Supporting Information

Synthesis of Medium-sized Heterocycles from Oxetanes Based on an Allylic Amination/Ring-opening Strategy

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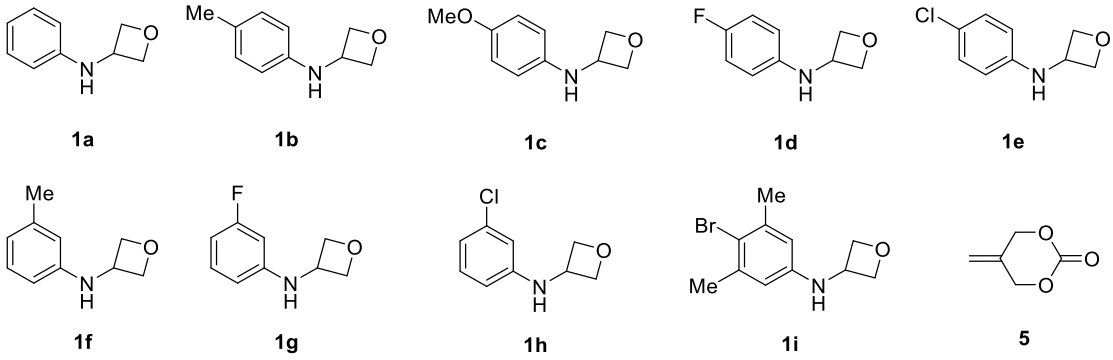
NMR Specturm

I. General Information

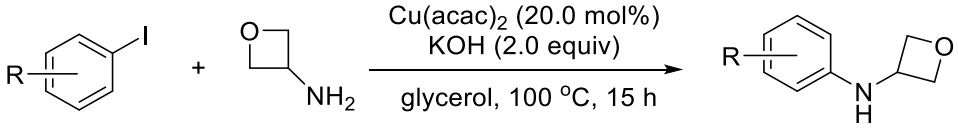
Flash column chromatography was performed over silica gel (200-300 mesh) purchased from Qindao Puke Co. Lit., China. All air or moisture sensitive reactions were conducted in oven-dried glassware under nitrogen atmosphere using anhydrous solvents. Anhydrous toluene, acetonitrile, dichloromethane, chloroform, dimethyl sulfoxide and tetrahydrofuran were purified by the Innovative® solvent purification system. Other anhydrous solvents were purchased from J&K Scientific. ^{1}H, ^{13}C and ^{19}F NMR spectra were collected on a Bruker AV 400 MHz NMR spectrometer using residue solvent peaks as an internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm, Acetone-d_6 at 2.05 ppm, Benzene-d_6 at 7.16 ppm,; ^{13}C NMR: $CDCl_3$ at 77.15 ppm, Acetone-d_6 at 206.26 ppm, Benzene-d_6 at 128.06 ppm). Mass spectra were collected on an Agilent GC/MS 5975C system, a MALDI Micro MX mass spectrometer, or an API QSTAR XL System with TOF as mass analyzer.

II. Preparation of Substrates and characterization

Substrates **1a**-**1i** and **5** are known compounds, were synthesized according to the literature procedure.



General Procedure A: Preparation of 3-Aminooxetanes.[1]



Under N_2 atmosphere, to a solution of iodobenzenes (5.0 mmol, 1.0 equiv), 3-aminooxetane (548.2 mg, 7.5 mmol, 1.5 equiv) and KOH (561.1 mg, 10.0 mmol, 2.0 equiv) in glycerol (25 mL) was added $Cu(acac)_2$ (26.2 mg, 1.0 mmol, 0.2 equiv). Then the reaction mixture was stirred at 100 ˚C on an oil bath for 15 h. The reaction mixture was diluted with H_2O (30 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic layers dried over Na_2SO_4, concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired 3-aminooxetanes **1**.

***N*-Phenyloxetan-3-amine (1a)** was prepared according to the General Procedure A as a white solid (chromatography eluent: petroleum ether/ ethyl acetate = 4:1) in 58% yield (335.8 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 7.23-7.17 (m, 2H), 6.78 (t, *J* = 5.5 Hz, 1H),

[1] Zhang J.; Hu, X. *Org. Chem. Front.*, **2021**, *8*, 6616-6621.

6.52-6.50 (m, 2H), 5.00 (t, *J* = 4.9 Hz, 2H), 4.67-4.61 (m, 1H), 4.53 (t, *J* = 4.7 Hz, 2H) ppm.

***N*-(*p*-Tolyl) oxetan-3-amine (1b)** was prepared according to the General Procedure A as a yellow solid (chromatography eluent: petroleum ether/ ethyl acetate = 4:1) in 26% yield (213.5 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 7.01 (d, *J* = 6.0 Hz, 2H), 6.46-6.42 (m, 2H), 4.99 (t, *J* = 4.9 Hz, 2H), 4.64-4.58 (m, 1H), 4.52 (t, *J* = 4.6 Hz, 2H), 2.25 (s, 3H) ppm.

***N*-(4-Methoxyphenyl) oxetan-3-amine (1c)** was prepared according to the General Procedure A as a brown solid (chromatography eluent: petroleum ether/ethyl acetate = 4:1) in 36% yield (324.3 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 6.80-6.76 (m, 2H), 6.50-6.46 (m, 2H), 4.98 (t, *J* = 4.7 Hz, 2H), 4.60-4.54 (m, 1H), 4.50 (t, *J* = 4.6 Hz, 2H) 3.74 (s, 3H) ppm.

***N*-(4-Fluorophenyl) oxetan-3-amine (1d)** was prepared according to the General Procedure A as a yellow solid (chromatography eluent: petroleum ether/ethyl acetate = 4:1) in 42% yield (352.7 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 6.93-6.87 (m, 2H), 6.46-6.41 (m, 2H), 4.99 (t, *J* = 4.7 Hz, 2H), 4.59-4.53 (m, 1H), 4.50 (t, *J* = 4.6 Hz, 2H) ppm.

***N*-(4-Chlorophenyl) oxetan-3-amine (1e)** was prepared according to the General Procedure A as a brown solid (chromatography eluent: petroleum ether/ethyl acetate = 4:1) in 49% yield (447.9 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 7.15-7.11 (m, 2H), 6.43-6.40 (m, 2H), 4.99 (t, *J* = 4.8 Hz, 2H), 4.61-4.55 (m, 1H), 4.50 (t, *J* = 4.6 Hz, 2H) ppm.

***N*-(*m*-Tolyl) oxetan-3-amine (1f)** was prepared according to the General

Procedure A as a yellow oil (chromatography eluent: petroleum ether/ ethyl acetate = 4:1) in 30% yield (348.2 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 7.09 (td, *J* = 5.6, 0.5 Hz, 1H), 6.61 (d, *J* = 5.6 Hz, 1H), 6.34-6.31 (m, 2H), 5.01 (t, *J* = 4.9 Hz, 2H), 4.66-4.60 (m, 1H), 4.53 (t, *J* = 4.6 Hz, 2H), 2.29 (s, 3H) ppm.

***N*-(3-Fluorophenyl) oxetan-3-amine (1g)** was prepared according to the General Procedure A as a white solid (chromatography eluent: petroleum ether/ ethyl acetate = 4:1) in 53% yield (443.6 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 7.11 (td, *J* = 6.1, 5.0 Hz, 1H), 6.45 (tdd, *J* = 6.6, 1.8, 0.5 Hz, 1H), 6.27 (ddd, *J* = 6.1, 1.6, 0.5 Hz, 1H), 6.17 (dt, *J* = 8.4, 1.7 Hz, 1H), 5.00 (t, *J* = 4.8 Hz, 2H), 4.62-4.56 (m,1H), 4.52 (t, *J* = 5.1 Hz, 2H) ppm.

***N*-(3-Chlorophenyl) oxetan-3-amine (1h)** was prepared according to the General Procedure A as a yellow solid (chromatography eluent: petroleum ether/ethyl acetate = 4:1) in 52% yield (476.6 mg).

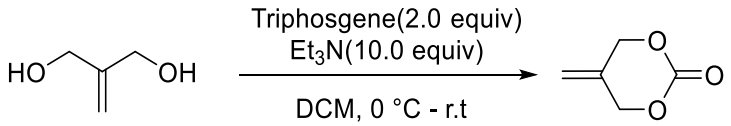
^{1}H NMR (300MHz, $CDCl_3$) δ 7.09 (t, *J* = 6.0 Hz, 1H), 6.73 (ddd, *J* = 5.9, 1.2, 0.6 Hz, 1H), 6.45 (t, *J* = 1.6 Hz, 1H), 6.37 (ddd, *J* = 6.1, 1.7, 0.5 Hz, 1H), 5.00 (t, *J* = 4.9 Hz, 2H), 4.62-4.56 (m, 1H), 4.51 (t, *J* = 4.6 Hz, 2H) ppm.

***N*-(4-Bromo-3,5-dimethylphenyl) oxetan-3-amine(1i)** was prepared according to the General Procedure A as a yellow solid (chromatography eluent: petroleum ether/ethyl acetate = 4:1) in 59% yield (755.6 mg).

^{1}H NMR (300MHz, $CDCl_3$) δ 6.25 (s, 2H), 4.99 (t, *J* = 4.9 Hz, 2H), 4.61-4.55 (m, 1H), 4.49 (t, *J* = 4.6 Hz, 2H), 2.33 (s, 6H) ppm.

General Procedure B: Preparation of the 2-Alkylidenetrimethylenes 5.[2]

[2] Zhang, S.; Zhang, L. *Org. Lett.* **2021**, *23*, 15, 5719-5723.



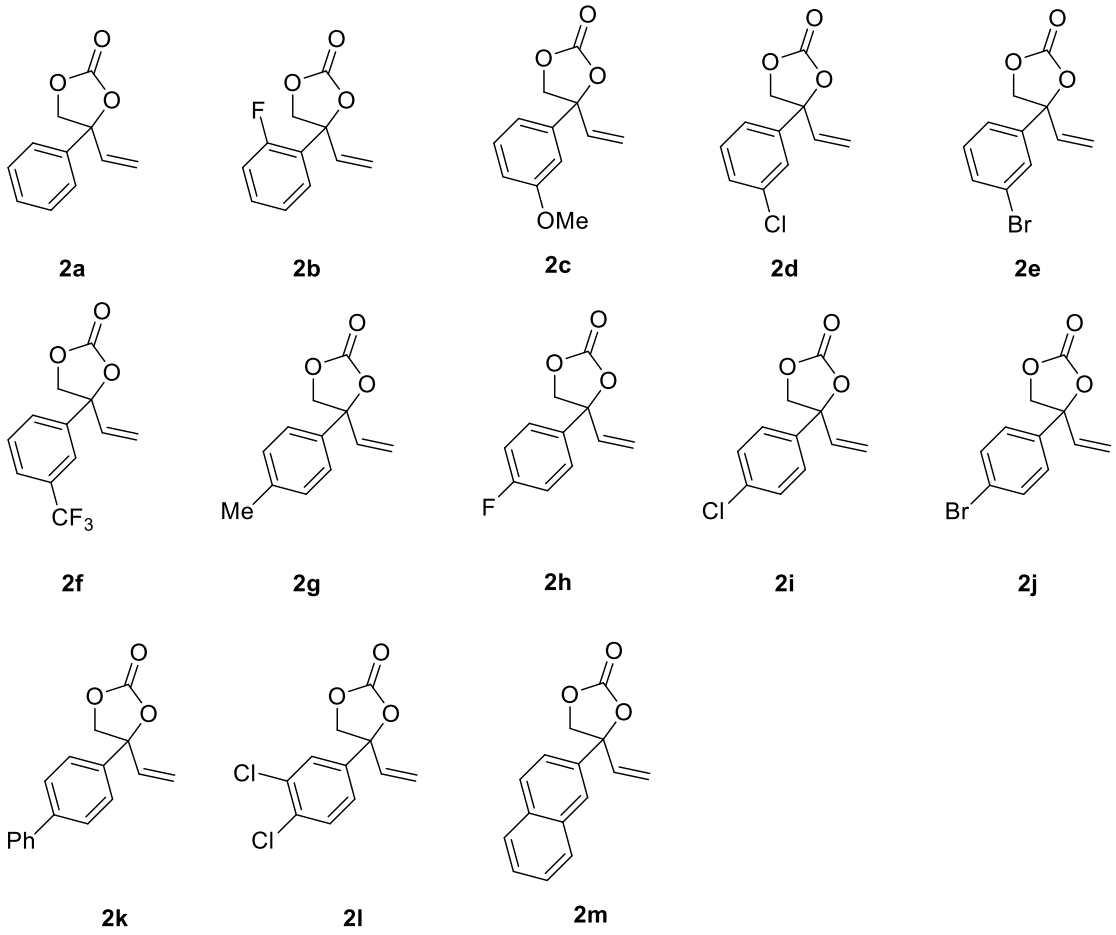
5

Under N_2 atmosphere at 0 °C, to a solution of 2-methylenepropane-1,3-diol (1.14 g, 10.0 mmol, 1.0 equiv) and Et_3N (13.8 mL, 10.0 equiv) in DCM (40 mL) was added dropwise triphosgene (50 mL, 2.5 M in DCM, 2.0 equiv). Then the reaction mixture was stirred at room temperature for 1 h. The reaction mixture was quenched in an ice bath with saturated ammonium chloride solution and extracted with DCM (30 mL × 3). The combined organic layers were washed with water, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate= 4:1) to afford **5** in 57% yield (656 mg).

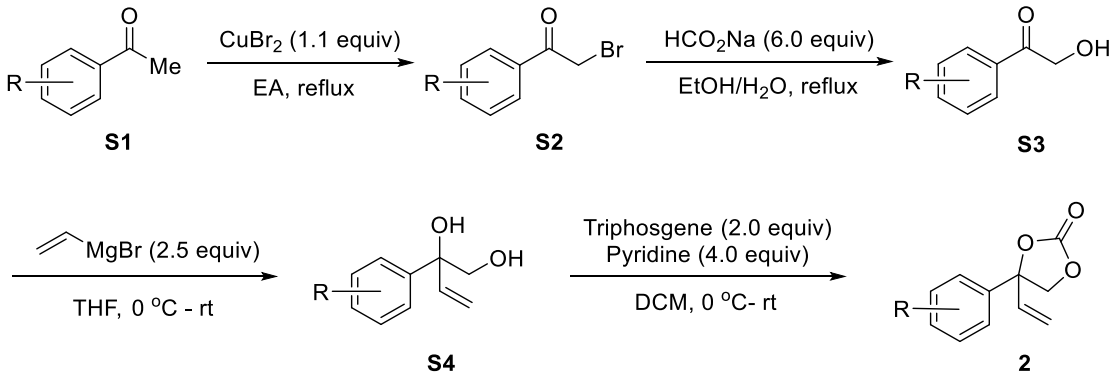
^{1}H NMR (300MHz, $CDCl_3$) δ 5.31 (s, 2H), 4.85 (s, 4H) ppm.

Substrates **2a**-**2m** are known compounds, were synthesized according to the literature procedure.[3]

[3] a) Ke, M.; Chen, F. *Org. Lett.* **2020**, *22*, 11, 4135-4140; b) Huang, Q.; Li, J. *ACS Catal.* **2021**, *11*, 16, 10148-10158; c) Ajmal K.; Zhang, Y. *Angew. Chem. Int. Ed.* **2014**, *53*, 6439-6442.



General Procedure C: Preparation of the Substituted Vinylethylene Carbonates (VECs) 2.



Under N_2 atmosphere, to a solution of acetophenones **S1** (5.00 mmol, 1.0 equiv) in ethyl acetate (15 mL) was added copper (II) bromide (1.23 g, 5.50 mmol, 1.1 equiv). The reaction mixture was warmed up to reflux and kept stirring for 5 h. Then the mixture was filtered through Celite, and the filter

cake was washed with ethyl acetate. Subsequently, the combined filtrates were concentrated under reduced pressure, then the residue was washed with diethyl ether to afford crude products **S2**.

Under an N_2 atmosphere, to a solution of **S2** (5.0 mmol, 1.0 equiv) in 85% of ethanol (15 mL) was added sodium formate (2.04g, 30.0 mmol). The reaction mixture was warmed up to reflux and kept stirring for 12 h. Then the reaction was quenched with H_2O (30 mL) and extracted with ethyl acetate (50 mL x 3). The combined organic extracts were washed with brine, dried over Na_2SO_4, concentrated under reduced pressure to afford crude products **S3**.

Under N_2 atmosphere at 0 ^oC, to a solution of **S3** (5.0 mmol, 1.0 equiv) in THF (20 mL) was added vinylmagnesium bromide (20 mL, 1.0 M in THF, 2.5 equiv). Then the reaction was stirred at room temperature for 3 h. The reaction was quenched by aq. NH_4Cl (15 mL) at 0 °C, and extracted with ethyl acetate (10 mL × 3). The combined organic phases were dried over Na_2SO_4, concentrated under reduced pressure. Then the residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired products **S4.**

Under N_2 atmosphere at 0 °C, to a solution of **S4** (5.0 mmol, 1.0 equiv) and triphosgene (10 mmol, 2.97 g 2.0 equiv) in DCM (15 mL) was added pyridine (1.6 ml, 20 mmol, 4.0 equiv). Then the reaction was stirred at room temperature for 10 h. The reaction was quenched by aq. NH_4Cl (15 mL) and extracted with DCM (10 mL × 3). Then organic phase was dried over Na_2SO_4, concentrated under reduced pressure. Then the residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired products **2**.

4-Phenyl-4-vinyl-1,3-dioxolan-2-one (2a) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 83% yield (789.3 mg).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.44-7.35 (m, 5H), 6.16 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.43 (s, 1H), 5.39 (d, *J* = 6.2 Hz, 1H), 4.66 (d, *J* = 8.5 Hz, 1H), 4.57 (d, *J* = 8.5 Hz, 1H) ppm.

4-(2-Fluorophenyl)-4-vinyl-1,3-dioxolan-2-one (2b) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 20.5% yield (213.4 mg).
^{1}H NMR (400MHz, $CDCl_3$) δ 7.55 (td, *J* = 7.7, 1.7 Hz, 1H), 7.41-7.35 (m, 1H), 7.22 (td, *J* = 7.6, 1.1 Hz, 1H), 7.11 (ddd, *J* = 11.1, 8.2, 1 Hz, 1H), 6.17 (ddd, *J* = 17.1, 10.6, 1.3 Hz, 1H), 5.40 (dd, *J* = 17.1, 1.2 Hz, 1H), 5.35 (d, *J* = 10.6 Hz, 1H), 4.74 (dd, *J* = 8.8, 2.6 Hz, 1H), 4.59 (dd, *J* = 8.8, 1.6 Hz, 1H)ppm.

4-(3-Methoxyphenyl)-4-vinyl-1,3-dioxolan-2-one (2c) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 71% yield (781.7 mg).
^{1}H NMR (400MHz, $CDCl_3$) δ 7.35-7.31 (m, 1H), 6.90-6.88 (m, 3H), 6.13 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.44-5.39 (m, 2H), 4.64 (d, *J* = 8.5 Hz, 1H), 4.55 (d, *J* = 8.5 Hz, 1H), 3.81 (s, 3H) ppm.

4-(3-Chlorophenyl)-4-vinyl-1,3-dioxolan-2-one (2d) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 64% yield (718.8 mg).
^{1}H NMR (400MHz, $CDCl_3$) δ 7.39-7.23 (m, 4H), 6.12 (dd, *J* = 17.1, 10.8 Hz, 1H), 5.46-5.40 (m, 2H), 4.65 (d, *J* = 8.6 Hz, 1H) 4.54 (d, *J* = 8.5 Hz, 1H) ppm.

4-(3-Bromophenyl)-4-vinyl-1,3-dioxolan-2-one (2e) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 56% yield (753.4 mg).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.52-7.50 (m, 2H), 7.33 -7.27 (m, 2H), 6.12 (dd, *J* = 17.1, 10.8 Hz, 1H), 5.46-5.40 (m, 2H), 4.65 (d, *J* = 8.6 Hz, 1H). 4.53 (d, *J* = 8.6 Hz, 1H) ppm.

4-(3-(Trifluoromethyl)phenyl)-4-vinyl-1,3-dioxolan-2-one(2f) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 72% yield (929.5 mg).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.66-7.57 (m, 4H), 6.16 (dd, *J* = 17.1, 10.7 Hz, 1H), 5.49-5.42 (m, 2H), 4.71 (d, *J* = 8.6 Hz, 1H), 4.56 (d, *J* = 8.6 Hz, 1H) ppm.

4-(*p*-Tolyl)-4-vinyl-1,3-dioxolan-2-one (2g) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 65% yield (663.7 mg).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.25-7.21 (m, 4H), 6.14 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.42-5.38 (m, 2H), 4.63 (d, *J* = 8.5 Hz, 1H), 4.56 (d, *J* = 8.4 Hz, 1H), 2.36 (s, 3H) ppm.

4-(4-Fluorophenyl)-4-vinyl-1,3-dioxolan-2-one(2h) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ ethyl acetate = 10:1) in 57% yield (593.3 mg).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.36-7.33 (m, 2H), 7.14-7.09 (m, 2H), 6.13 (dd, *J* = 17.1, 10.7 Hz, 1H), 5.45-5.38 (m, 2H), 4.64 (d, *J* = 8.6 Hz, 1H), 4.55 (d, *J* = 8.5 Hz, 1H) ppm.

4-(4-Chlorophenyl)-4-vinyl-1,3-dioxolan-2-one(2i) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 62% yield (696.3 mg).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.41-7.39 (m, 2H), 7.31-7.29 (m, 2H), 6.12 (dd, (dd, *J* = 17.1, 10.7 Hz, 1H), 5.45-5.38 (m, 2H), 4.64 (d, *J* = 8.6 Hz, 1H), 4.53 (d, *J* = 8.6 Hz, 1H) ppm.

4-(4-Bromophenyl)-4-vinyl-1,3-dioxolan-2-one(2j) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 53% yield (707.8 mg).
^{1}H NMR (400MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.5 Hz, 2H), 6.12 (dd, *J* = 17.1, 10.7 Hz, 1H), 5.45-5.38 (m, 2H), 4.64 (d, *J* = 8.5 Hz, 1H), 4.53 (d, *J* = 8.6 Hz, 1H) ppm.

4-([1,1'-Biphenyl]-4-yl)-4-vinyl-1,3-dioxolan-2-one(2k) was prepared according to the General Procedure C as a white solid (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 45.7% yield (608.7 mg).
^{1}H NMR (400MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 7.3 Hz, 2H), 7.48-7.37 (m, 5H), 6.20 (dd, *J* = 17.1, 10.7 Hz, 1H), 5.49-5.44 (m, 2H), 4.69 (d, *J* = 8.5 Hz, 1H), 4.62 (d, *J* = 8.5 Hz, 1H) ppm.

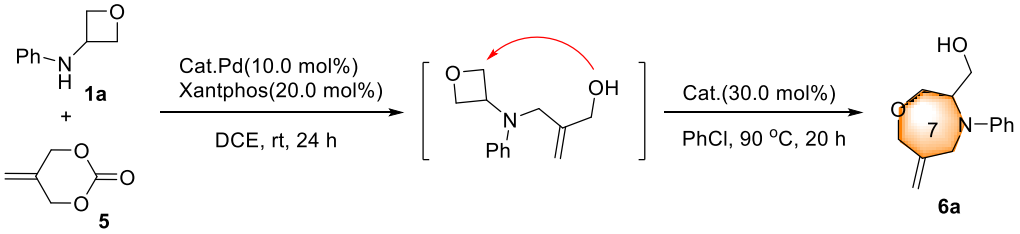
4-(3,4-Dichlorophenyl)-4-vinyl-1,3-dioxolan-2-one(2l) was prepared according to the General Procedure C as a light yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 55% yield (706.9 mg).
^{1}H NMR (400MHz, $CDCl_3$) δ 7.51-7.46 (m, 2H), 7.20 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.10 (dd, *J* = 17.1, 10.7 Hz, 1H), 5.48-5.40 (m, 2H), 4.65 (d, *J* = 8.6 Hz, 1H), 4.52 (d, *J* = 8.6 Hz, 1H) ppm.

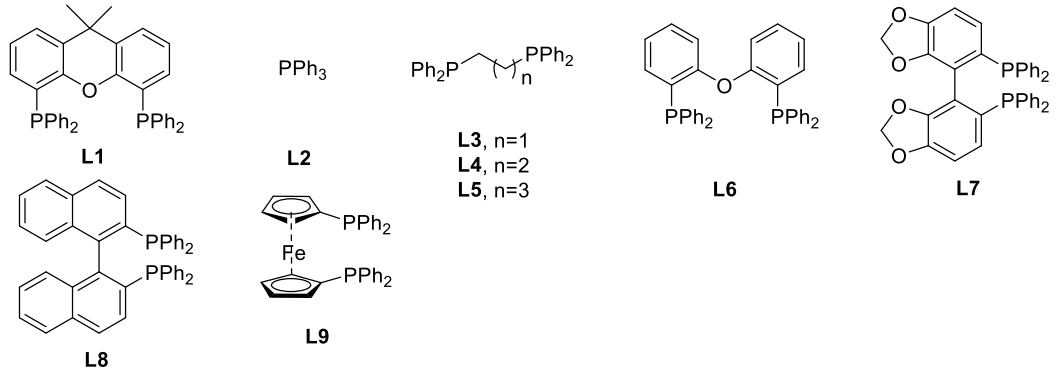
4-(Naphthalen-2-yl)-4-vinyl-1,3-dioxolan-2-one(2m) was prepared according to the General Procedure C as a brawn solid (chromatography eluent: petroleum ether/ethyl acetate = 10:1) in 87% yield (1.05 g).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.92-7.84 (m, 4H), 7.56-7.52 (m, 2H), 7.39 (dd, *J* = 8.6, 1.8 Hz, 1H), 6.24 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.48-5.44 (m, 2H), 4.73 (d, *J* = 8.5 Hz, 1H), 4.68 (d, *J* = 8.5 Hz, 1H) ppm.

III. The Optimization of Reaction Conditions for 1,4-Oxazepanes

Table S1. The Optimization of Reaction Conditions for 1,4-Oxazepanes.





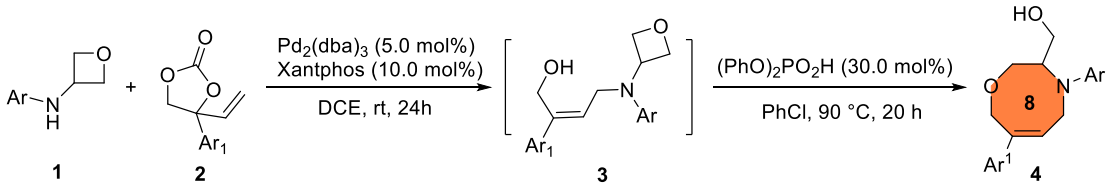
Entry	Cat. Pd	L	Cat.	Yield of **7a**[b]	Yield of **6a**[c]
1	$Pd(OAc)_2$	**L1**	--	85%	--
2	$PdCl_2$	**L1**	--	<10%	--
3	$Pd(PPh_3)_4$	**L1**	--	72%	--
4	Pd_2dba_3	**L1**	--	68%	--
5	$Pd(OAc)_2$	**L2**	--	NR	--
6	$Pd(OAc)_2$	**L3**	--	NR	--
7	$Pd(OAc)_2$	**L4**	--	NR	--
8	$Pd(OAc)_2$	**L5**	--	NR	--
9	$Pd(OAc)_2$	**L6**	--	32%	--
10	$Pd(OAc)_2$	**L7**	--	<10%	--
11	$Pd(OAc)_2$	**L8**	--	<10%	--
12	$Pd(OAc)_2$	**L9**	--	NR	--
15	$Pd(OAc)_2$	**L1**	$B(C_6F_5)_3$	--	41%
16	$Pd(OAc)_2$	**L1**	$(PhO)_2PO_2H$	--	55%
17	$Pd(OAc)_2$	**L1**	TFA	--	ND
18	$Pd(OAc)_2$	**L1**	TsOH	--	ND

[a] Reaction conditions: **1a** (0.2 mmol), **5** (0.3 mmol), Cat. Pd (0.01 mmol, 10.0 mol%) and **L** (0.04 mmol, 20.0 mol%) in DCE (2.0 mL) at rt for 24 h; then Cat. (0.06 mmol, 30.0 mol%) in PhCl (3.0

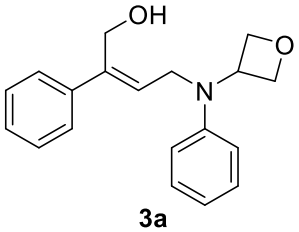
mL) at 90 °C for 20 h; NR = No Reaction; ND = Not determined. [b] Yield was determined based on crude NMR analysis using CH_2Br_2 as internal standard. [c] Total isolated yield.

IV. Synthesis of Meidum-sized Heterocycles

General Procedure D: The synthesis of tetrahydro-2*H*-1,4-oxazocines 4.



Under N_2 atmosphere, the solution of 3-aminooxetanes **1** (0.2 mmol, 1.0 equiv), vinylethylene carbonate **2** (0.6 mmol, 3.0 equiv) and Xantphos (11.6 mg, 0.02 mmol, 10.0 mol%) in DCE (2.0 mL) was added $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol, 5.0 mol%). Then the reaction mixture was stirred at room temperature for 24 h. The reaction mixture was filtrated through a short column to remove the polar impurities and concentrated to afford crude products **3**. Then the crude products **3** was added to the solution of $(PhO)_2PO_2H$ (15.0 mg, 0.06 mmol, 30 mol%) in PhCl (3.0 mL). The reaction mixture was stirred at 90 °C for 20 h, then concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired products **4**.



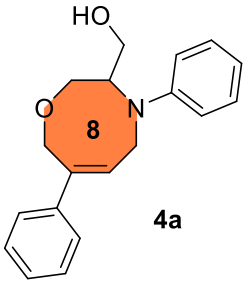
3a

(*Z*)-4-(Oxetan-3-yl(phenyl)amino)-2-phenylbut-2-en-1-ol (3a):

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.23 (m, 7H), 6.88 (t, J = 7.3 Hz, 1H), 6.72 (m, 2H), 5.85 (t, J = 6.5 Hz, 1H), 4.87-4.82(m, 2H), 4.74-4.69 (m, 3H), 4.55 (s, 2H), 4.15 (d, J = 6.5 Hz, 2H) ppm.

^{13}C NMR (400 MHz, $CDCl_3$) δ 148.3, 141.6, 140.2, 129.5, 128.7, 128.3, 127.8, 126.5, 120.4, 117.0, 76.5, 59.9, 53.7, 47.9 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{21}NO_2Na$: 318.1470; found 318.1465.



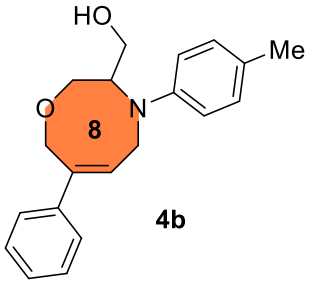
4a

(*Z*)-(4,7-Diphenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4a) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ ethyl acetate = 3:1) in 72% yield (42.5 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.35-7.29 (m, 4H), 7.26-7.22 (m, 1H), 7.20-7.15 (m, 2H), 6.88 (d, J = 8 Hz, 2H), 6.63 (tt, J = 7.2, 0.9 Hz, 1H), 6.31 (t, J = 5.7 Hz, 2H), 4.59 (dd, J = 5.7, 17.6 Hz, 1H), 4.53 (s, 2H), 4.21 (dd, J = 12.4, 5.7 Hz, 1H), 4.03-3.93 (m, 2H), 3.81-3.76 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 150.2, 143.5, 138.8, 129.9 (2C), 129.1, 127.8, 126.8, 117.1, 113.3, 70.1, 67.5, 60.5, 59.9, 44.5 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{21}NO_2Na$: 318.1470; found 318.1463.



4b

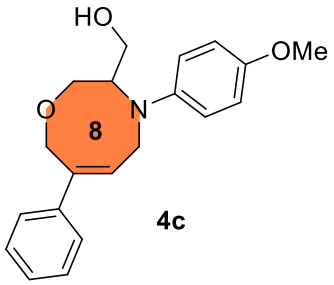
(*Z*)-(7-Phenyl-4-(*p*-tolyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4b) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ ethyl acetate = 3:1) in 61% yield (37.3 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.35 - 7.24 (m, 5H), 6.99 (d, J = 8.3 Hz, 2H), 6.77 (d, J = 8.6 Hz, 2H), 6.28 (t, J = 5.2 Hz, 1H), 4.57 - 4.51 (m, 3H), 4.17 (dd, J =

12.4, 5.9 Hz, 1H), 3.98 - 3.89 (m, 2H), 3.81 - 3.73 (m, 3H), 2.18 (s, 3H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 148.2, 143.7, 138.6, 130.52, 130.51, 129 .2, 127.9, 126.9, 126.0, 113.6, 70.0, 67.4, 60.7, 60.4, 45.0, 20.3 ppm.

HRMS (ESI) m/z: [M + Na]$^+$ calcd for $C_{20}H_{23}NO_2Na$ calcd.: 332.1598; found: 332.1617.



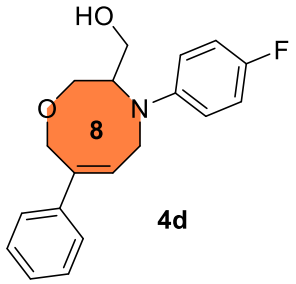
4c

(*Z*)-(4-(4-Methoxyphenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4c) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 47% yield (30.5 mg).

^{1}H NMR (400 MHz, *d*$_6$-Acetone) δ 7.35-7.24 (m, 5H), 7.85-6.79 (m, 4H), 6.26 (t, *J* = 5.4 Hz, 1H), 4.60-4.47 (m, 3H), 4.14 (dd, *J* = 12.2, 5.8 Hz, 1H), 3.95-3.83 (m, 2H), 3.82-3.72 (m, 3H), 3.69 (s, 3H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 152.6, 144.9, 143.8, 138.6, 130.8, 129.2, 127.9, 126.9, 115.6, 115.2, 69.9, 67.5, 61.0, 60.9, 55.8, 45.6 ppm.

HRMS (ESI) m/z: [M + Na]$^+$ calcd for $C_{20}H_{23}NO_3Na$ calcd.: 348.1598; found: 348.1566.



4d

(*Z*)-(4-(4-Fluorophenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl)

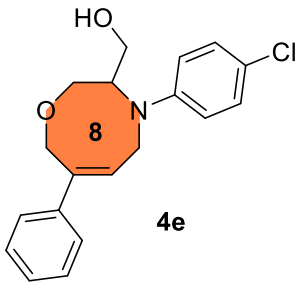
methanol (4d) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 71% yield (44.5 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 - 7.23 (m, 5H), 6.97 - 6.91 (m, 2H), 6.86 – 6.82 (m, 2H), 6.21 (t, *J* = 6.6 Hz, 1H), 4.67 (d, *J* = 15.7 Hz, 1H), 4.57 (dd, *J* = 16.6, 6.9 Hz, 1H), 4.42 (d, *J* = 15.7 Hz, 1H), 4.15 (dd, *J* = 12.3, 3.8 Hz, 1H), 3.91 - 3.75 (m, 5H) ppm.

^{13}C NMR (400 MHz, $CDCl_3$) δ 155.9(d, *J* = 237.4 Hz), 146.1 (d, *J* = 1.7 Hz), 141.7, 139.8, 128.5, 127.6, 127.5, 126.3, 115.9 (d, *J* = 22.3 Hz), 114.9 (d, *J* = 7.3 Hz), 71.1, 68.1, 60.6, 59.2, 43.3 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -127.6 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{20}FNO_2Na$ calcd.: 336.1376; found: 336.1368.



4e

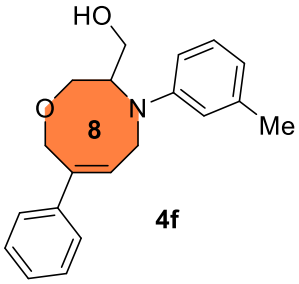
(*Z*)-(4-(4-Chlorophenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl)methanol (4e) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 70% yield (45.9 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 7.22 (m, 5H), 7.16 (d, *J* = 8.9 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.22 (t, *J* = 6.6 Hz, 1H), 4.70 - 4.38 (m, 3H), 4.16 (dd, *J* = 12.2, 3.3 Hz, 1H), 3.90 - 3.73 (m, 5H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.1, 143.2, 139.5, 129.60, 129.1, 129.0, 128.0, 126.9, 121.1, 114.7, 70.5, 67.9, 60.5, 59.9, 44.3 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{20}ClNO_2$ Na calcd.: 352.1081; found:

352.1081.



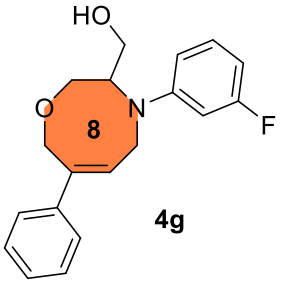
4f

(*Z*)-(7-Phenyl-4-(m-tolyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4f) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 53% yield (32.8 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.35-7.23 (m, 5H), 7.05 (t, J = 7.8 Hz, 1H), 6.71 (s, 1H), 6.67 (dd, J = 8.3, 2.3 Hz, 1H), 6.46 (d, J = 7.4 Hz, 1H), 6.30 (t, J = 5.6 Hz, 1H), 4.59-4.52 (m, 3H), 4.20 (dd, J = 12.4, 5.8 Hz, 1H), 3.99-3.93 (m, 2H), 3.81-3.74 (m, 3H), 2.25 (s, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 150.3, 143.6, 139.3, 138.7, 130.2, 129.9, 129.2, 127.9, 126.9, 118.2, 114.1, 110.7, 70.0, 67.5, 60.5, 60.0, 44.7, 22.0 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{20}H_{23}NO_2Na$ calcd.: 332.1598; found: 332.1618.



4g

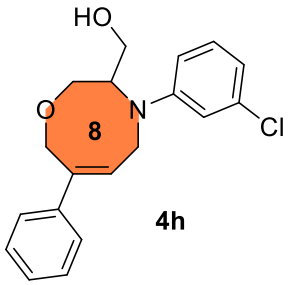
(*Z*)-(4-(3-Fluorophenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4g) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 63% yield (39.4 mg).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 - 7.14 (m, 5H), 7.06 (q, J = 8.0 Hz, 1H), 6.56 – 6.47 (m, 2H), 6.33 (t, J = 6.7 Hz, 1H), 6.16 (t, J = 6.6 Hz, 1H), 4.63-4.30 (m, 3H), 4.10 (dd, J = 12.2, 3.1 Hz, 1H), 3-81-3.64 (m, 5H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 165.0 (d, J = 240.4 Hz), 152.2 (d, J = 10.8 Hz), 143.1, 139.6, 131.2 (d, J = 10.5 Hz), 129.2, 128.8, 128.0, 126.9, 109.1 (d, J = 2.0 Hz), 103.1 (d, J = 21.8 Hz), 100.0 (d, J = 26.4 Hz), 70.6, 68.0, 60.4, 59.9, 44.1 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -113.9 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{20}FNO_2Na$ calcd.: 336.1398; found: 336.1371.



