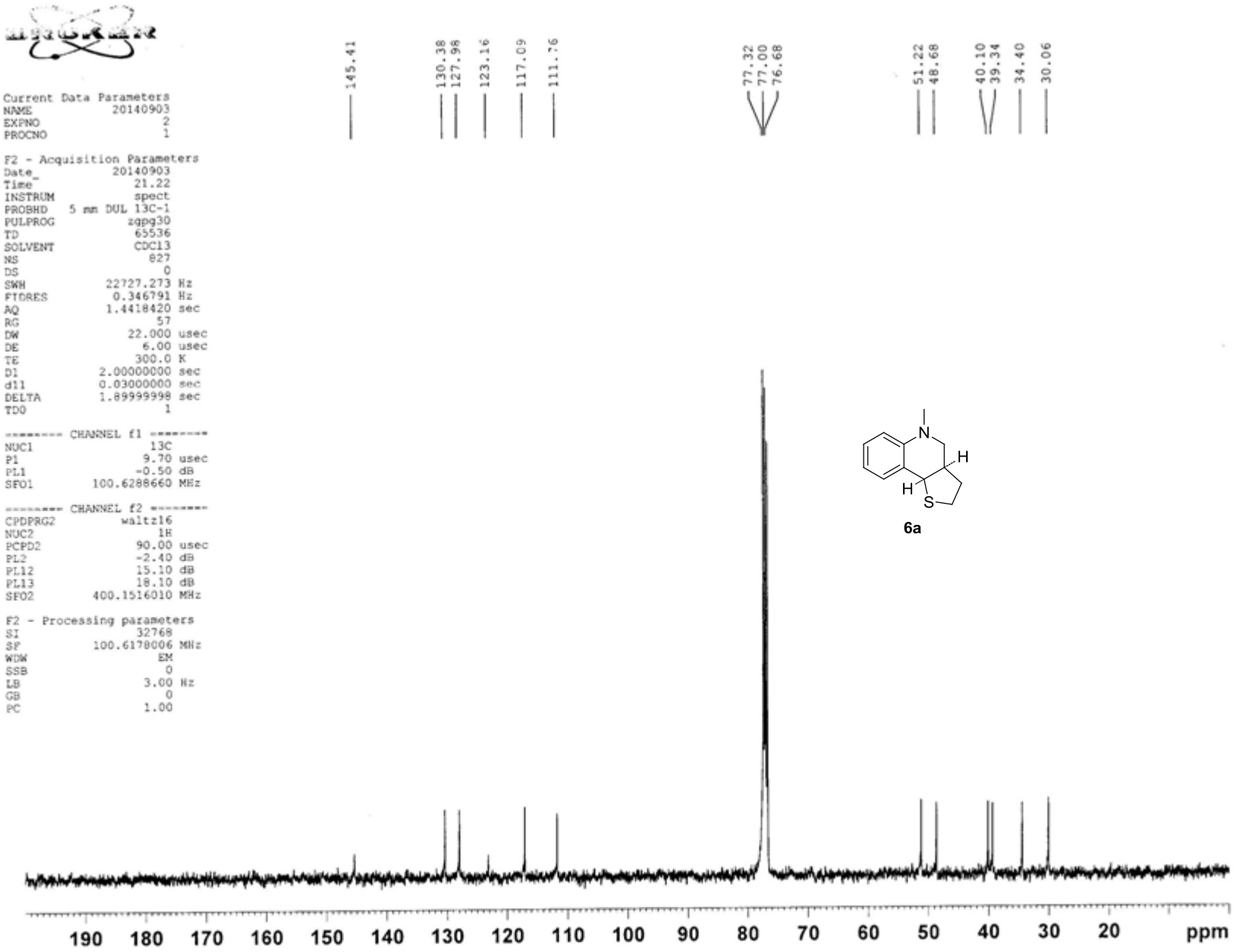

7.240 6.933 6.927 6.918 6.912 6.835 6.830 6.820 6.815 6.806 6.801 6.482 6.475 6.467 6.460 4.391 4.386 3.880 3.861 3.644 3.639 3.628 3.624 3.623 3.620 3.609 3.604 3.474 3.455 3.438 3.231 3.220 3.209 3.206 3.195 3.155 3.143 3.140 3.129 3.118 2.983 2.978 2.964 2.959 2.065 2.057 2.048 1.838 1.834 1.815 1.753 1.745 1.737 1.733 1.728 1.725 1.720 1.718 1.713 1.539 1.536 1.531 1.528 1.524 1.476 1.473 1.468 1.459 1.452 1.356 1.353 1.344 1.343 1.331 1.327 1.319 1.308 1.306 1.300 1.295 1.288 1.283 1.274 1.269 1.267 0.904 0.893 0.880
Current Data Parameters
NAME Jean-129
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140831
Time 19.06
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 33556
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.250008 Hz
AQ 1.9999876 sec
RG 512
DW 59.600 usec
DE 6.50 usec
TE 303.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 598.7035922 MHz
F2 - Processing parameters
SI 32768
SF 598.7000259 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 2.333 ppm
F1 1396.63 Hz
F2P 0.566 ppm
F2 338.94 Hz
PPMCM 0.08833 ppm/cm
HZCM 52.88489 Hz/cm
4.0
3.5
3.0
ppm
1.07
1.18
1.27
1.21
1.26
1.14
1.17
N
H
F
H
O
5h
1.00
1.03
1.15
1.07
1.18
1.27
1.21
1.26
1.14
1.17
1.07
1.28
2.38
1.40
1.18
5.48
3.38
10
9
8
7
6
5
4
3
2
1
ppm

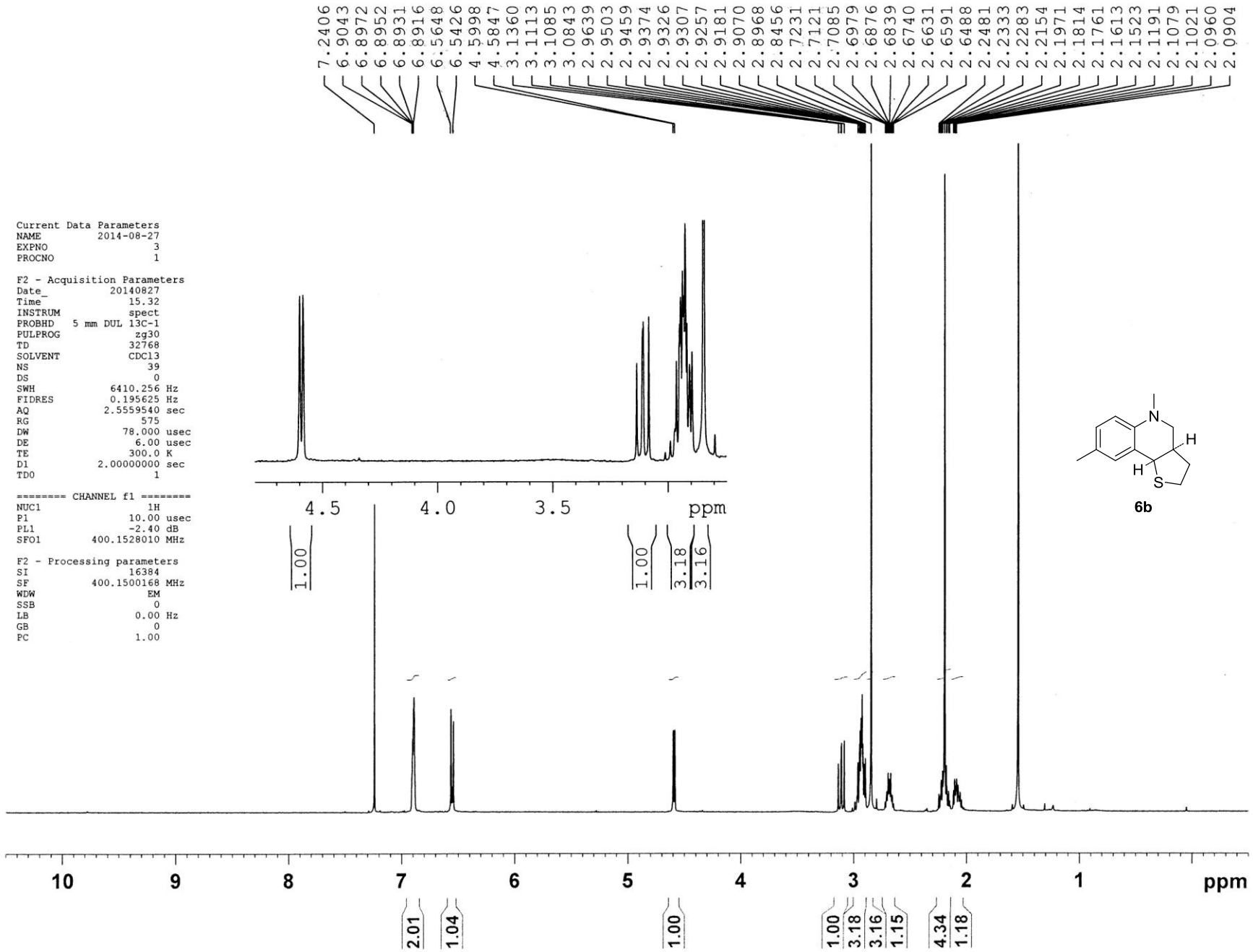

155.34
153.78
141.87
122.03
121.99
116.69
116.55
115.76
115.61
111.68
111.63
77.21
77.00
76.78
73.72
66.67
51.89
49.49
32.26
29.42
25.82
25.14
22.82
22.59
14.05
Current Data Parameters
NAME Jean-129
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date 20140901
Time 8.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 528
DS 0
SWH 45045.047 Hz
FIDRES 1.374668 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 303.1 K
D1 3.50000000 sec
d11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.80 usec
PL1 0 dB
SFO1 150.5597948 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 598.7029935 MHz
F2 - Processing parameters
SI 65536
SF 150.5432356 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
F
H
H
N
O
5h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

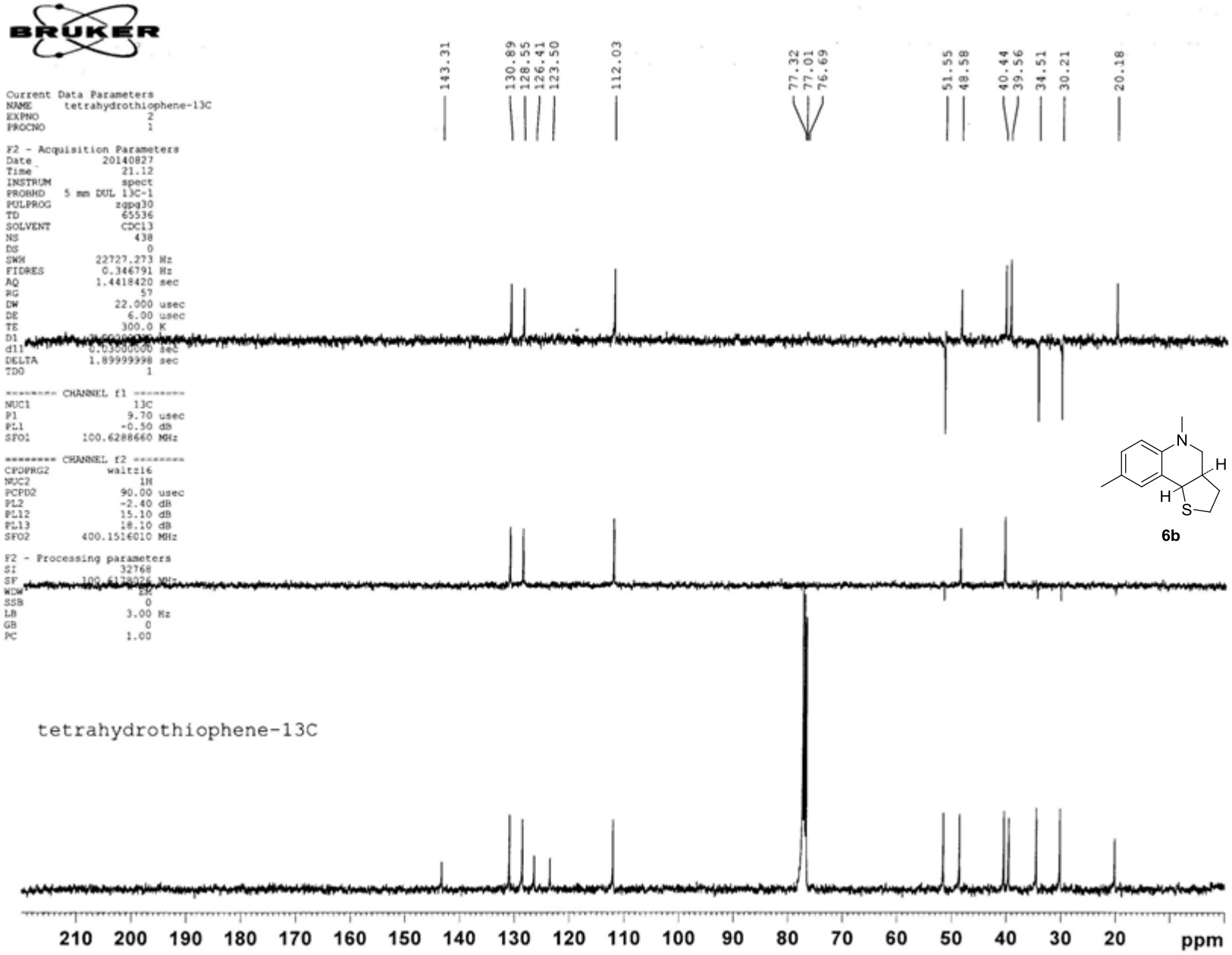

7.2402
7.1639
7.1577
7.0532
7.0469
7.0309
7.0246
6.6190
6.5965
4.3597
4.3523
4.0782
4.0618
4.0453
4.0288
4.0123
3.8864
3.8579
3.6500
3.6430
3.6207
3.5970
3.5917
3.2691
3.2419
3.2146
2.9890
2.9804
2.9606
2.9518
1.9836
1.9736
1.9628
1.9483
1.9382
1.8439
1.8390
1.8256
1.8094
1.7966
1.7649
1.7540
1.7440
1.7365
1.7191
1.7095
1.5635
1.4800
1.4697
1.4575
P-5-78
Current Data Parameters
NAME 01092014
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140901
Time 23.41
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 34
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 228
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.4
4.2
4.0
3.8
3.6
3.4
3.2
ppm
1.060
1.095
1.074
1.075
1.081
1.085
Cl
N
H
H
O
5i
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.09
1.06
1.10
1.07
1.07
1.08
1.08
1.08
3.31
1.03
3.16
3.07