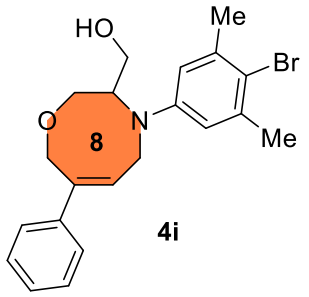
4h

(*Z*)-(4-(3-Chlorophenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4h) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 68% yield (44.8 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.35-7.23 (m, 5H), 7.15 (t, J = 8.2 Hz, 1H), 6.88 (t, J = 2.2 Hz, 1H), 6.84 (dd, J = 8.4, 2.4 Hz, 1H), 6.62 (dd, J = 7.8, 1.2 Hz, 1H), 6.32 (t, J = 6.1 Hz, 1H), 4.68-4.46 (m, 3H), 4.22 (dd, J = 12.4, 5.0 Hz, 1H), 3.97-3.92 (m, 2H), 3.80-3.76 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 151.6, 143.2, 139.8, 135.5, 131.2, 129.2, 128.7, 128.1, 127.0, 116.6, 112.9, 111.9, 70.7, 68.1, 60.4, 59.8, 44.1 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{20}ClNO_2Na$ calcd.: 352.1098; found: 352.1076.



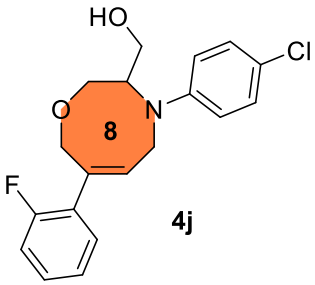
4i

(*Z*)-(4-(4-Bromo-3,5-dimethylphenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4i) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 77% yield (61.9 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.34-7.23 (m, 5H), 6.72 (s, 2H), 6.30 (t, J = 5.9 Hz, 1H), 4.59-4.47 (m, 3H), 4.19 (dd, J = 12.4, 5.4 Hz, 1H), 3.97-3.92 (m, 2H), 3.79-3.74 (m, 3H), 2.31 (s, 6H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 148.9, 143.2, 139.1, 138.8, 129.3, 129.1, 127.9, 126.8, 113.9, 113.5, 70.3, 67.6, 60.3, 59.6, 44.2, 24.2 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{21}H_{24}BrNO_2$ Na calcd.: 424.0888; found: 424.0886.



4j

(*Z*)-(4-(4-Chlorophenyl)-7-(2-fluorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4j) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 64% yield (44.5 mg).

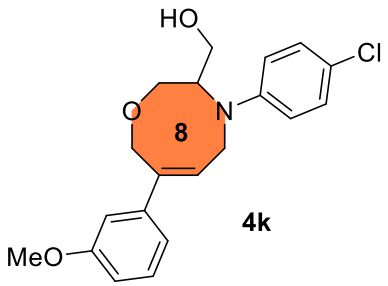
^{1}H NMR (400 MHz, d_6-Acetone) δ 7.34-7.24 (m, 2H), 7.17-7.07 (m, 4H), 6.89 (d, J = 8.9 Hz, 2H), 6.23 (t, J = 6.0 Hz, 1H), 4.64 (dd, J = 17.2, 6.3 Hz, 1H), 4.42 (dd, J = 68.1, 15.4 Hz, 2H), 4.22 (dd, J = 12.4, 4.6 Hz, 1H), 3.94-3.88 (m, 2H), 3.81-3.78

(m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 160.5 (d, J = 245.5 Hz), 149.0, 136.2, 131.6, 131.2 (d, J = 4.2 Hz), 130.7, 130.1 (d, J = 8.2 Hz), 129.6, 125.3 (d, J = 3.4 Hz), 121.1, 116.3 (d, J = 22.5 Hz), 114.7, 71.0 (d, J = 3.8 Hz), 68.2, 60.4, 59.6, 43.8 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -115.4 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{19}ClFNO_2H$ calcd.: 348.1167; found: 348.1162.



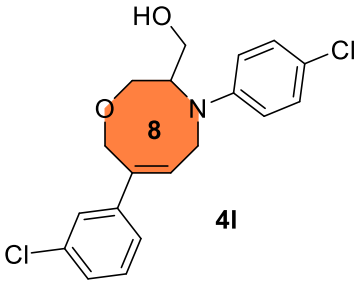
4k

(*Z*)-(4-(4-Chlorophenyl)-7-(3-methoxyphenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4k) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 74% yield (53.2 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.23 (t, J = 7.9 Hz, 1H), 7.17-7.13 (m, 2H), 6.91-6.86 (m, 4H), 6.83 (dd, J = 8.2, 2.5 Hz, 1H), 6.32 (t, J = 5.8 Hz, 1H), 4.63 – 4.44 (m, 3H), 4.19 (dd, J = 12.4, 5.3 Hz, 1H), 3.94-3.89 (m, 2H), 3.78-3.74 (m, 6H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 160.5, 149.0, 144.6, 139.3, 130.1, 129.5, 129.1, 121.0, 119.2, 114.7, 113.2, 112.6, 70.4, 67.8, 60.4, 59.9, 55.3, 44.2 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{20}H_{22}ClNO_3H$ calcd.: 360.1367; found: 360.1363.



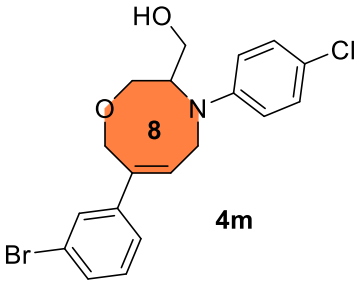
4l

(*Z*)-(7-(3-Chlorophenyl)-4-(4-chlorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazoci n-3-yl) methanol (4l) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 66% yield (48.0 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.37-7.27 (m, 4H), 7.16 (d, J = 9.1 Hz, 2H), 6.89 (d, J = 9.1 Hz, 2H), 6.37 (t, J = 5.6 Hz, 1H), 4.64-4.46 (m, 3H), 4.18 (dd, J = 12.6, 5.7 Hz, 1H), 3.99-3.91 (m, 2H), 3.80-3.75 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 150.0, 145.5, 137.8, 134.5, 130.9, 130.8, 129.6, 127.8, 126.8, 125.4, 121.2, 144.7, 69.7, 67.5, 60.4, 60.1, 44.5 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{19}Cl_2NO_2H$ calcd.: 364.0872; found: 364.0864.



4m

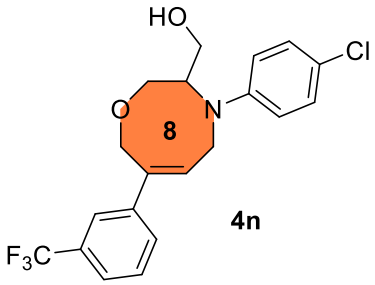
(*Z*)-(7-(3-Bromophenyl)-4-(4-chlorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazoci n-3-yl) methanol (4m) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 51% yield (41.6 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.51 (s, 1H), 7.44 (d, J = 7.8 Hz, 1H), 7.35-7.26 (m, 2H), 7.16 (d, J = 8.8 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 6.36 (t, J = 5.4 Hz, 1H), 4.61(dd, J = 17.7, 5.6 Hz, 1H), 4.50 (d, J = 4.6 Hz, 2H), 4.18(dd, J = 12.5,

5.6 Hz, 1H), 3.99-3.93 (m, 2H), 3.81-3.76 (m, 3H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 149.1, 145.9, 137.8, 131.2, 131.1, 130.8, 129.9, 129.7, 125.9, 122.9, 121.3, 114.9, 69.9, 67.6, 60.6, 60.3, 44.7 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{19}BrClNO_2H$ calcd.: 408.0367; found: 408.0361.



4n

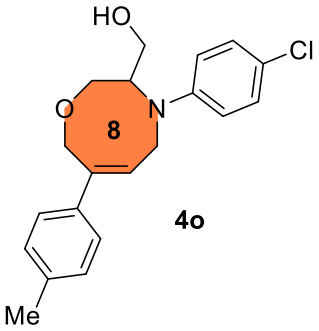
(*Z*)-(4-(4-Chlorophenyl)-7-(3-(trifluoromethyl)phenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl)methanol (4n) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 51% yield (40.5 mg).

^{1}H NMR (400 MHz, *d*$_6$-Acetone) δ 7.66-7.55 (m, 4H), 7.16 (d, *J* = 9.0 Hz, 2H), 6.90 (d, *J* = 9.0 Hz, 2H), 6.43 (t, *J* = 5.4 Hz, 1H), 4.63 (dd, *J* = 17.9, 5.4 Hz, 1H), 4.55 (s, 2H), 4.19 (dd, *J* = 12.6, 5.8 Hz, 1H), 4.03-3.93 (m, 2H), 3.84-3.77 (m, 3H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 149.1, 144.6, 137.8, 131.8, 131.1, 130.8, 130.2, 129.7, 125.3 (q, *J* = 273.0 Hz), 124.6 (q, *J* = 3.8 Hz), 123.6 (q, *J* = 3.9 Hz), 121.3, 114.9, 69.7, 67.4, 60.6, 60.4, 44.8 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -62.6 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{20}H_{19}ClF_3NO_2H$ calcd.: 398.1135; found: 398.1134.



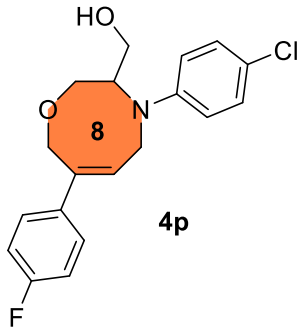
4o

(*Z*)-(4-(4-Chlorophenyl)-7-(p-tolyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4o) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 68% yield (46.7 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.21 (d, J = 8.2 Hz, 2H), 7.16-7.11 (m, 4H), 6.90-6.86 (m, 2H), 6.27 (t, J = 6.0 Hz, 1H), 4.64-4.42 (m, 3H), 4.20 (dd, J = 12.4, 5.0 Hz, 1H), 3.92-3.87 (m, 2H), 3.78-3.73 (m, 3H), 2.28 (s, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.0, 140.2, 139.4, 137.5, 129.7, 129.5, 128.0, 126.7, 120.9, 114.6, 70.6, 68.0, 60.4, 59.8, 44.0, 20.9 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{20}H_{22}ClNO_2Na$ calcd.: 366.1198; found: 366.1227.



4p

(*Z*)-(4-(4-Chlorophenyl)-7-(4-fluorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4p) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 63% yield (43.8 mg).

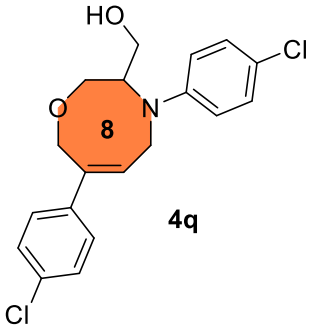
^{1}H NMR (400 MHz, d_6-Acetone) δ 7.39-7.34 (m, 2H), 7.17-7.05(m, 4H), 6.90-6.86 (m, 2H), 6.29 (t, J = 5.9 Hz, 1H), 4.64-4.43 (m, 3H), 4.20 (dd, J = 12.5,

5.3 Hz, 1H), 3.95-3.89 (m, 2H), 3.79-3.74 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 162.8 (d, J = 245.0 Hz), 149.0, 139.5 (d, J = 3.0 Hz), 138.4, 129.5, 129.2, 128.8 (d, J = 8.1 Hz), 121.0, 115.8 (d, J = 21.5 Hz), 114.7, 70.4, 67.8, 60.4, 59.9, 44.2 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -114.7 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{19}ClFNO_2Na$ calcd.: 370.0998; found: 370.0976.



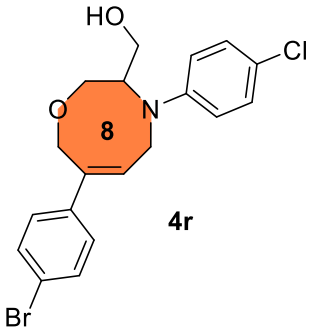
4q

(*Z*)-(4,7-Bis(4-chlorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4q) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 61% yield (44.4 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.34 (s, 4H), 7.16-7.14 (m, 2H), 6.89-6.87 (m, 2H), 6.34 (t, J = 5.7 Hz, 1H), 4.63-4.44 (m, 3H), 4.18 (dd, J = 12.5, 5.4 Hz, 1H), 3.97-3.91 (m, 2H), 3.78-3.75 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.0, 142.0, 138.0, 133.3, 130.1, 129.6, 129.1, 128.5, 121.1, 114.7, 70.0, 67.7, 60.4, 60.0, 44.4 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{19}Cl_2NO_2Na$ calcd.: 386.0698; found: 386.0701.



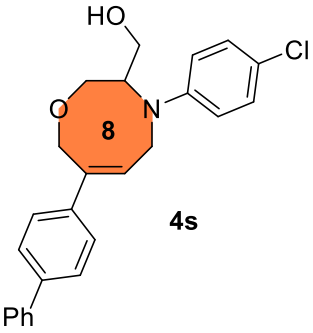
4r

(*Z*)-(7-(4-Bromophenyl)-4-(4-chlorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4r) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 51% yield (41.6 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.49 (d, J = 8.6 Hz, 2H), 7.29 (d, J = 8.5 Hz, 2H), 7.15 (d, J = 9.1 Hz, 2H), 6.88 (d, J = 8.6 Hz, 2H), 6.35 (t, J = 5.7 Hz, 1H), 4.60 (dd, J = 17.7, 5.8 Hz, 1H), 4.50 (q, J = 28.0, 15.0 Hz, 2H), 4.18 (dd, J = 12.5, 5.5 Hz, 1H), 3.97-3.91 (m, 2H), 3.77 (t, J = 6.3 Hz, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.0, 142.5, 138.1, 132.2, 130.2, 129.6, 128.9, 121.4, 121.1, 114.7, 69.9, 67.6, 60.4, 60.1, 44.4 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{19}BrClNO_2H$ calcd.: 408.0367; found: 408.0357.



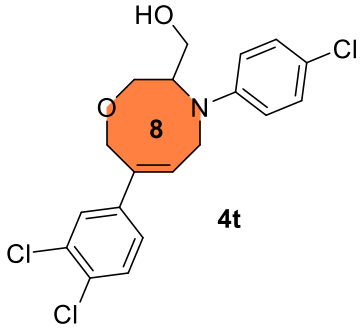
4s

(*Z*)-(7-([1,1'-Biphenyl]-4-yl)-4-(4-chlorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4s) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 79% yield (64.1 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.66-7.61 (m, 4H), 7.47-7.42 (m, 4H), 7.3 (t, J = 7.2 Hz, 1H), 7.16 (d, J = 8.9 Hz, 2H), 6.91 (d, J = 8.9 Hz, 2H), 6.39 (t, J = 5.8 Hz, 1H), 4.67-4.50 (m, 3H), 4.22 (dd, J = 12.4, 5.2 Hz, 1H), 3.99-3.93 (m, 2H), 3.80-3.78 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.1, 142.2, 141.3, 140.6, 139.0, 129.7, 129.6, 129.2, 128.2, 127.7, 127.54, 127.46 121.1, 114.8, 70.4, 68.0, 60.5, 60.0, 44.4 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{25}H_{24}ClNO_2H$ calcd.: 406.1575; found: 406.1566.



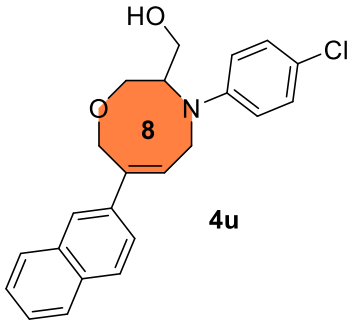
4t

(*Z*)-(4-(4-Chlorophenyl)-7-(3,4-dichlorophenyl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl) methanol (4t) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 56% yield (44.7 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.53 (s, 1H), 7.51 (d, J = 8.5 Hz, 1H), 7.322 (d, J = 8.4 Hz, 1H), 7.15 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 6.42 (t, J = 5.4 Hz, 1H), 4.61 (dd, J = 17.8, 5.5 Hz, 1H), 4.50 (s, 2H), 4.17 (dd, J = 12.5, 5.7 Hz, 1H), 4.00-3.93 (m, 2H), 3.81-3.76 (m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.1, 144.1, 136.9, 132.6, 131.8, 131.3, 131.2, 129.7, 128.9, 127.0, 121.4, 114.9, 69.6, 67.5, 60.6, 60.3, 44.8 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{18}Cl_3NO_2H$ calcd.: 398.0482; found: 398.0485.



4u

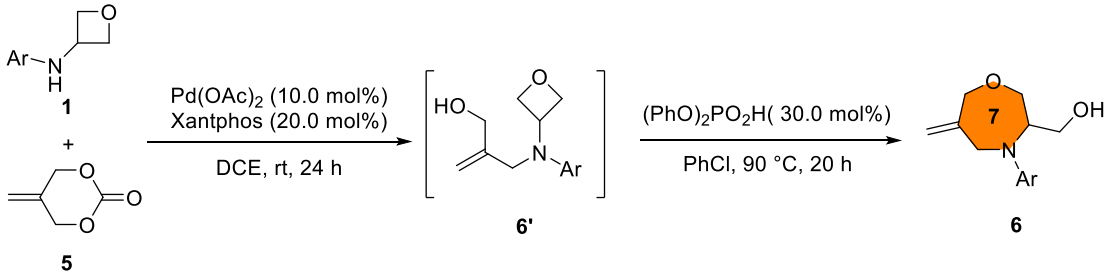
(*Z*)-(4-(4-Chlorophenyl)-7-(naphthalen-2-yl)-3,4,5,8-tetrahydro-2*H*-1,4-oxazoc in-3-yl) methanol (4u) was prepared according to the General Procedure D as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 63% yield (47.8 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.88-7.82 (m, 4H), 7.53-7.44 (m, 3H), 7.17 (d, J = 8.9 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 6.46 (t, J = 5.6 Hz, 1H), 4.72-4.57 (m, 3H), 4.24 (dd, J = 5.3, 12.5 Hz, 1H), 4.01-3.95 (m, 2H), 3.84-3.79 (m, 3H) ppm.

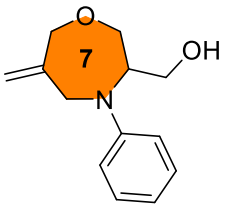
^{13}C NMR (400 MHz, d_6-Acetone) δ 149.1, 140.5, 139.1, 134.3, 133.5, 129.9, 129.6, 128.8, 128.7, 128.3, 127.0, 126.6, 125.5, 125.3, 121.1, 114.8, 70.3, 67.8, 60.5, 60.1, 44.5 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{23}H_{22}ClNO_2H$ calcd.: 380.1418; found: 380.1413.

General Procedure E: The synthesis of 1,4-oxazepanes 6.



Under N_2 atmosphere, the solution of 3-aminooxetanes **1** (0.2 mmol, 1.0 equiv), 5-methylene-1,3-dioxan-2-one **5** (0.3 mmol, 1.5 equiv) and Xantphos (23.1 mg, 0.04 mmol, 20.0 mol%) in DCE (2.0 mL) was added $Pd(OAc)_2$ (4.5 mg, 0.02 mmol, 10.0 mol%). Then the reaction mixture was stirred at room temperature for 24 h. The reaction mixture was filtrated through a short column to remove the polar impurities and concentrated to afford crude products **6′**. Then the crude products **6′** was added to the solution of $(PhO)_2PO_2H$ (15.0 mg, 0.06 mmol, 30 mol%) in PhCl (3.0 mL). The reaction mixture was stirred at 90 °C for 20 h, then concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired products **6**.



6a

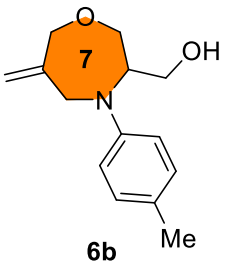
(6-Methylene-4-phenyl-1,4-oxazepan-3-yl) methanol(6a) was prepared according to the General Procedure E as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 55% yield (24.1 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.18-7.14 (m, 2H), 6.90-6.87 (m, 2H), 6.64 (tt, J = 7.2, 0.9 Hz, 1H), 5.08 (q, J = 1.2 Hz, 1H), 4.97 (q, J = 1.2 Hz, 1H), 4.27 (d, J = 14.4 Hz, 1H), 4.14 (s, 2H), 4.02-3.98 (m, 1H), 3.90-3.84 (m, 4H), 3.76 (dd, J = 12.6,

3.4 Hz, 1H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 150.3, 149.2, 129.8, 117.4, 113.9, 110.9, 76.1, 72.5, 60.9, 60.6, 48.8 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{13}H_{17}NO_2Na$ calcd.: 242.1157; found: 242.1156.



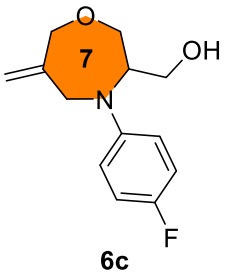
6b

(6-Methylene-4-(p-tolyl)-1,4-oxazepan-3-yl) methanol (6b) was prepared according to the General Procedure E as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 51% yield (23.8 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 6.97 (d, J = 8.4 Hz, 2H), 6.79 (d, J = 8.7 Hz, 2H), 5.05 (d, J = 1 Hz, 1H), 4.95 (d, J = 1 Hz, 1H), 4.26 (d, J = 14.4 Hz, 1H), 4.10 (s, 2H), 4.01-3.97 (m, 2H), 3.84-3.83 (m, 3H), 3.76-3.72 (m, 1H), 2.18 (s, 3H)ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 149.6, 148.2, 130.4, 126.3, 114.3, 110.8, 76.2, 72.5, 61.1, 60.8, 49.0, 20.2 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{14}H_{19}NO_2Na$ calcd.: 256.1298; found: 256.1316.



6c

(4-(4-Fluorophenyl)-6-methylene-1,4-oxazepan-3-yl) methanol (6c) was prepared according to the General Procedure E as a yellow oil

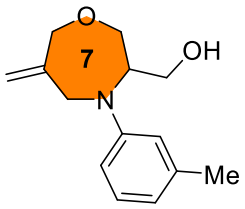
(chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 57% yield (27.0 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 6.96-6.86 (m, 4H), 5.07 (s, 1H), 4.97 (s, 1H), 4.26 (d, J = 14.4 Hz, 1H), 4.19-4.05 (m, 2H), 4.00-3.95 (m, 2H), 3.89-3.82 (m, 3H), 3.79-3.75 (m, 1H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 156.1(d, J = 234.2 Hz), 149.1, 147.3 (d, J = 1.7 Hz), 116.1, 115.9, 115.5 (d, J = 7.2 Hz), 111.2, 76.2, 72.5, 61.2 (d, J = 33.3 Hz), 49.5 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -130.4 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{13}H_{16}FNO_2Na$ calcd.: 260.1063; found: 260.1061.



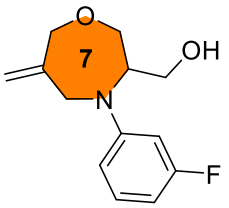
6d

(6-Methylene-4-(m-tolyl)-1,4-oxazepan-3-yl) methanol (6d) was prepared according to the General Procedure E as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 59% yield (27.5 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.03 (t, J = 7.9 Hz, 1H), 6.72-6.66 (m, 2H), 6.47 (d, J = 7.4 Hz, 1H), 5.08 (d, J = 1.1Hz, 1H), 4.96 (d, J = 1.1Hz, 1H), 4.26 (d, J = 14.3 Hz, 1H), 4.12 (s, 2H), 4.01-3.97 (m, 1H), 3.88-3.82 (m, 4H), 3.77-3.73 (m, 1H), 2.23 (s, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 150.4, 149.4, 139.2, 129.8, 118.4, 114.7, 111.3, 110.8, 76.2, 72.6, 61.0, 60.6, 48.9, 21.9 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{14}H_{19}NO_2Na$ calcd.: 256.1298; found: 256.1317.



6e

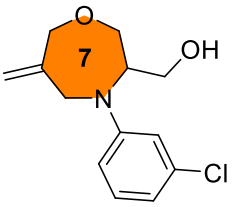
(4-(3-Fluorophenyl)-6-methylene-1,4-oxazepan-3-yl) methanol (6e) was prepared according to the General Procedure E as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 61% yield (28.9 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.15 (q, J = 15.8, 8.2 Hz, 1H), 6.70 (dd, J = 8.5, 2.4 Hz, 1H), 6.61 (dt, J = 13.6, 2.4 Hz, 1H), 6.36 (td, J = 18.2, 2.0 Hz, 1H), 5.13 (s, 1H), 5.01 (s, 1H), 4.30 – 4.10 (m, 3H), 4.01-3.97 (m, 2H), 3.92-3.84 (m, 3H), 3.81-3.77 (m, 1H)ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 165.0 (d, J = 240.6 Hz), 152.4 (d, J = 10.8 Hz), 148.4, 131.1 (d, J = 10.5 Hz), 111.5, 109.6 (d, J = 2.0 Hz), 103.4 (d, J = 21.9Hz), 100.5 (d, J = 26.6 Hz), 76.3, 72.6, 60.9, 60.8, 48.9 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -114.0 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{13}H_{16}FNO_2Na$ calcd.: 260.1063; found: 260.1073.



6f

(4-(3-Chlorophenyl)-6-methylene-1,4-oxazepan-3-yl) methanol (6f) was prepared according to the General Procedure E as a yellow oil (chromatography eluent: petroleum ether/ethyl acetate = 3:1) in 51% yield (25.9 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 7.14 (t, J = 8.1 Hz, 1H), 6.88-6.83 (m, 2H),

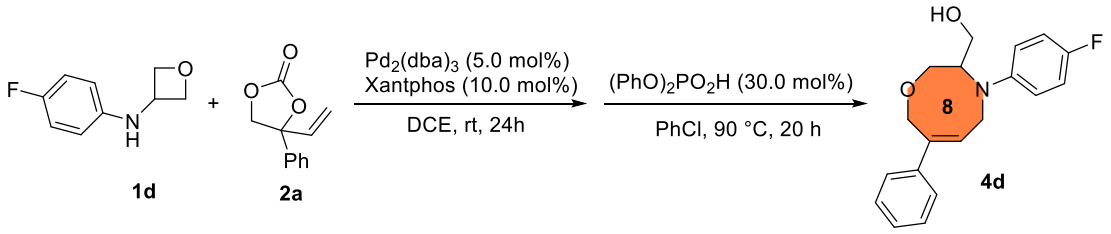
6.63 (d, J = 7.8 Hz, 1H), 5.13 (s, 1H), 5.01 (s, 1H), 4.31-4.10 (m, 3H), 4.01-3.90 (m, 3H), 3.87-3.77(m, 3H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 151.7, 148.2, 135.3, 131.1, 116.9, 113.4, 112.2, 111.5, 76.2, 72.5, 60.8, 60.7, 48.8 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{13}H_{16}ClNO_2Na$ calcd.: 276.0768; found: 276.0775.

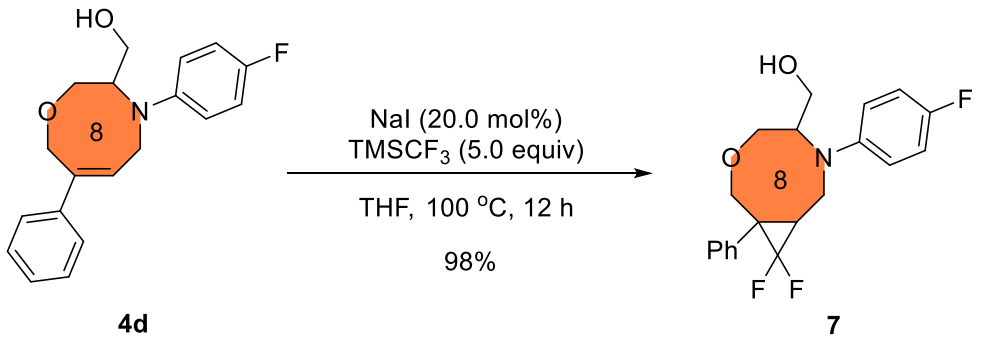
V. Gram-scale Synthesis and Derivation of Products

Gram-scale synthesis:



Under an N_2 atmosphere, to a solution of 4-phenyl-4-vinyl-1,3-dioxolan-2-one **2a** (1.1412 g, 6.0 mmol, 2.0 equiv.), ***N***-(4-fluorophenyl)oxetan-3-amine **1d** (501.5 mg, 3.0 mmol, 1.0 equiv), and Xantphos (173.6 mg, 0.3 mmol, 10 mol%) in DCE (30 mL) was added $Pd_2(dba)_3$ (137.4 mg, 0.15 mmol, 5.0 mol%). Then the reaction mixture was stirred at room temperature for 24 h. The reaction mixture was filtrated through a short column to remove the polar impurities and concentrated to afford crude products **3d**. Then the crude products **3d** was added to the solution of $(PhO)_2PO_2H$ (225.2 mg, 0.9 mmol, 30 mol%) in PhCl (45.0 mL). The reaction mixture was stirred at 90 °C for 20 h, then concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 3:1) to afford the desired product **4d** (752 mg, 80% yield).

Derivation of Product:



(9,9-Difluoro-6-(4-fluorophenyl)-1-phenyl-3-oxa-6-azabicyclo[6.1.0]nonan-5-yl)methanol (7): Under N_2 atmosphere, to a solution of anhydrous **4d**

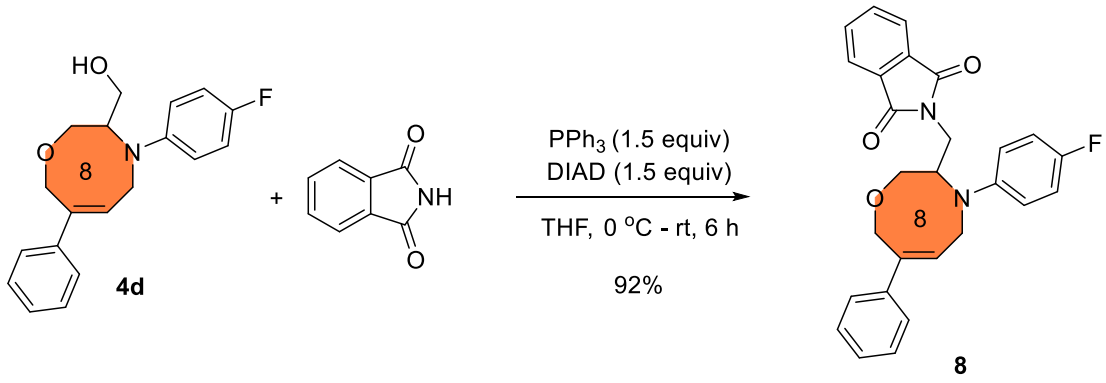
(62.7 mg, 0.2 mmol, 1.0 equiv) and NaI (9.1 mg, 0.06 mmol, 30 mol%) in anhydrous THF (1.8 mL) was added $TMSCF_3$ (0.55 mL). Then the reaction mixture was heated up to 100 °C and kept stirred for 12 h. The reaction mixture was concentrated under reduced pressure. Then the residue was purified by silica gel column chromatography (Hexanes/DCM = 2:1) to afford the desired product **7** as a yellow oil in 98% yield (30.7 mg).

^{1}H NMR (400 MHz, *d*$_6$-Acetone) δ 7.25-7.03 (m, 9H), 4.22-4.01 (m, 6H), 3.91-3.82 (m, 1H), 3.74-3.62 (m, 2H), 2.56 (t, J = 11.8 Hz, 1H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 156.0 (d, J = 234.5 Hz), 145.7 (dd, J = 17.0, 1.6 Hz), 139.9, 129.8 (t, J = 2.2 Hz), 129.4, 128.3, 116.5 (dd, J = 22.1, 6.3 Hz), 114.6 (dd, J = 19.0, 7.2 Hz), 74.2 (d, J = 10.2 Hz), 73.5 (dd, J = 14.6, 4.6 Hz), 61.0, 59.9 (d, J = 120.2 Hz), 59.8 (d, J = 109.4 Hz), 59.0, 40.5 (t, J = 9.6 Hz), 40.1 (dd, J = 21.6, 3.4 Hz) ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -127.7 (dd, J = 163.3, 4.2 Hz), -130.8 (d, J = 8.7 Hz), -144.1 (dd, J = 163.4, 28.1 Hz) ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{20}H_{20}F_3NO_2H$ calcd.: 364.1525; found: 364.1522.



(*Z*)-2-((4-(4-Fluorophenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl)methyl)isoindoline-1,3-dione (8): At 0 °C, to a solution of **4d** (31.3 mg, 0.1 mmol, 1.5 equiv), isoindoline-1,3-dione (22.1 mg, 0.15 mmol, 1.5 equiv) and PPh_3 (39.3 mg, 0.15mmol, 1.5 equiv) in THF (1.0 mL) was added dropwise

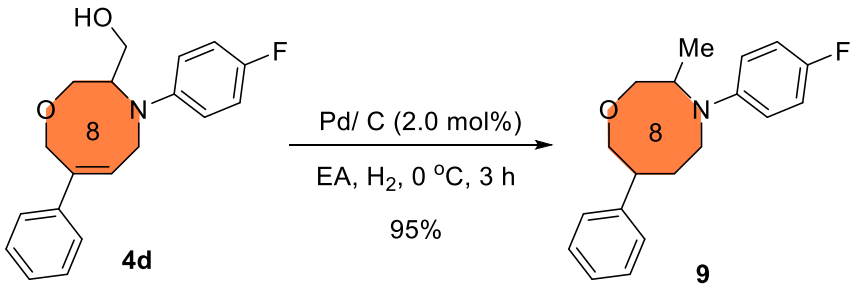
diisopropyl diazene-1,2-dicarboxylate (DIAD) (30.3 mg, 0.15 mmol, 1.5 equiv). Then the reaction mixture was stirred at room temperature for 6 h. the reaction mixture was concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 10: 1) to afford the desired product **8** in 92% yield (40.7 mg).

^{1}H NMR (400 MHz, d_6-Benzene) δ 7.42-7.32 (m, 2H), 7.09-7.03 (m, 5H), 6.83-6.71 (m, 4H), 6.68-6.63 (m, 2H), 5.96 (t, J = 6.6 Hz, 1H), 4.68 (dd, J = 16.8, 7.1 Hz, 1H), 4.56-4.19 (m, 3H), 4.12-4.06 (m, 1H), 3.77 (dd, J = 12.3, 4.4 Hz, 1H), 3.68-3.61 (m, 2H), 3.37 (dd, J = 12.4, 2.2 Hz, 1H) ppm.

^{13}C NMR (400 MHz, d_6-Benzene) δ 168.3, 155.8 (d, J = 236.1 Hz), 145.9 (d, J = 1.9 Hz), 142.2, 139.5, 133.6, 132.3, 128.6, 127.9, 127.6, 126.6, 123.0, 116.1 (d, J = 22.1 Hz), 113.7 (d, J = 7.2 Hz), 70.8, 68.5, 55.5, 42.8, 36.8 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -128.9 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{27}H_{23}FN_2O_3Na$ calcd.: 465.1591; found: 465.1589.



4-(4-Fluorophenyl)-3-methyl-7-phenyl-1,4-oxazocane(9): To a solution of **4d** (62.7 mg, 0.2 mmol, 1.0 equiv) in ethyl acetate (2.0 mL) was added Pd/C (12.5 mg, 20 wt.%, 2.0 mol%) and hydrogen gas was bubbled from a balloon at 0 °C. After 3 h, the reaction mixture was filtered through a pad of celite and washed with ethyl acetate (5.0 mL) and diethyl ether (5.0 mL). The combined organic phase was dried with Na_2SO_4, concentrated under reduce pressure, then the residue was purified by silica gel column chromatography (chromatography eluent: petroleum ether/ ethyl acetate = 2:1) to afford the

desired product **9** as a yellow oil in 95% yield (56.8 mg, ~ 1 : 1 dr).

Isomer I

^{1}H NMR (400 MHz, *d*$_6$-Acetone) δ 7.32-7.18 (m, 5H), 6.88-6.84 (m, 2H), 6.63-6.58 (m, 2H), 3.63-3.47 (m, 7H), 2.99 (s, 1.2H), 2.79-2.72 (m, 1H), 1.88-1.80 (m, 1H), 1.62-1.50 (m, 1H), 0.78 (d, *J* = 7.6 Hz, 3H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 155.9 (d, *J* = 232.8 Hz), 145.5, 144.0 (d, *J* = 5.3 Hz), 128.9, 128.7, 126.9, 115.9 (d, *J* = 21.9 Hz), 114.48 (d, *J* = 8.0 Hz), 76.2, 70.6, 62.0, 55.70, 48.5, 25.98, 12.1 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -127.1 ppm.

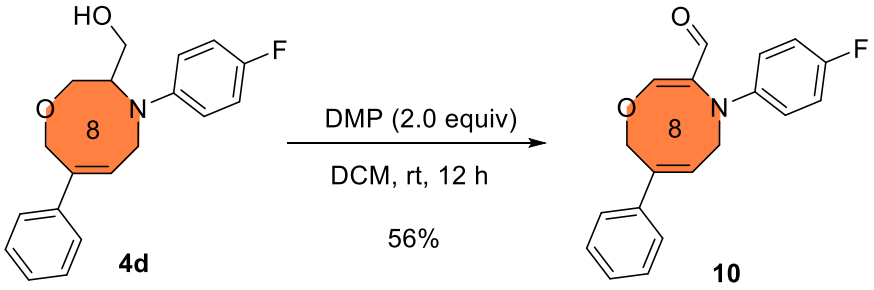
Isomer I

^{1}H NMR (400 MHz, *d*$_6$-Acetone) δ 7.32-7.18 (m, 5H), 6.88-6.84 (m, 2H), 6.63-6.58 (m, 2H), 3.63-3.47 (m, 7H), 2.99 (s, 1.2H), 2.79-2.72 (m, 1H), 1.88-1.80 (m, 1H), 1.62-1.50 (m, 1H), 0.77 (d, *J* = 7.2 Hz, 3H) ppm.

^{13}C NMR (400 MHz, *d*$_6$-Acetone) δ 155.9 (d, *J* = 232.8 Hz), 145.5, 144.0 (d, *J* = 5.3 Hz), 128.9, 128.7, 126.9, 115.9 (d, *J* = 21.9 Hz), 114.46 (d, *J* = 7.0 Hz), 76.2, 70.6, 62.0, 55.66, 48.5, 25.97, 12.1 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -127.2 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{22}FNO_2H$ calcd.: 322.1583; found: 322.1592.



(2*Z*,6*Z*)-4-(4-Fluorophenyl)-7-phenyl-5,8-dihydro-4*H*-1,4-oxazocine-3-carbaldehyde (10): At 0 °C, to a solution of **4d** (31.3 mg, 0.1 mmol, 1.0 equiv) in anhydrous CH_2Cl_2 (4.0 mL) was added Dess-Martin periodinane (DMP) (254.5 mg, 0.6 mmol, 2.0 equiv). The reaction mixture was spontaneously warmed to

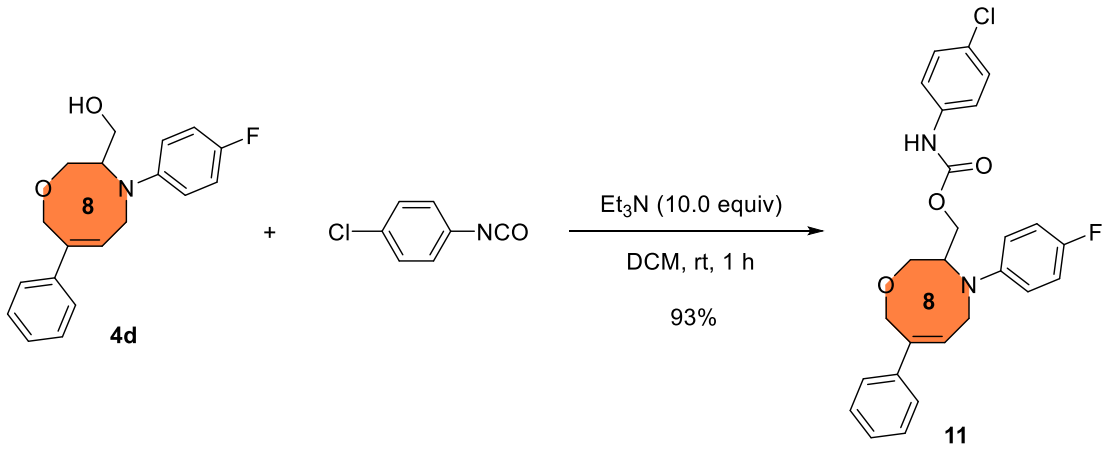
room temperature and kept stirring for 12 h. The mixture was diluted with CH_2Cl_2 (15 mL), washed with water and brine, dried over anhydrous Na_2SO_4, concentrated under reduced pressure. Then the residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 10:1 to 5:1) to afford the desired product **10** as yellow oil in 56% yield (52.0 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 9.42 (s, 1H), 7.73 (s, 1H), 7.45-7.42 (m, 2H), 7.39-7.35 (m, 2H), 7.31-7.27 (m, 1H), 7.00-6.95 (m, 2H), 6.77-6.73 (m, 2H), 6.32 (t, J = 3.4 Hz, 1H), 5.23 (s, 2H), 4.46 (d, J = 3.4 Hz, 2H) ppm.

^{13}C NMR (400 MHz, d_6-Acetone) δ 189.9, 167.0, 156.7 (d, J = 235.0 Hz), 145.4, 143.2, 136.0, 133.9, 129.4, 128.2 (d, J = 2.2 Hz), 126.8, 116.5 (d, J = 22.6 Hz), 114.8 (d, J = 7.5 Hz), 69.8, 52.6 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -127.6 ppm.

HRMS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{16}FNO_2$ Na calcd.: 332.1063; found: 332.1066.



(*Z*)-(4-(4-Fluorophenyl)-7-phenyl-3,4,5,8-tetrahydro-2*H*-1,4-oxazocin-3-yl)methyl (4-chlorophenyl)carbamate (11): To a solution of **4d** (31.3 mg, 0.1 mmol, 1.0 equiv) and 4-chlorophenyl isocyanate (23.0 mg, 0.15 mmol, 1.5 equiv) in DCM (1.0 mL) was added Et_3N (0.14 mL, 1.0 mmol, 10.0 equiv). The solution was stirred at room temperature for 1 h. The reaction mixture was concentrated under reduced pressure. The residue was purified by silica gel

column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the desired product **11** in 93% yield (43.4 mg).

^{1}H NMR (400 MHz, d_6-Acetone) δ 8.95 (s, 1 H), 7.58 (d, J = 8.4 Hz, 2H), 7.33-7.26 (m, 7H), 6.95 (d, J = 6.9 Hz, 4H), 6.34 (t, J = 6.2 Hz, 1H), 4.70-4.64 (m, 2H), 4.54-4.47 (m, 2H), 4.29-4.13 (m, 3H), 3.90 (dd, J = 17.0, 6.0 Hz, 1H), 3.79 (d, J = 12.2 Hz, 1H) ppm.

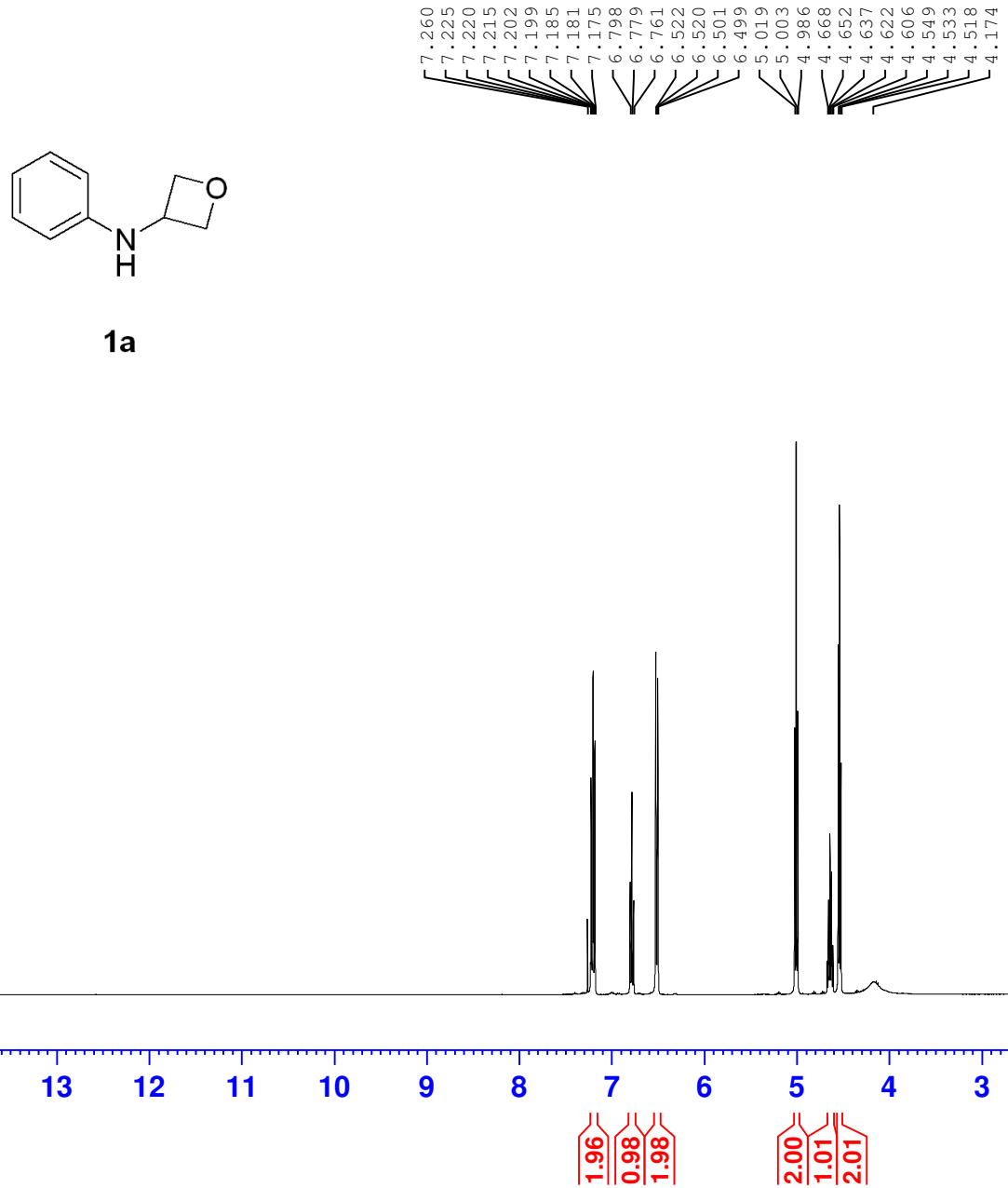
^{13}C NMR (400 MHz, d_6-Acetone) δ 156.0 (d, J = 234.3 Hz), 154.3, 146.7 (d, J = 1.3 Hz), 142.9, 139.7, 138.9, 129.5, 129.1, 128.9, 128.1, 127.8, 126.9, 120.6, 116.2 (d, J = 22.1 Hz), 114.6 (d, J = 7.3 Hz), 70.8, 68.0, 62.5, 57.0, 44.2 ppm.

^{19}F NMR (282MHz, $CDCl_3$) δ -128.3 ppm.

HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{26}H_{24}ClFN_2O_3H$ calcd.: 467.1538; found: 467.1539.

NMR spectra

LJX-1a

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PROCNO                 1

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TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                290.6 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1         23.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
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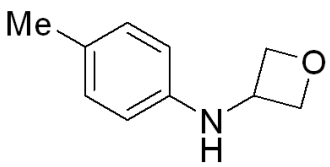
LJX-1b



1b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Current Data Parameters
NAME       20230709-400M
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230708
Time               16.02
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                290.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
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SF          400.1900140 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
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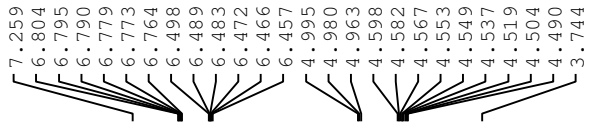
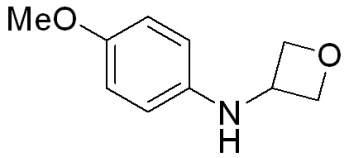
LJX-1c





1c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EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
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Time              16.05
INSTRUM           spect
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PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9845889 sec
RG               113.67
DW               60.800 usec
DE                6.50 usec
TE               290.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                65536
SF          400.1900142 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
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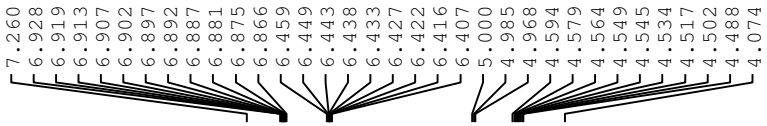
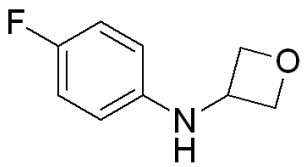
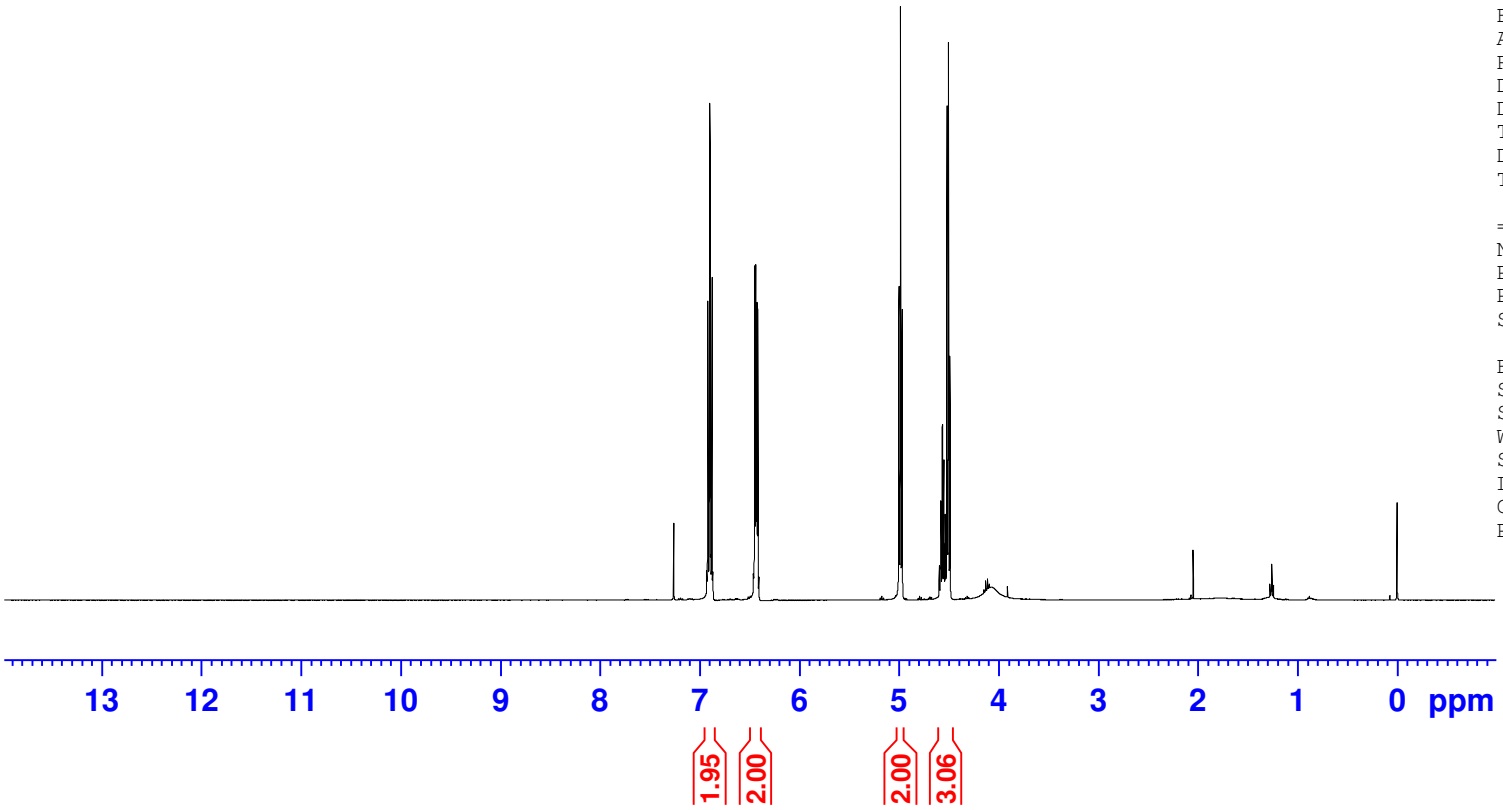
LJX-1d





1d

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PROCNO                 1

F2 - Acquisition Parameters
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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                290.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900140 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
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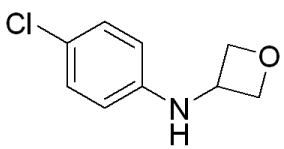
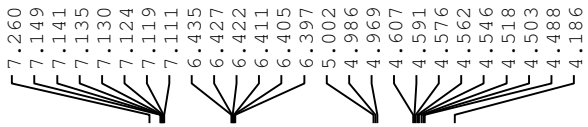
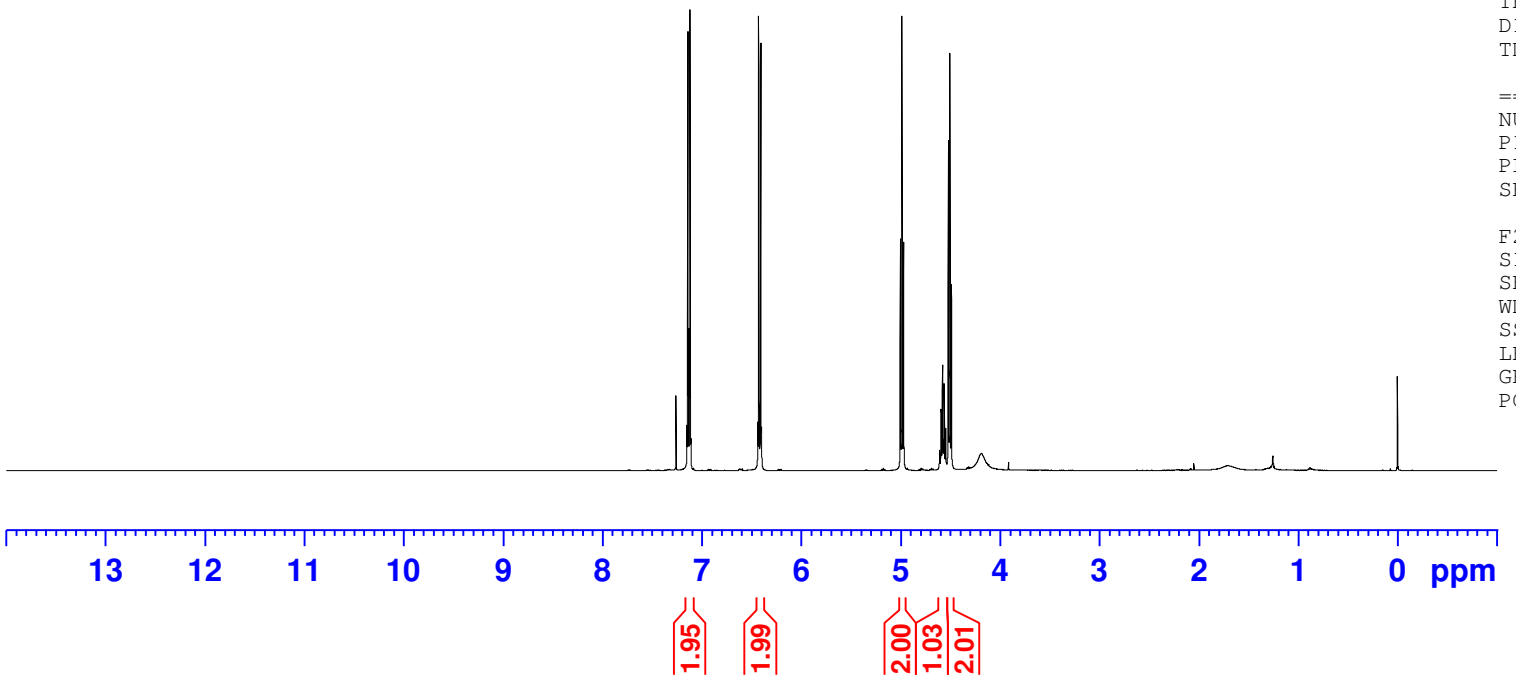
LJX-1e



1e



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Current Data Parameters
NAME       20230709-400M
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230708
Time               16.13
INSTRUM            spect
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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
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RG                113.67
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D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
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SF          400.1900140 MHz
WDW                   EM
SSB                    0
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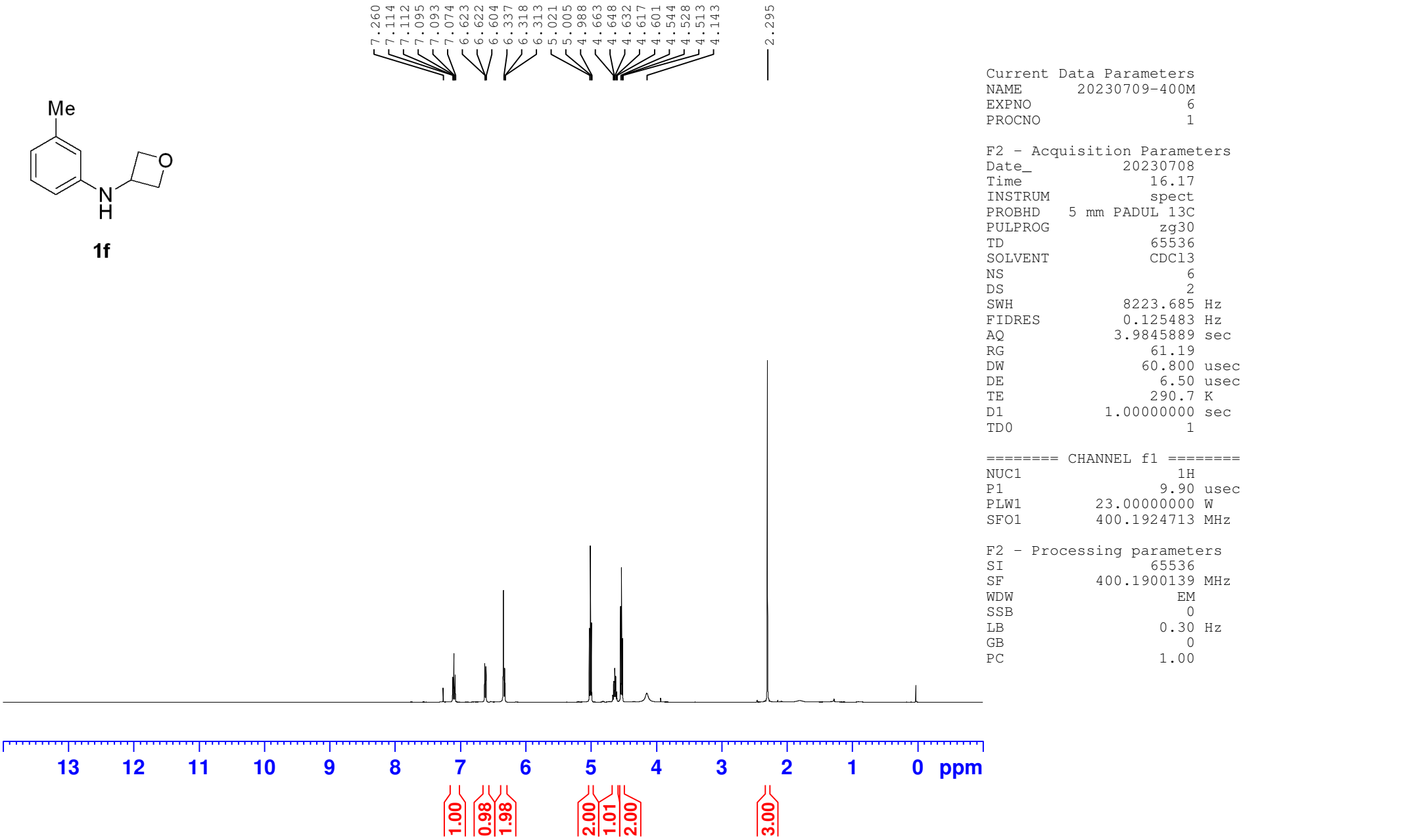
LJX-1f

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Current Data Parameters
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EXPNO                 6
PROCNO                1

F2 - Acquisition Parameters
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Time              16.17
INSTRUM           spect
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PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9845889 sec
RG                61.19
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TE                290.7 K
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TD0                   1

======== CHANNEL f1 ========
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P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
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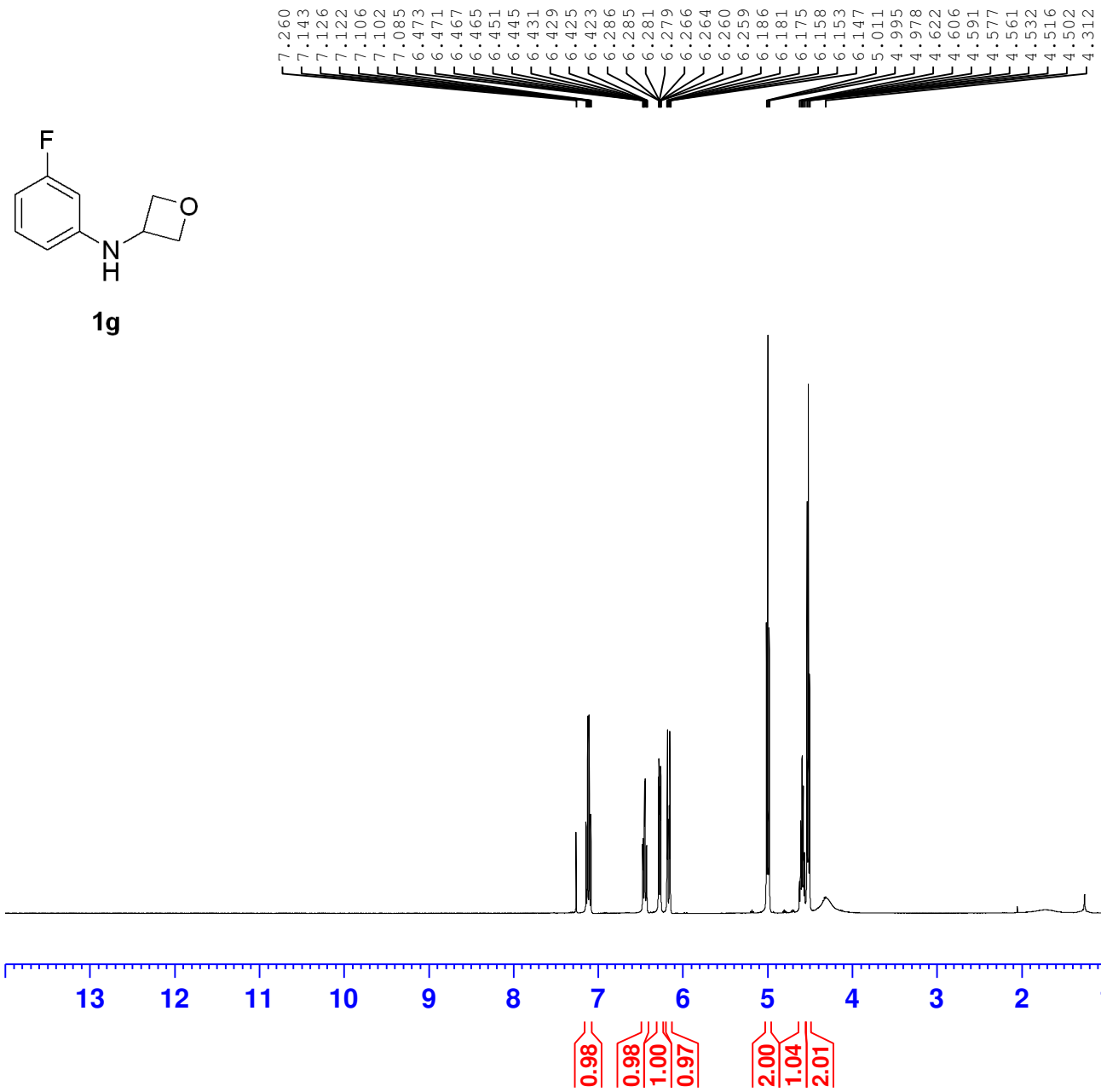
LJX-1g

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PROCNO                 1

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TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
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FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                290.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
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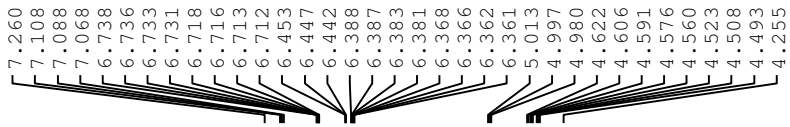
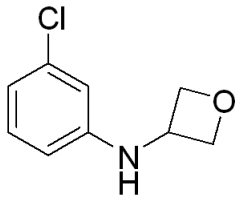
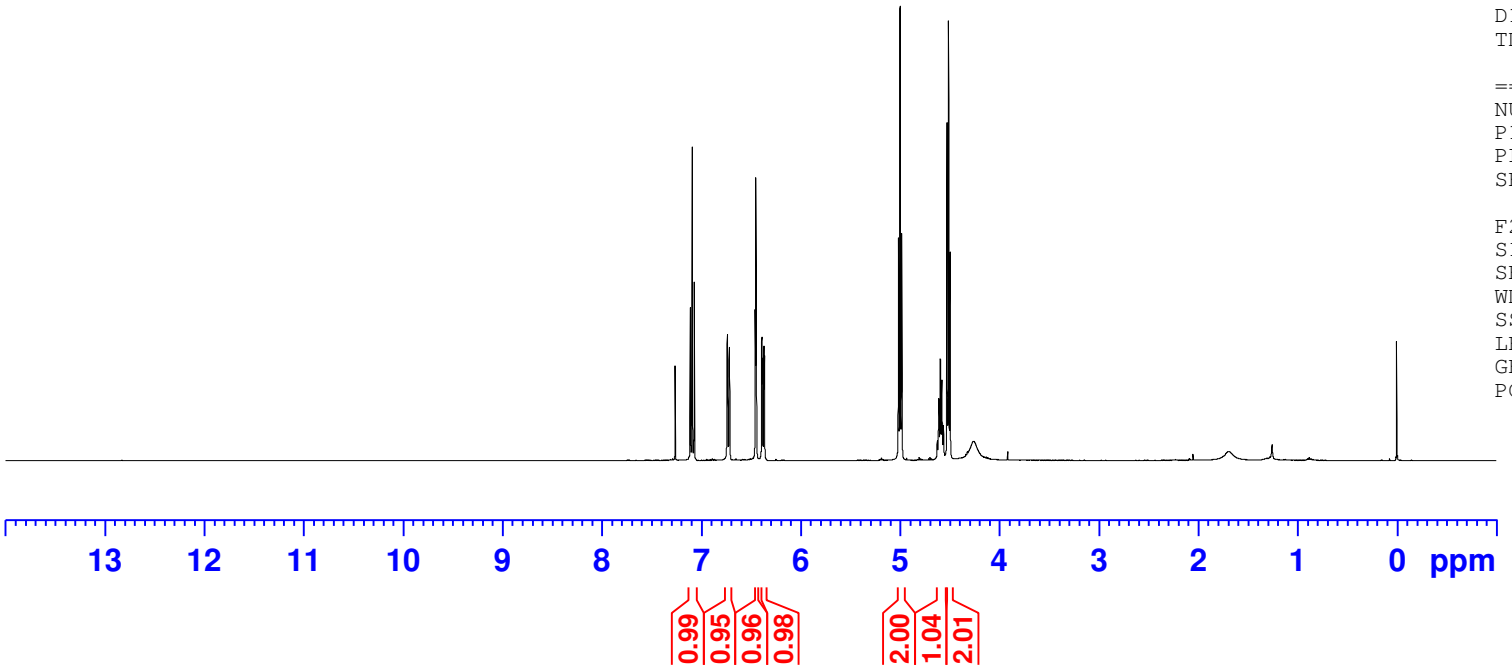
LJX-1h





1h

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Current Data Parameters
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PROCNO                 1

F2 - Acquisition Parameters
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Time               16.25
INSTRUM            spect
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PULPROG             zg30
TD                 65536
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DS                     2
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FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                125.76
DW                60.800 usec
DE                 6.50 usec
TE                290.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
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SF          400.1900141 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
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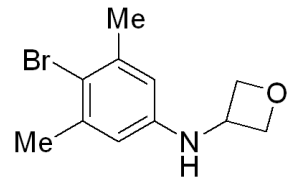
LJX-1i



1i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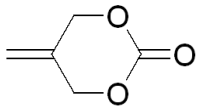
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230708
Time               16.29
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                290.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900140 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-5



5

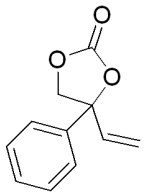

```
Current Data Parameters
NAME      20230720-300M
EXPNO               342
PROCNO                1

F2 - Acquisition Parameters
Date_          20230720
Time              11.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6009.615 Hz
FIDRES        0.091699 Hz
AQ            5.4525952 sec
RG                  203
DW               83.200 usec
DE                6.50 usec
TE                297.7 K
D1           1.00000000 sec
TD0                   1

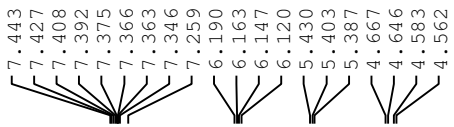
======== CHANNEL f1 ========
SFO1        300.1318534 MHz
NUC1                  1H
P1                10.00 usec
PLW1        14.00000000 W

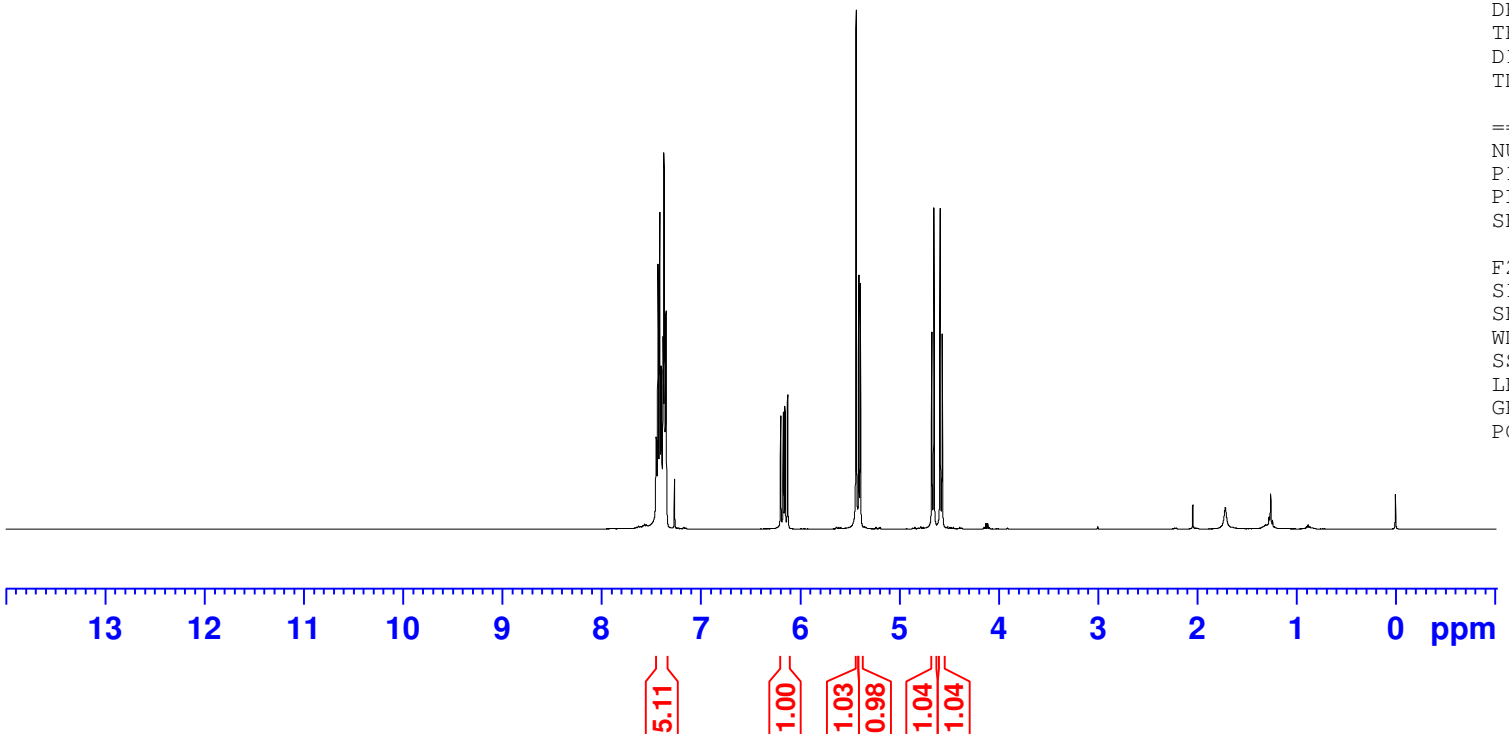
F2 - Processing parameters
SI                65536
SF          300.1300065 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

ljx-2a



2a





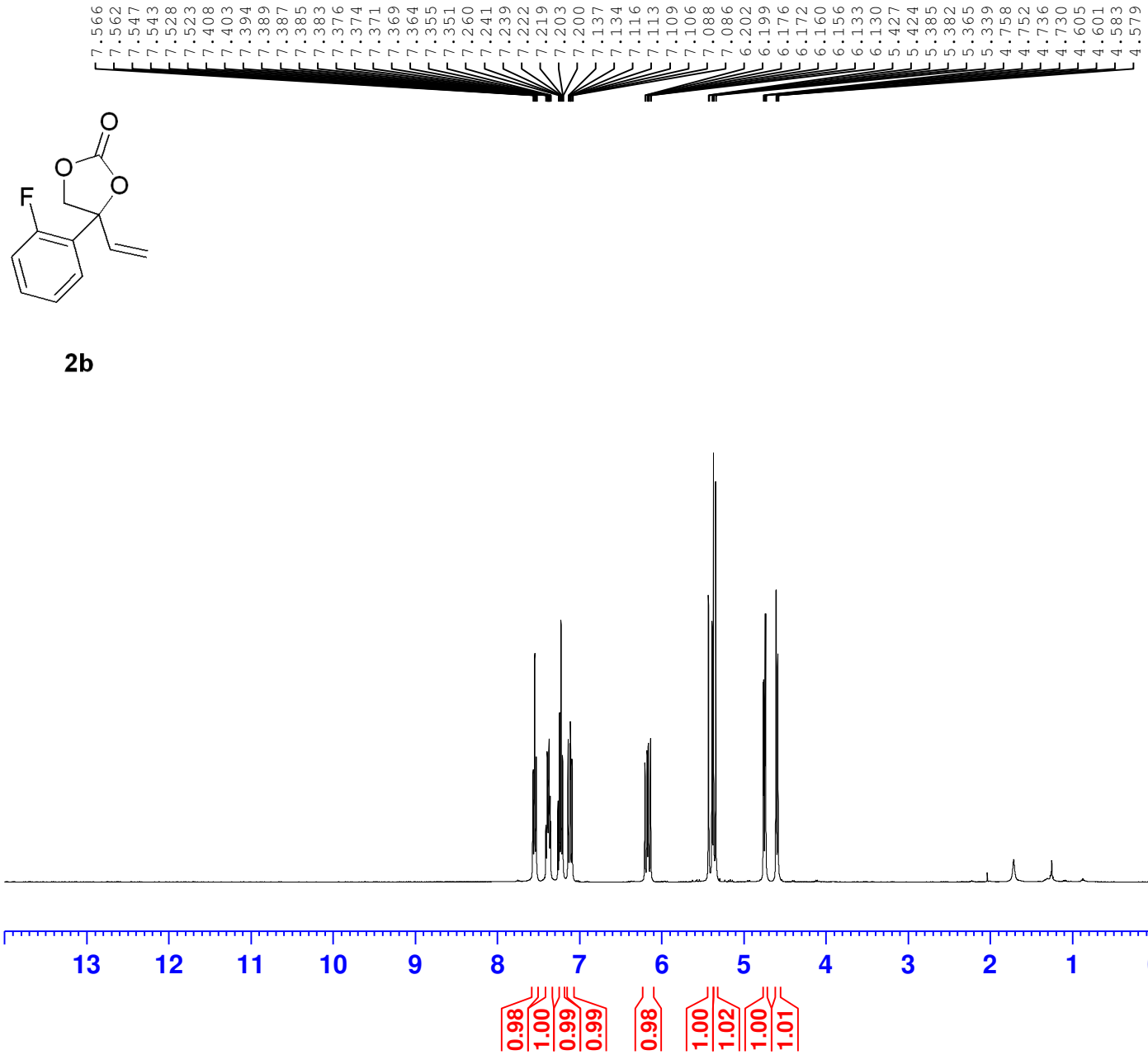