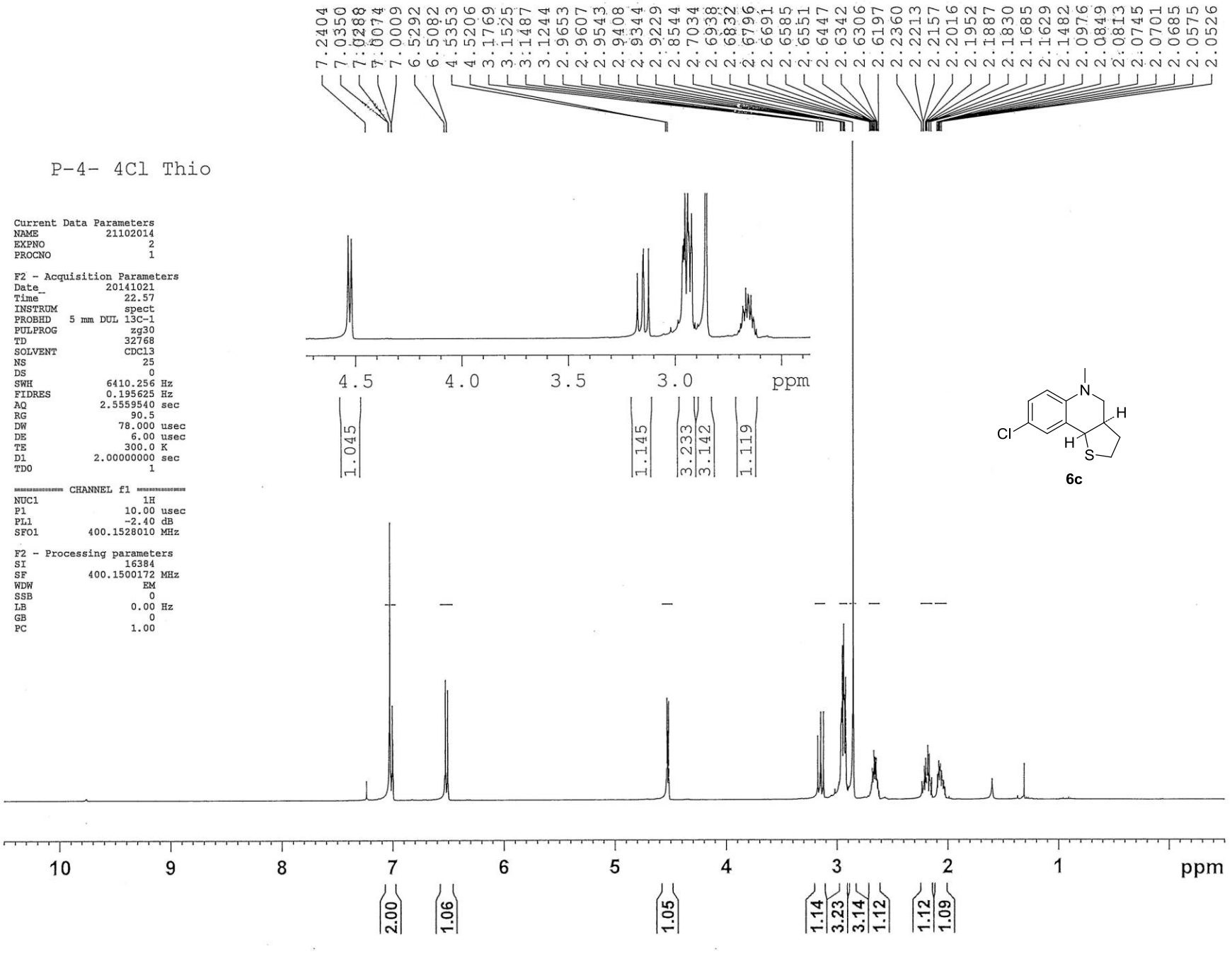

143.93
130.44
128.89
123.06
120.01
112.11
77.31
76.99
76.67
73.65
66.70
46.70
39.85
31.94
25.23
22.70
19.76
18.21
Current Data Parameters
NAME 02092014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140902
Time 22.17
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 377
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178020 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-4-78
N
H
Cl
H
O
5i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

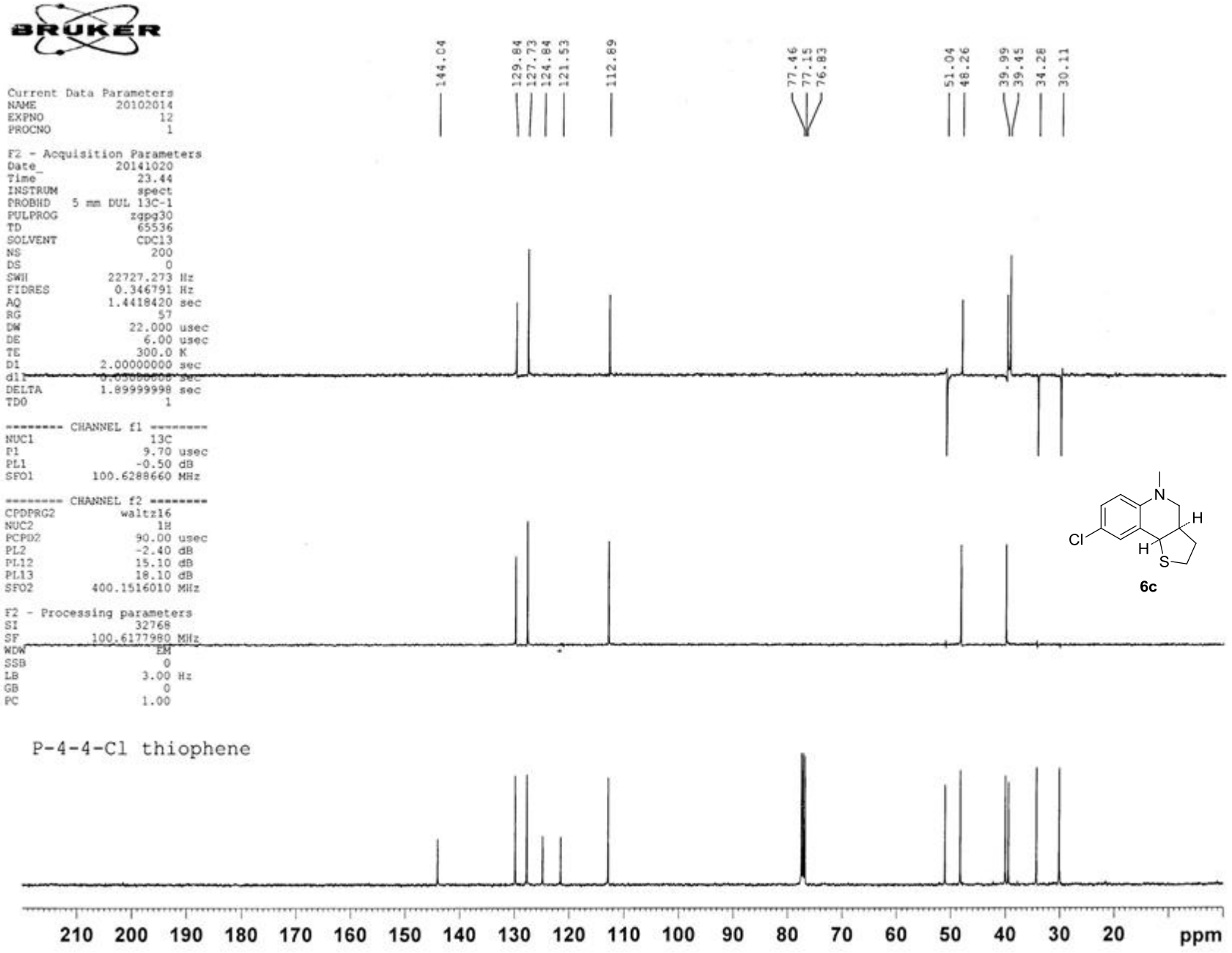

7.2408
7.1115
7.0895
7.0704
6.7142
6.6725
6.6541
6.6411
4.6342
4.6198
3.2249
3.2086
3.1830
3.1564
2.9740
2.9625
2.9506
2.9354
2.8856
2.7339
2.7097
2.6981
2.6852
2.6745
2.6607
2.2576
2.2371
2.2237
2.2046
2.1898
2.1698
2.1534
2.1212
2.1106
2.0939
2.0816
2.0510
1.5671
Current Data Parameters
NAME 20140903
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140903
Time 21.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 228
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500165 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
N
H
H
S
6a
9
8
7
6
5
4
3
2
1
ppm
2.06
2.14
1.00
0.97
3.07
3.12
1.11
1.15
1.25

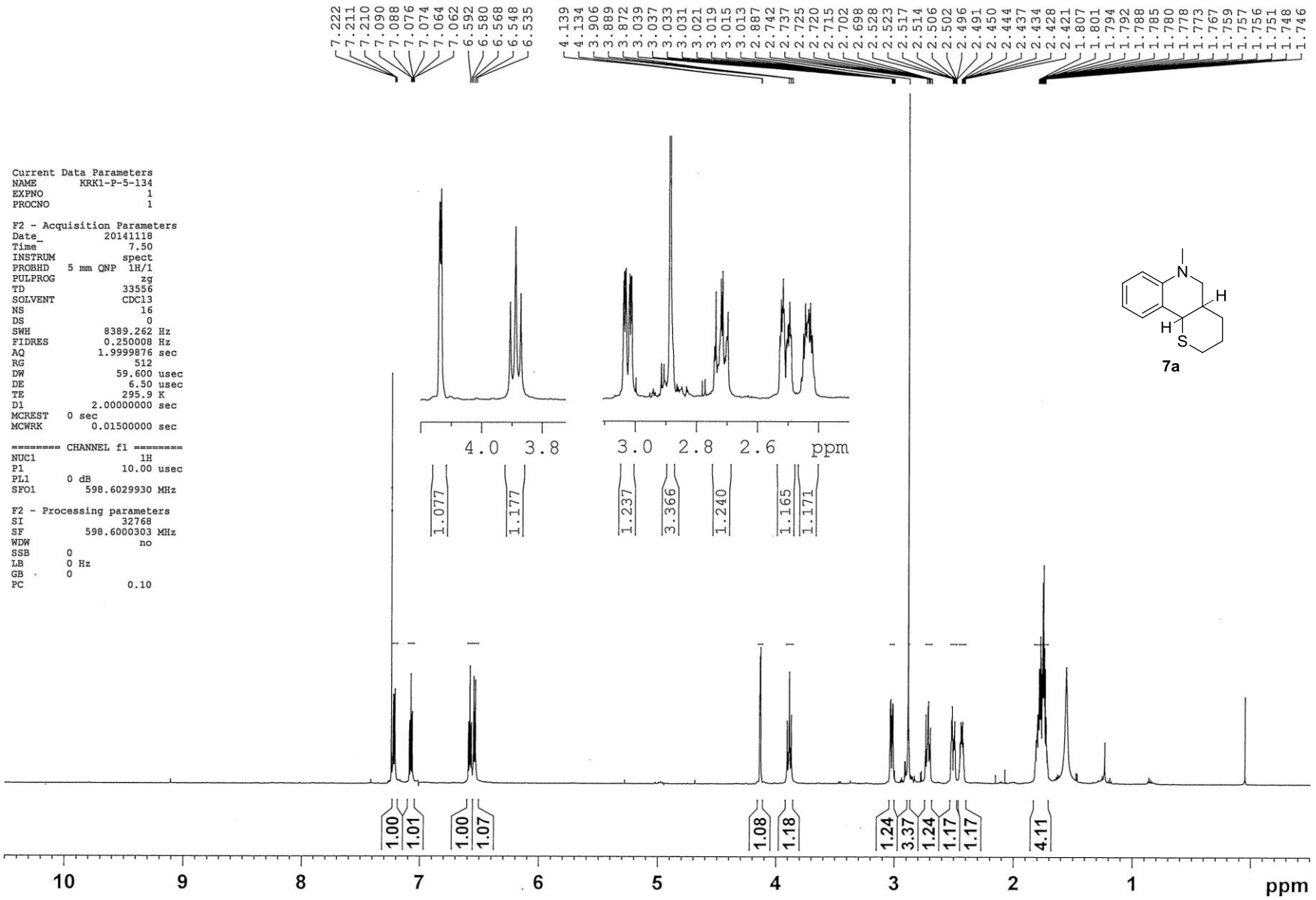

Current Data Parameters
NAME 20140903
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140903
Time 21.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 827
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178006 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
145.41
130.38
127.96
123.16
117.09
111.76
77.32
77.00
76.68
51.22
48.68
40.10
39.34
34.40
30.06
N
H
H
S
6a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

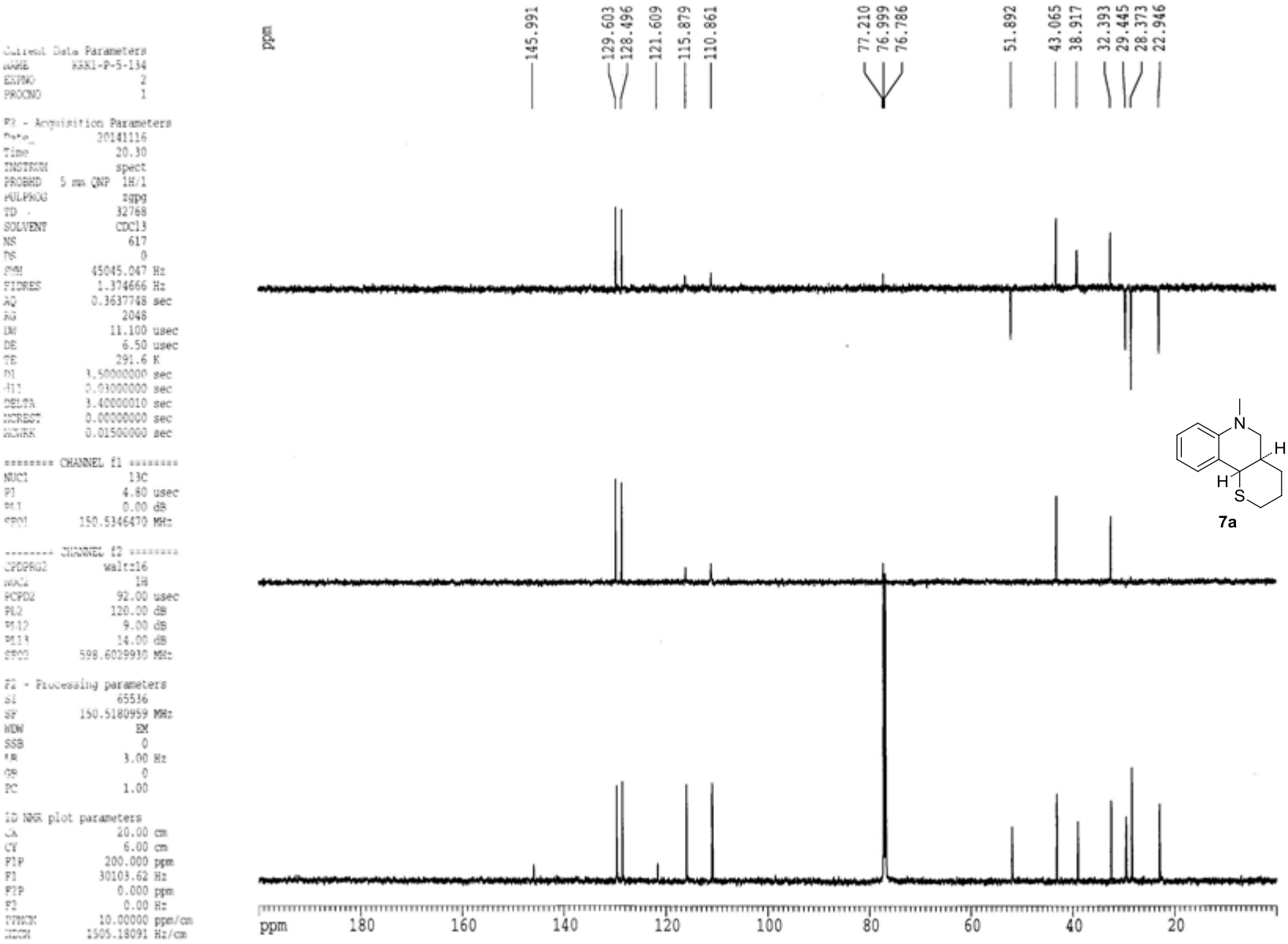

7.2406
6.9043
6.8972
6.8952
6.8931
6.8916
6.5648
6.5426
4.5998
4.5847
3.1360
3.1113
3.1085
3.0843
2.9639
2.9503
2.9459
2.9374
2.9326
2.9307
2.9257
2.9181
2.9070
2.8968
2.8456
2.7231
2.7121
2.7085
2.6979
2.6876
2.6839
2.6740
2.6631
2.6591
2.6488
2.2481
2.2333
2.2283
2.2154
2.1971
2.1814
2.1761
2.1613
2.1523
2.1191
2.1079
2.1021
2.0960
2.0904
Current Data Parameters
NAME 2014-08-27
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140827
Time 15.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 39
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 575
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.5
4.0
3.5
ppm
1.00
1.00
3.18
3.16
N
H
H
S
6b
10
9
8
7
6
5
4
3
2
1
ppm
2.01
1.04
1.00
1.00
3.18
3.16
1.15
4.34
1.18