```
Current Data Parameters
NAME       20230721-400M
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230720
Time               22.17
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 75.43
DW                60.800 usec
DE                 6.50 usec
TE                294.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900143 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-2b



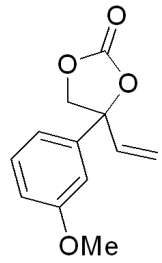

```
Current Data Parameters
NAME       20230721-400M
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230720
Time               22.21
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 75.43
DW                60.800 usec
DE                 6.50 usec
TE                294.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900140 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-2c



2c

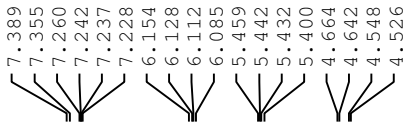

```
Current Data Parameters
NAME       20230721-400M
EXPNO                 11
PROCNO                 1

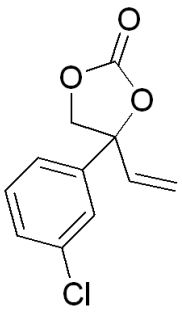
F2 - Acquisition Parameters
Date_           20230720
Time               22.26
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 68.24
DW                60.800 usec
DE                 6.50 usec
TE                294.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

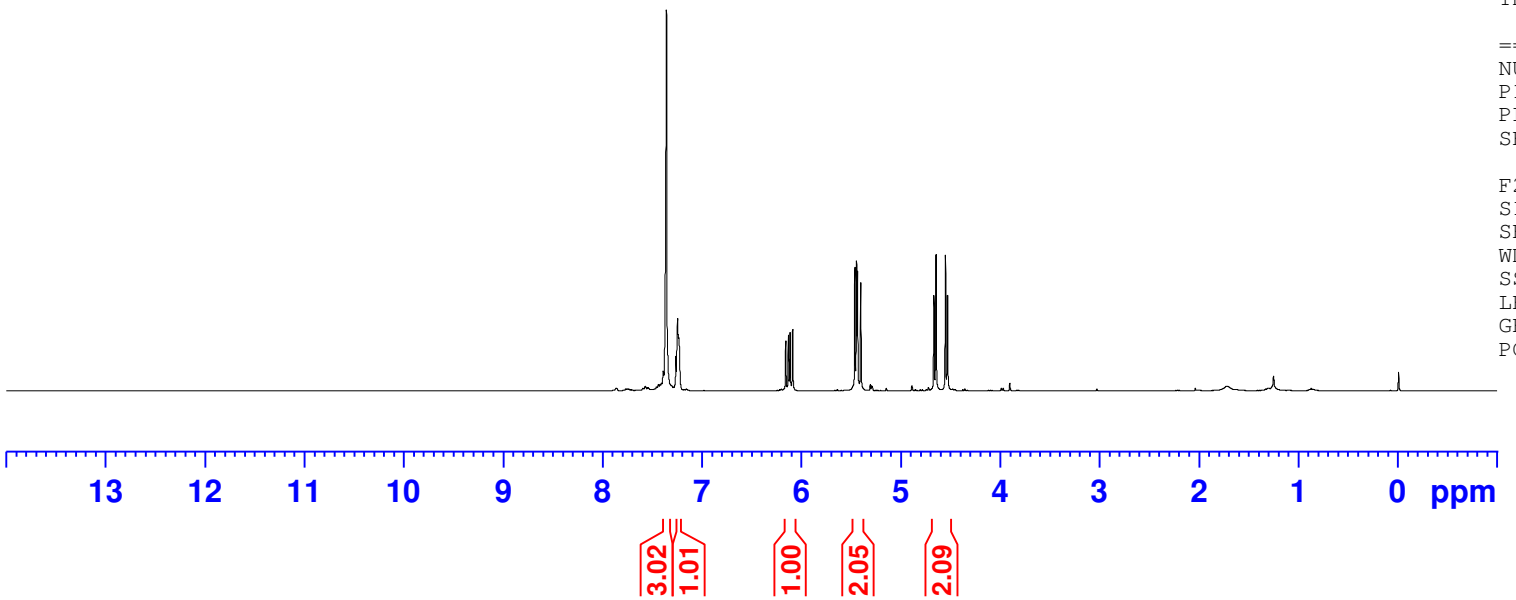
F2 - Processing parameters
SI                 65536
SF          400.1900141 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

ljx-2d





2d



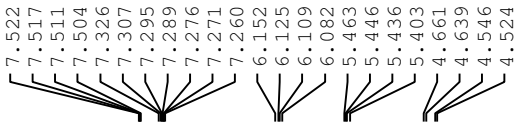

```
Current Data Parameters
NAME       20230721-400M
EXPNO                 12
PROCNO                 1

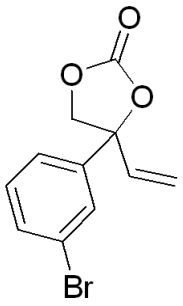
F2 - Acquisition Parameters
Date_           20230720
Time               22.30
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 75.43
DW                60.800 usec
DE                 6.50 usec
TE                294.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

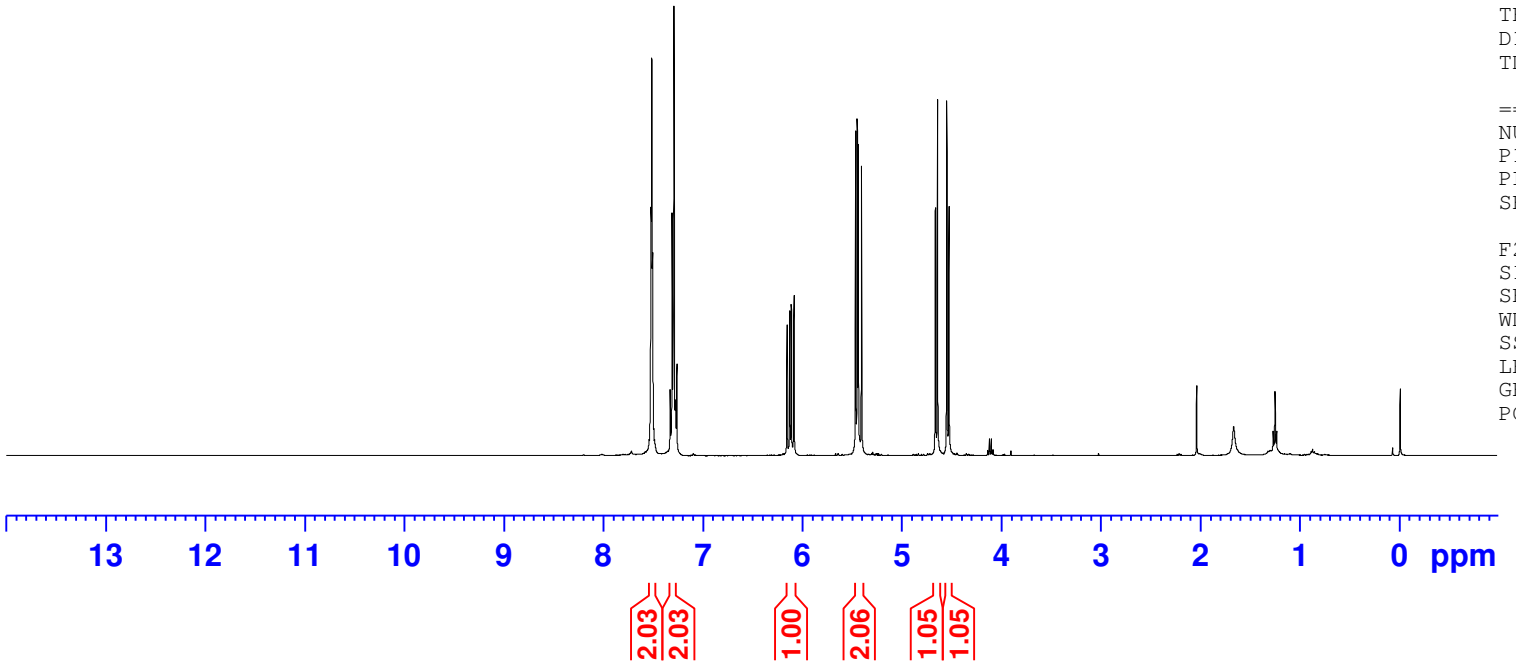
F2 - Processing parameters
SI                 65536
SF          400.1900142 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

ljx-2e





2e



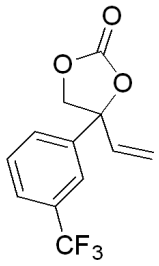

```
Current Data Parameters
NAME       20230721-400M
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230720
Time               22.34
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                294.2 K
D1           1.00000000 sec
TD0                    1

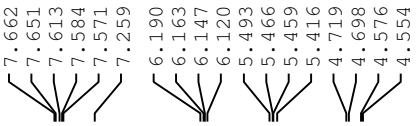
======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

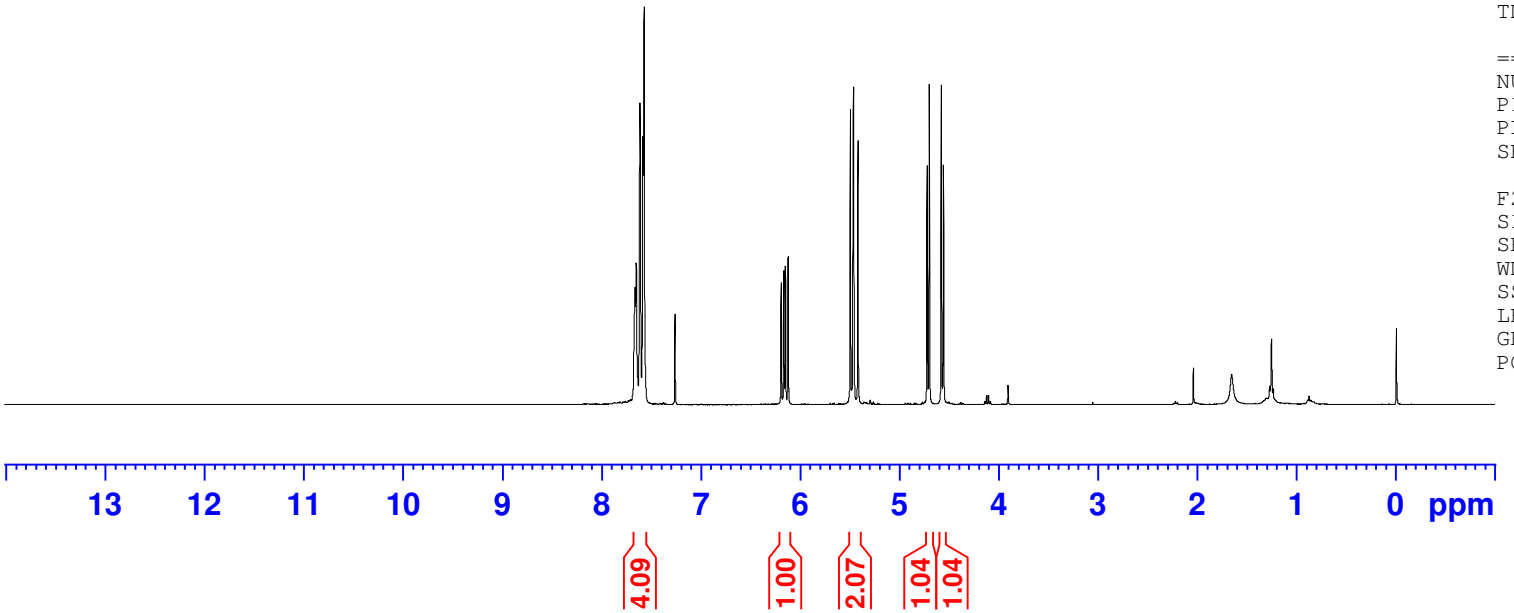
F2 - Processing parameters
SI                 65536
SF          400.1900142 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

ljx-2f



2f





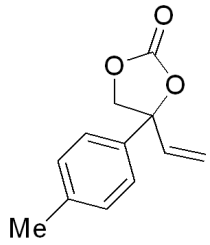

```
Current Data Parameters
NAME       20230721-400M
EXPNO                 23
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230720
Time               23.15
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                294.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900144 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

ljx-2g



2g

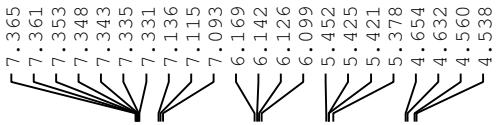

```
Current Data Parameters
NAME       20230721-400M
EXPNO                 24
PROCNO                 1

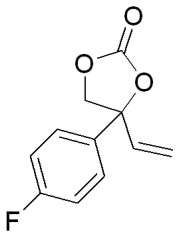
F2 - Acquisition Parameters
Date_           20230720
Time               23.19
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 90.23
DW                60.800 usec
DE                 6.50 usec
TE                294.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

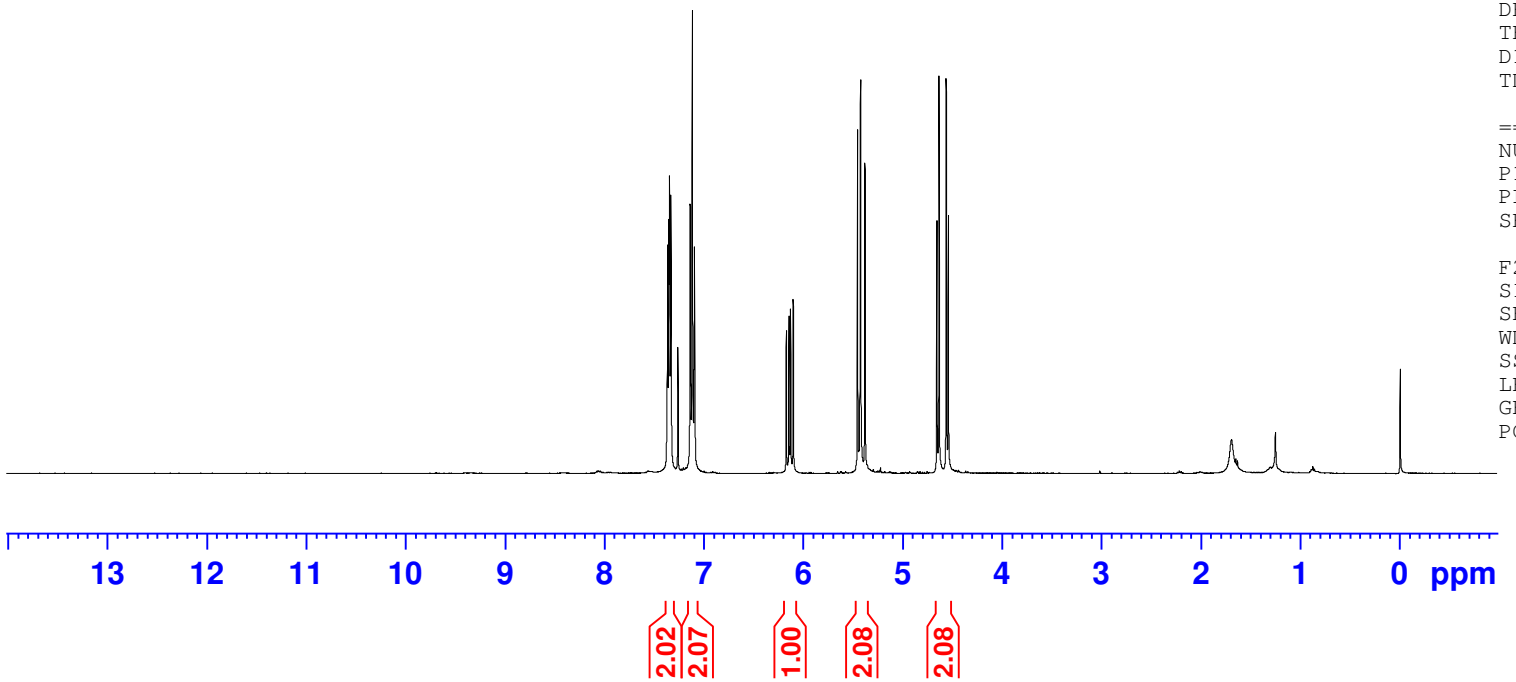
F2 - Processing parameters
SI                 65536
SF          400.1900138 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

ljx-2h





2h



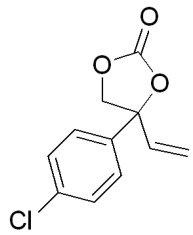

```
Current Data Parameters
NAME       20230722-400M
EXPNO                 28
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230721
Time               23.10
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                294.0 K
D1           1.00000000 sec
TD0                    1

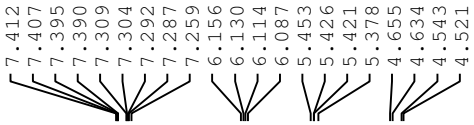
======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

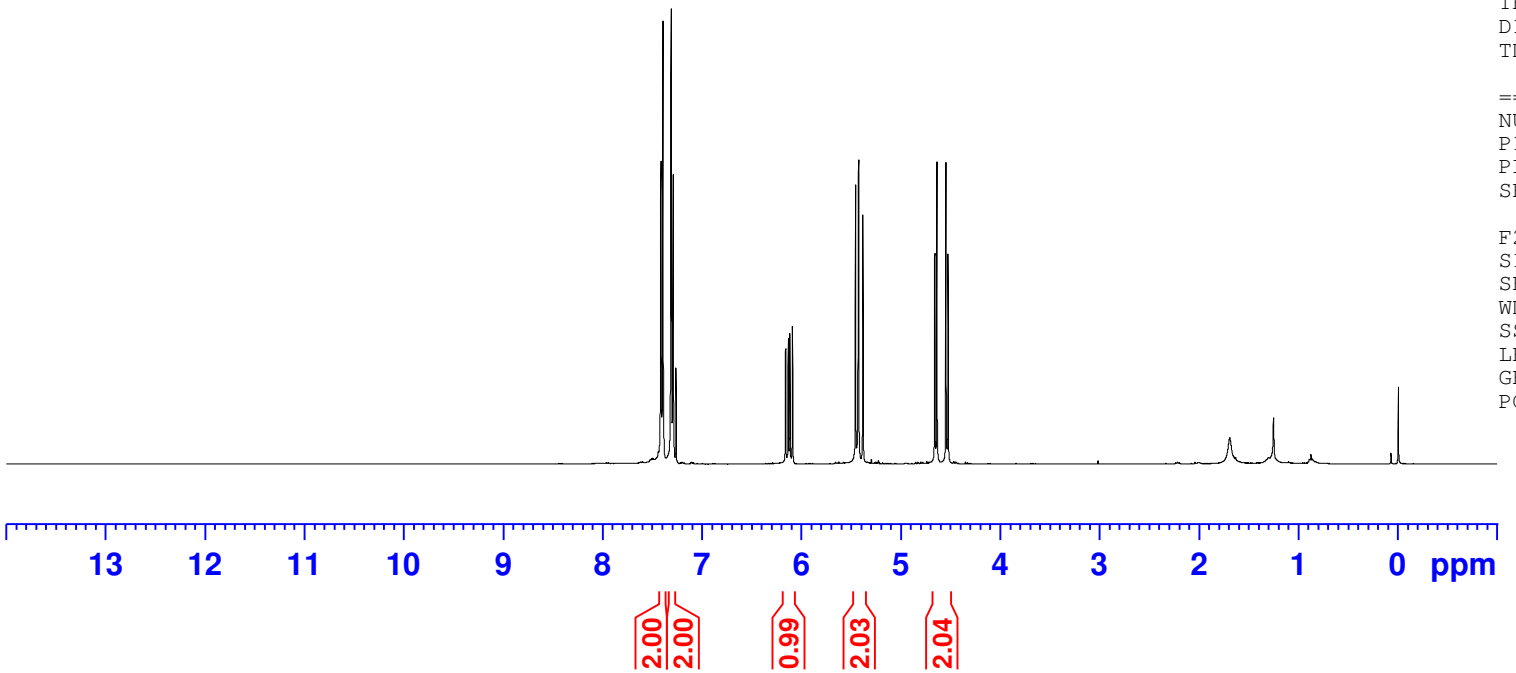
F2 - Processing parameters
SI                 65536
SF          400.1900141 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

ljx-2i



2i





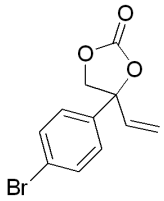

```
Current Data Parameters
NAME       20230722-400M
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230721
Time               21.34
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                293.7 K
D1           1.00000000 sec
TD0                    1

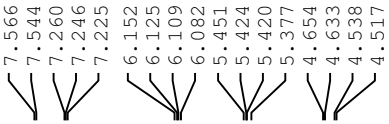
======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

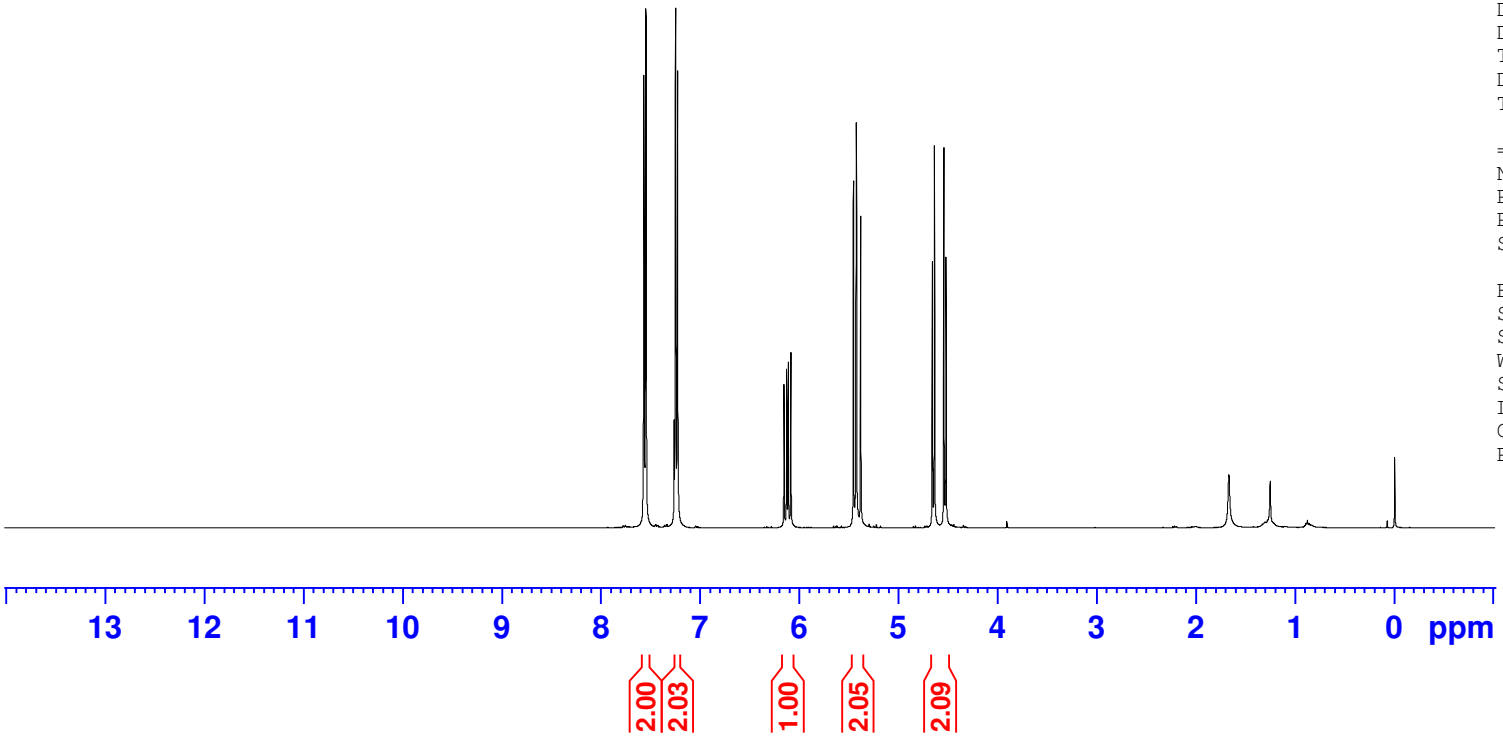
F2 - Processing parameters
SI                 65536
SF          400.1900143 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-2j



2j





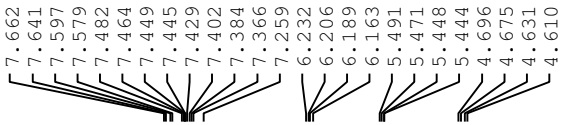

```
Current Data Parameters
NAME       20230722-400M
EXPNO                  4
PROCNO                 1

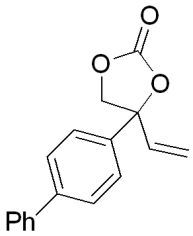
F2 - Acquisition Parameters
Date_           20230721
Time               21.38
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                293.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

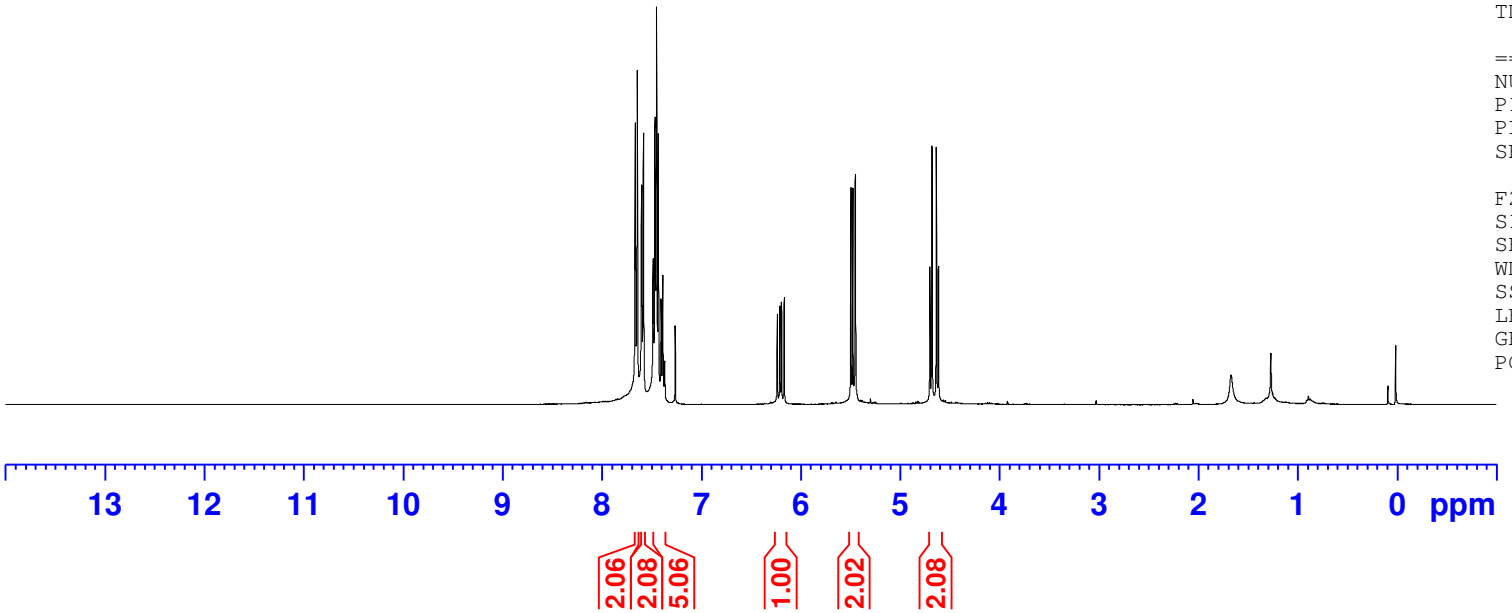
F2 - Processing parameters
SI                 65536
SF          400.1900140 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-2k





2k



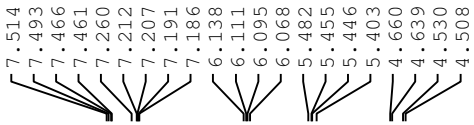

```
Current Data Parameters
NAME       20230722-400M
EXPNO                 10
PROCNO                 1

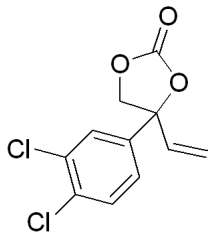
F2 - Acquisition Parameters
Date_           20230721
Time               22.01
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 75.43
DW                60.800 usec
DE                 6.50 usec
TE                293.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

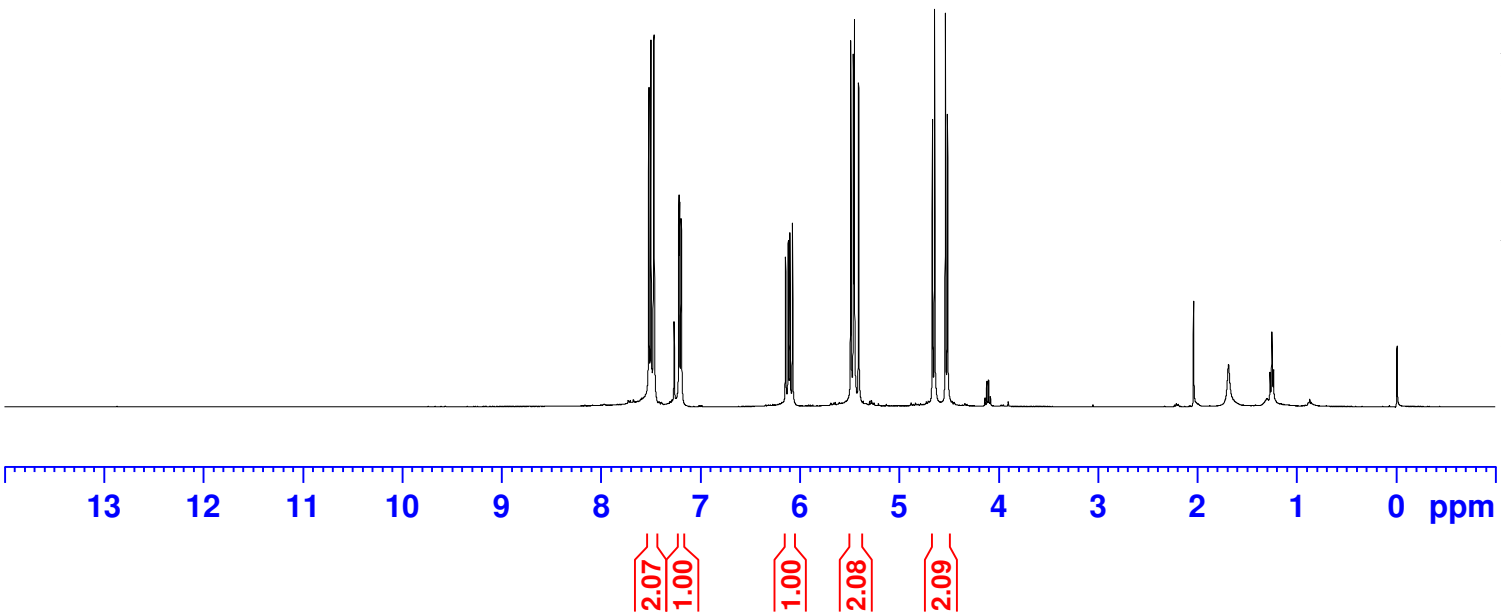
F2 - Processing parameters
SI                 65536
SF          400.1900142 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-2l





2l



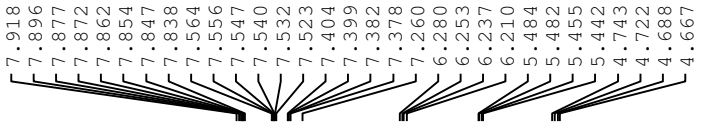

```
Current Data Parameters
NAME       20230722-400M
EXPNO                 14
PROCNO                 1

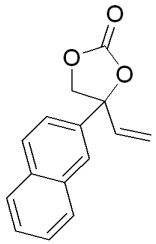
F2 - Acquisition Parameters
Date_           20230721
Time               22.18
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 90.23
DW                60.800 usec
DE                 6.50 usec
TE                294.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

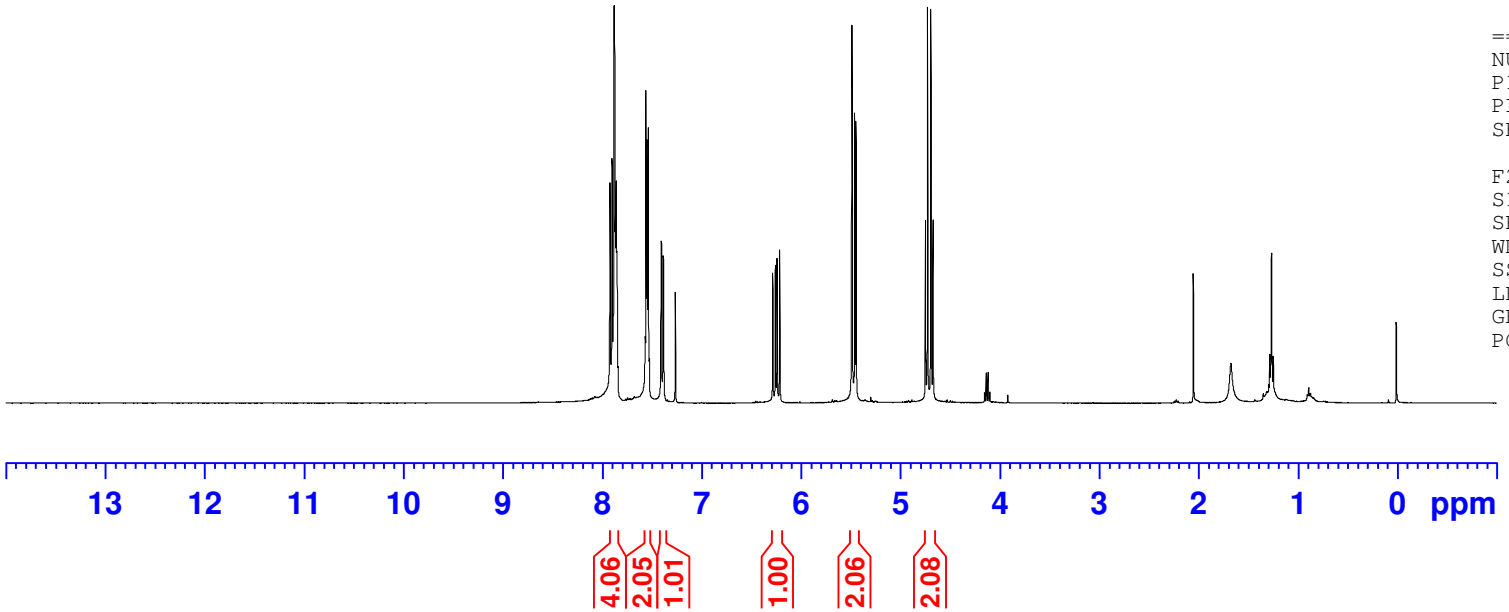
F2 - Processing parameters
SI                 65536
SF          400.1900142 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-2m





2m

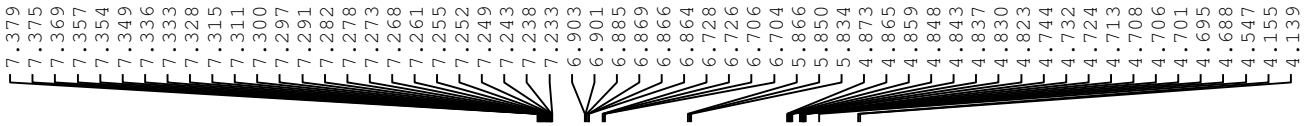


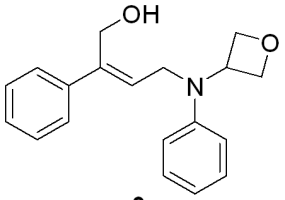

```
Current Data Parameters
NAME       20230722-400M
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230721
Time               22.13
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 90.23
DW                60.800 usec
DE                 6.50 usec
TE                293.8 K
D1           1.00000000 sec
TD0                    1

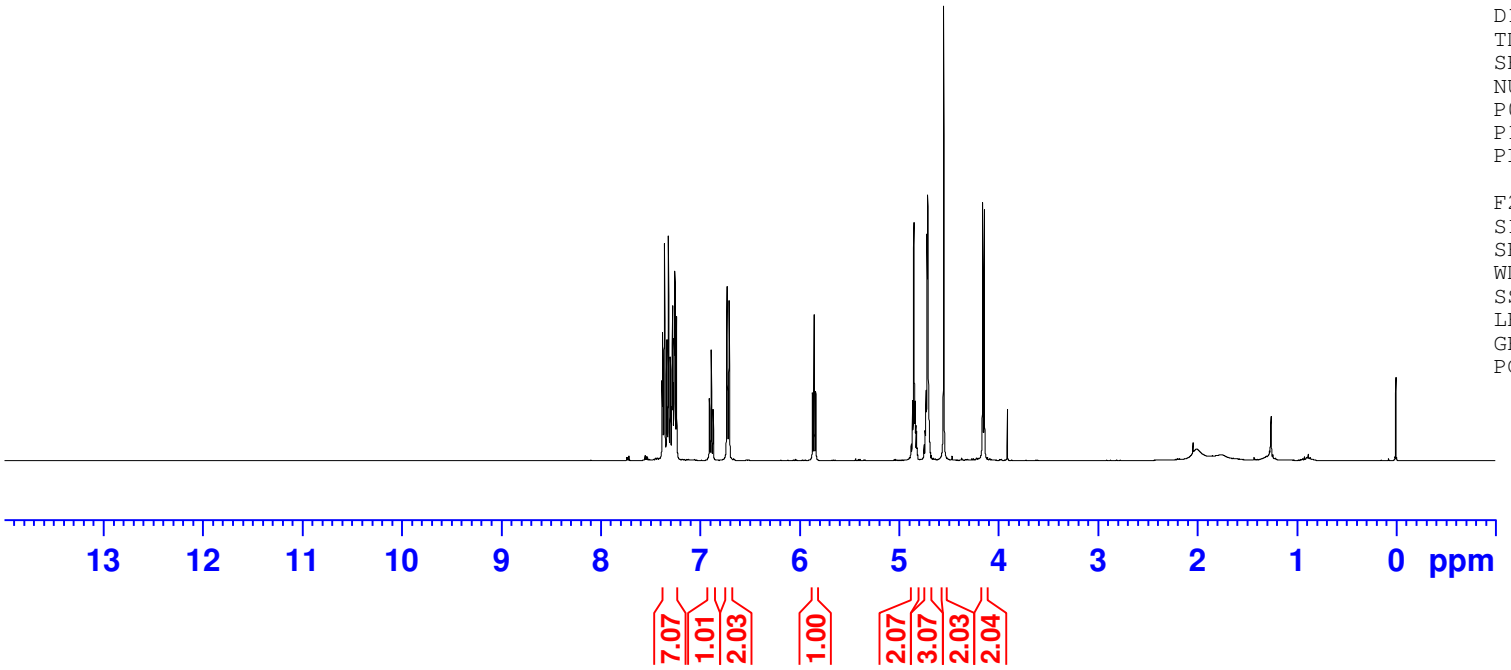
======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900141 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```





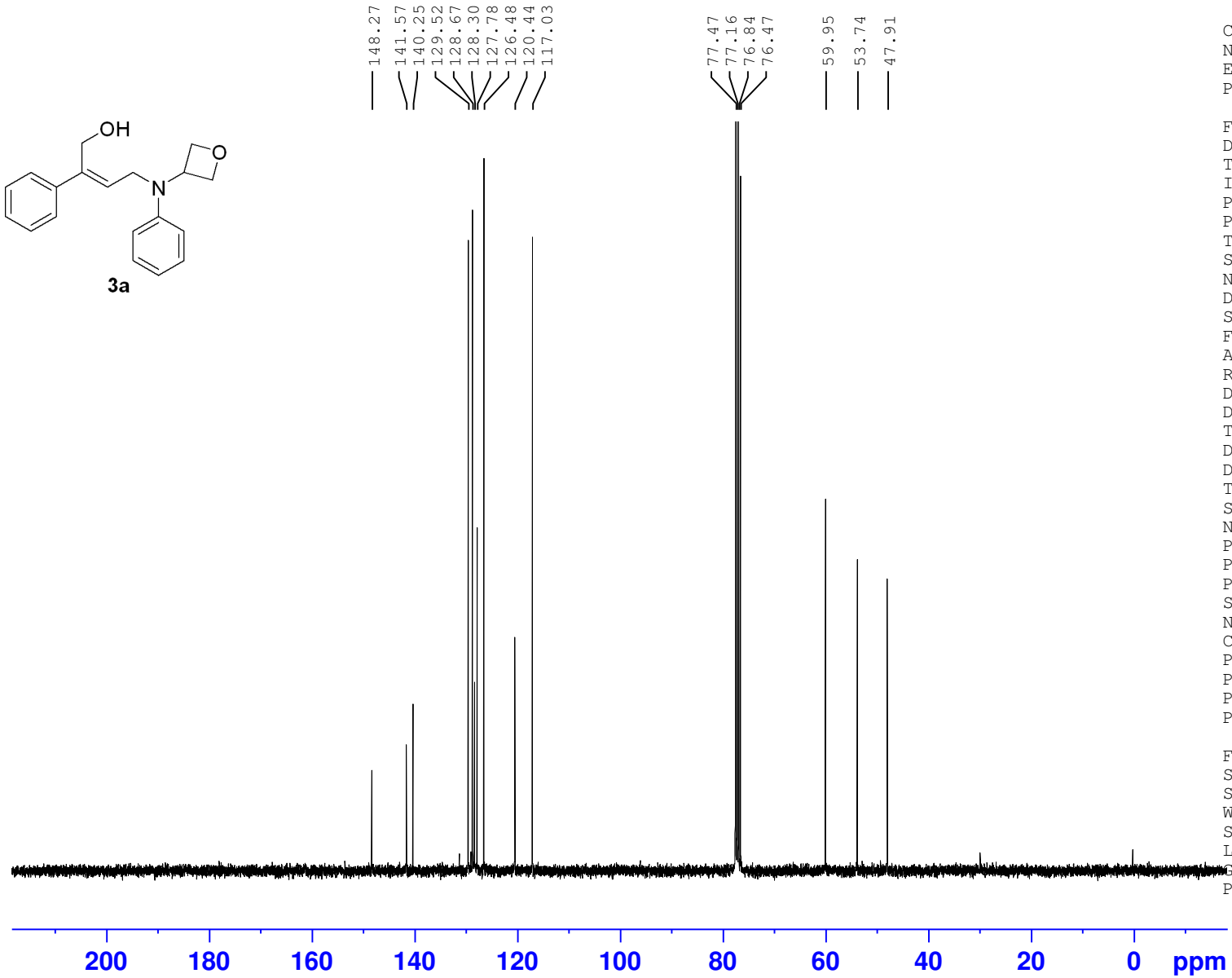
3a




```
Current Data Parameters
NAME            LJX-1-39
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230729
Time                4.05 h
INSTRUM           Avance
PROBHD   Z116098_0833 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ             3.9976959 sec
RG                   101
DW                61.000 usec
DE                 13.54 usec
TE                 294.3 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1         20.73200035 W

F2 - Processing parameters
SI                 65536
SF           400.1300142 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

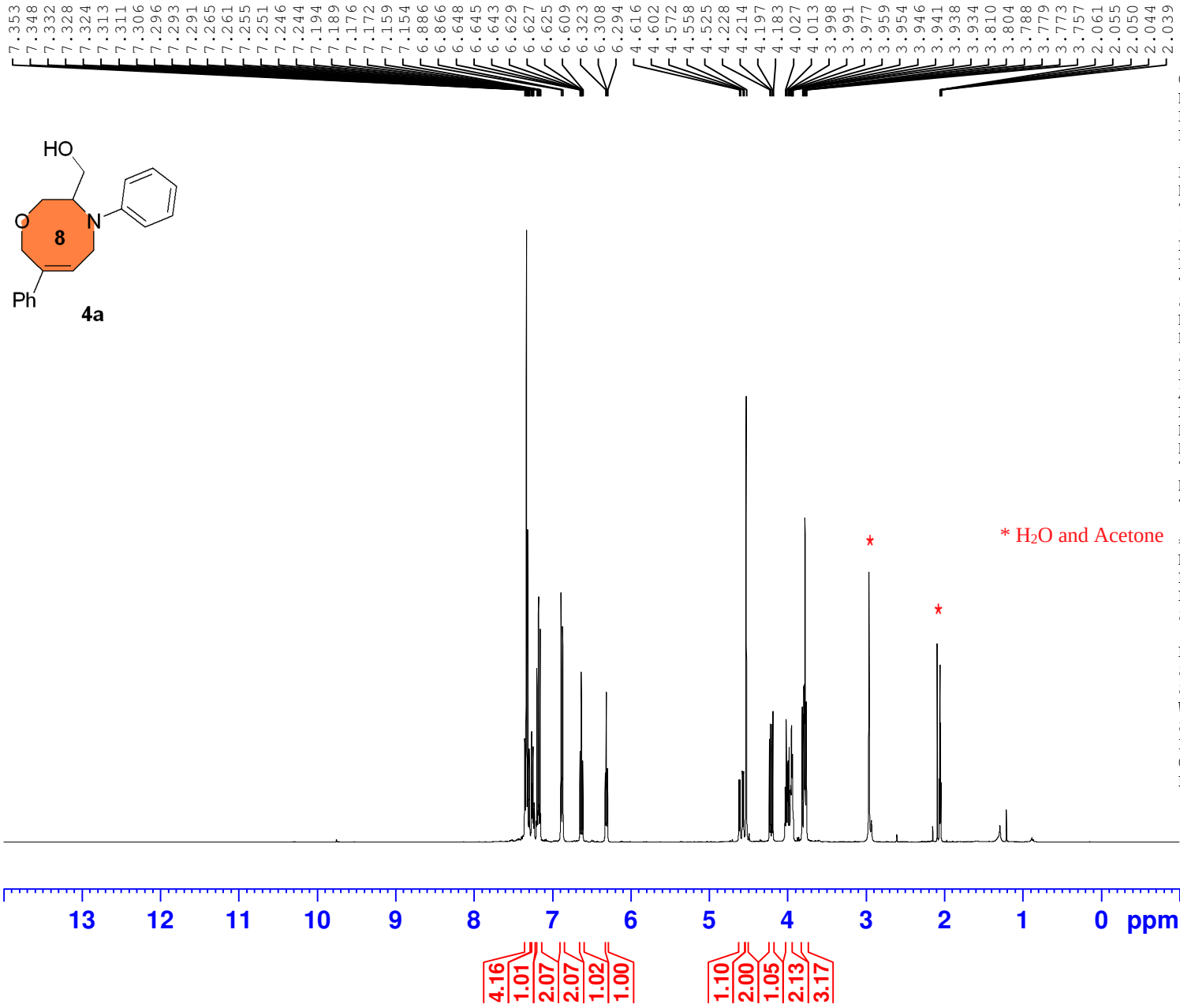



```
Current Data Parameters
NAME            LJX-1-39
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230729
Time                4.35 h
INSTRUM           Avance
PROBHD   Z116098_0833 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     4
SWH            23809.523 Hz
FIDRES          0.726609 Hz
AQ             1.3762560 sec
RG               46.0295
DW                21.000 usec
DE                 6.50 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1         87.89900208 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         20.73200035 W
PLW12         0.25595000 W
PLW13         0.12874000 W

F2 - Processing parameters
SI                 32768
SF           100.6127603 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-2-44



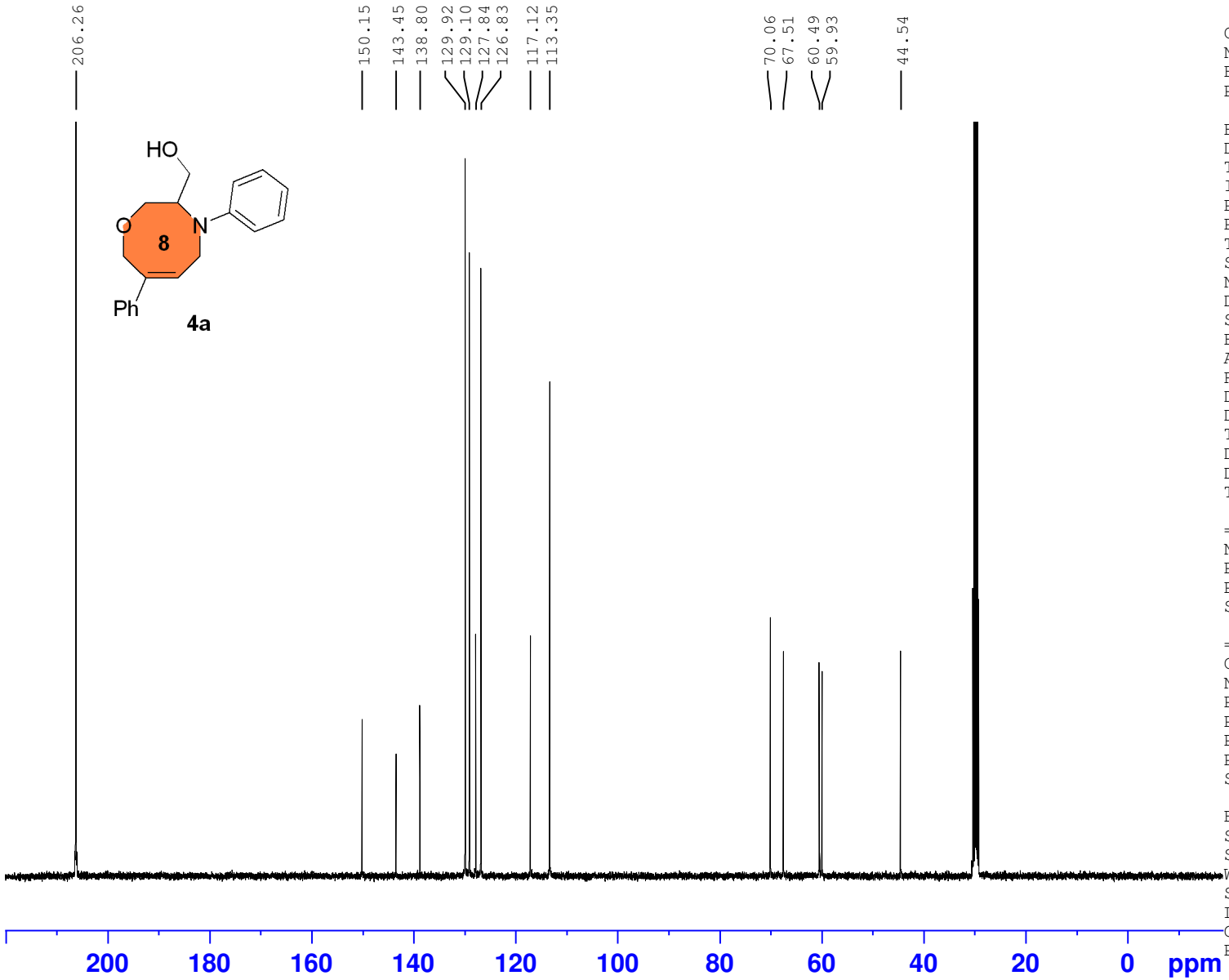

```
Current Data Parameters
NAME        20230610-400
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230609
Time               23.11
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                  53.3
DW                60.800 usec
DE                 6.50 usec
TE                291.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900111 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

ljx-2-44




```
Current Data Parameters
NAME      20230615-400m
EXPNO                39
PROCNO                1

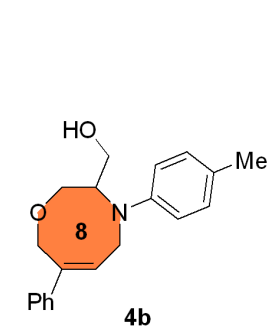
F2 - Acquisition Parameters
Date_          20230615
Time               3.10
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 50.16
DW               20.800 usec
DE                6.50 usec
TE               292.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.80 usec
PLW1        47.40000153 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        23.00000000 W
PLW12        0.30712000 W
PLW13        0.24877000 W
SFO2       400.1916008 MHz

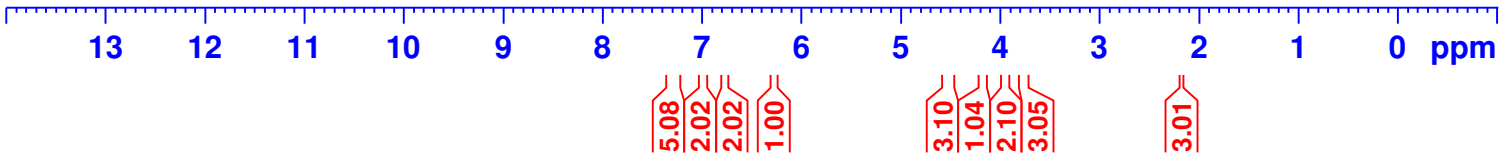
F2 - Processing parameters
SI                32768
SF         100.6277790 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-3-12



4b

7.350 7.345 7.328 7.325 7.308 7.303 7.293 7.288 7.261 7.256 7.239 7.000 6.979 6.786 6.764 6.294 6.281 6.268 4.572 4.558 4.528 4.513 4.507 4.195 4.180 4.164 4.149 3.977 3.963 3.932 3.918 3.893 3.890 3.808 3.801 3.777 3.770 3.760 3.746 3.730 2.181 2.060 2.055 2.049 2.044 2.038



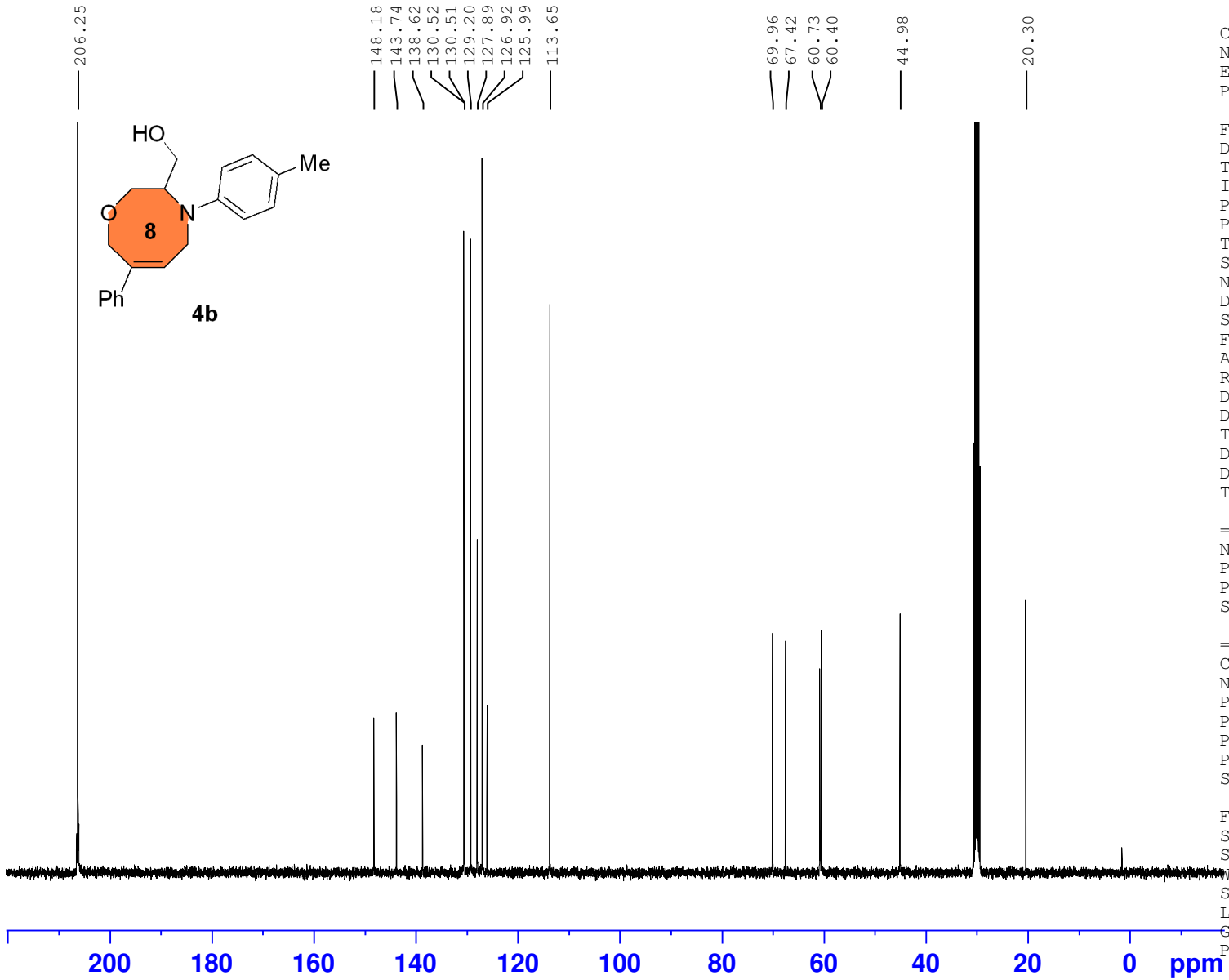

```
Current Data Parameters
NAME                  3-12
EXPNO                   33
PROCNO                   1

F2 - Acquisition Parameters
Date_             20230316
Time                  5.12
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT           Acetone
NS                       6
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9845889 sec
RG                   90.23
DW                  60.800 usec
DE                   6.50 usec
TE                  293.1 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.68 usec
PLW1          14.00000000 W
SFO1          400.1924713 MHz

F2 - Processing parameters
SI                   65536
SF            400.1900125 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

ljx-3-12




```
Current Data Parameters
NAME                  3-12
EXPNO                   34
PROCNO                   1

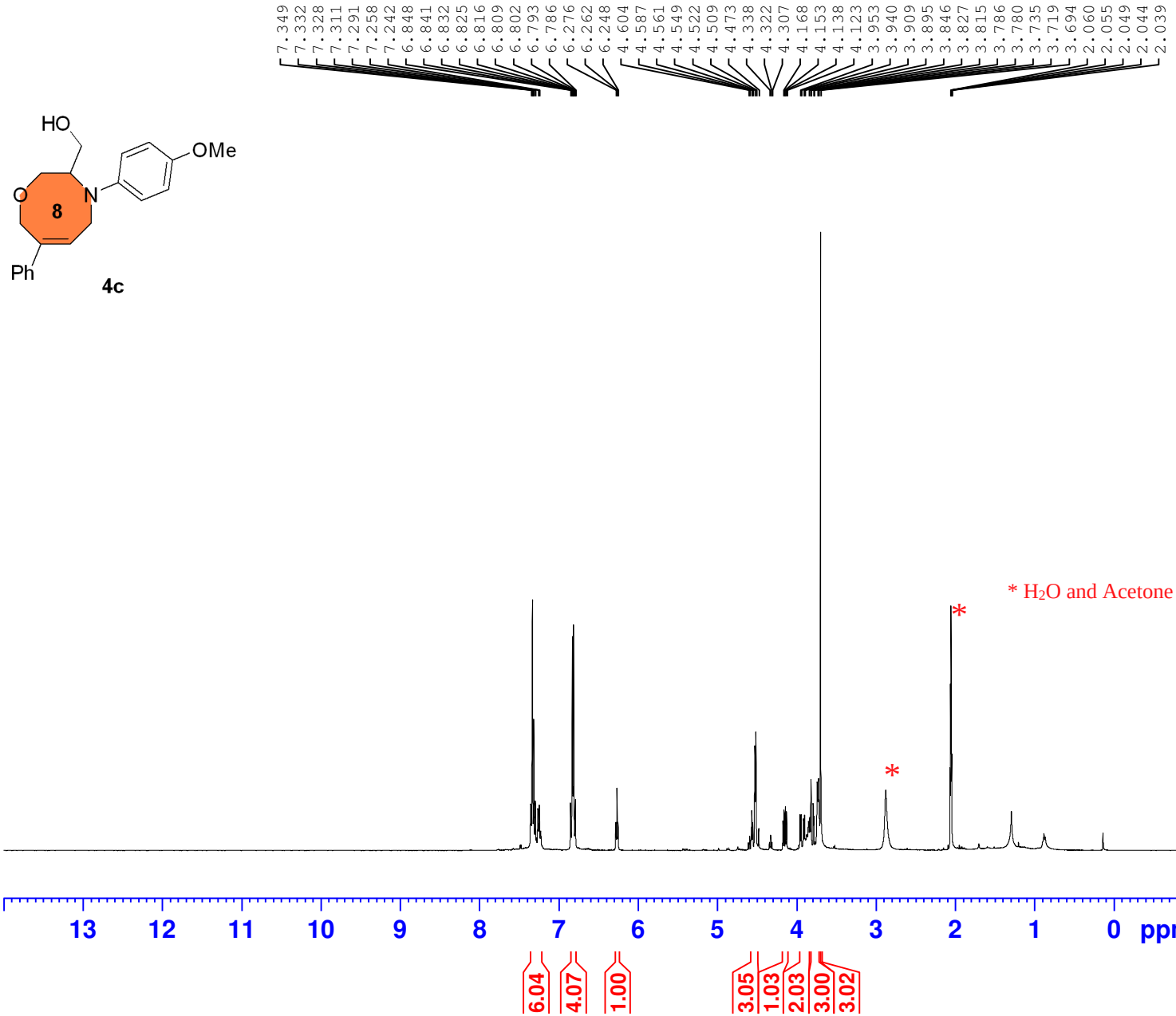
F2 - Acquisition Parameters
Date_              20230316
Time                   5.48
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                       600
DS                         4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                   193.13
DW                  20.800 usec
DE                   6.50 usec
TE                  293.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.00 usec
PLW1           53.00000000 W
SFO1          100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           14.00000000 W
PLW12           0.37246999 W
PLW13           0.30170000 W
SFO2          400.1916008 MHz

F2 - Processing parameters
SI                      32768
SF            100.6277672 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

ljx-3-8



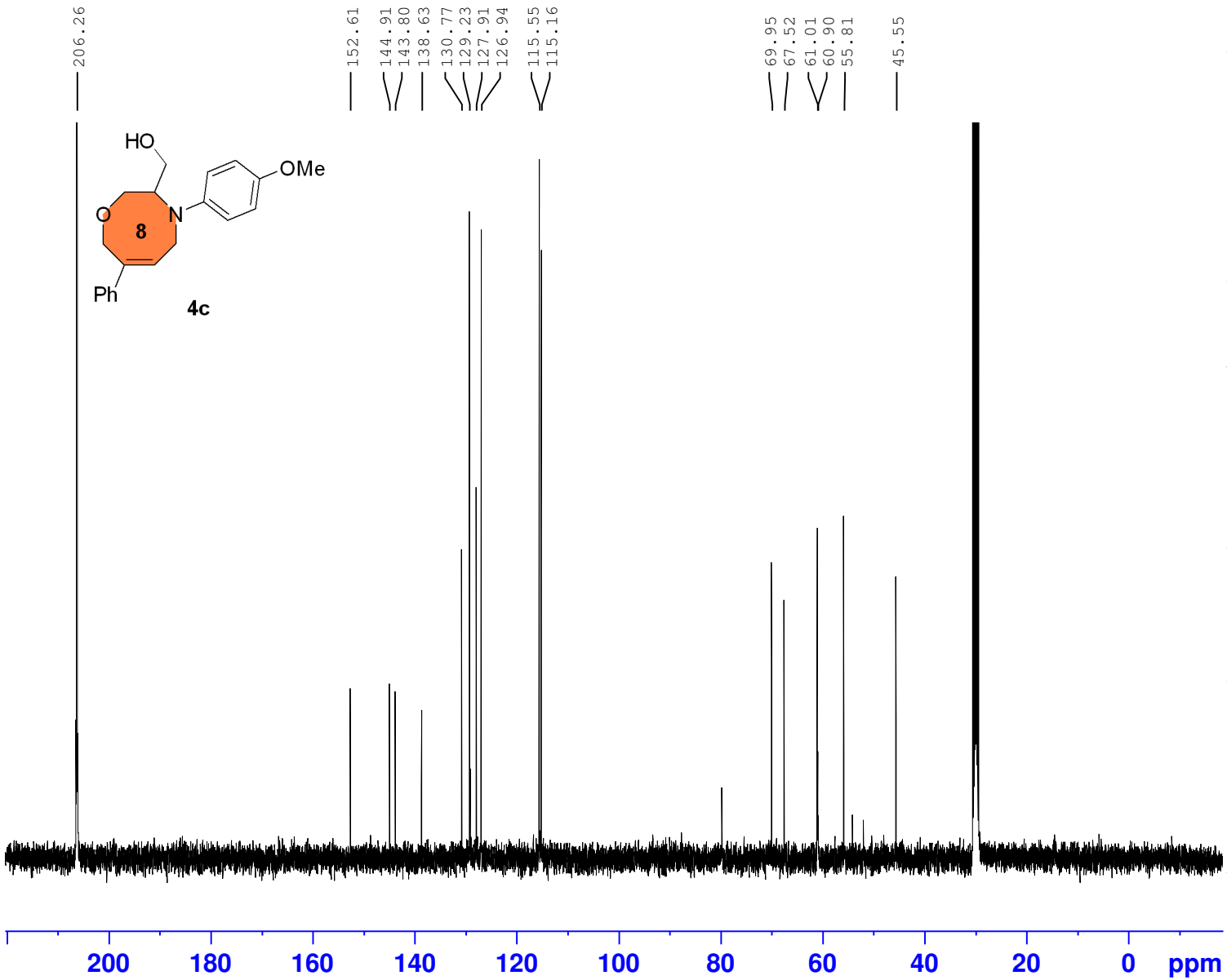

```
Current Data Parameters
NAME       20230316-400M
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230316
Time                4.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                140.02
DW                60.800 usec
DE                 6.50 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1        14.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900125 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-3-8




```
Current Data Parameters
NAME       20230316-400M
EXPNO                 32
PROCNO                 1

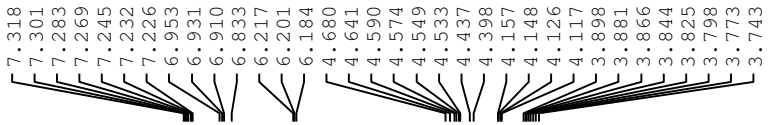
F2 - Acquisition Parameters
Date_           20230316
Time                5.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  600
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               293.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

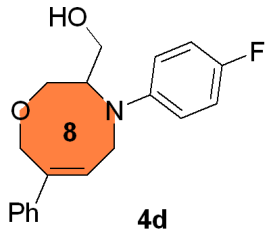
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

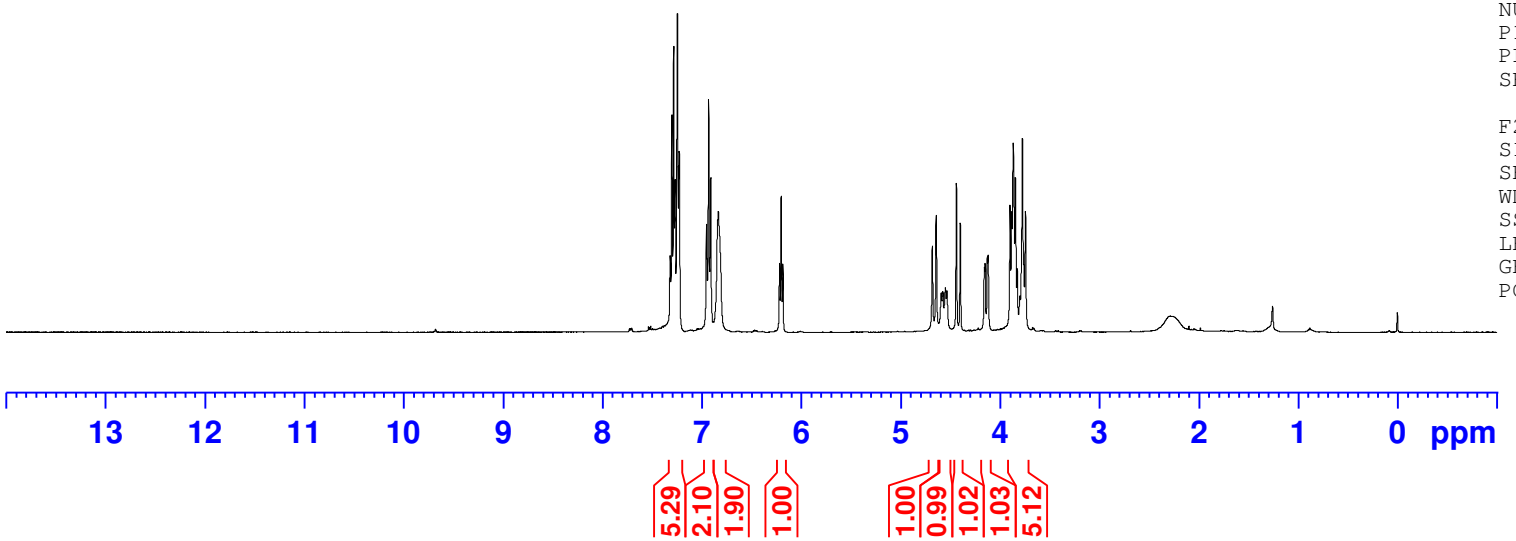
F2 - Processing parameters
SI                32768
SF         100.6277636 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-3-11





4d

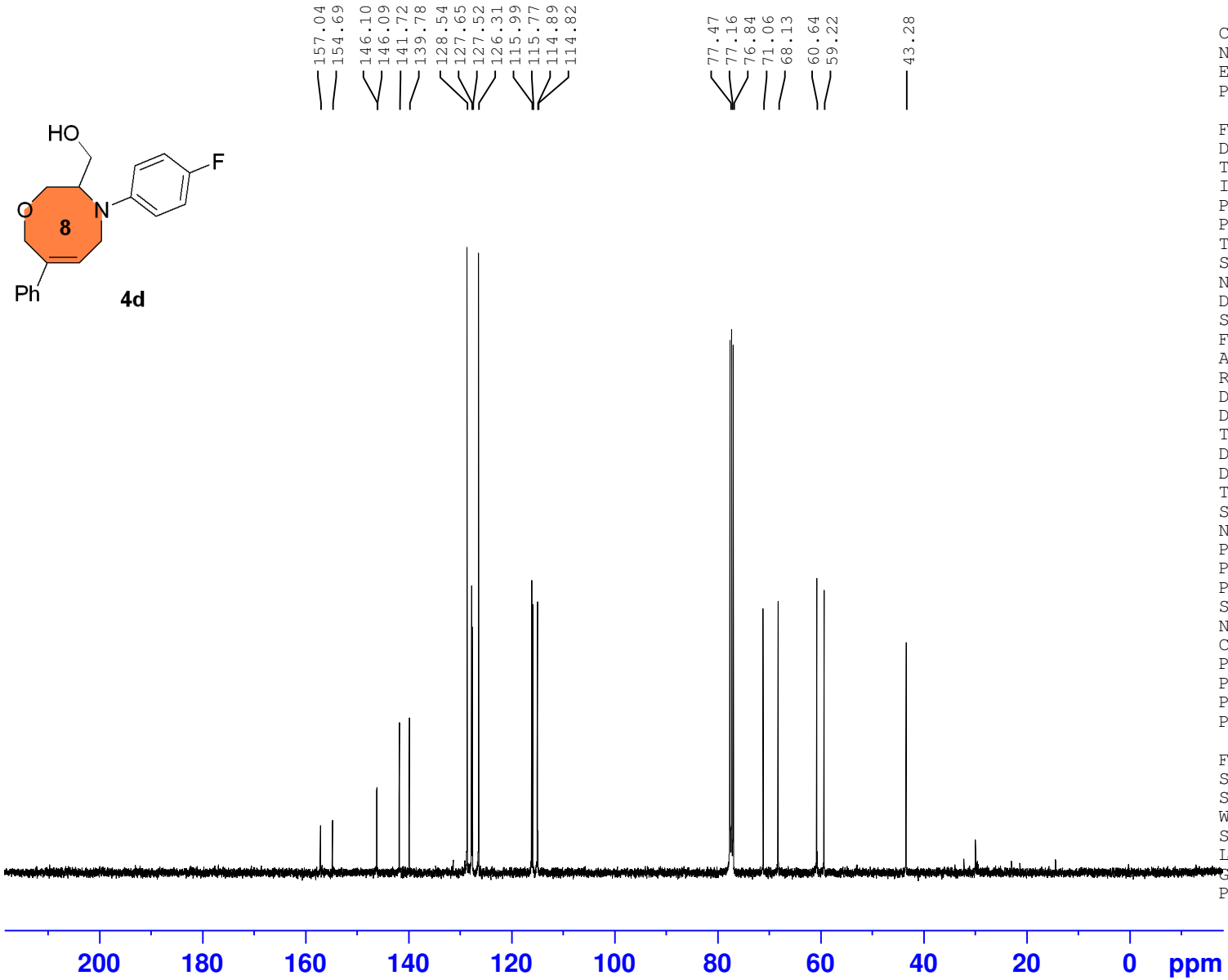


Current Data Parameters
NAME 20231021-400M
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231020
Time 22.41
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 29.75
DW 60.800 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900248 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

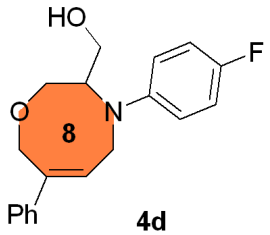



```
Current Data Parameters
NAME                  LJX-3-11
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                20230729
Time                     5.11 h
INSTRUM                Avance
PROBHD   Z116098_0833 (
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        500
DS                          4
SWH                 23809.523 Hz
FIDRES               0.726609 Hz
AQ                   1.3762560 sec
RG                     46.0295
DW                     21.000 usec
DE                      6.50 usec
TE                     295.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1
SFO1               100.6228298 MHz
NUC1                       13C
P0                      3.33 usec
P1                     10.00 usec
PLW1               87.89900208 W
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2              waltz65
PCPD2                  90.00 usec
PLW2               20.73200035 W
PLW12               0.25595000 W
PLW13               0.12874000 W

F2 - Processing parameters
SI                      32768
SF                 100.6127606 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

^{19}F NMR



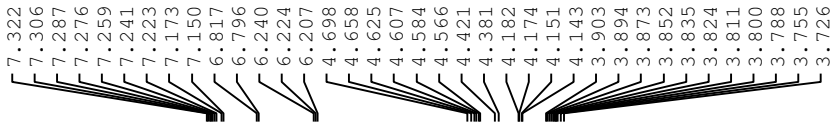
4d

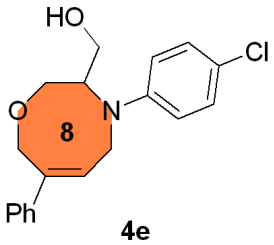

```
Current Data Parameters
NAME            LJX-3-11
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230729
Time                5.12 h
INSTRUM           Avance
PROBHD   Z116098_0833 (
PULPROG             zgig
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                294.6 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         376.4607164 MHz
NUC1                19F
P1                18.00 usec
PLW1         16.73100090 W
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         20.73200035 W
PLW12         0.25595000 W