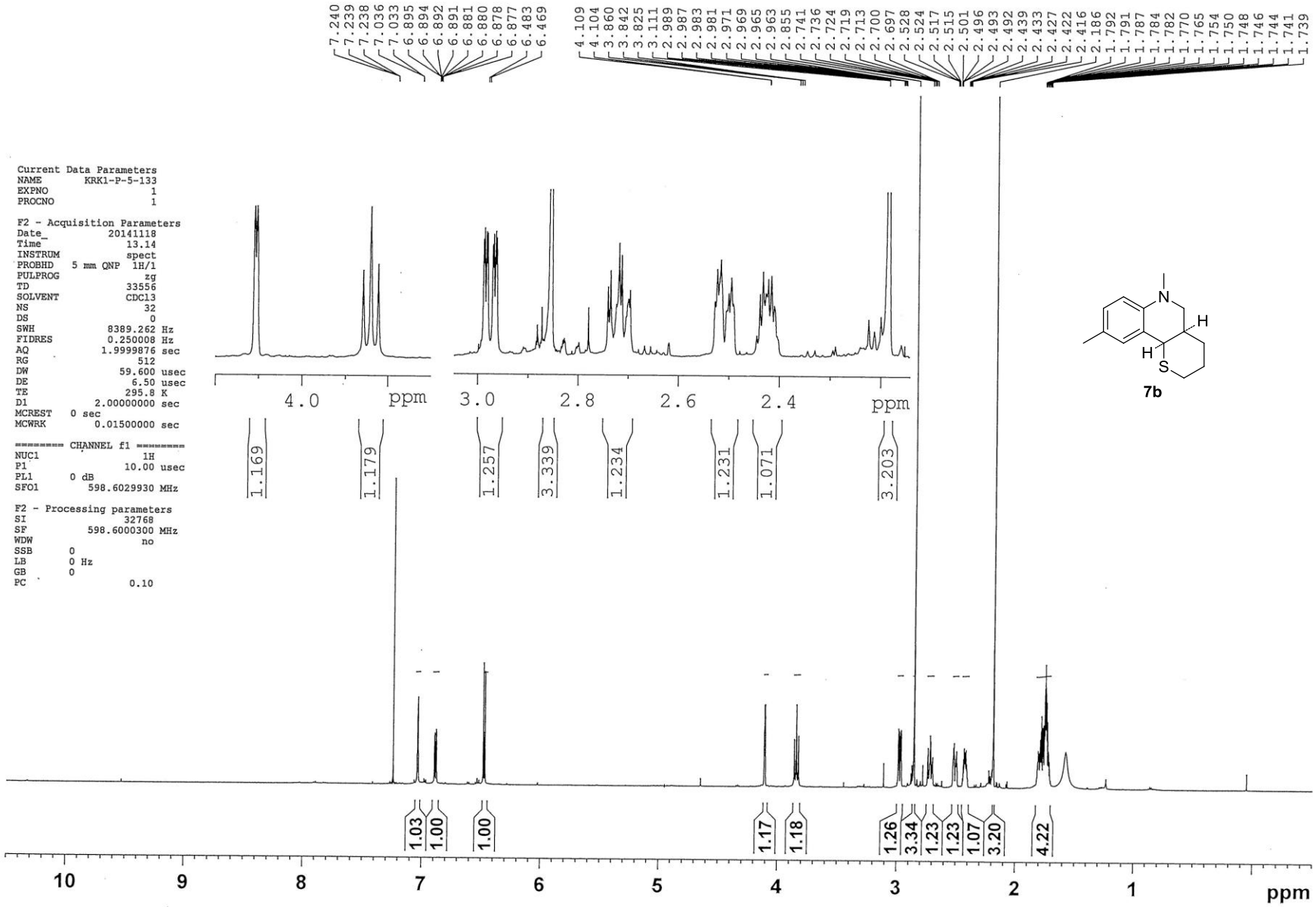

143.31
130.89
128.55
126.41
123.50
112.03
77.32
77.01
76.69
51.55
48.58
40.44
39.56
34.51
30.21
20.18
Current Data Parameters
NAME tetrahydrothiophene-13C
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date 20140827
Time 21.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 438
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6128026 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
H
H
S
6b
tetrahydrothiophene-13C
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

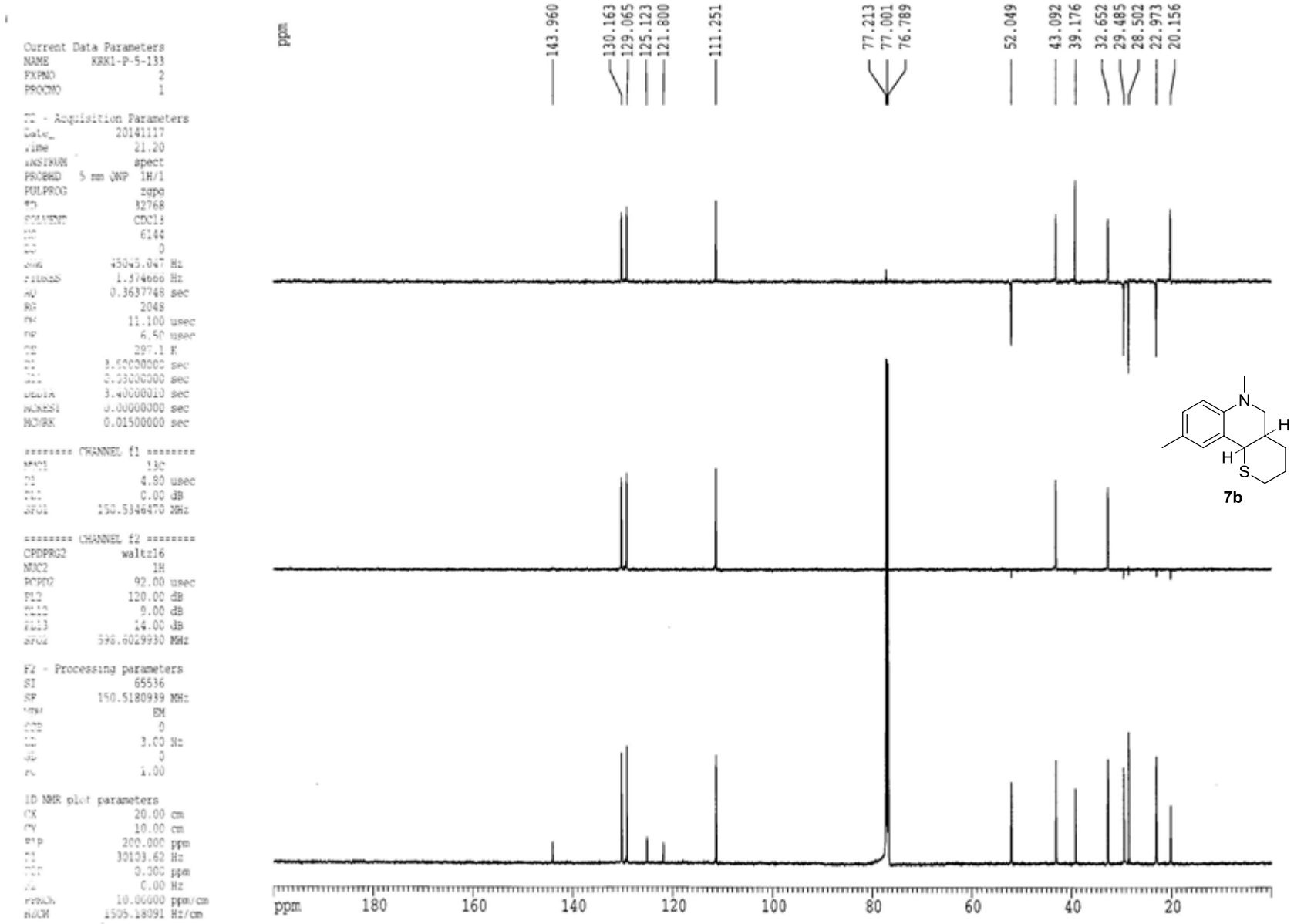

P-4- 4Cl Thio
Current Data Parameters
NAME 21102014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141021
Time 22.57
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 25
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.2404 7.0350 7.0288 7.0074 7.0009 6.5292 6.5082 4.5353 4.5206 3.1769 3.1525 3.1487 3.1244 2.9653 2.9607 2.9543 2.9408 2.9344 2.9229 2.8544 2.7034 2.6938 2.6832 2.6796 2.6691 2.6585 2.6551 2.6447 2.6342 2.6306 2.6197 2.2360 2.2213 2.2157 2.2016 2.1952 2.1887 2.1830 2.1685 2.1629 2.1482 2.0976 2.0849 2.0813 2.0745 2.0701 2.0685 2.0575 2.0526
4.5 4.0 3.5 3.0 ppm
1.045 1.145 3.233 3.142 1.119
10 9 8 7 6 5 4 3 2 1 ppm
2.00 1.06 1.05 1.14 3.23 3.14 1.12 1.12 1.09
N H Cl H S
6c

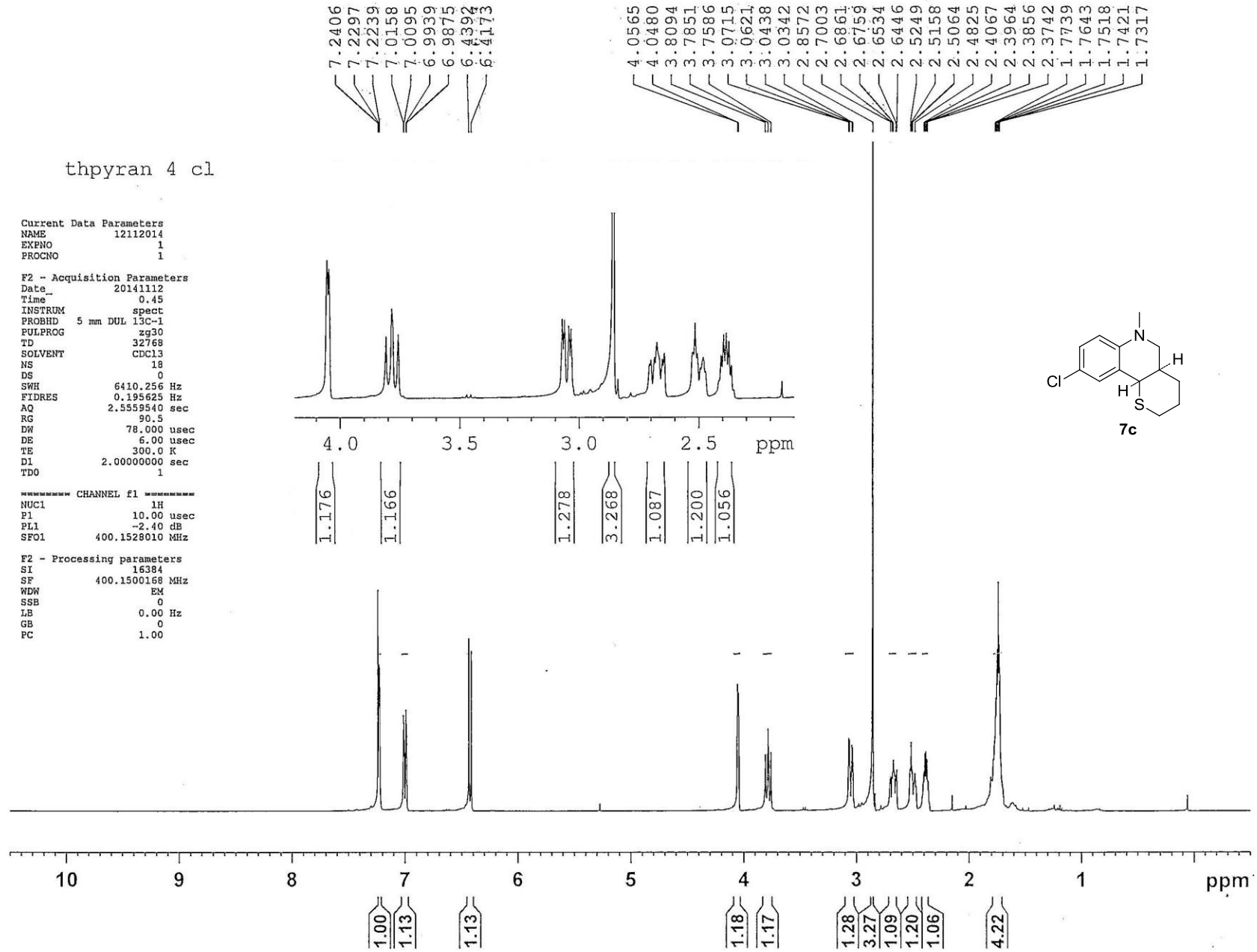

144.04
129.84
127.73
124.84
121.53
112.89
77.46
77.15
76.83
51.04
48.26
39.99
39.45
34.28
30.11
Current Data Parameters
NAME 20102014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141020
Time 23.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
N
H
Cl
H
S
6c
P-4-4-Cl thiophene
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

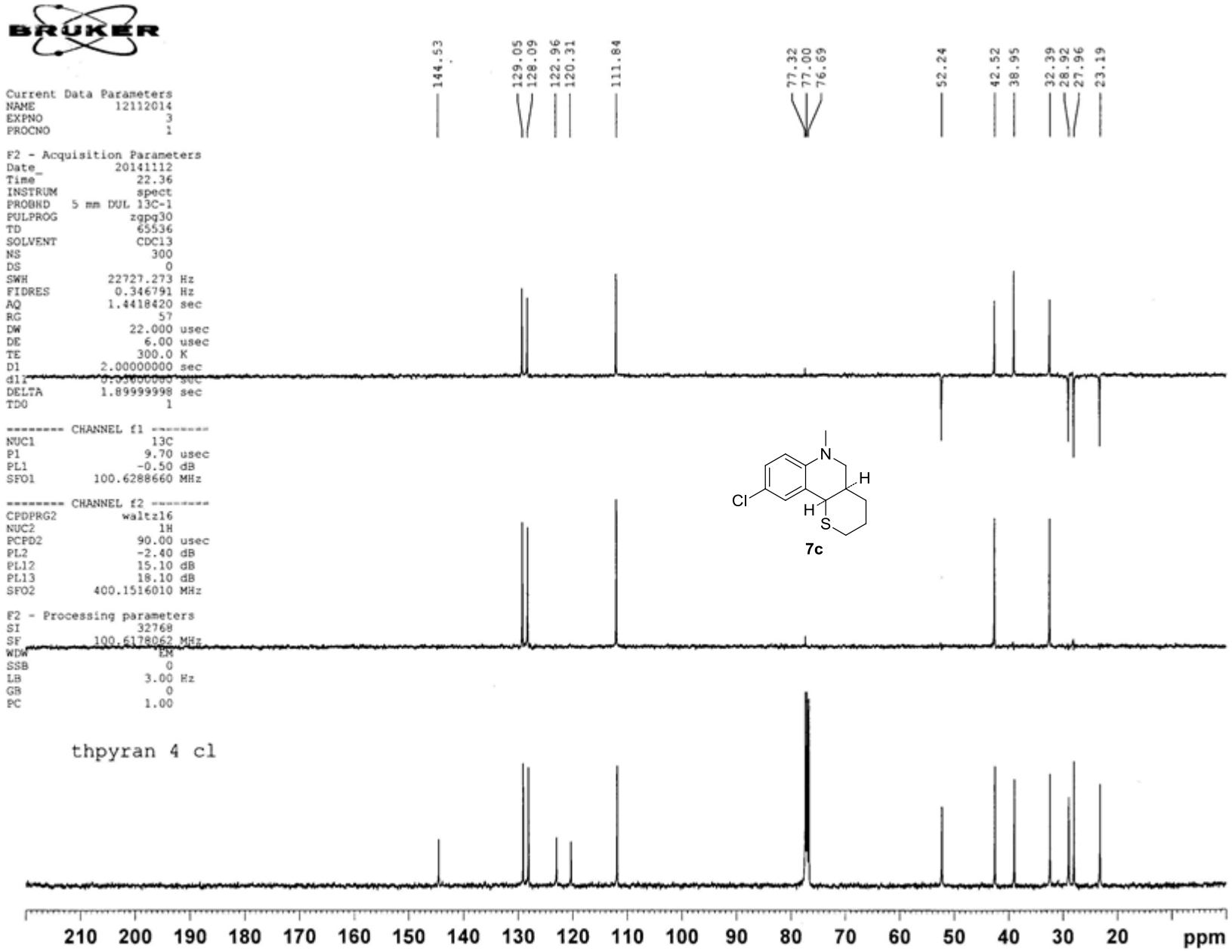

Current Data Parameters
NAME KRK1-P-5-134
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141118
Time 7.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 33556
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.250008 Hz
AQ 1.9999876 sec
RG 512
DW 59.600 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0 dB
SFO1 598.6029930 MHz
F2 - Processing parameters
SI 32768
SF 598.6000303 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 0.10
7.222 7.211 7.210 7.090 7.088 7.076 7.074 7.064 7.062 6.592 6.580 6.568 6.548 6.535
4.139 4.134 3.906 3.889 3.872 3.039 3.037 3.033 3.031 3.021 3.019 3.015 3.013 2.887 2.742 2.737 2.725 2.720 2.715 2.702 2.698 2.528 2.523 2.517 2.514 2.506 2.502 2.496 2.491 2.450 2.444 2.437 2.434 2.428 2.421 1.807 1.801 1.794 1.792 1.788 1.785 1.780 1.778 1.773 1.767 1.759 1.757 1.756 1.751 1.748 1.746
4.0 3.8
3.0 2.8 2.6 ppm
1.077 1.177 1.237 3.366 1.240 1.165 1.171
1.00 1.01 1.00 1.07 1.08 1.18 1.24 3.37 1.24 1.17 1.17 4.11
10 9 8 7 6 5 4 3 2 1 ppm
N
H
H
S
7a