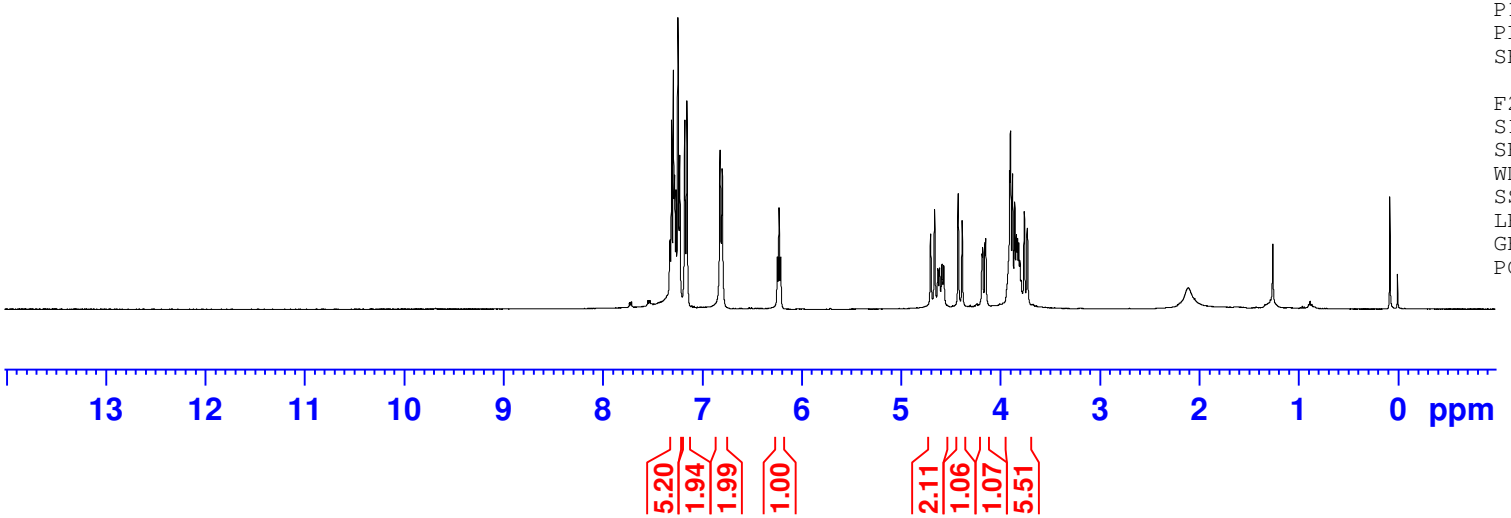
F2 - Processing parameters
SI                 65536
SF           376.4983662 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

ljx-3-10





4e



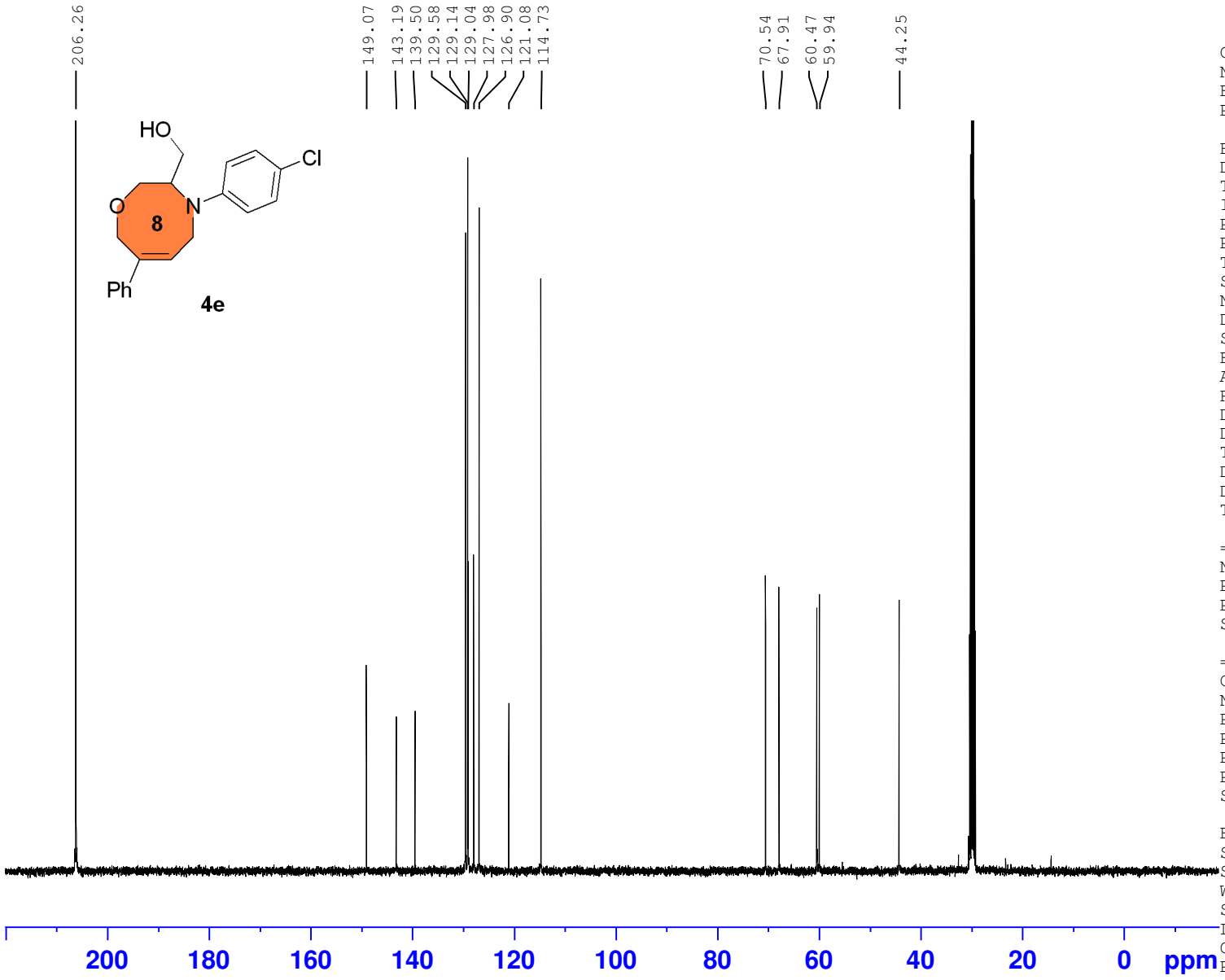

```
Current Data Parameters
NAME       20231021-400M
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231020
Time               22.18
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 61.19
DW                60.800 usec
DE                 6.50 usec
TE                293.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900215 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-3-10




```
Current Data Parameters
NAME                  3-10
EXPNO                   17
PROCNO                   1

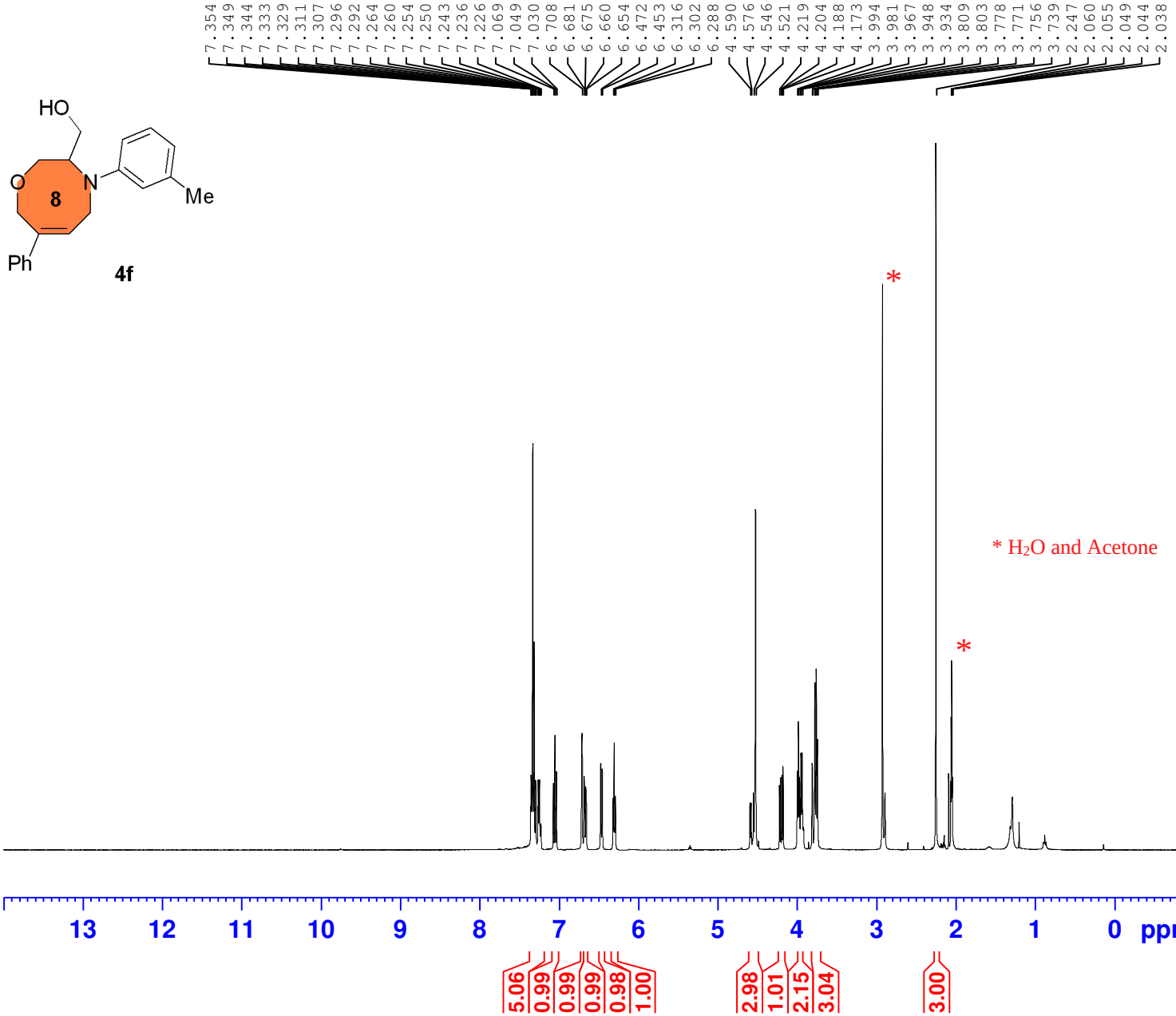
F2 - Acquisition Parameters
Date_              20230310
Time                   0.36
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                      300
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   193.13
DW                  20.800 usec
DE                   6.50 usec
TE                  295.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.00 usec
PLW1           53.00000000 W
SFO1          100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           14.00000000 W
PLW12           0.37246999 W
PLW13           0.30170000 W
SFO2          400.1916008 MHz

F2 - Processing parameters
SI                    32768
SF            100.6277765 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

ljx-3-17



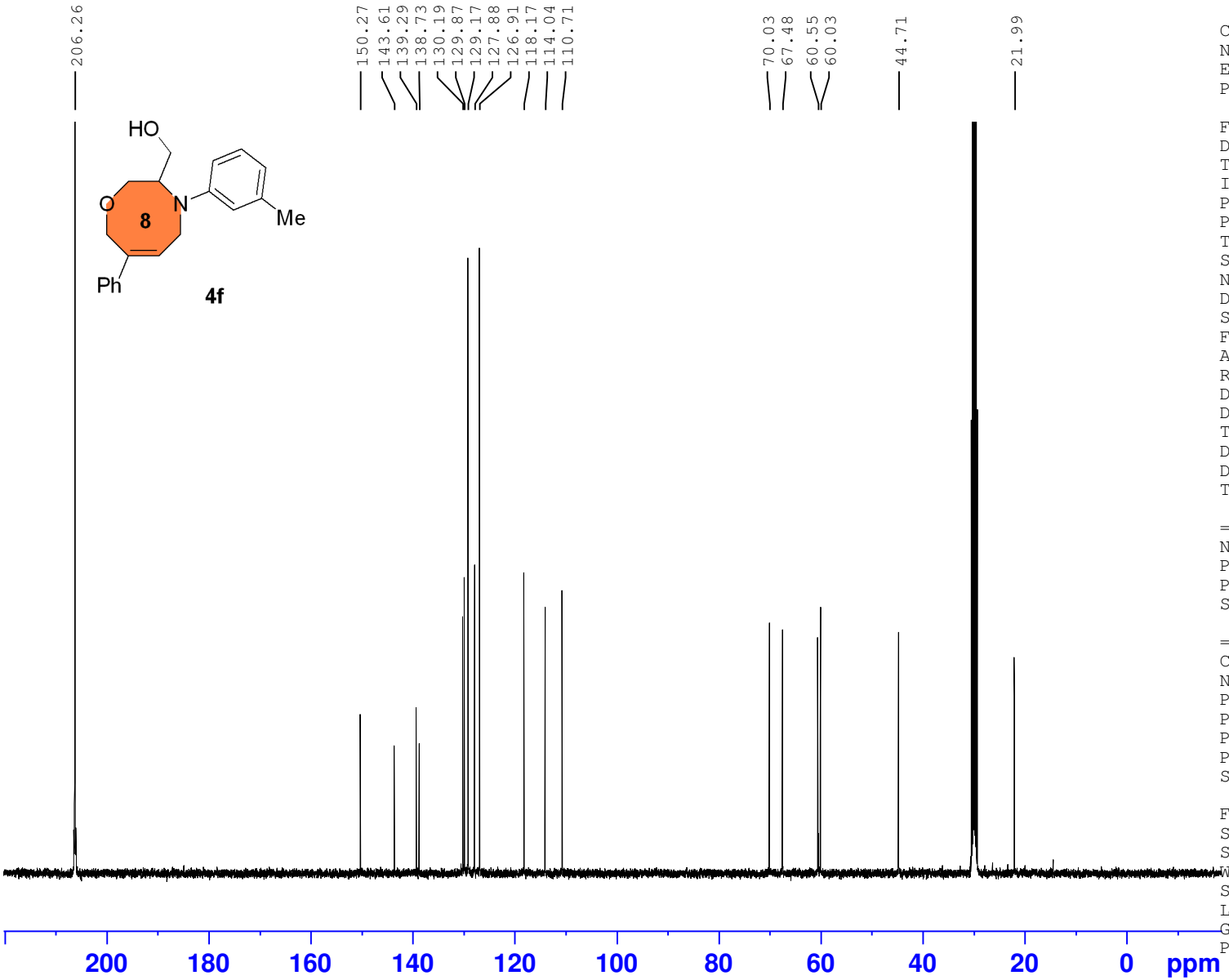

```
Current Data Parameters
NAME                  3-17
EXPNO                   24
PROCNO                   1

F2 - Acquisition Parameters
Date_            20230322
Time                 2.43
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           Acetone
NS                      8
DS                      2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                  90.23
DW                 60.800 usec
DE                  6.50 usec
TE                 291.6 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                  65536
SF           400.1900127 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

ljx-3-17



Current Data Parameters
NAME 3-17
EXPNO 25
PROCNO 1

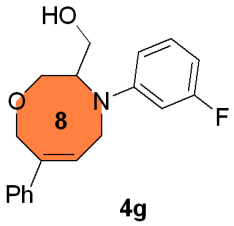
F2 - Acquisition Parameters
Date_ 20230322
Time 3.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 800
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 193.13
DW 20.800 usec
DE 6.50 usec
TE 292.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PLW1 53.00000000 W
SFO1 100.6379178 MHz

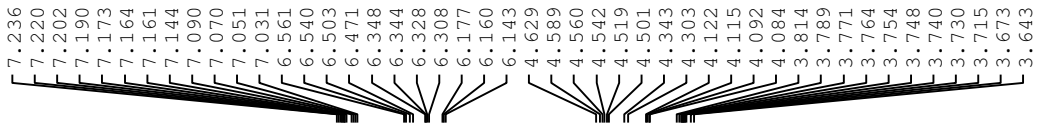
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.37246999 W
PLW13 0.30170000 W
SFO2 400.1916008 MHz

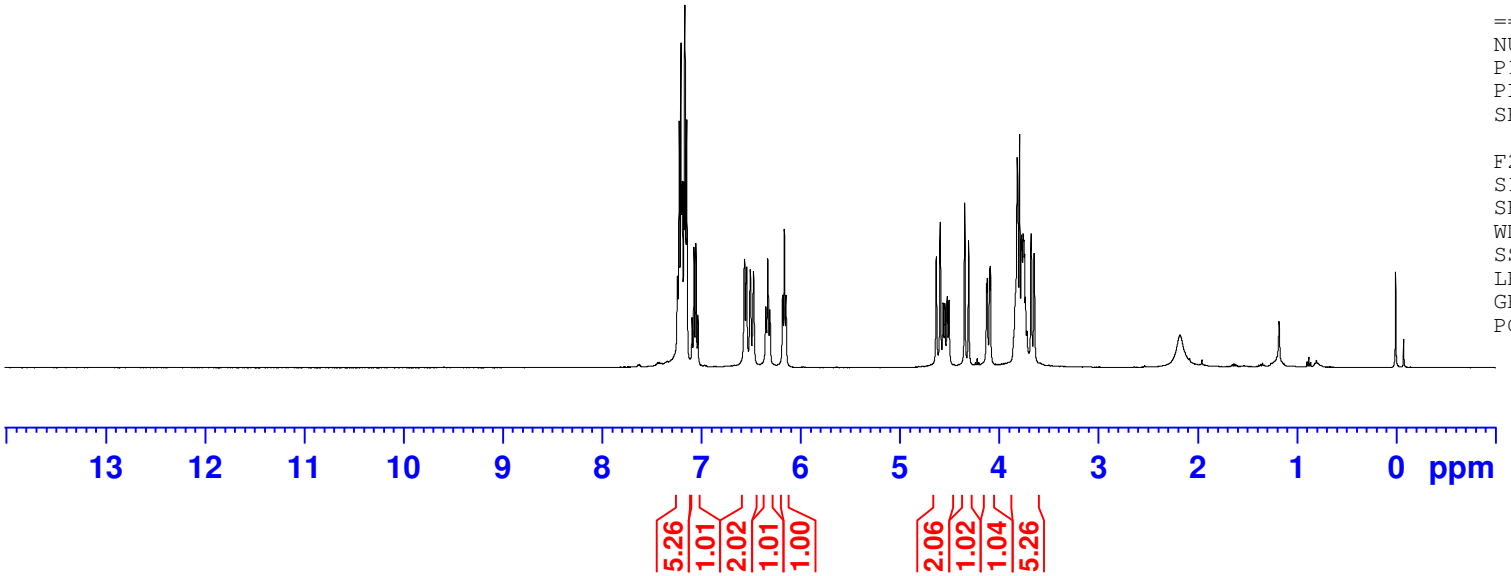
F2 - Processing parameters
SI 32768
SF 100.6277712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ljx-5-14



4g





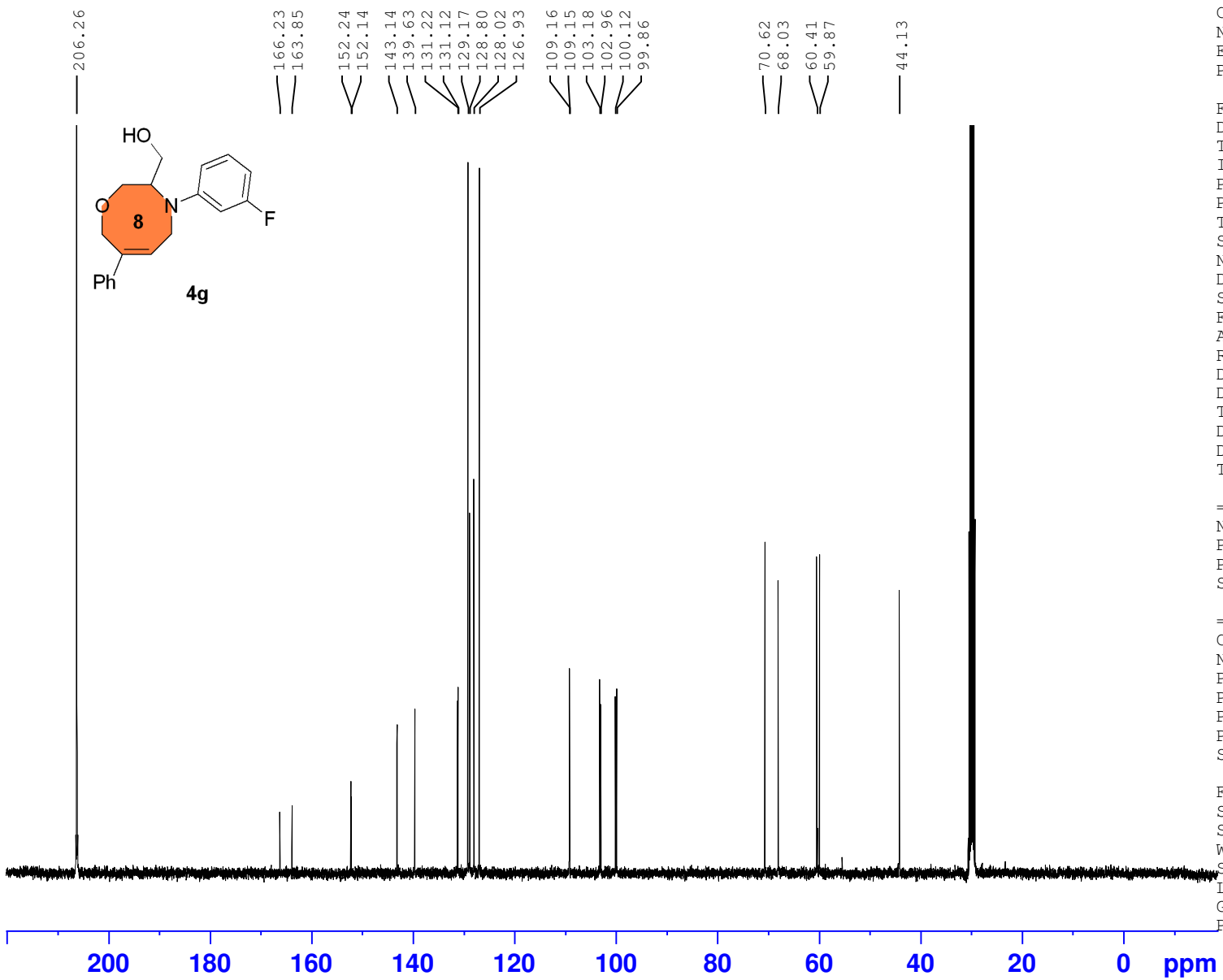
Current Data Parameters
NAME 20231021-400M
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231020
Time 22.37
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 29.75
DW 60.800 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900598 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ljx-3-31




```
Current Data Parameters
NAME      2023-3-30-400
EXPNO               167
PROCNO                1

F2 - Acquisition Parameters
Date_          20230330
Time               3.27
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               291.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

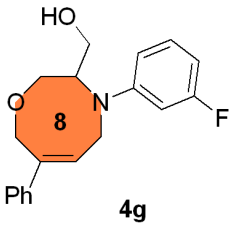
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                32768
SF          100.6277746 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR

LJX-5-14



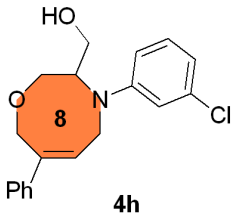
4g


```
Current Data Parameters
NAME                  0927HH
EXPNO                      5
PROCNO                     1

F2 - Acquisition Parameters
Date_              20230927
Time                  16.29 h
INSTRUM              Avance
PROBHD    Z116098_0833 (
PULPROG                zgig
TD                   131072
SOLVENT             Acetone
NS                        16
DS                         4
SWH              90909.094 Hz
FIDRES            1.387163 Hz
AQ               0.7208960 sec
RG                       101
DW                   5.500 usec
DE                   6.50 usec
TE                   294.4 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                        1
SFO1           376.4607164 MHz
NUC1                    19F
P1                   18.00 usec
PLW1           16.73100090 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           20.73200035 W
PLW12           0.25595000 W

F2 - Processing parameters
SI                     65536
SF             376.4983662 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00
```

ljx-5-15



4h

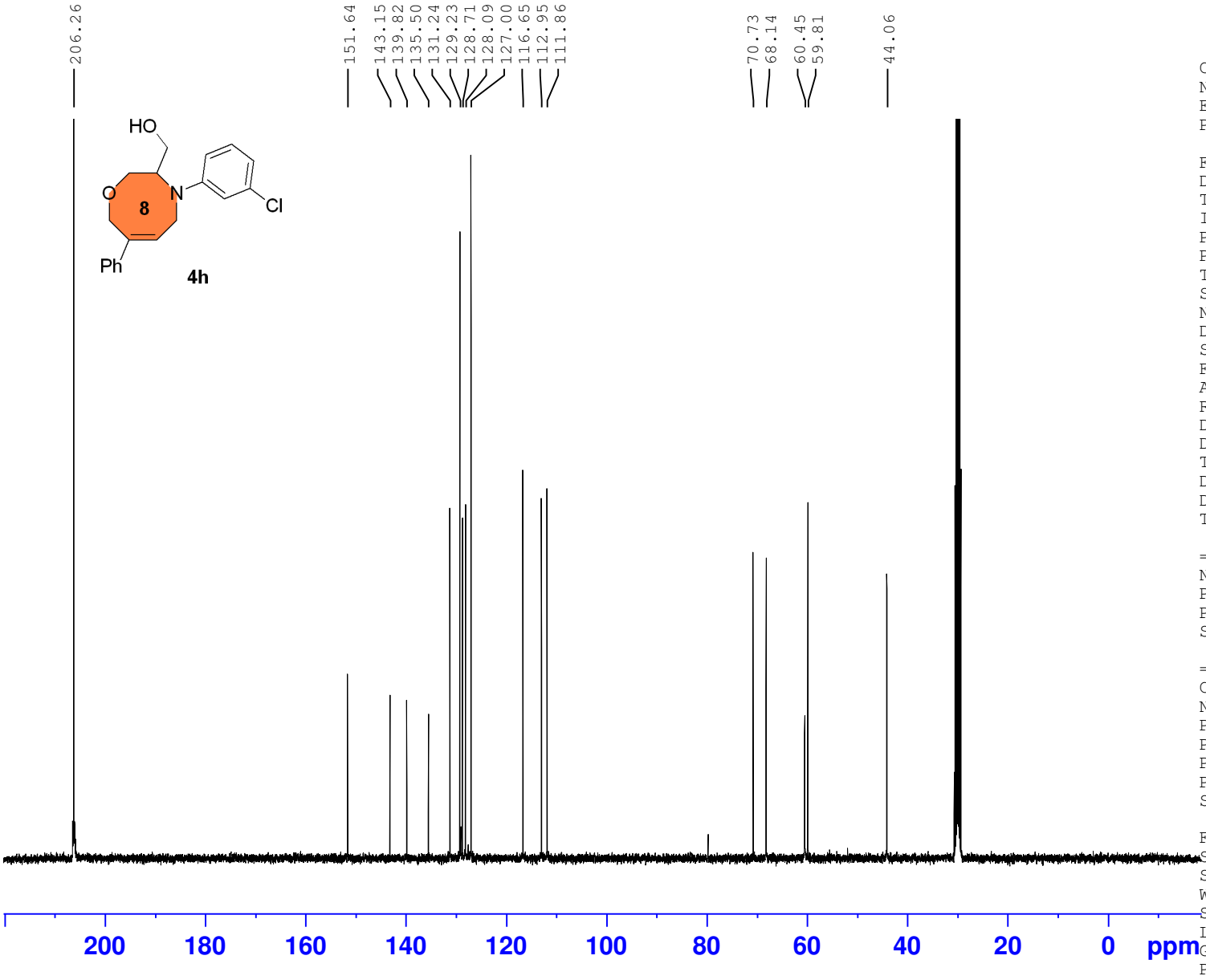

```
Current Data Parameters
NAME       2023-9-27-400
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230926
Time               23.25
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 29.75
DW                60.800 usec
DE                 6.50 usec
TE                291.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900111 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-2-99




```
Current Data Parameters
NAME      20230316-400M
EXPNO                30
PROCNO                1

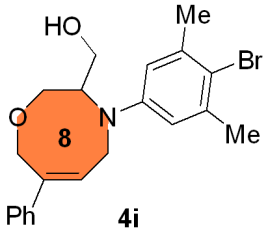
F2 - Acquisition Parameters
Date_          20230316
Time               4.30
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  600
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               293.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                32768
SF          100.6277693 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-4-12



4i

7.344 7.338 7.331 7.322 7.317 7.306 7.300 7.291 7.286 7.264 7.258 7.251 7.245 7.242 7.234 7.227 6.720 6.319 6.304 6.289 4.592 4.576 4.572 4.548 4.534 4.504 4.467 4.216 4.203 4.185 4.172 4.056 4.041 4.027 3.975 3.960 3.943 3.931 3.925 3.918 3.788 3.781 3.778 3.762 3.758 3.751 3.745 2.313 2.061 2.055 2.050 2.044 2.039

* H_2O and Acetone

13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

5.06 2.00 1.00 3.07 1.02 2.04 3.01 6.01

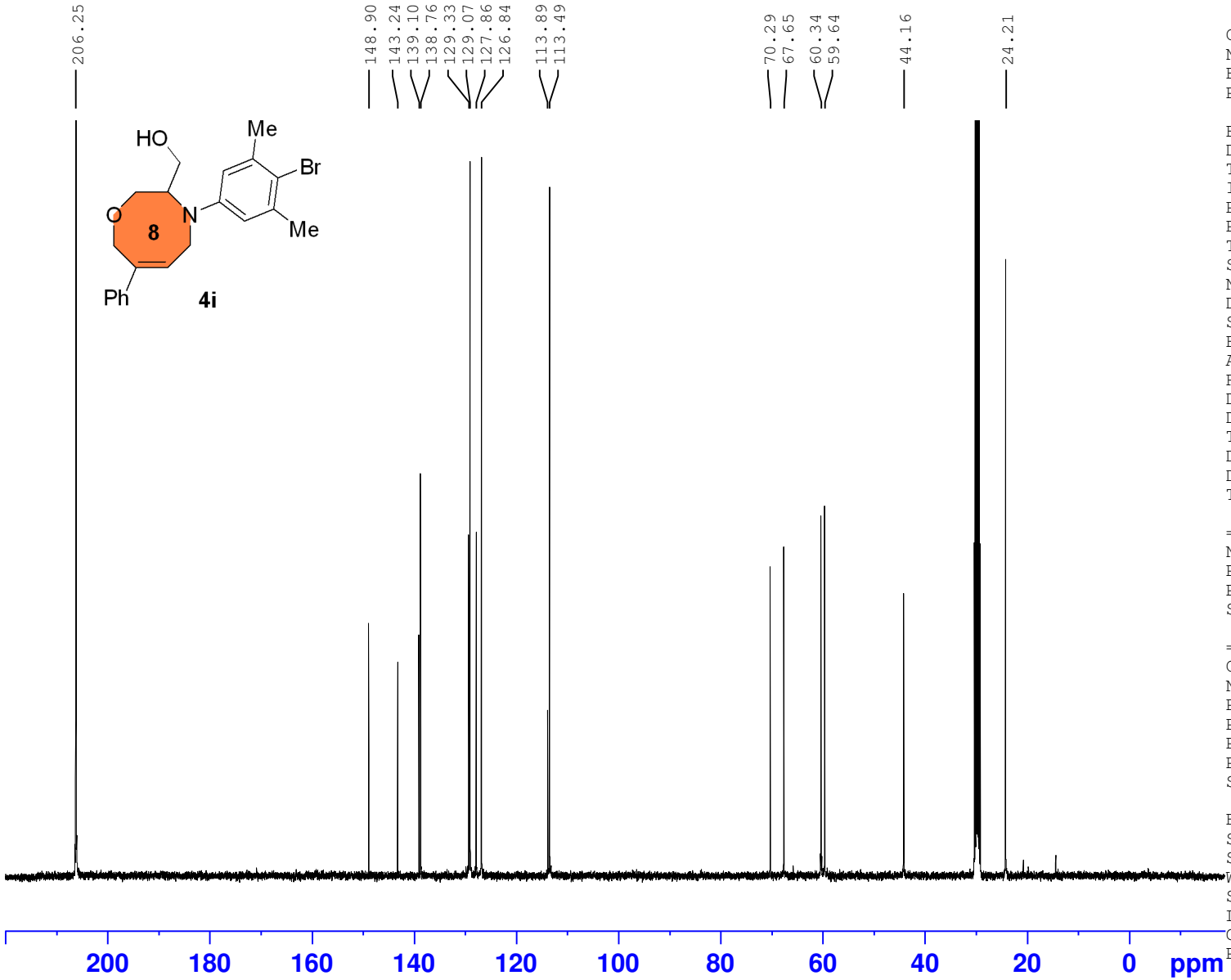
```
Current Data Parameters
NAME       20230614-400M
EXPNO                 49
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230614
Time                7.32
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 29.75
DW                60.800 usec
DE                 6.50 usec
TE                292.6 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900111 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

ljx-4-12




```
Current Data Parameters
NAME       20230614-400M
EXPNO                 50
PROCNO                 1

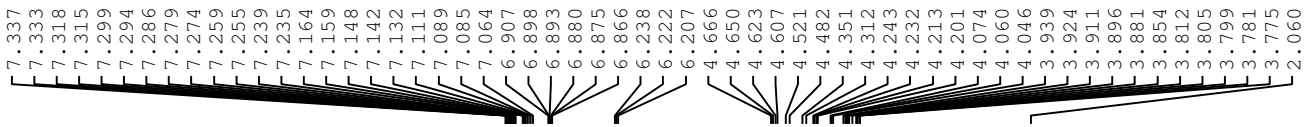
F2 - Acquisition Parameters
Date_           20230614
Time                8.02
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                35.06
DW               20.800 usec
DE                6.50 usec
TE                293.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

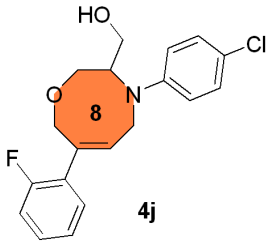
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.80 usec
PLW1        47.40000153 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        23.00000000 W
PLW12        0.30712000 W
PLW13        0.24877000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6277834 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

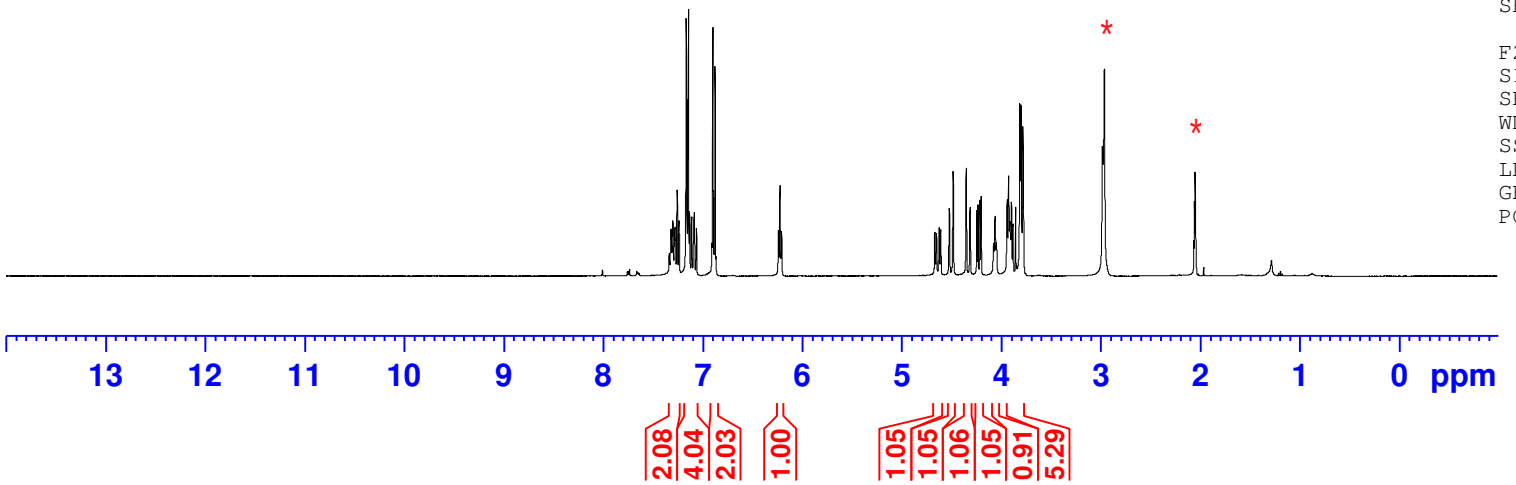
ljx-4-85





4j

* H_2O and Acetone



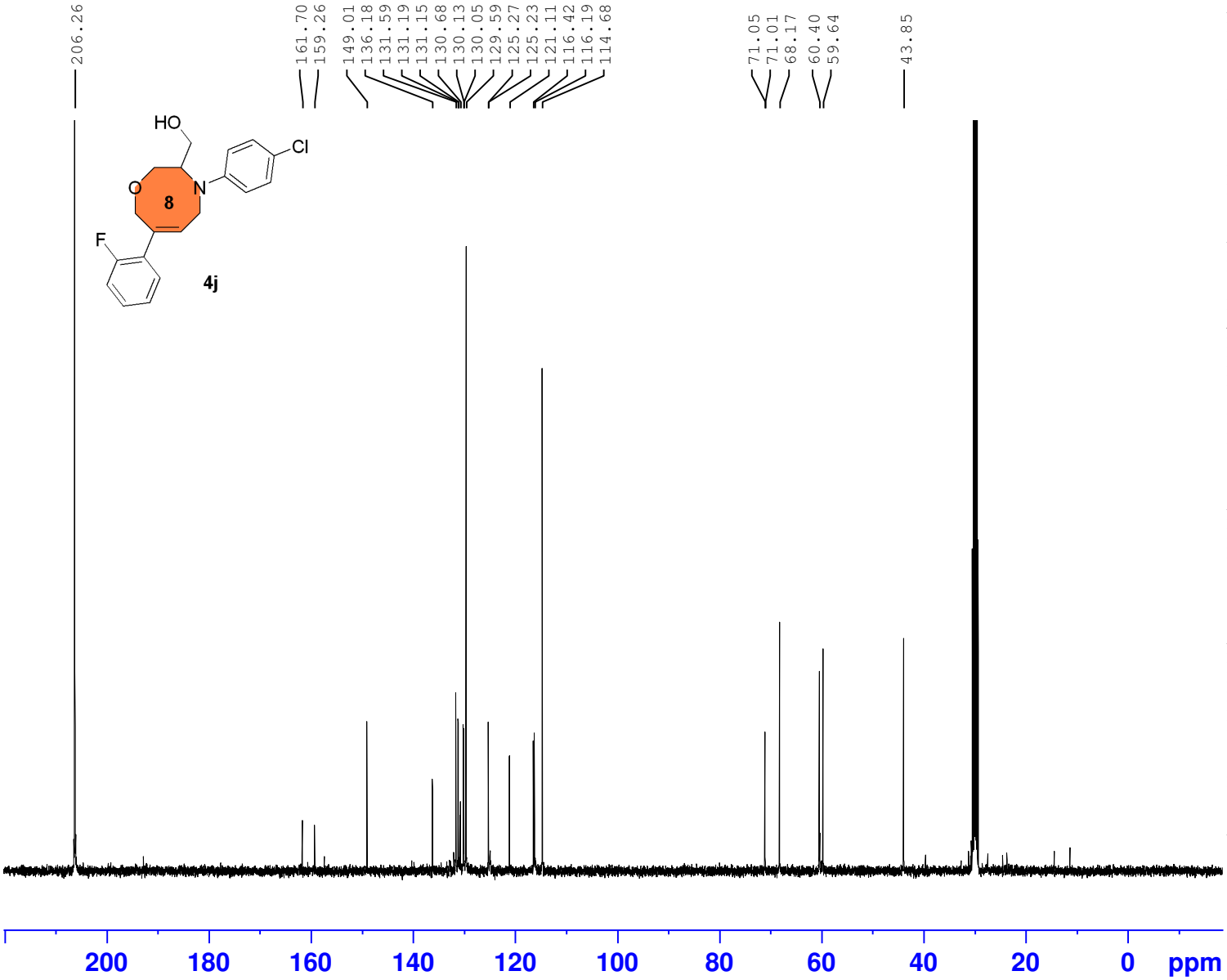
Current Data Parameters
NAME 20231026-400m
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231025
Time 22.05
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 53.3
DW 60.800 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ljx-3-85




```
Current Data Parameters
NAME       2023-5-27-400
EXPNO                 25
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230527
Time                1.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE                293.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

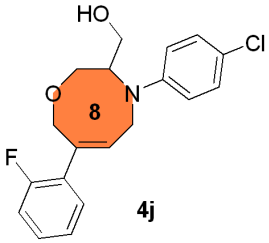
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6277744 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR

LJX-3-85



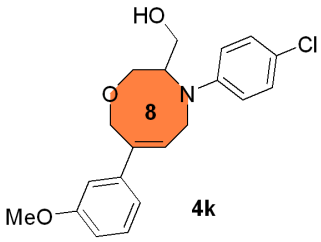
4j


```
Current Data Parameters
NAME                  0927HH
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20230927
Time                  15.48 h
INSTRUM              Avance
PROBHD    Z116098_0833 (
PULPROG                zgig
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              90909.094 Hz
FIDRES            1.387163 Hz
AQ               0.7208960 sec
RG                      101
DW                    5.500 usec
DE                    6.50 usec
TE                    294.5 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           376.4607164 MHz
NUC1                   19F
P1                   18.00 usec
PLW1           16.73100090 W
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2           20.73200035 W
PLW12           0.25595000 W

F2 - Processing parameters
SI                    65536
SF             376.4983662 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

LJX-3-75



7.246 7.226 7.206 7.172 7.163 7.157 7.145 7.140 7.131 6.905 6.903 6.893 6.886 6.884 6.877 6.870 6.861 6.843 6.837 6.822 6.816 6.333 6.318 6.303 4.635 4.621 4.591 4.577 4.539 4.475 4.437 4.214 4.201 4.183 4.169 3.942 3.927 3.900 3.885 3.778 3.758 3.747 3.741 2.061 2.055 2.050 2.044 2.039

* H_2O and Acetone

13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

1.05 2.02 4.04 1.01 1.00 1.00 2.07 1.04 2.09 6.02

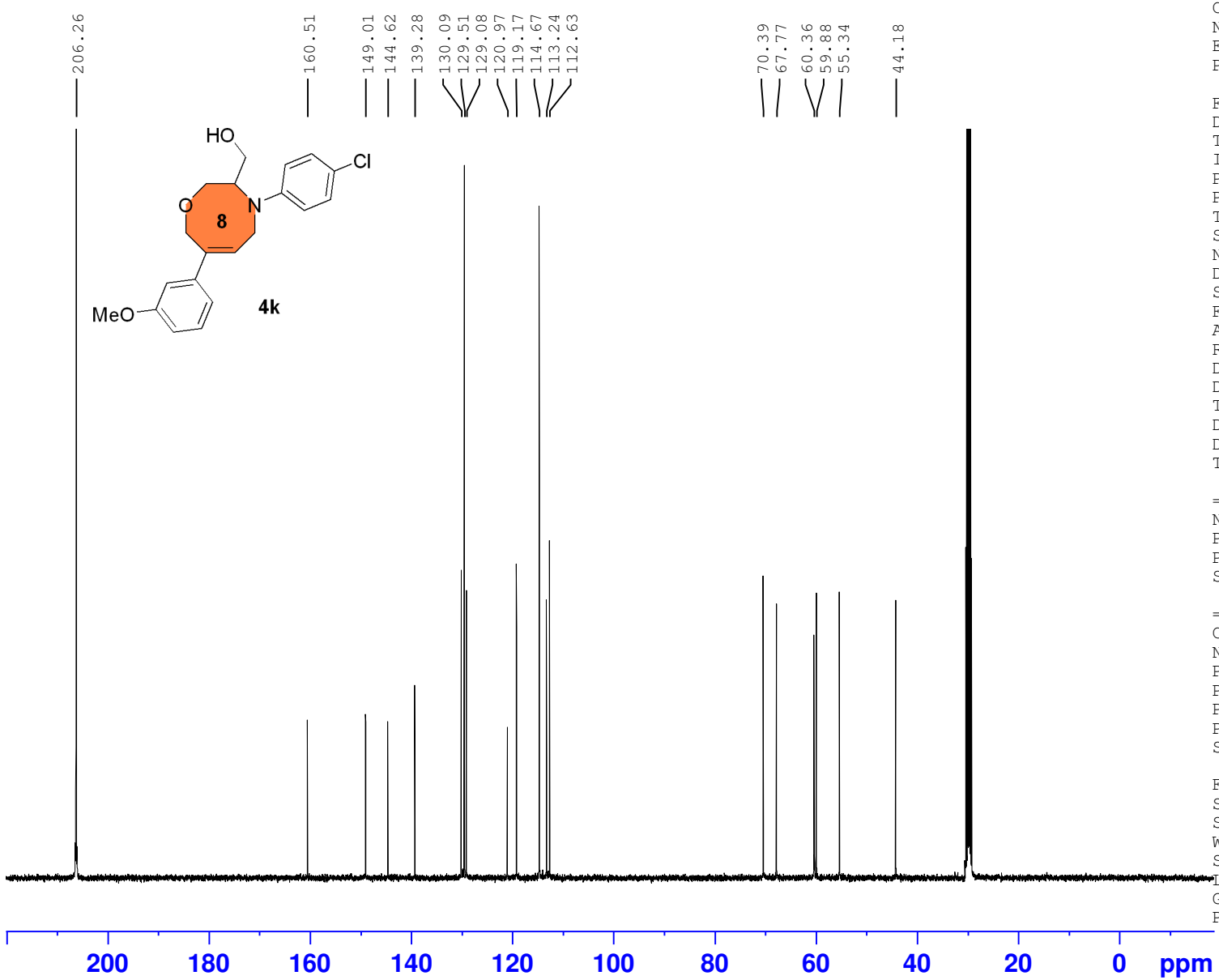
```
Current Data Parameters
NAME       2023-5-20-400
EXPNO                 27
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230520
Time                0.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    10
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 61.19
DW                60.800 usec
DE                 6.50 usec
TE                288.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900121 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

LJX-3-75




```
Current Data Parameters
NAME      2023-5-20-400
EXPNO                28
PROCNO                1

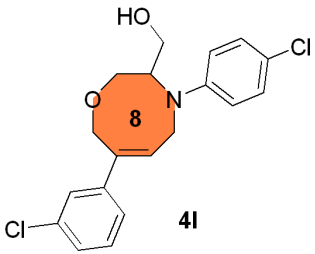
F2 - Acquisition Parameters
Date_          20230520
Time               1.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 1000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               289.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

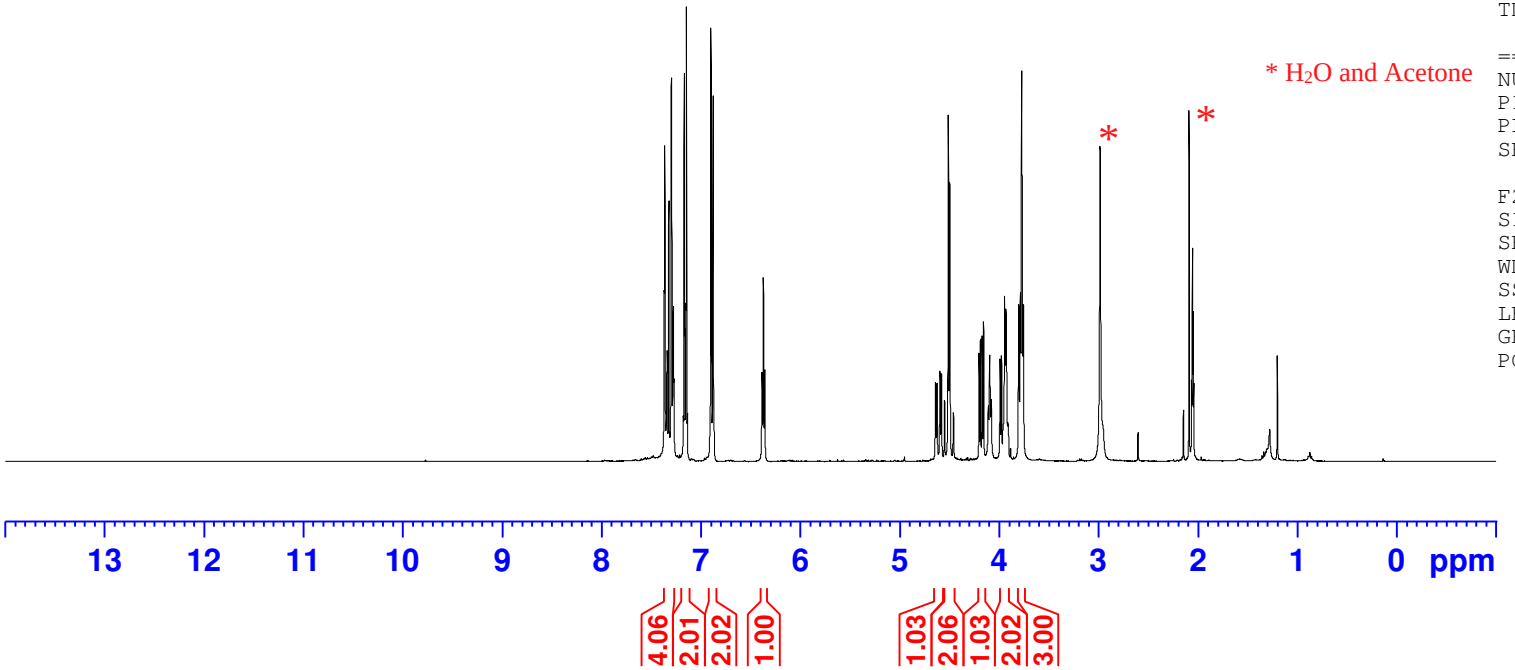
F2 - Processing parameters
SI                32768
SF          100.6277826 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

LJX-3-74



4l

7.368 7.363 7.346 7.340 7.337 7.322 7.296 7.281 7.277 7.274 7.270 7.167 7.145 6.898 6.875 6.384 6.370 6.356 4.637 4.623 4.593 4.579 4.546 4.509 4.495 4.457 4.199 4.185 4.168 4.154 3.988 3.974 3.959 3.943 3.929 3.911 3.905 3.803 3.796 3.785 3.771 3.754 2.061 2.055 2.050 2.044 2.039



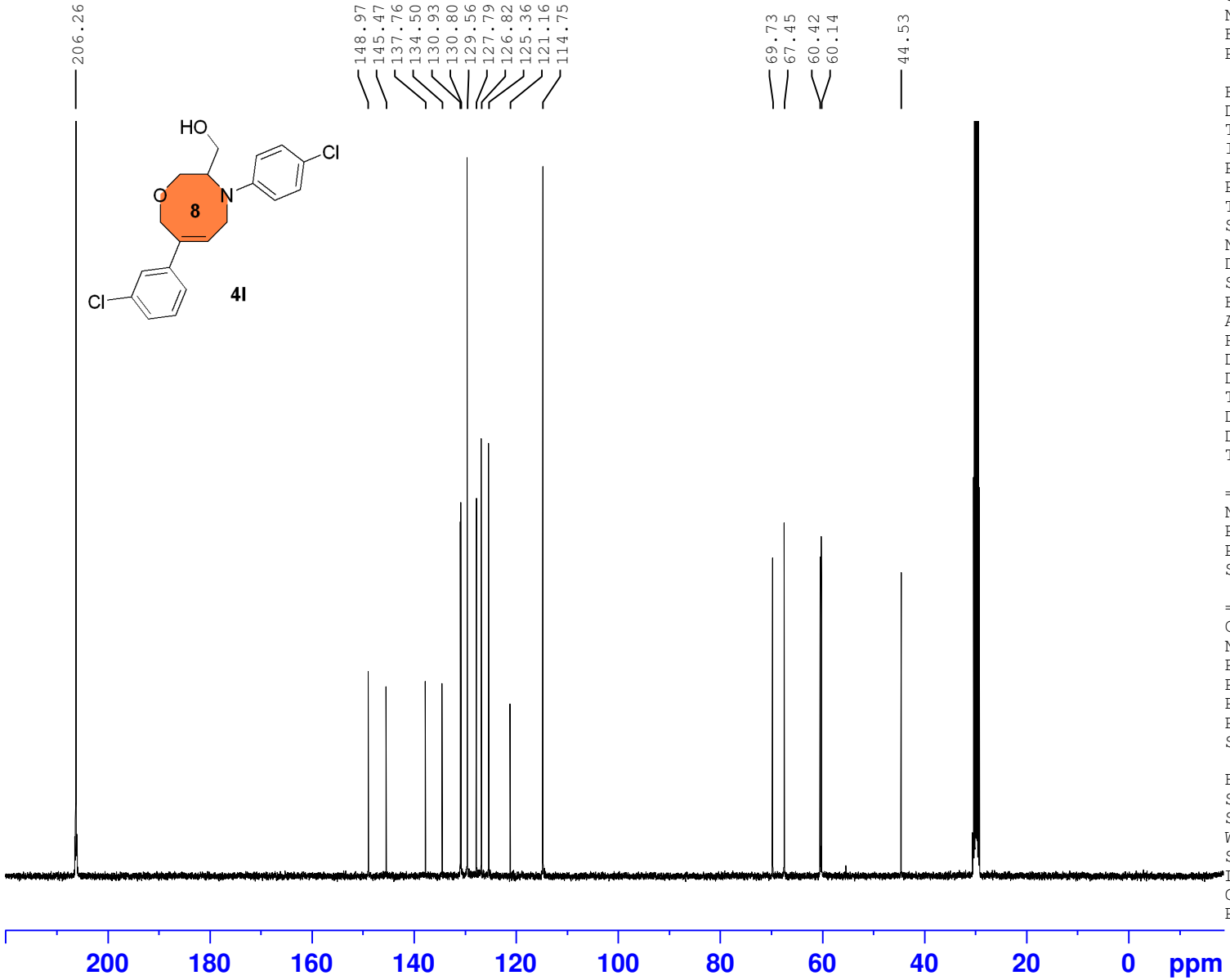

```
Current Data Parameters
NAME       2023-5-20-400
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230520
Time                2.42
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    10
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 68.24
DW                60.800 usec
DE                 6.50 usec
TE                288.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

LJX-3-74




```
Current Data Parameters
NAME      2023-5-20-400
EXPNO                32
PROCNO                1

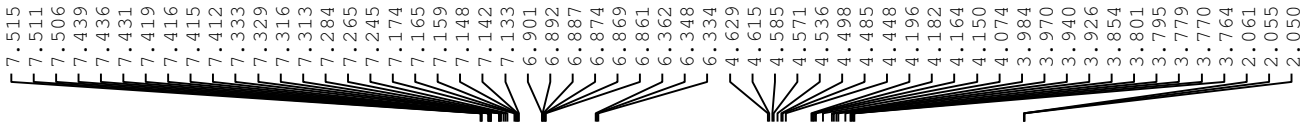
F2 - Acquisition Parameters
Date_          20230520
Time               3.40
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 1000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE                289.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

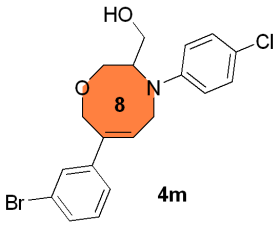
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                32768
SF           100.6277811 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

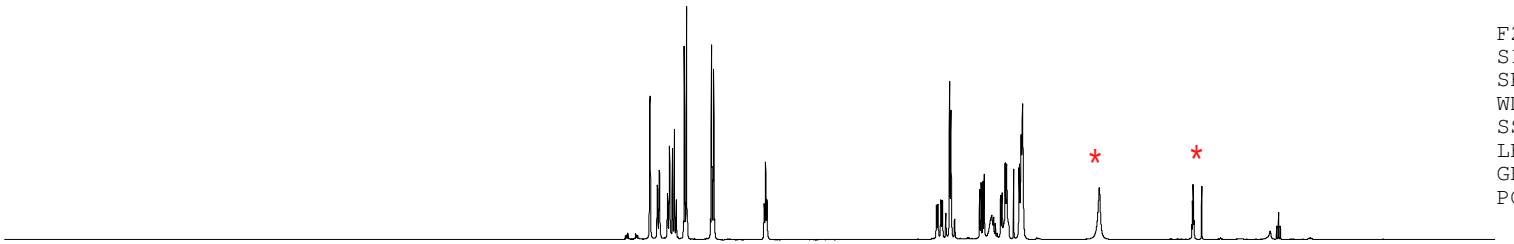
ljx-3-95

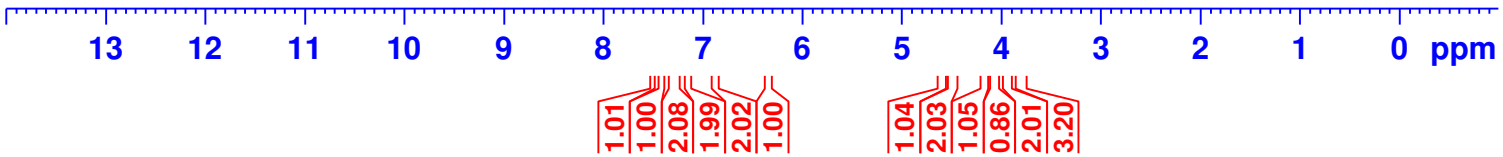




4m

* H_2O and Acetone





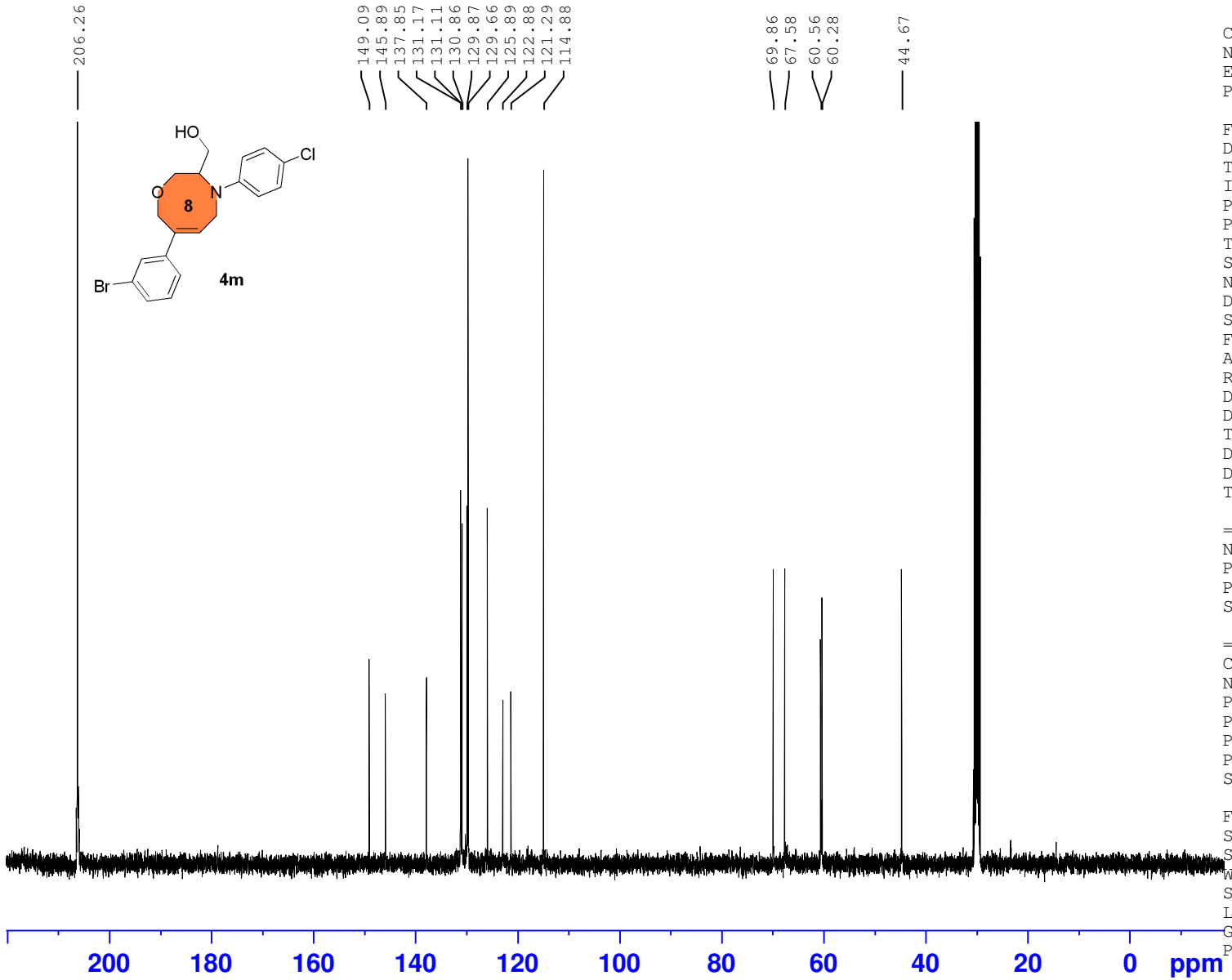

```
Current Data Parameters
NAME       20231028-400M
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231027
Time               22.03
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 29.75
DW                60.800 usec
DE                 6.50 usec
TE                292.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900111 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-3-95




```
Current Data Parameters
NAME      20230531-400M
EXPNO               210
PROCNO                1

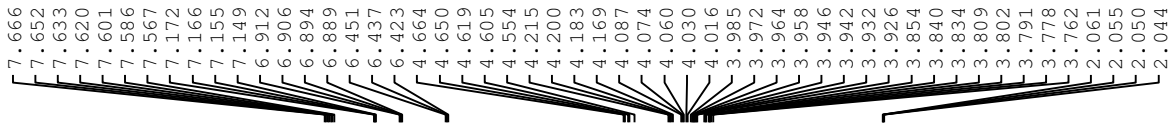
F2 - Acquisition Parameters
Date_          20230531
Time               7.53
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               294.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

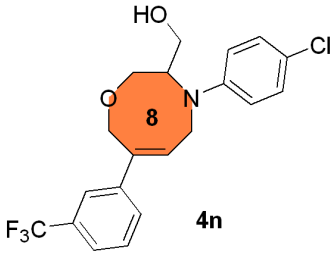
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

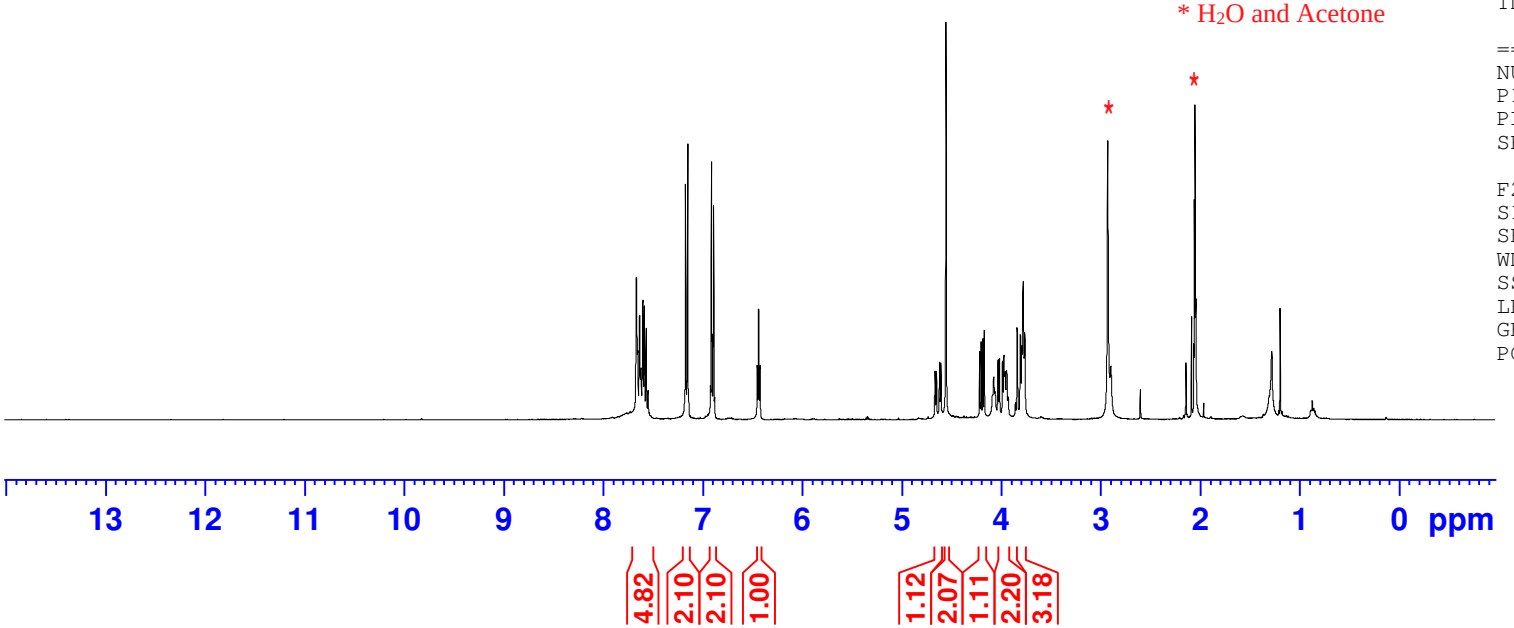
F2 - Processing parameters
SI                32768
SF         100.6277699 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-3-99





4n



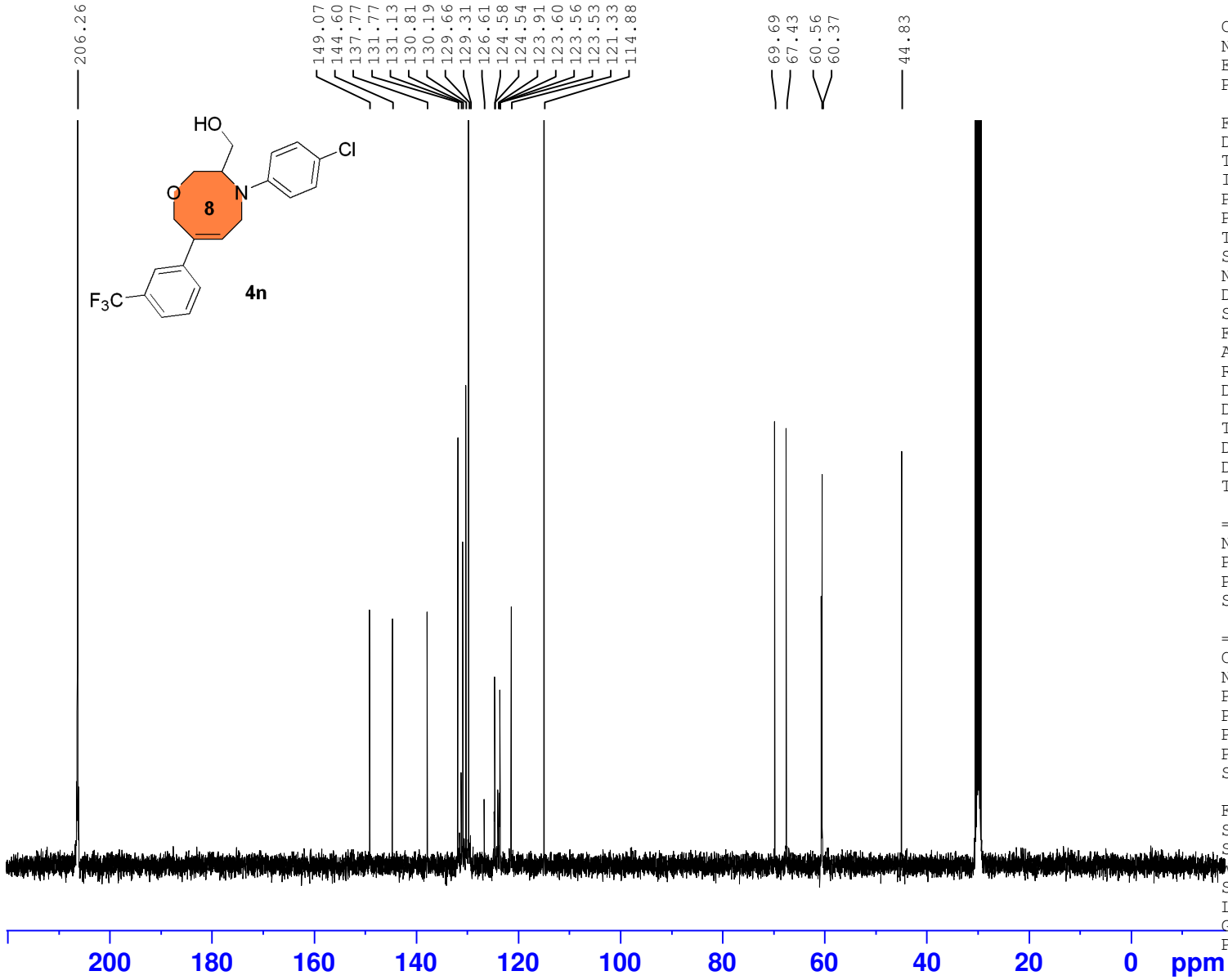
Current Data Parameters
NAME 2023-9-19-400
EXPNO 26
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230918
Time 23.22
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 100.49
DW 60.800 usec
DE 6.50 usec
TE 289.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900111 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ljx-3-99




```
Current Data Parameters
NAME       2023-5-27-400
EXPNO                 29
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230527
Time                2.12
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                   400
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               293.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

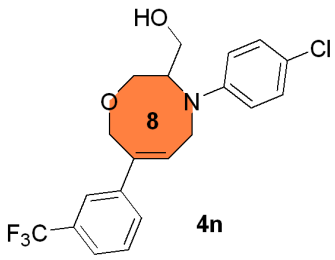
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6277713 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

^{19}F NMR

LJX-3-99



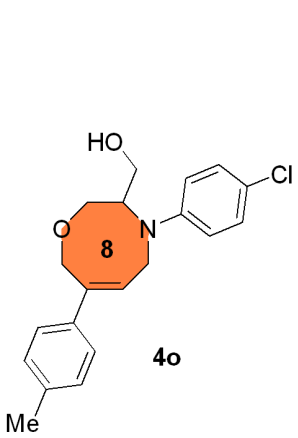
4n


```
Current Data Parameters
NAME                  0927HH
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_              20230927
Time                  16.10 h
INSTRUM              Avance
PROBHD    Z116098_0833 (
PULPROG                zgig
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              90909.094 Hz
FIDRES            1.387163 Hz
AQ               0.7208960 sec
RG                      101
DW                    5.500 usec
DE                    6.50 usec
TE                    294.5 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           376.4607164 MHz
NUC1                   19F
P1                   18.00 usec
PLW1           16.73100090 W
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           20.73200035 W
PLW12           0.25595000 W

F2 - Processing parameters
SI                    65536
SF             376.4983662 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

LJX-3-49



4o

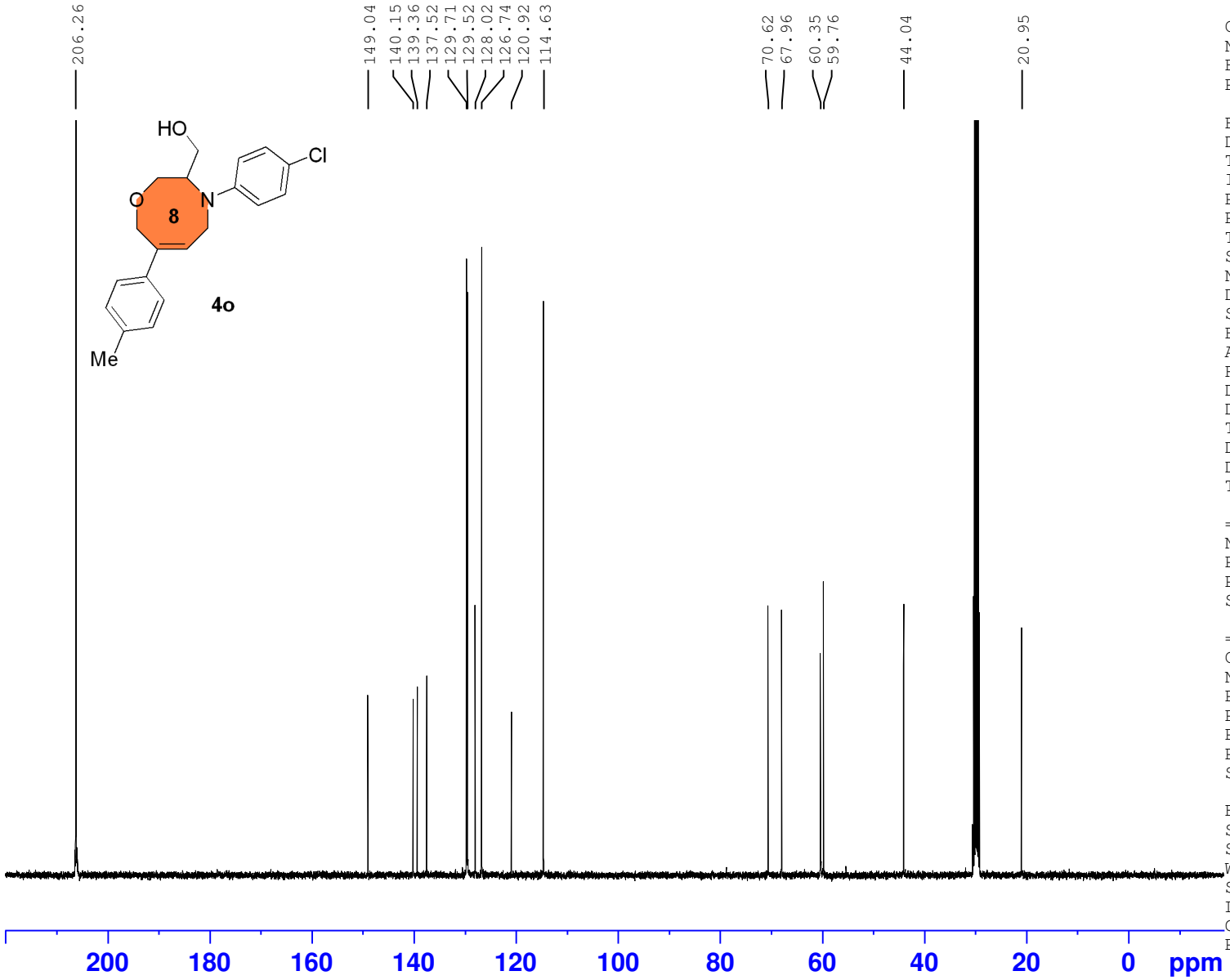

```
Current Data Parameters
NAME         230407-400M
EXPNO                 24
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time                6.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 61.19
DW                60.800 usec
DE                 6.50 usec
TE                289.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900121 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

LJX-3-49




```
Current Data Parameters
NAME         230407-400M
EXPNO                 25
PROCNO                 1

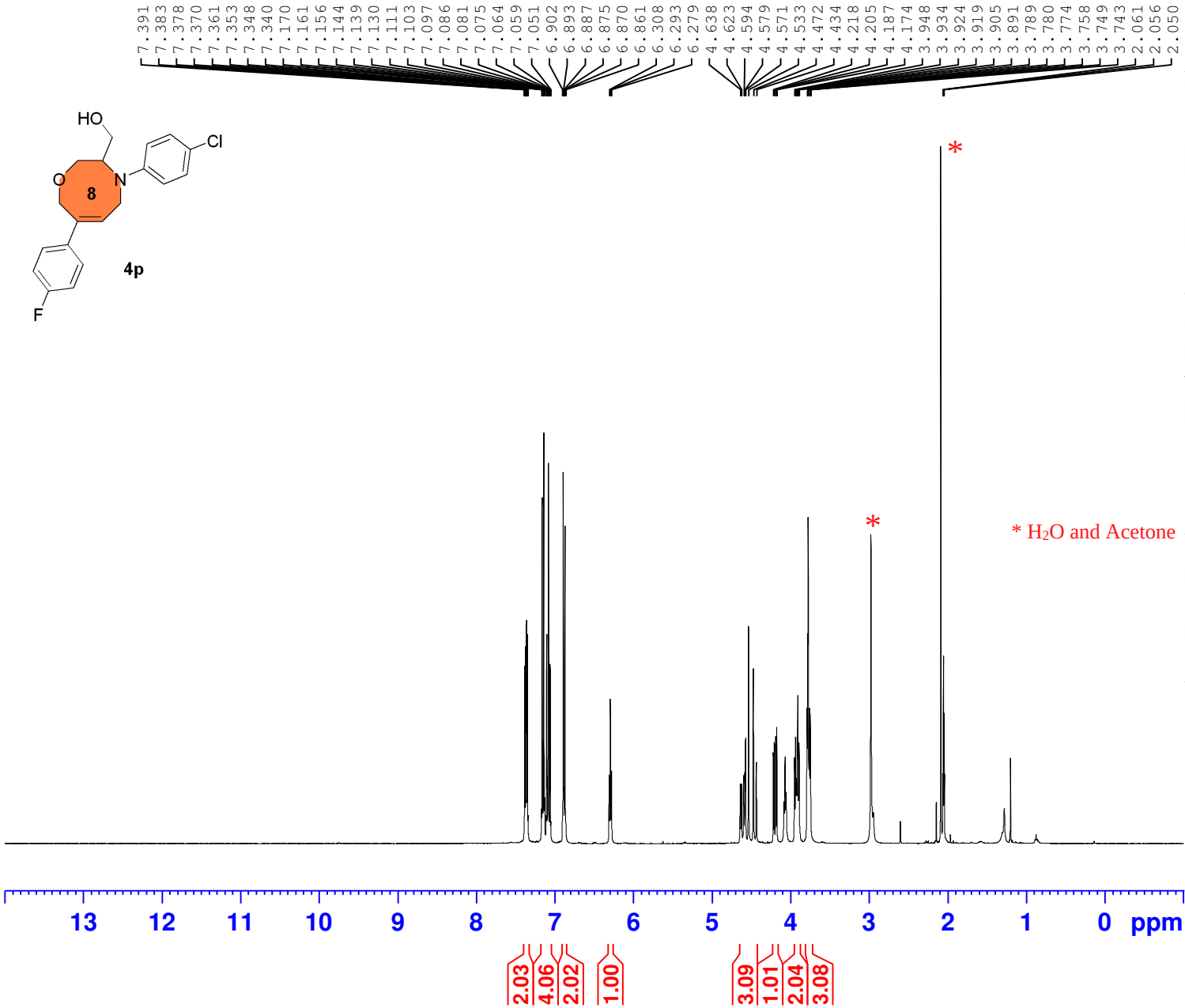
F2 - Acquisition Parameters
Date_           20230408
Time                7.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               290.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6277810 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