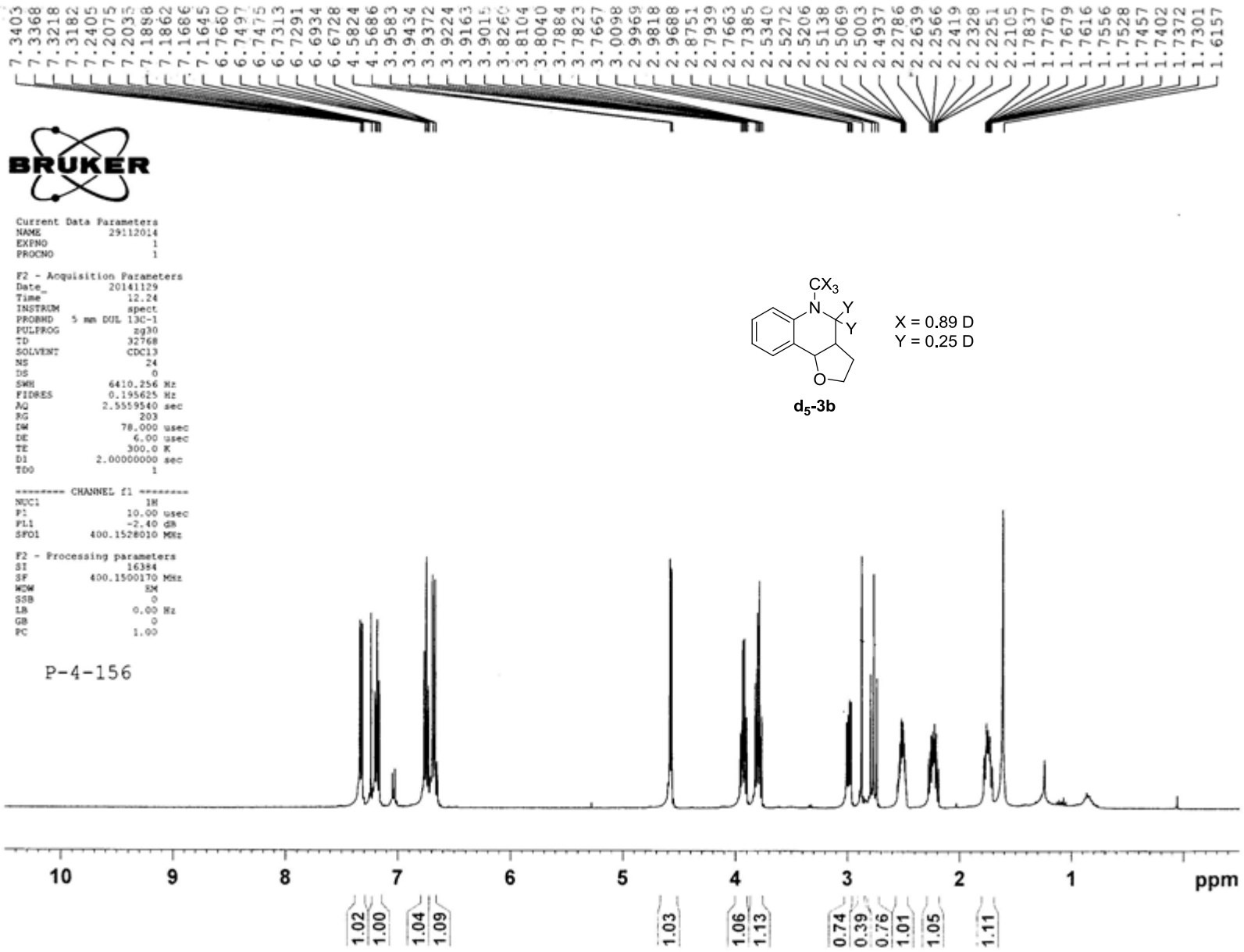

Current Data Parameters
NAME KKK1-P-5-134
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141116
Time 20.30
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 617
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 291.6 K
D1 3.50000000 sec
D11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.80 usec
PL1 0.00 dB
SFO1 150.5346470 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 598.6029930 MHz
F2 - Processing parameters
SI 65536
SF 150.5180959 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.000 ppm
F1 30103.62 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1505.18091 Hz/cm
ppm
145.991
129.603
128.496
121.609
115.879
110.861
77.210
76.999
76.786
51.892
43.065
38.917
32.393
29.445
28.373
22.946
N
H
H
S
7a
ppm
180
160
140
120
100
80
60
40
20


7.240
7.239
7.238
7.036
7.033
6.895
6.894
6.892
6.891
6.881
6.880
6.878
6.877
6.483
6.469
4.109
4.104
3.860
3.842
3.825
3.111
2.989
2.987
2.983
2.981
2.971
2.969
2.965
2.963
2.855
2.741
2.736
2.724
2.719
2.713
2.700
2.697
2.528
2.524
2.517
2.515
2.501
2.496
2.493
2.492
2.439
2.433
2.427
2.422
2.416
2.186
1.792
1.791
1.787
1.784
1.782
1.770
1.765
1.754
1.750
1.748
1.746
1.744
1.741
1.739
Current Data Parameters
NAME KRK1-P-5-133
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141118
Time 13.14
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 33556
SOLVENT CDCl3
NS 32
DS 0
SWH 8389.262 Hz
FIDRES 0.250008 Hz
AQ 1.9999876 sec
RG 512
DW 59.600 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
MCREST 0 sec
MCWRK 0.01500000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 10.00 usec
PL1 0 dB
SFO1 598.6029930 MHz
F2 - Processing parameters
SI 32768
SF 598.6000300 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 0.10
4.0
ppm
3.0
2.8
2.6
2.4
ppm
1.169
1.179
1.257
3.339
1.234
1.231
1.071
3.203
N
H
H
S
7b
1.03
1.00
1.00
1.17
1.18
1.26
3.34
1.23
1.23
1.07
3.20
4.22
10
9
8
7
6
5
4
3
2
1
ppm


Current Data Parameters
NAME KRK1-P-5-133
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141117
Time 21.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 6144
DS 0
SWH 45045.047 Hz
FIDRES 1.374666 Hz
AQ 0.3637748 sec
RG 2048
DW 11.100 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 3.40000010 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 4.80 usec
PL1 0.00 dB
SFO1 150.5346470 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 599.6029930 MHz
F2 - Processing parameters
SI 65536
SF 150.5180939 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.000 ppm
F1 30103.62 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1505.18091 Hz/cm
ppm
143.960
130.163
129.065
125.123
121.800
111.251
77.213
77.001
76.789
52.049
43.092
39.176
32.652
29.485
28.502
22.973
20.156
N
H
H
S
7b
ppm
180
160
140
120
100
80
60
40
20


thpyran 4 cl
Current Data Parameters
NAME 12112014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141112
Time 0.45
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 18
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.2406
7.2297
7.2239
7.0158
7.0095
6.9939
6.9875
6.4392
6.4173
4.0565
4.0480
3.8094
3.7851
3.7586
3.0715
3.0621
3.0438
3.0342
2.8572
2.7003
2.6861
2.6759
2.6534
2.6446
2.5249
2.5158
2.5064
2.4825
2.4067
2.3964
2.3856
2.3742
1.7739
1.7643
1.7518
1.7421
1.7317
4.0
3.5
3.0
2.5
ppm
1.176
1.166
1.278
3.268
1.087
1.200
1.056
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.13
1.13
1.18
1.17
1.28
3.27
1.09
1.20
1.06
4.22
Cl
N
H
H
S
7c


144.53
129.05
128.09
122.96
120.31
111.84
77.32
77.00
76.69
52.24
42.52
38.95
32.39
28.92
27.96
23.19
Current Data Parameters
NAME 12112014
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141112
Time 22.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178062 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
thpyran 4 cl
Cl
N
H
H
S
7c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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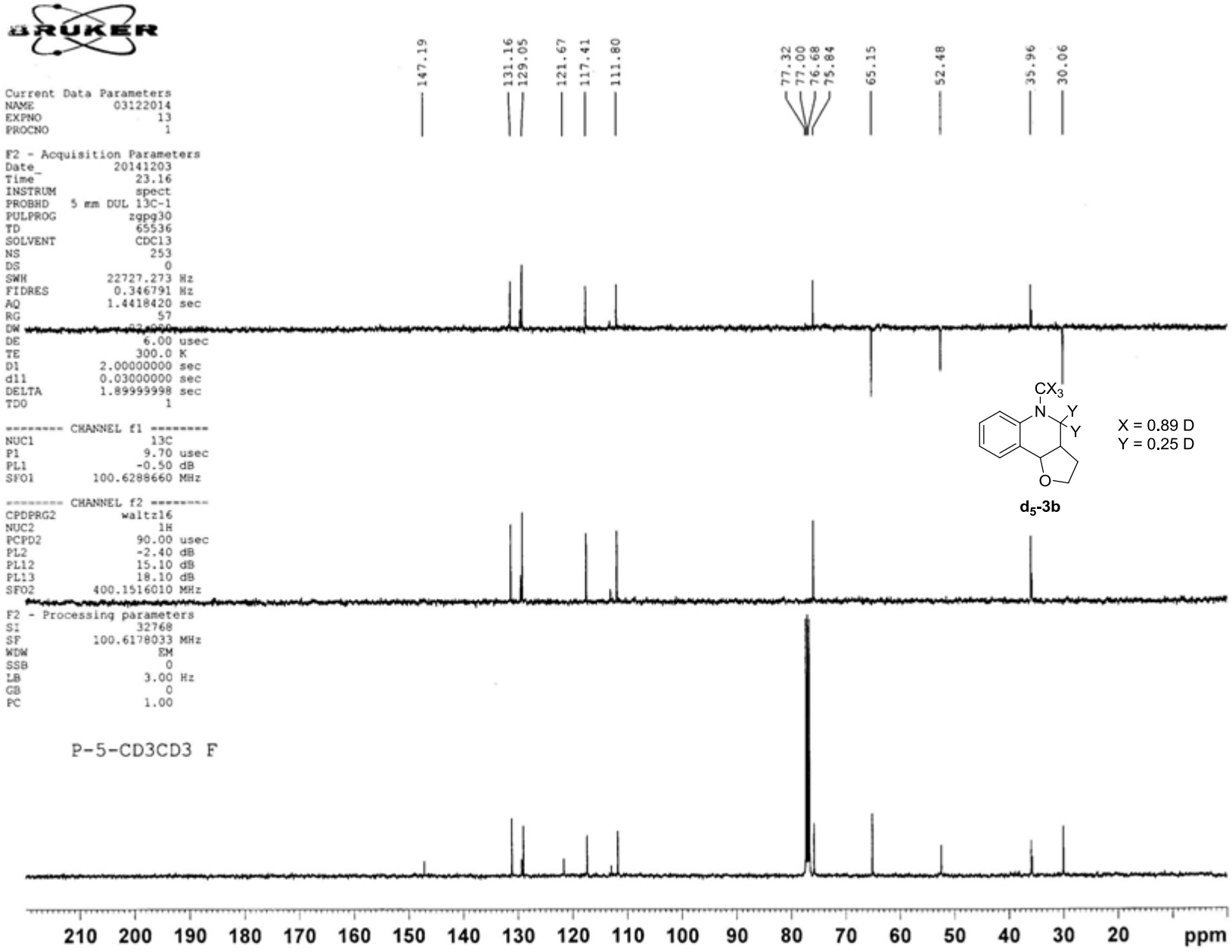
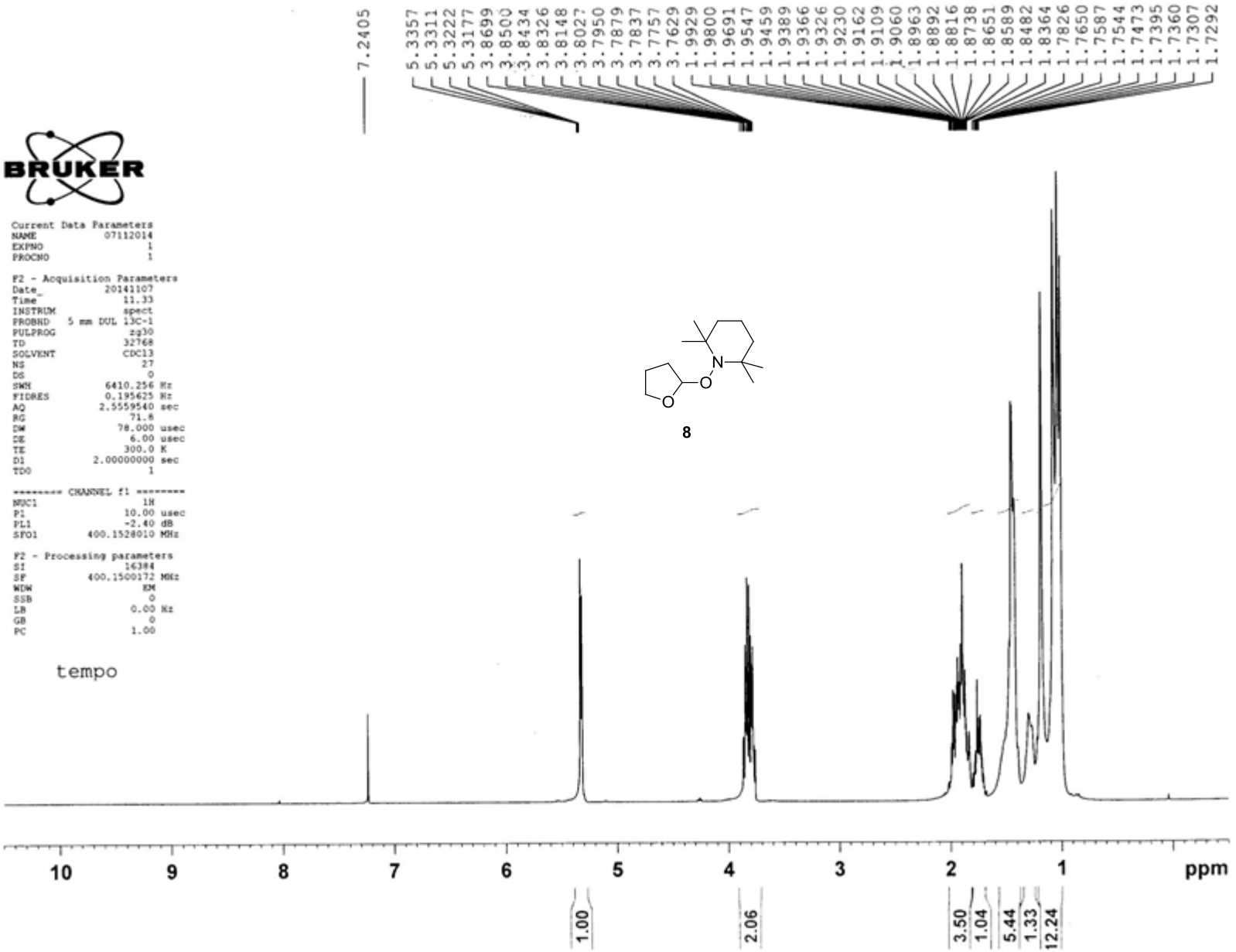
7.3368
7.3218
7.3182
7.2405
7.2075
7.2035
7.1898
7.1862
7.1686
7.1645
7.7660
6.7497
6.7475
6.7313
6.7291
6.6934
6.6728
4.5824
4.5686
3.9583
3.9434
3.9372
3.9224
3.9163
3.9015
3.8260
3.8104
3.8040
3.7884
3.7823
3.7667
3.0098
2.9969
2.9818
2.9688
2.8751
2.7939
2.7663
2.7385
2.5340
2.5272
2.5206
2.5138
2.5069
2.5003
2.4937
2.2786
2.2639
2.2566
2.2419
2.2328
2.2251
2.2105
1.7837
1.7767
1.7679
1.7616
1.7556
1.7528
1.7457
1.7402
1.7372
1.7301
1.6157
BRUKER
Current Data Parameters
NAME 29112014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141129
Time 12.24
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 203
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500170 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-4-156
CX3
N
Y
Y
O
X = 0.89 D
Y = 0.25 D
d5-3b
10
9
8
7
6
5
4
3
2
1
ppm
1.02
1.00
1.04
1.09
1.03
1.06
1.13
0.74
0.39
0.76
1.01
1.05
1.11


147.19
131.16
129.05
121.67
117.41
111.80
77.32
77.00
76.68
75.84
65.15
52.48
35.96
30.06
Current Data Parameters
NAME 03122014
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141203
Time 23.16
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 253
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178033 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
P-5-CD3CD3 F
CX3
N
Y
Y
O
X = 0.89 D
Y = 0.25 D
d5-3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.2405
5.3357
5.3311
5.3222
5.3177
3.8699
3.8500
3.8434
3.8326
3.8148
3.8027
3.7950
3.7879
3.7837
3.7757
3.7629
1.9929
1.9800
1.9691
1.9547
1.9459
1.9389
1.9366
1.9326
1.9230
1.9162
1.9109
1.9060
1.8963
1.8892
1.8816
1.8738
1.8651
1.8589
1.8482
1.8364
1.7826
1.7650
1.7587
1.7544
1.7473
1.7395
1.7360
1.7307
1.7292
BRUKER
Current Data Parameters
NAME 07112014
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141107
Time 11.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 27
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 71.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
tempo
N
O
O
8
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.06
3.50
1.04
5.44
1.33
12.24

tempo

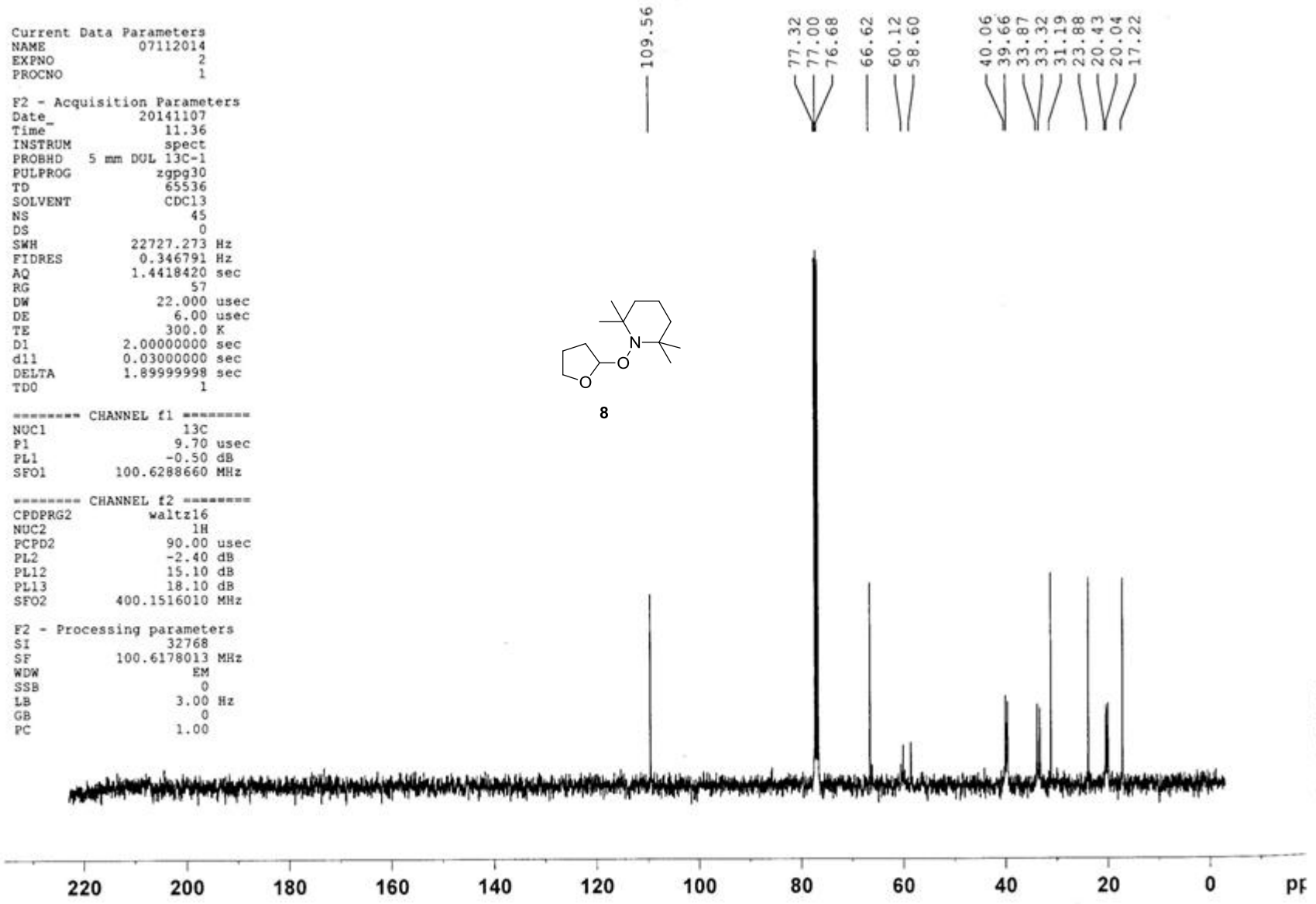
```
Current Data Parameters
NAME                 07112014
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20141107
Time                   11.36
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         45
DS                          0
SWH                22727.273 Hz
FIDRES              0.346791 Hz
AQ                  1.4418420 sec
RG                         57
DW                    22.000 usec
DE                     6.00 usec
TE                     300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                         1

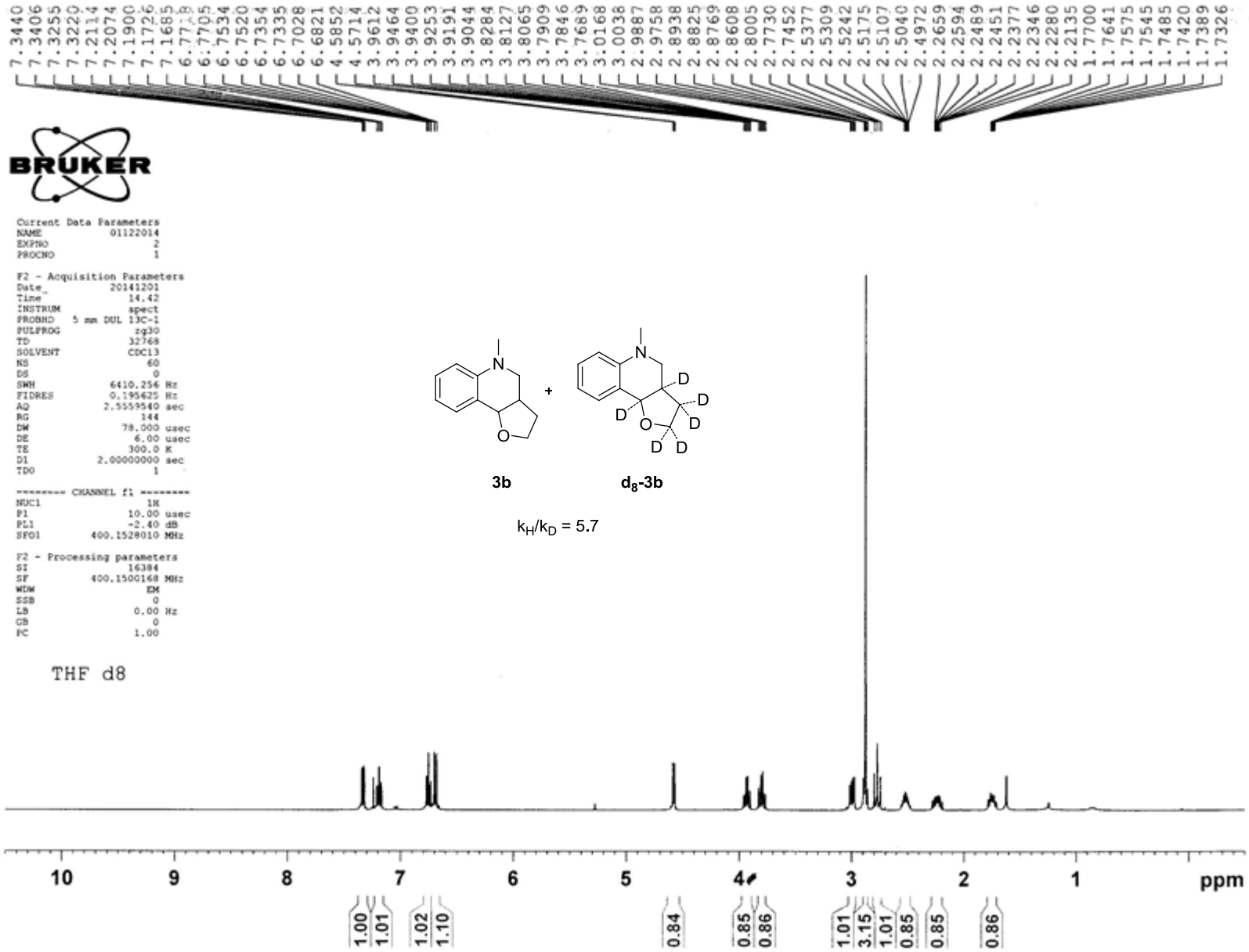
======== CHANNEL f1 ========
NUC1                      13C
P1                       9.70 usec
PL1                     -0.50 dB
SFO1             100.6288660 MHz

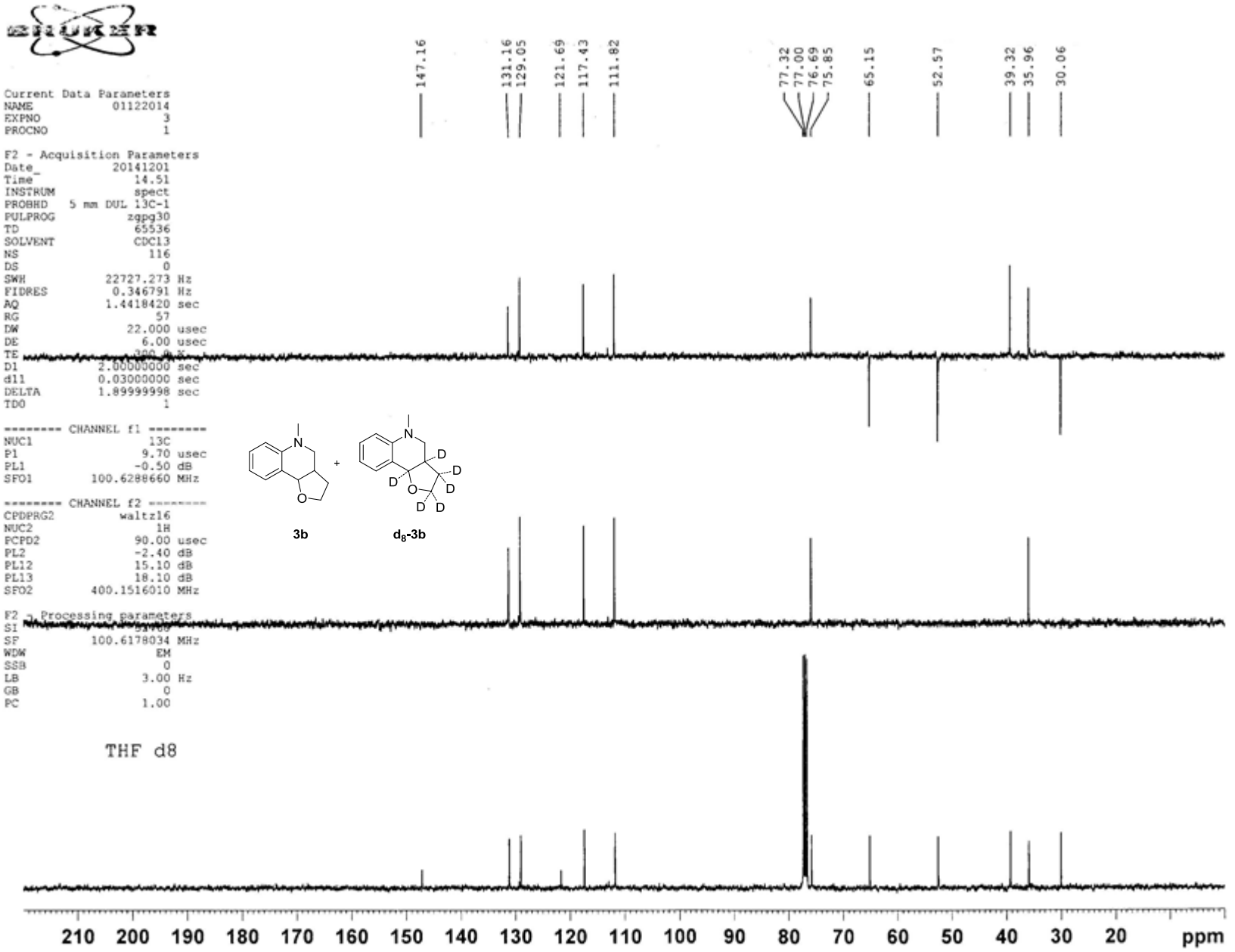
======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 90.00 usec
PL2                     -2.40 dB
PL12                    15.10 dB
PL13                    18.10 dB
SFO2             400.1516010 MHz

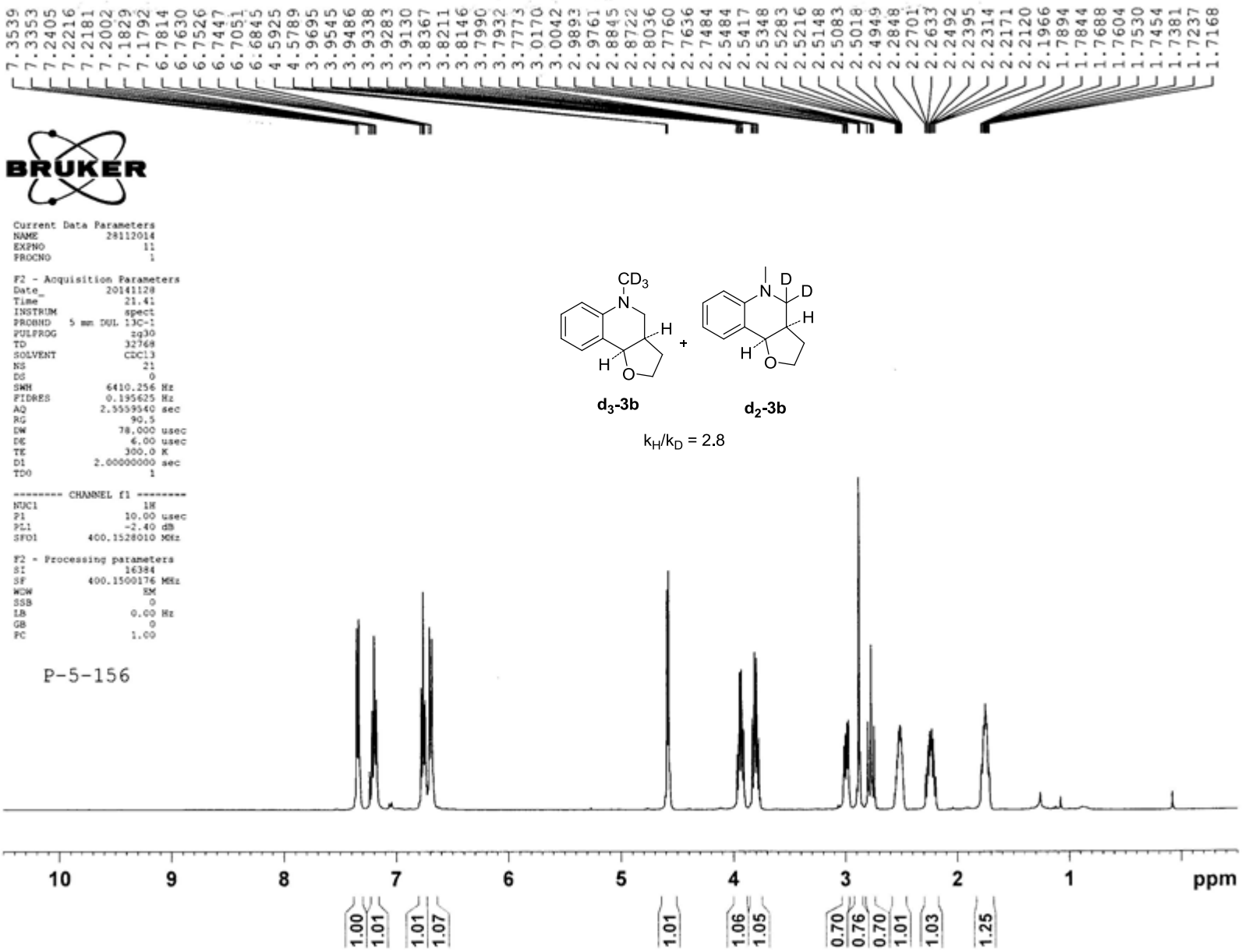
F2 - Processing parameters
SI                        32768
SF               100.6178013 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.00
```

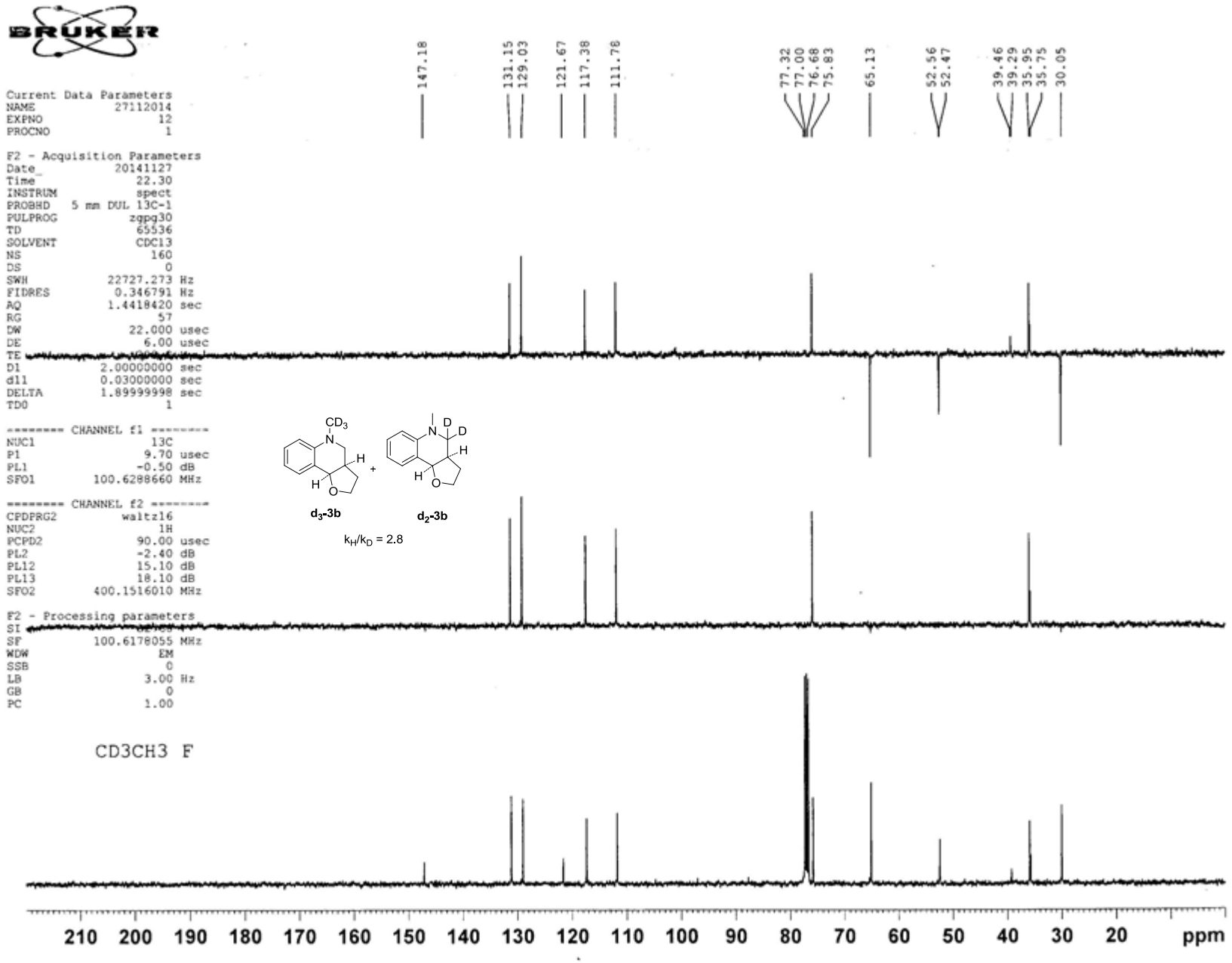


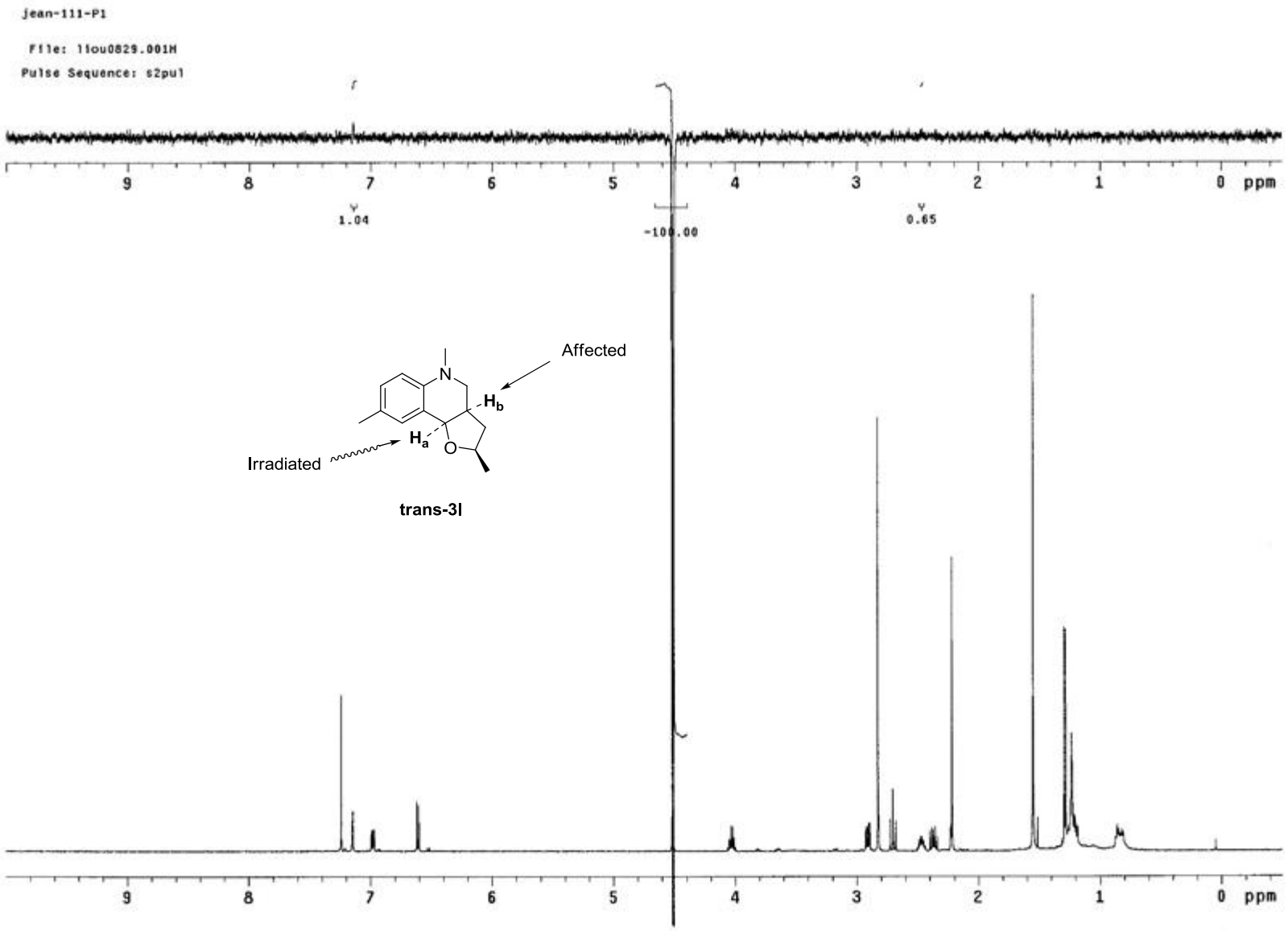
8

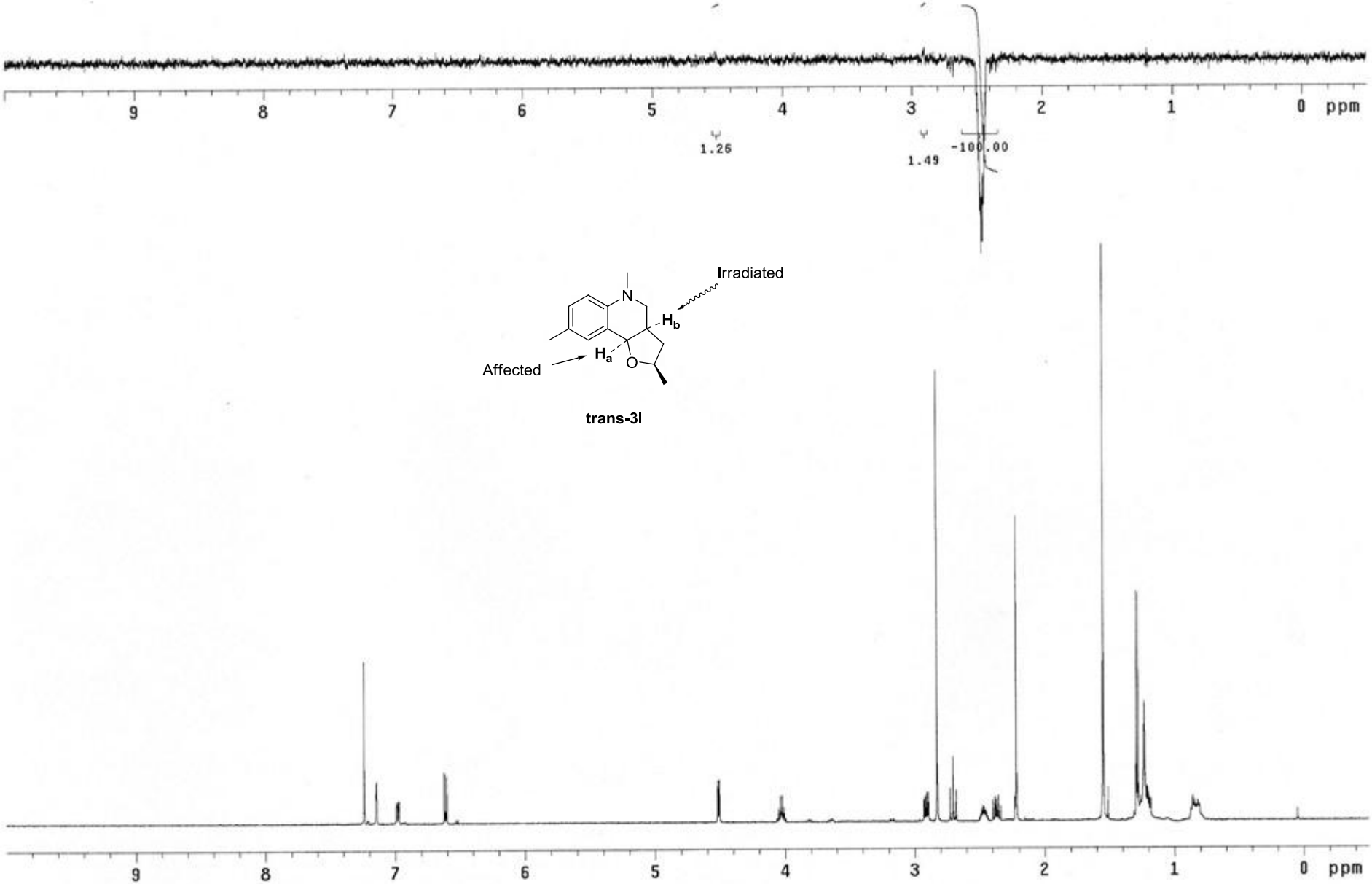

7.3440
7.3406
7.3255
7.3220
7.2114
7.2074
7.1900
7.1726
7.1685
6.7705
6.7534
6.7520
6.7354
6.7335
6.7028
6.6821
4.5852
4.5714
3.9612
3.9464
3.9400
3.9253
3.9191
3.9044
3.8284
3.8127
3.8065
3.7909
3.7846
3.7689
3.0168
3.0038
2.9887
2.9758
2.8938
2.8825
2.8769
2.8608
2.8005
2.7730
2.7452
2.5377
2.5309
2.5242
2.5175
2.5107
2.5040
2.4972
2.2659
2.2594
2.2489
2.2451
2.2377
2.2346
2.2280
2.2135
1.7700
1.7641
1.7575
1.7545
1.7485
1.7420
1.7389
1.7326
BRUKER
Current Data Parameters
NAME 01122014
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141201
Time 14.42
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 60
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 144
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
THF d8
N
O
+
N
D
D
D
D
O
D
D
3b
d8-3b
kH/kD = 5.7
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.02
1.10
0.84
0.85
0.86
1.01
3.15
1.01
0.85
0.85
0.86

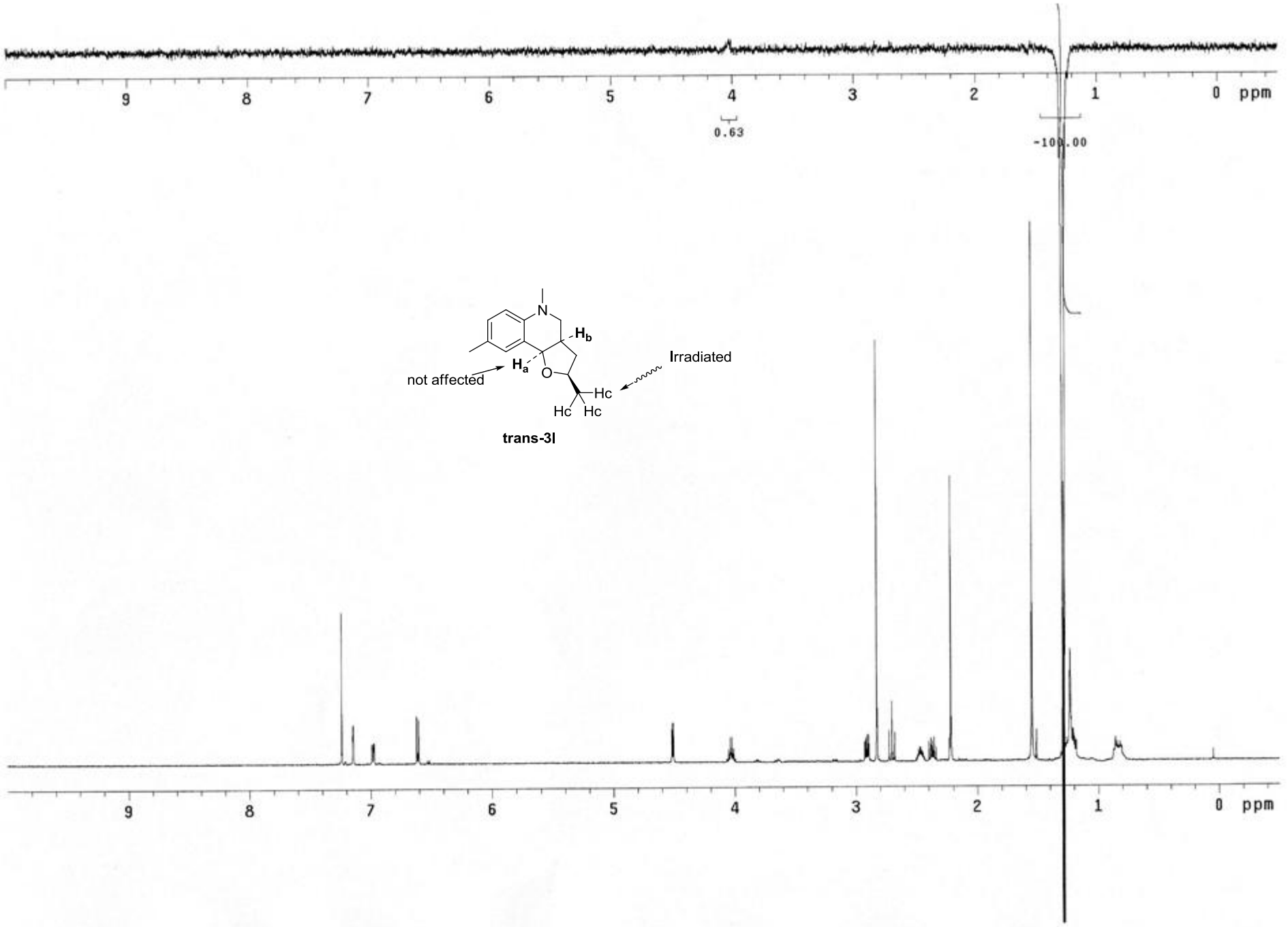

147.16
131.16
129.05
121.69
117.43
111.82
77.32
77.00
76.69
75.85
65.15
52.57
39.32
35.96
30.06
Current Data Parameters
NAME 01122014
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141201
Time 14.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 116
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SF 100.6178034 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
THF d8
3b
d8-3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

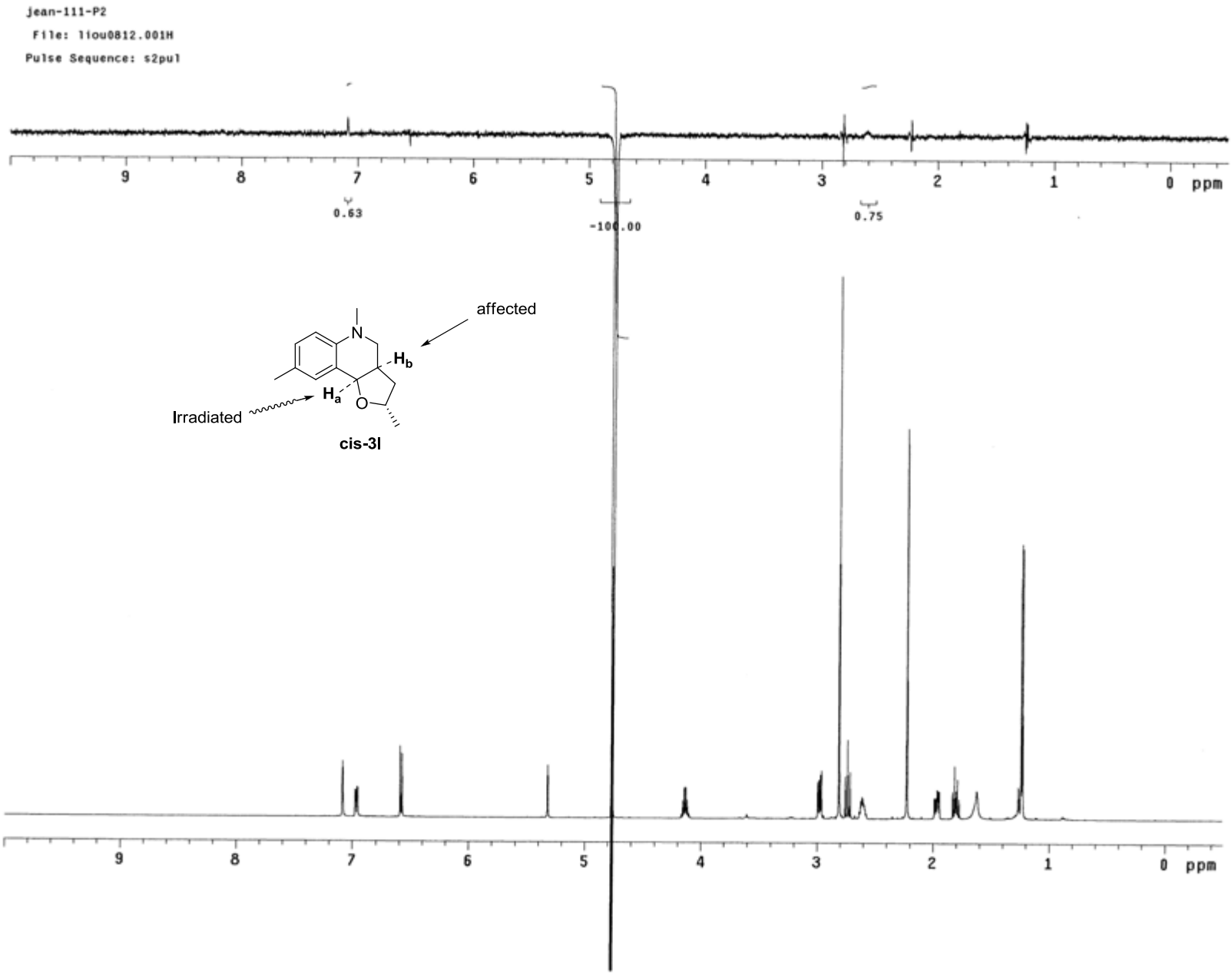

7.3539
7.3353
7.2405
7.2216
7.2181
7.2002
7.1829
7.1792
6.7814
6.7630
6.7526
6.7447
6.7051
6.6845
4.5925
4.5789
3.9695
3.9545
3.9486
3.9338
3.9283
3.9130
3.8367
3.8211
3.8146
3.7990
3.7932
3.7773
3.0170
3.0042
2.9893
2.9761
2.8845
2.8722
2.8036
2.7760
2.7636
2.7484
2.5484
2.5417
2.5348
2.5283
2.5216
2.5148
2.5083
2.5018
2.4949
2.2848
2.2701
2.2633
2.2492
2.2395
2.2314
2.2171
2.2120
2.1966
1.7894
1.7844
1.7688
1.7604
1.7530
1.7454
1.7381
1.7237
1.7168
BRUKER
Current Data Parameters
NAME 28112014
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141128
Time 21.41
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 21
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500176 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
P-5-156
CD3
N
H
H
O
+
N
D
D
H
H
O
d3-3b
d2-3b
kH/kD = 2.8
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.01
1.01
1.07
1.01
1.06
1.05
0.70
0.76
0.70
1.01
1.03
1.25

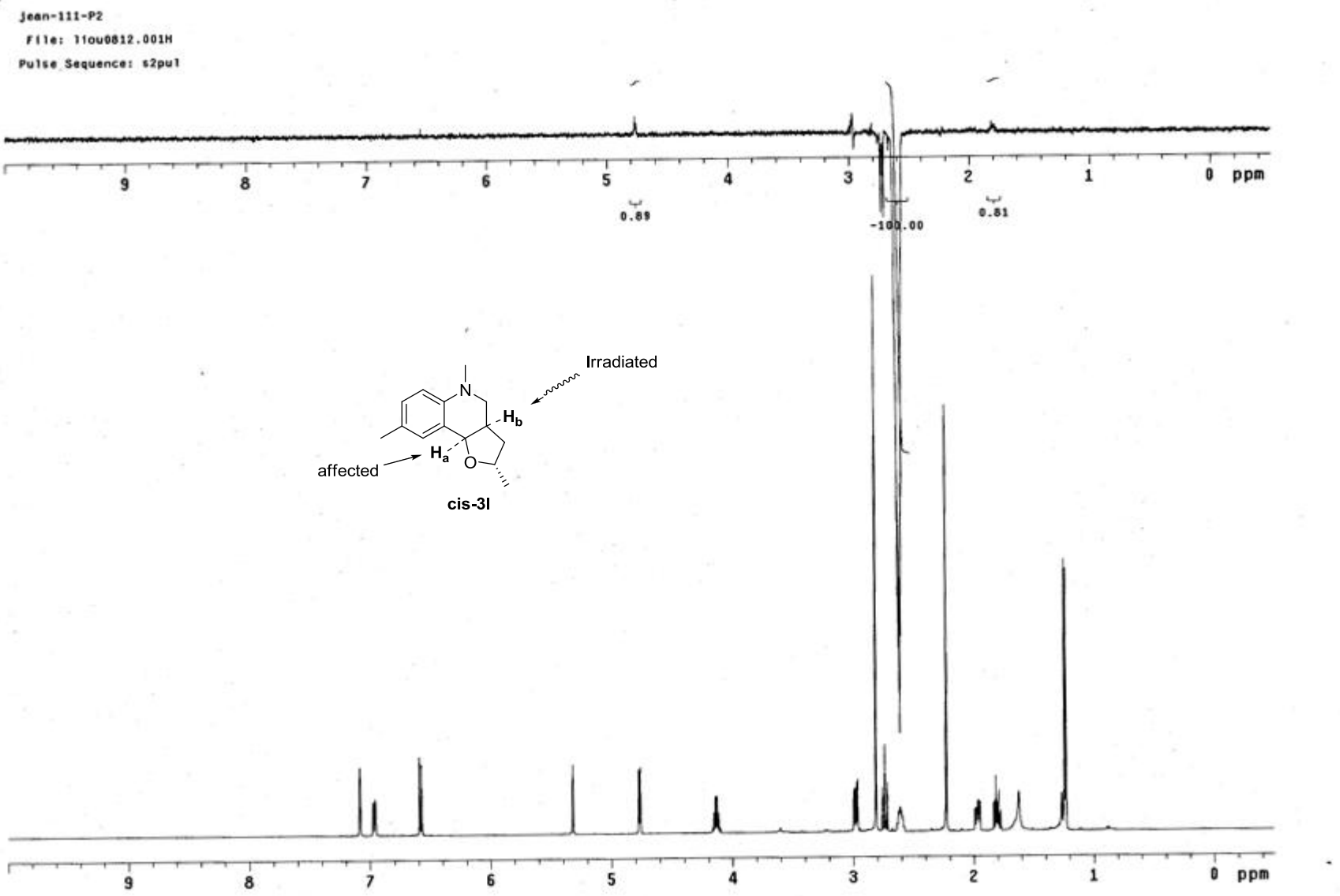

147.18
131.15
129.03
121.67
117.38
111.76
77.32
77.00
76.68
75.83
65.13
52.56
52.47
39.46
39.29
35.95
35.75
30.05
Current Data Parameters
NAME 27112014
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141127
Time 22.30
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178055 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
CD3CH3 F
d3-3b
d2-3b
kH/kD = 2.8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

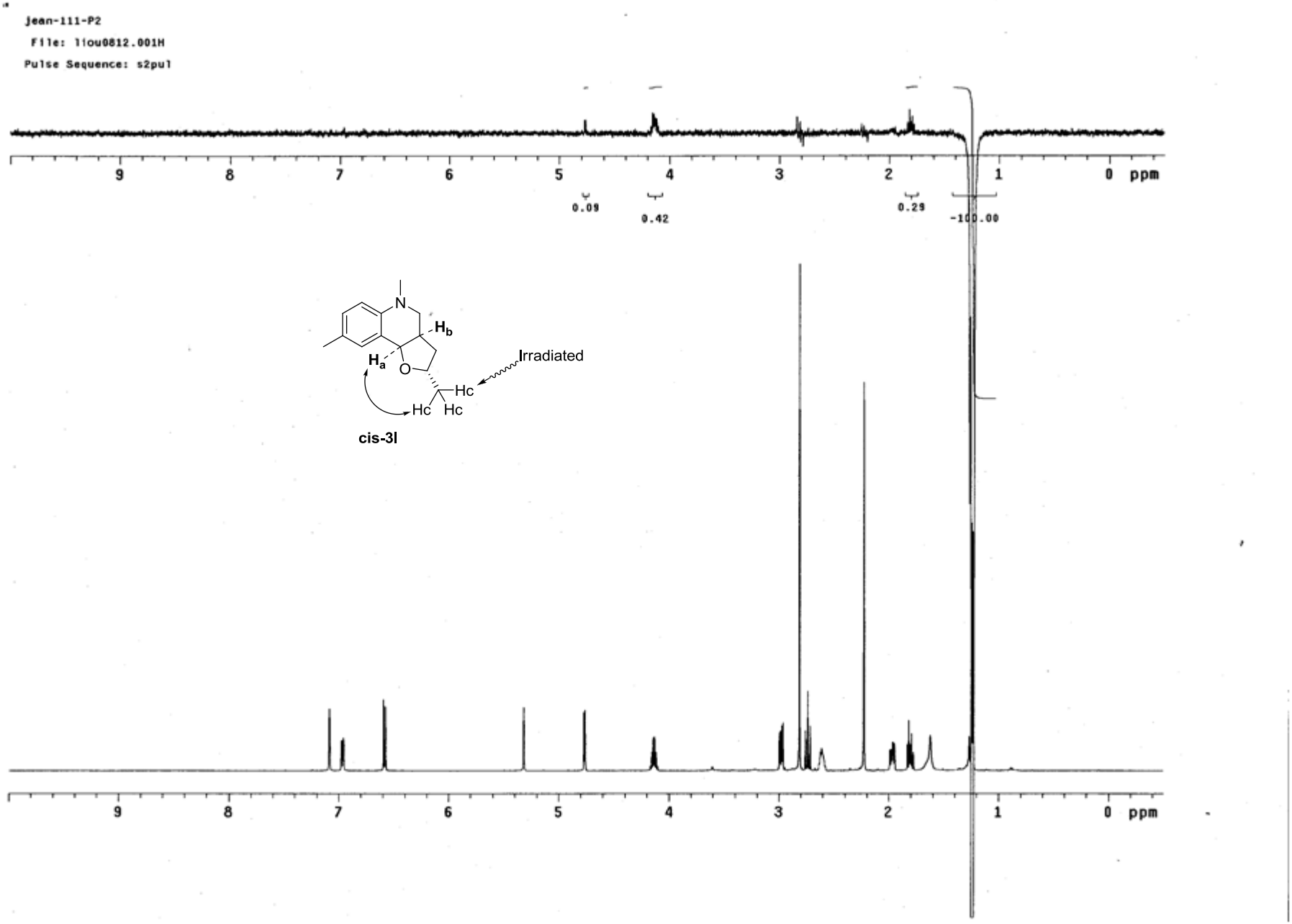

jean-111-P1
File: 11ou0829.001H
Pulse Sequence: s2pul
9 8 7 6 5 4 3 2 1 0 ppm
1.04
-100.00
0.65
Affected
H_b
H_a
Irradiated
trans-3l
9 8 7 6 5 4 3 2 1 0 ppm

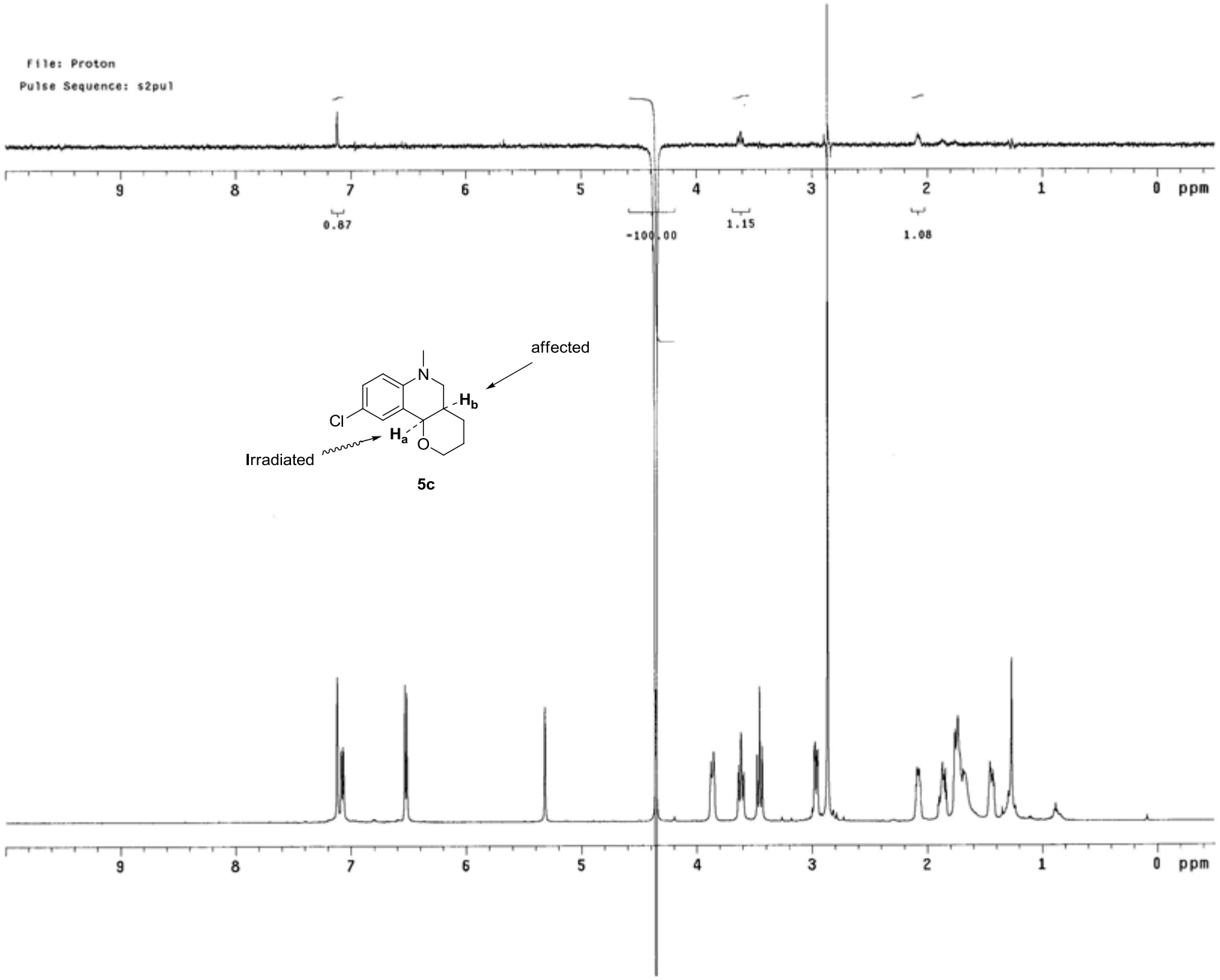

9
8
7
6
5
4
3
2
1
0 ppm
1.26
1.49
-100.00
Irradiated
Affected
H_b
H_a
N
O
trans-3l
9
8
7
6
5
4
3
2
1
0 ppm

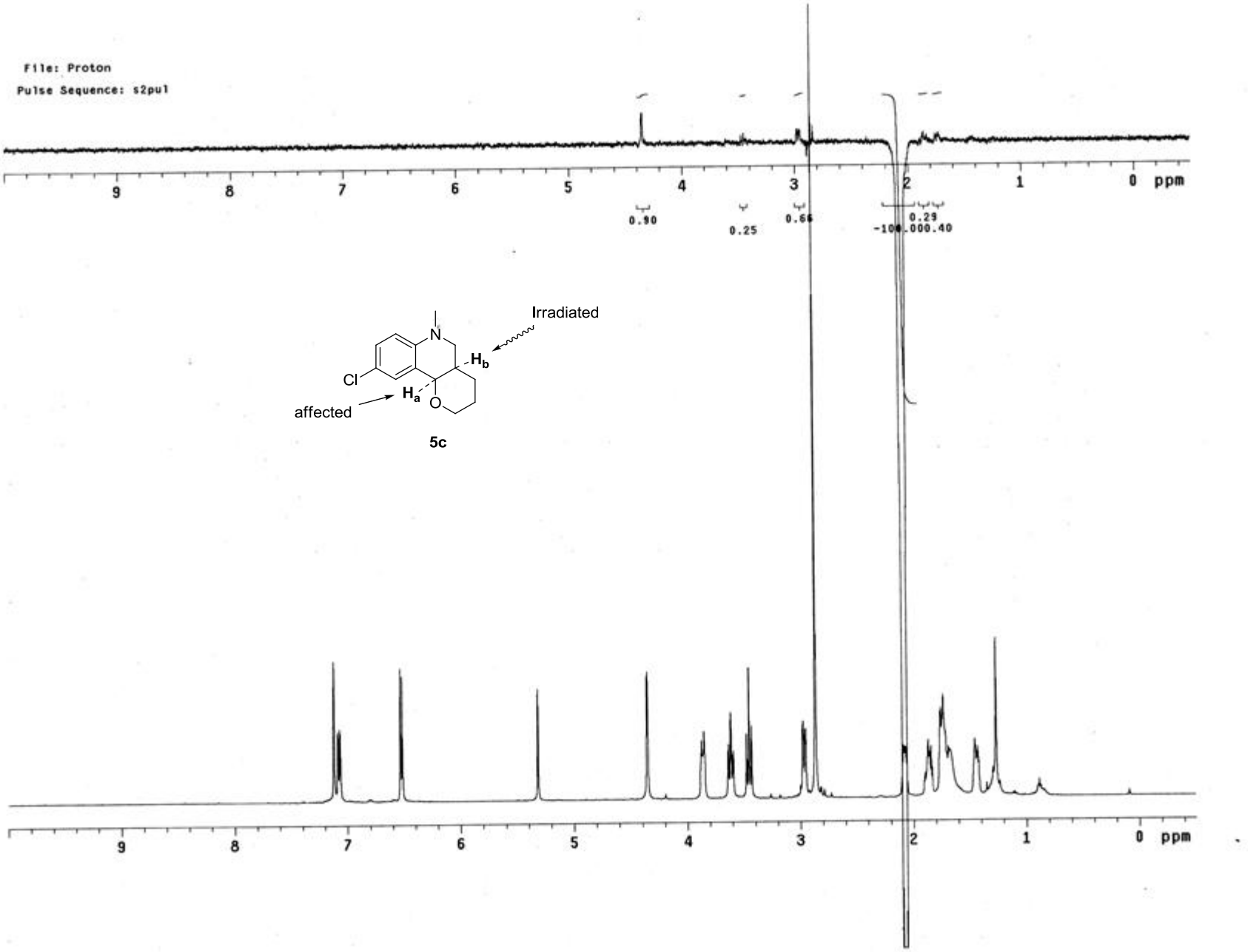

9
8
7
6
5
4
3
2
1
0 ppm
0.63
-100.00
N
Hb
Ha
O
Hc
Hc Hc
not affected
Irradiated
trans-3l
9
8
7
6
5
4
3
2
1
0 ppm

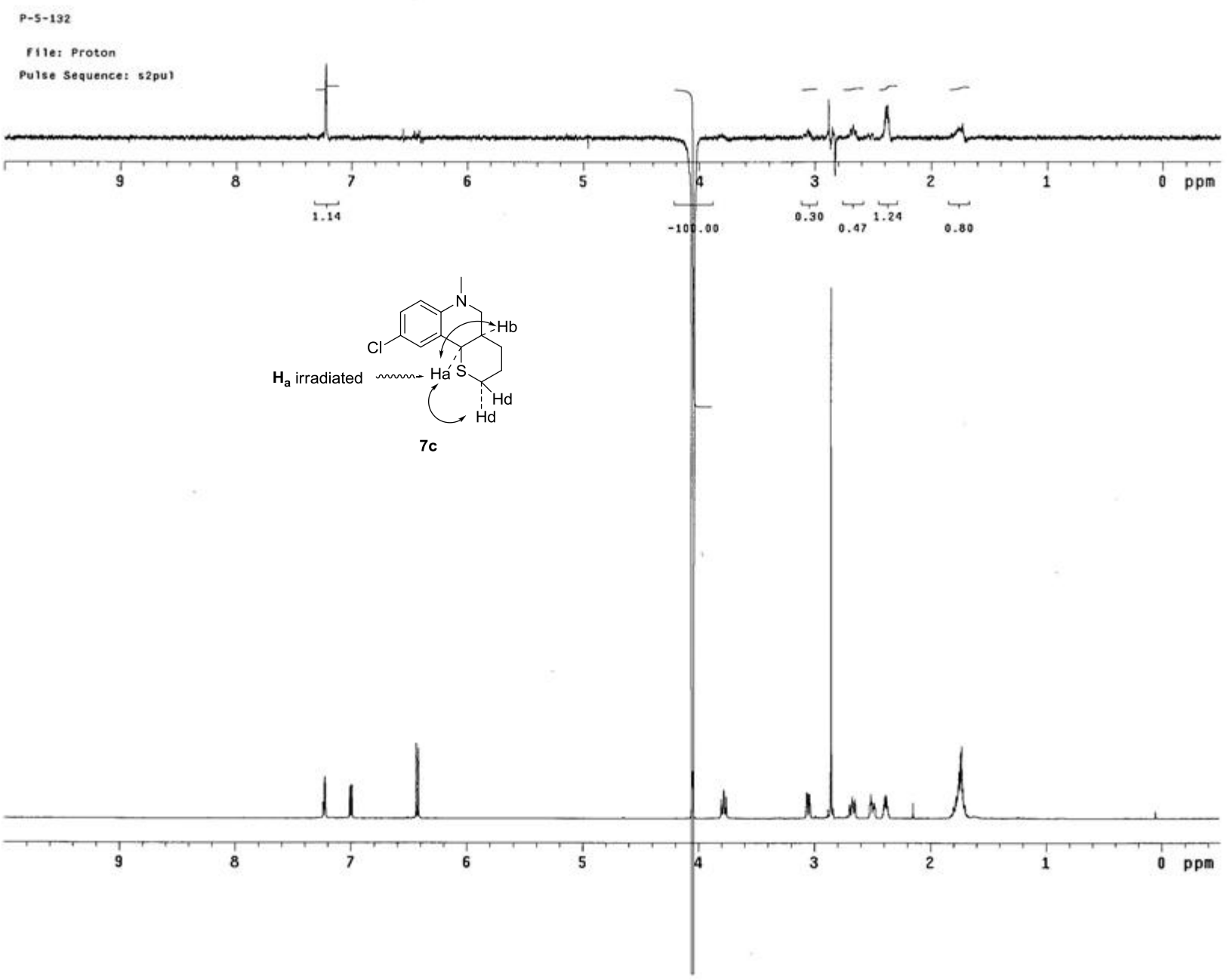

jean-111-P2
File: liou0812.001H
Pulse Sequence: s2pul
9 8 7 6 5 4 3 2 1 0 ppm
0.63
-100.00
0.75
affected
Irradiated
H_b
H_a
N
O
cis-3l
9 8 7 6 5 4 3 2 1 0 ppm

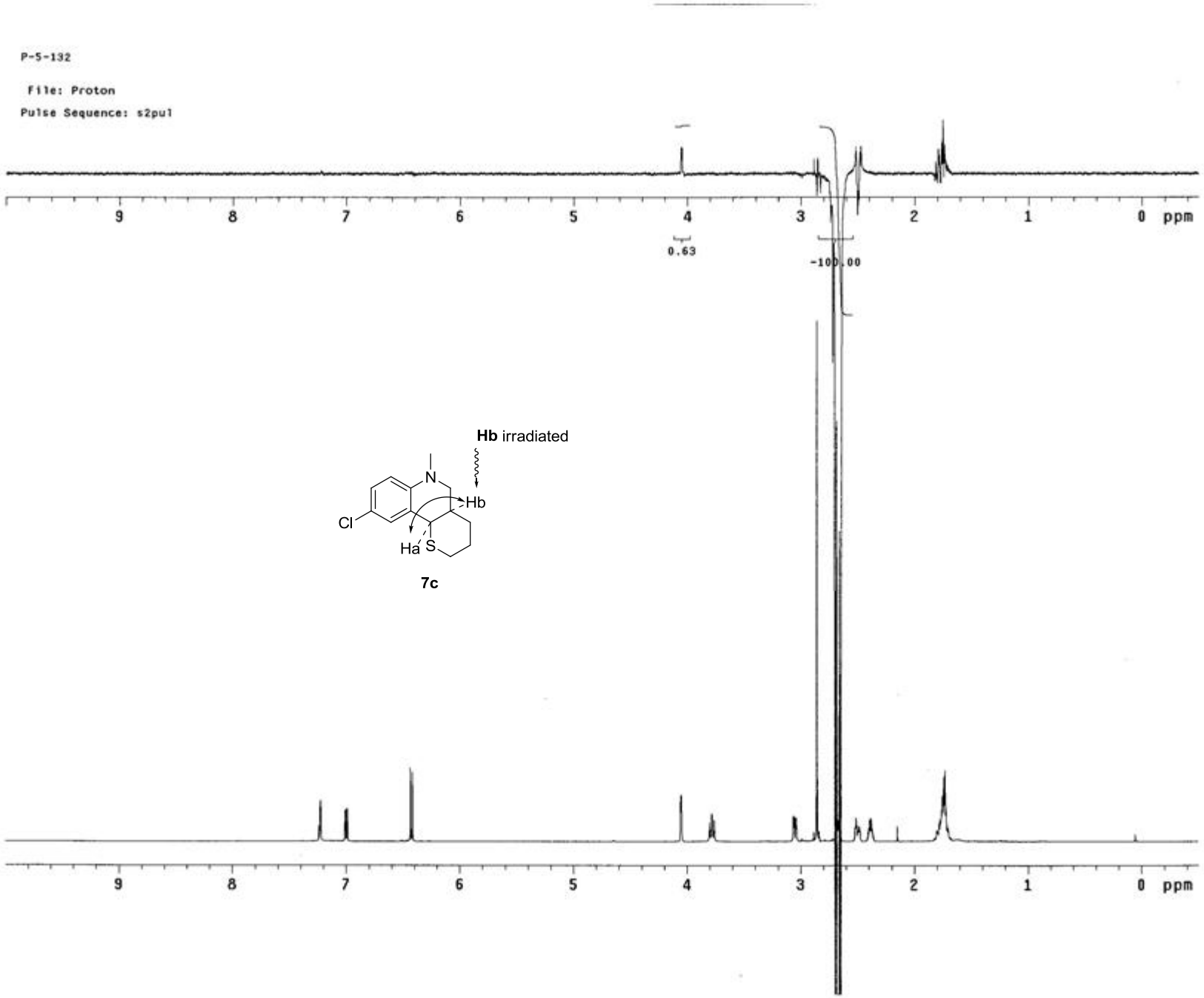

Jean-111-P2
File: 1iou0812.001H
Pulse Sequence: s2pul
9 8 7 6 5 4 3 2 1 0 ppm
0.89
-100.00
0.81
Irradiated
H_b
H_a
affected
O
N
cis-3l
9 8 7 6 5 4 3 2 1 0 ppm


jean-111-P2
File: 11ou0812.001H
Pulse Sequence: s2pul
9
8
7
6
5
4
3
2
1
0 ppm
0.09
0.42
0.29
-100.00
N
Hb
Ha
O
Hc
Hc
Hc
Irradiated
cis-3l
9
8
7
6
5
4
3
2
1
0 ppm


File: Proton
Pulse Sequence: s2pul
9 8 7 6 5 4 3 2 1 0 ppm
0.87
-100.00
1.15
1.08
affected
Irradiated
Cl
N
O
H_a
H_b
5c
9 8 7 6 5 4 3 2 1 0 ppm


File: Proton
Pulse Sequence: s2pul
9 8 7 6 5 4 3 2 1 0 ppm
0.90
0.25
0.66
0.29
-100.000.40
Irradiated
Cl
H_b
H_a
O
affected
5c
9 8 7 6 5 4 3 2 1 0 ppm


P-5-132
File: Proton
Pulse Sequence: s2pul
9
8
7
6
5
4
3
2
1
0 ppm
1.14
-100.00
0.30
0.47
1.24
0.80
N
Hb
Cl
Ha
S
Hd
Hd
Ha irradiated
7c


P-5-132
File: Proton
Pulse Sequence: s2pul
9
8
7
6
5
4
3
2
1
0 ppm
0.63
-100.00
Hb irradiated
N
Hb
Cl
Ha
S
7c
9
8
7
6
5
4
3
2
1
0 ppm