LJX-3-51



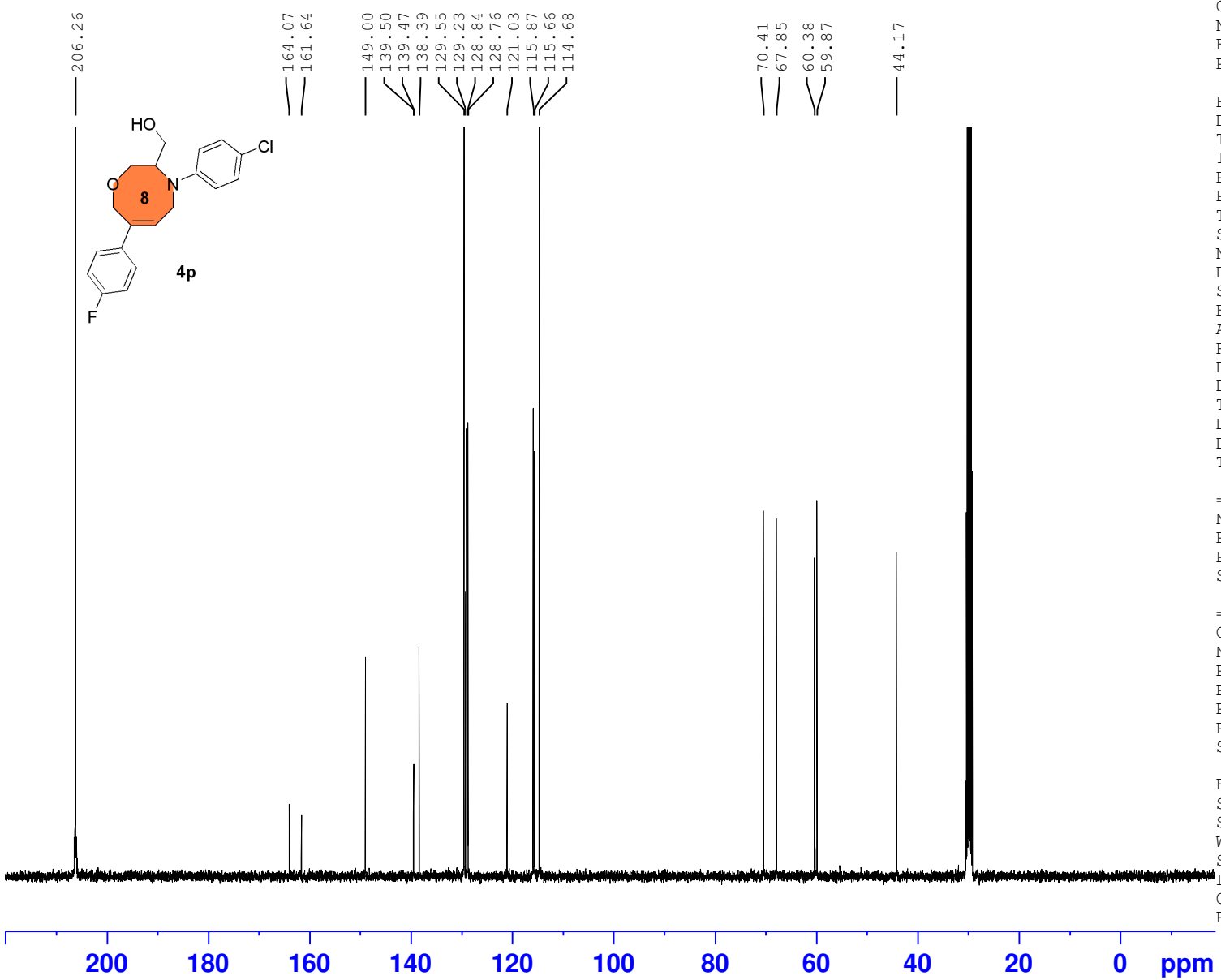

```
Current Data Parameters
NAME         230407-400M
EXPNO                 28
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time                8.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 68.24
DW                60.800 usec
DE                 6.50 usec
TE                289.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1        14.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900121 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

LJX-3-51




```
Current Data Parameters
NAME         230407-400M
EXPNO                 29
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time                8.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE                290.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

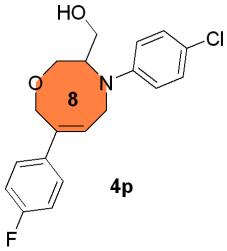
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6277800 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR

ljx-3-51



4p


```
Current Data Parameters
NAME      20230725-300M
EXPNO               406
PROCNO                1

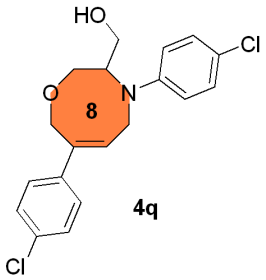
F2 - Acquisition Parameters
Date_          20230725
Time              11.57
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG      zgfhigqn.2
TD              131072
SOLVENT           CDCl3
NS                   20
DS                    4
SWH           66964.289 Hz
FIDRES         0.510897 Hz
AQ            0.9786710 sec
RG                  203
DW                7.467 usec
DE                6.50 usec
TE                298.3 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        282.3761148 MHz
NUC1                19F
P1                14.50 usec
PLW1        10.39999962 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.17284000 W

F2 - Processing parameters
SI                  65536
SF          282.4043552 MHz
WDW                    EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

LJX-3-50



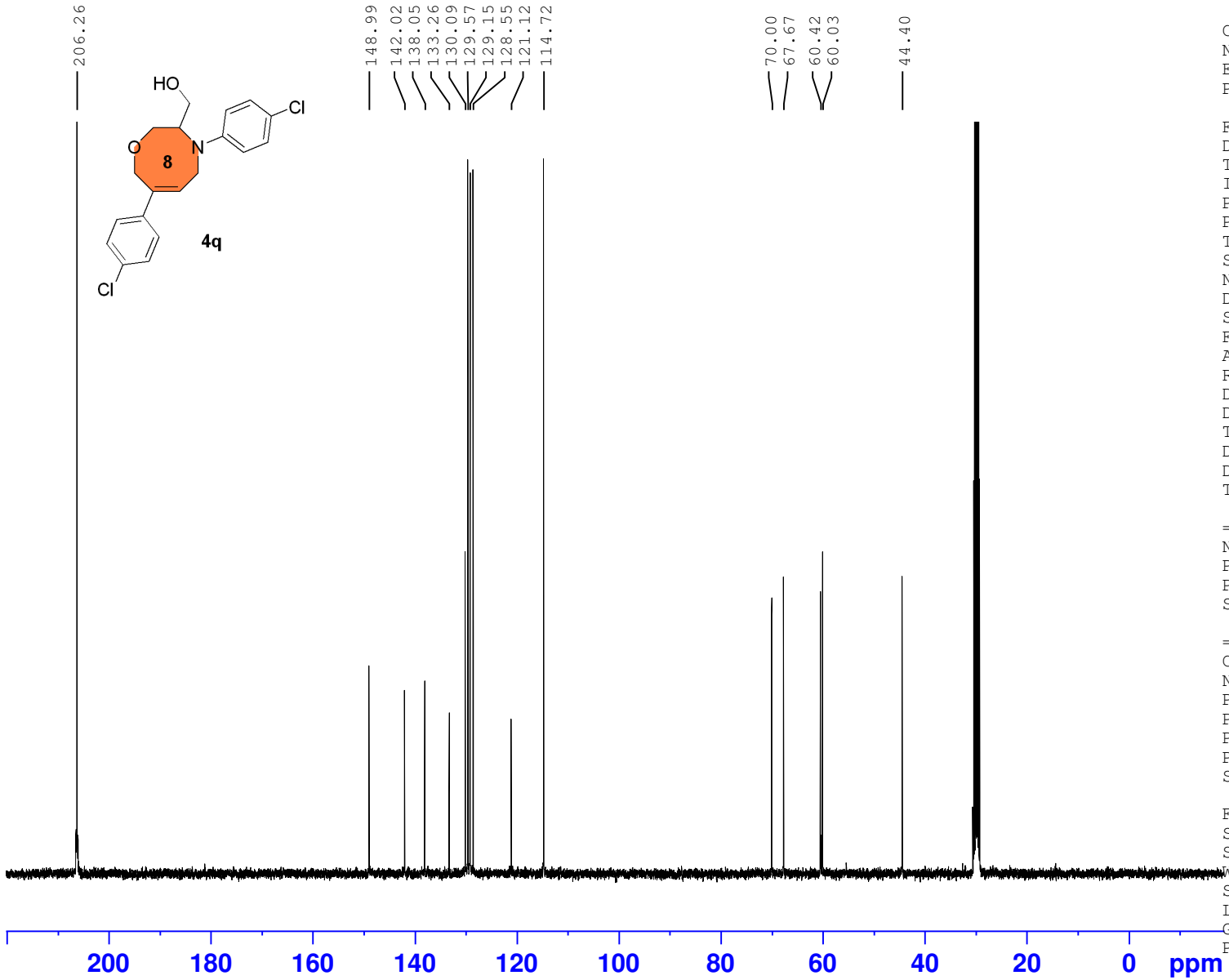

```
Current Data Parameters
NAME         230407-400M
EXPNO                 26
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time                7.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 75.43
DW                60.800 usec
DE                 6.50 usec
TE                 289.6 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900123 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

LJX-3-50



Current Data Parameters
NAME 230407-400M
EXPNO 27
PROCNO 1

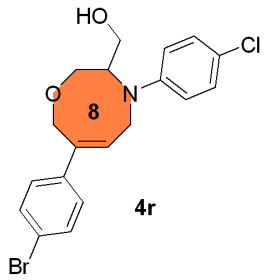
F2 - Acquisition Parameters
Date_ 20230408
Time 7.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 193.13
DW 20.800 usec
DE 6.50 usec
TE 290.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PLW1 53.00000000 W
SFO1 100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.37246999 W
PLW13 0.30170000 W
SFO2 400.1916008 MHz

F2 - Processing parameters
SI 32768
SF 100.6277786 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

LJX-3-68



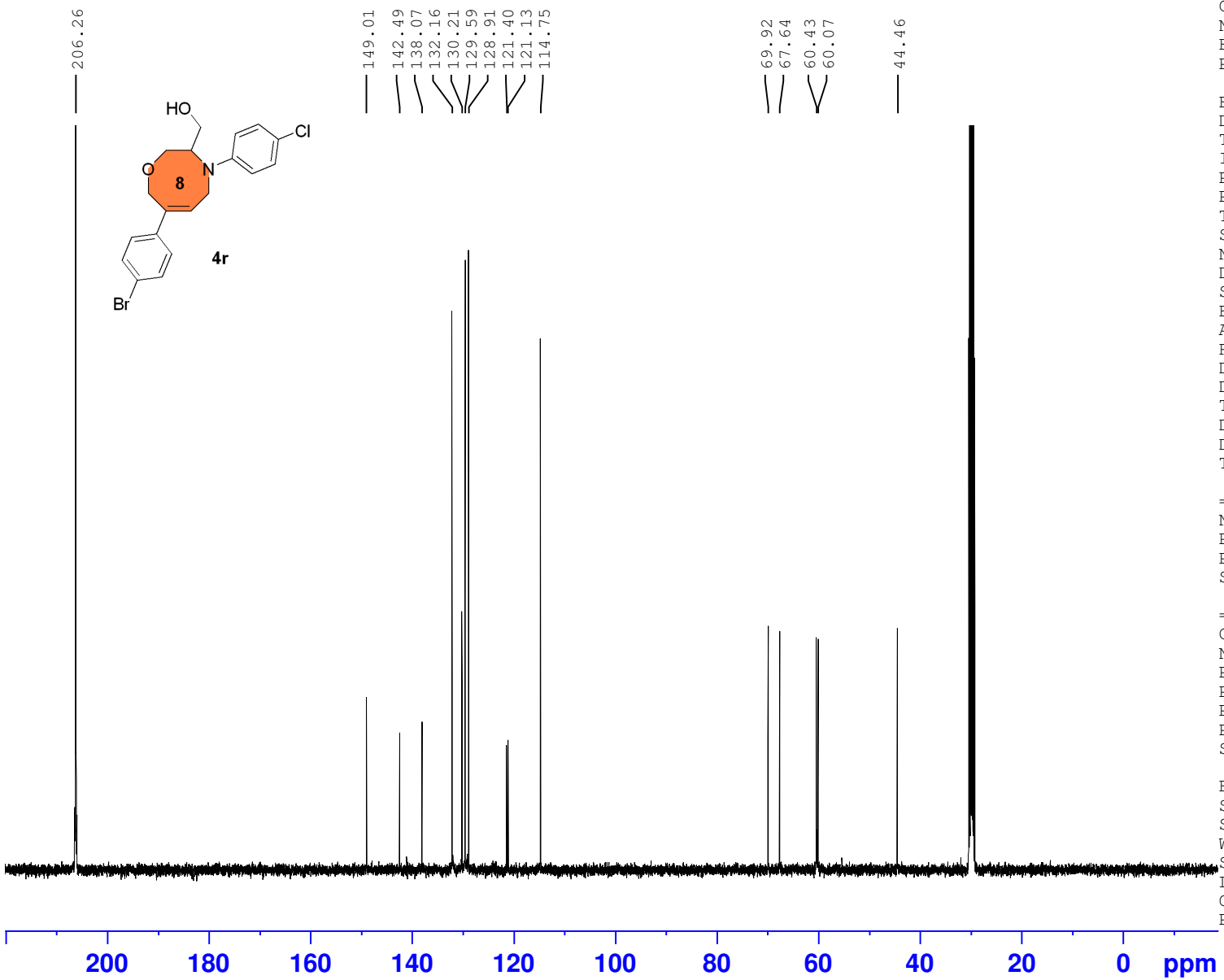

```
Current Data Parameters
NAME       2023-5-20-400
EXPNO                 25
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230519
Time               23.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    10
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                287.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

LJX-3-68




```
Current Data Parameters
NAME      2023-5-20-400
EXPNO                26
PROCNO                1

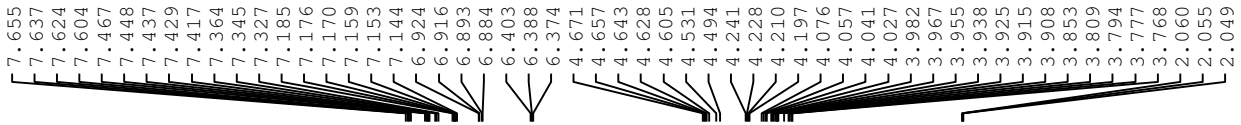
F2 - Acquisition Parameters
Date_          20230520
Time               0.32
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                 1000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               288.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

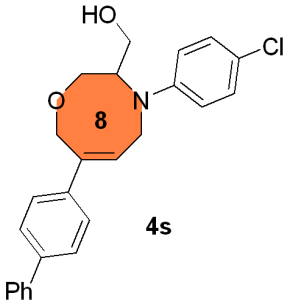
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

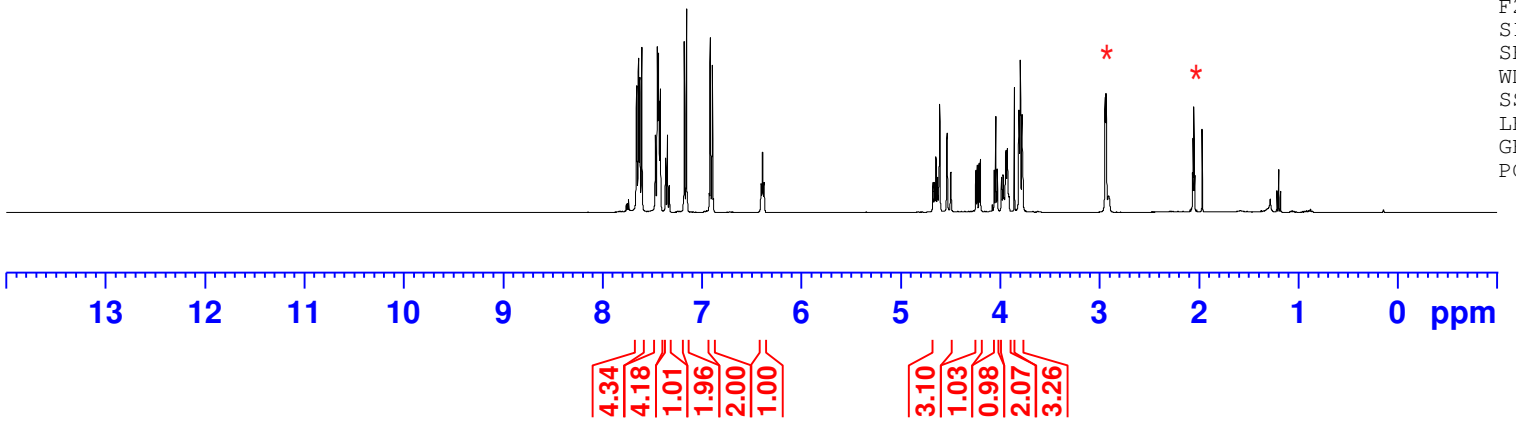
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF           100.6277763 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-5-16







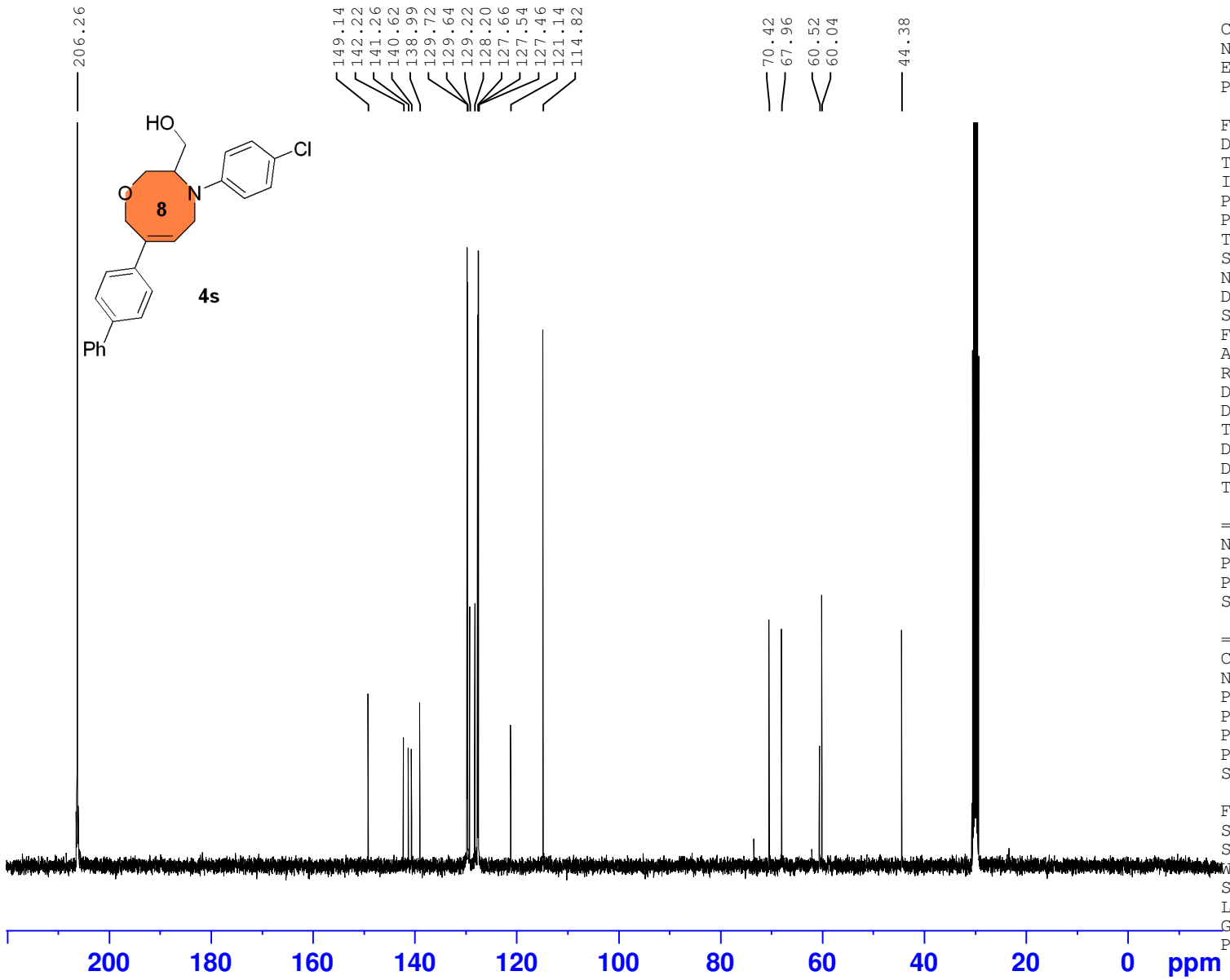

```
Current Data Parameters
NAME       20231028-400M
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20231027
Time               21.46
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 61.19
DW                60.800 usec
DE                 6.50 usec
TE                292.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900112 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

ljx-3-98




```
Current Data Parameters
NAME      20230531-400M
EXPNO               206
PROCNO                1

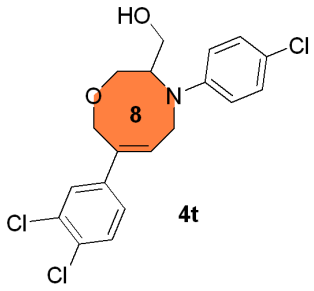
F2 - Acquisition Parameters
Date_          20230531
Time               6.58
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT        Acetone
NS                 400
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631488 sec
RG              193.13
DW              20.800 usec
DE               6.50 usec
TE              294.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

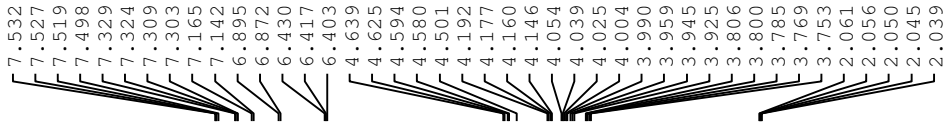
======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

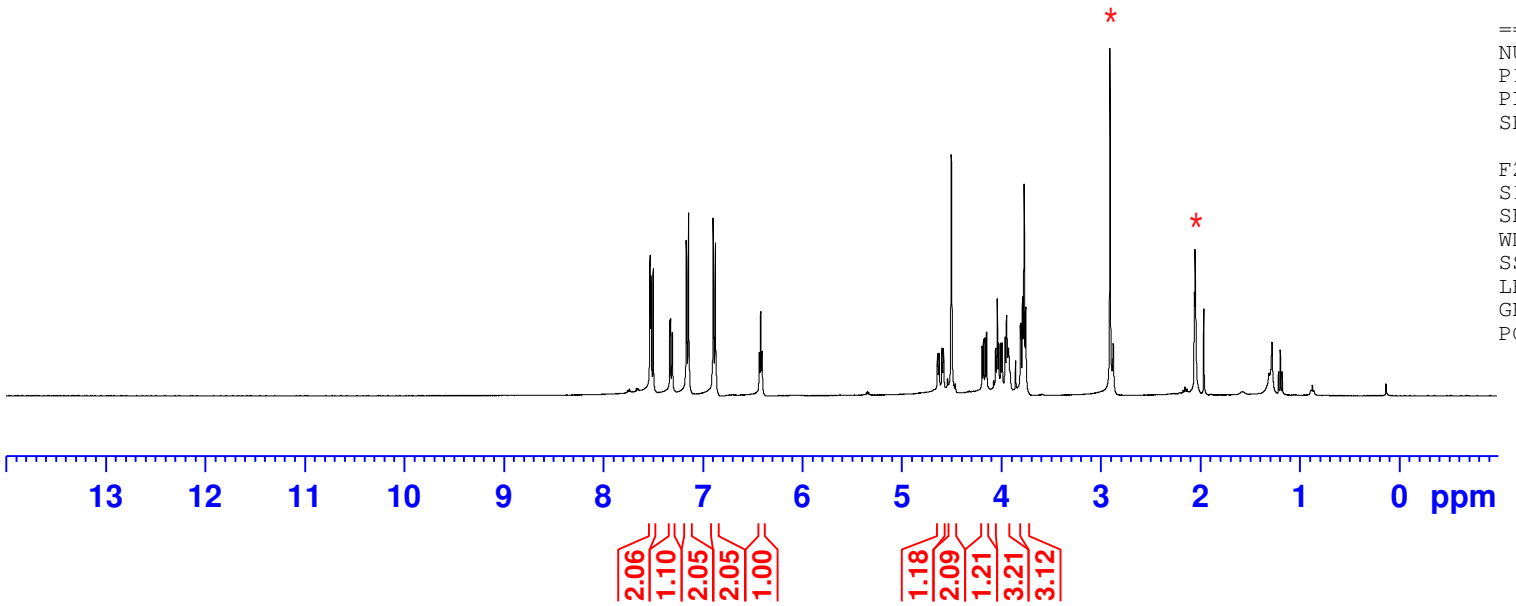
F2 - Processing parameters
SI                 32768
SF         100.6277710 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

ljx-5-17



4t





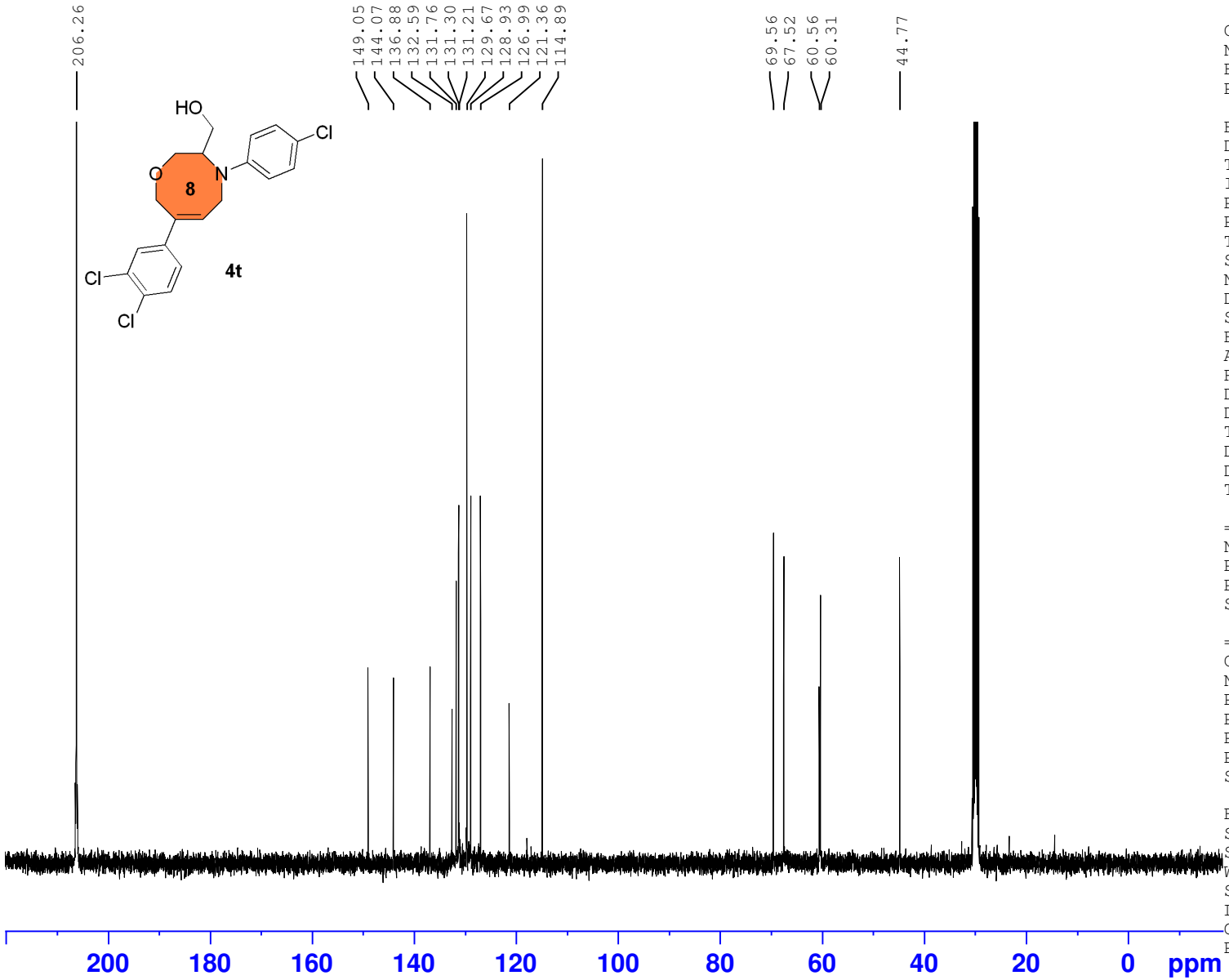
Current Data Parameters
NAME 20231026-400m
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231025
Time 21.57
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 100.49
DW 60.800 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900111 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ljx-3-96



Current Data Parameters
NAME 20230531-400M
EXPNO 202
PROCNO 1

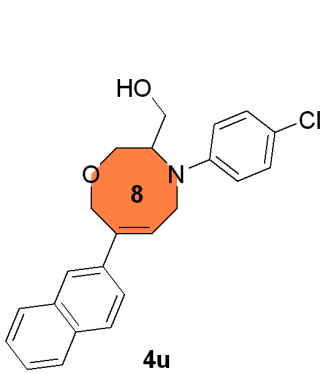
F2 - Acquisition Parameters
Date_ 20230531
Time 6.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 193.13
DW 20.800 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PLW1 53.00000000 W
SFO1 100.6379178 MHz

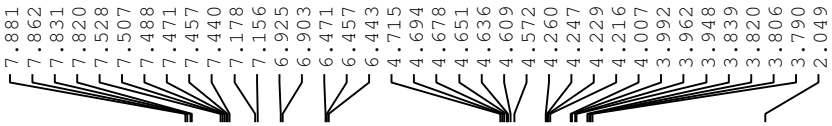
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.37246999 W
PLW13 0.30170000 W
SFO2 400.1916008 MHz

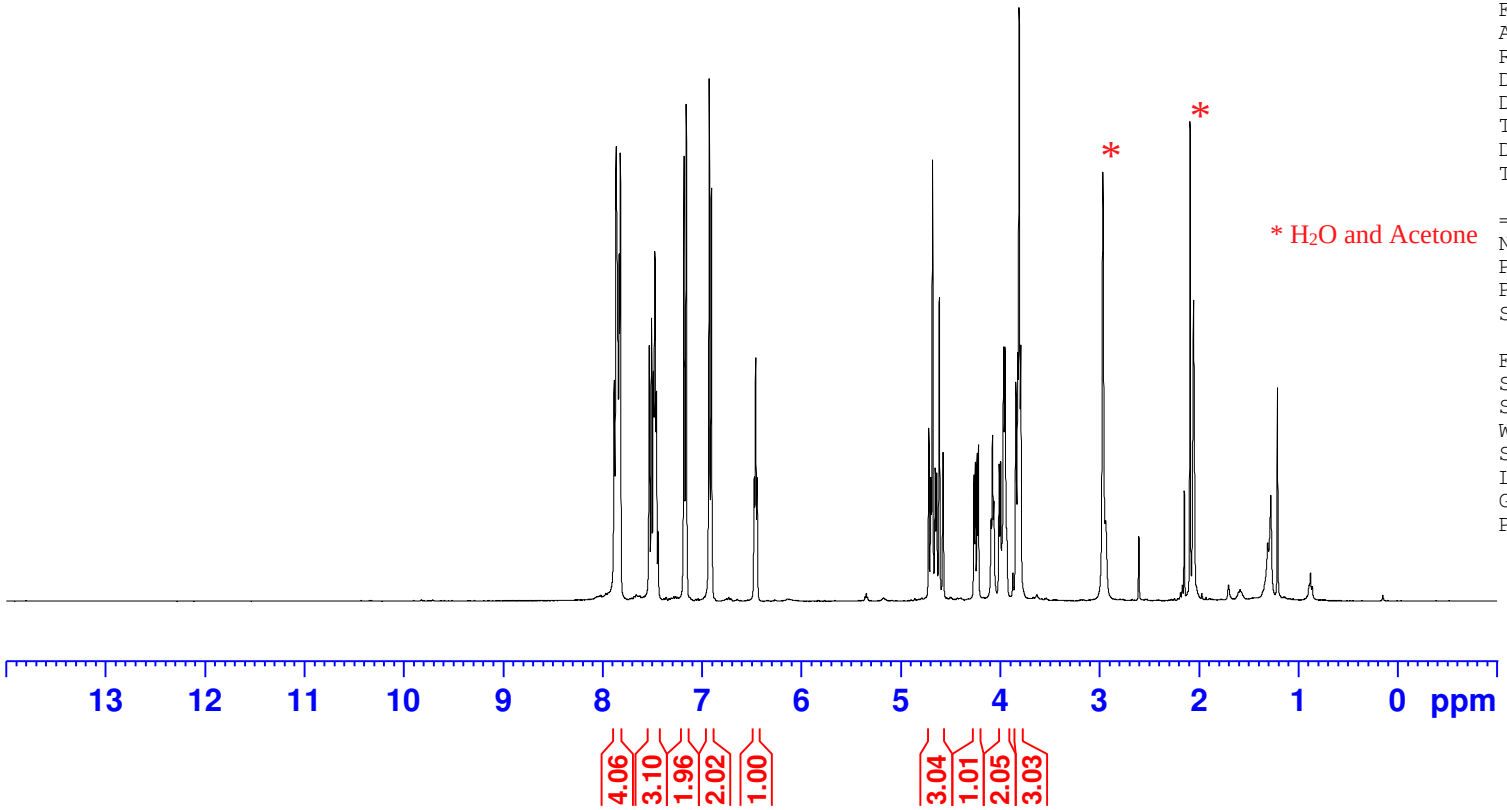
F2 - Processing parameters
SI 32768
SF 100.6277699 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ljx-3-94



4u





* H_2O and Acetone

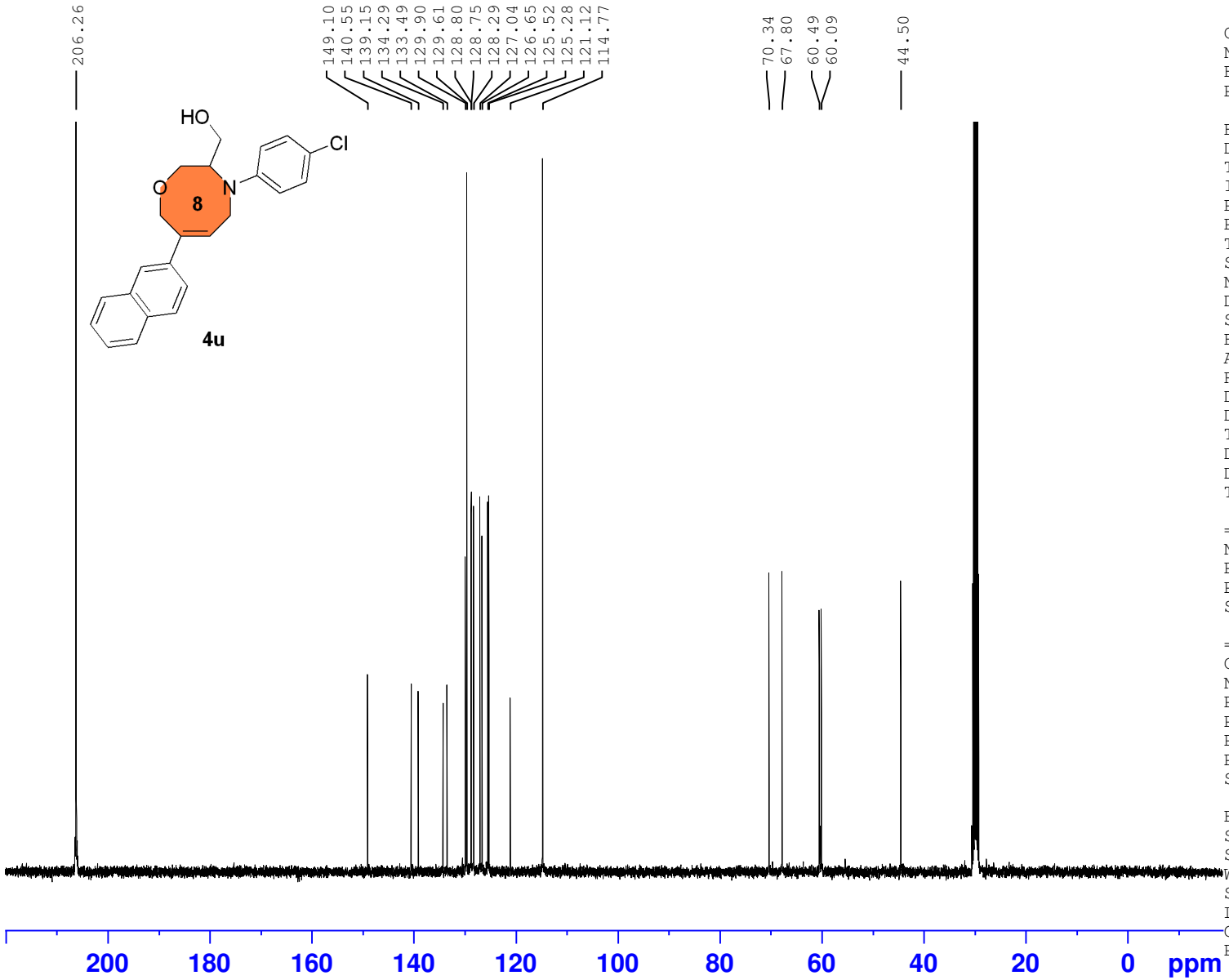
```
Current Data Parameters
NAME       20230531-400M
EXPNO                207
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230531
Time                7.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 68.24
DW                60.800 usec
DE                 6.50 usec
TE                293.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900131 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```

ljx-3-94




```
Current Data Parameters
NAME       20230531-400M
EXPNO                208
PROCNO                 1

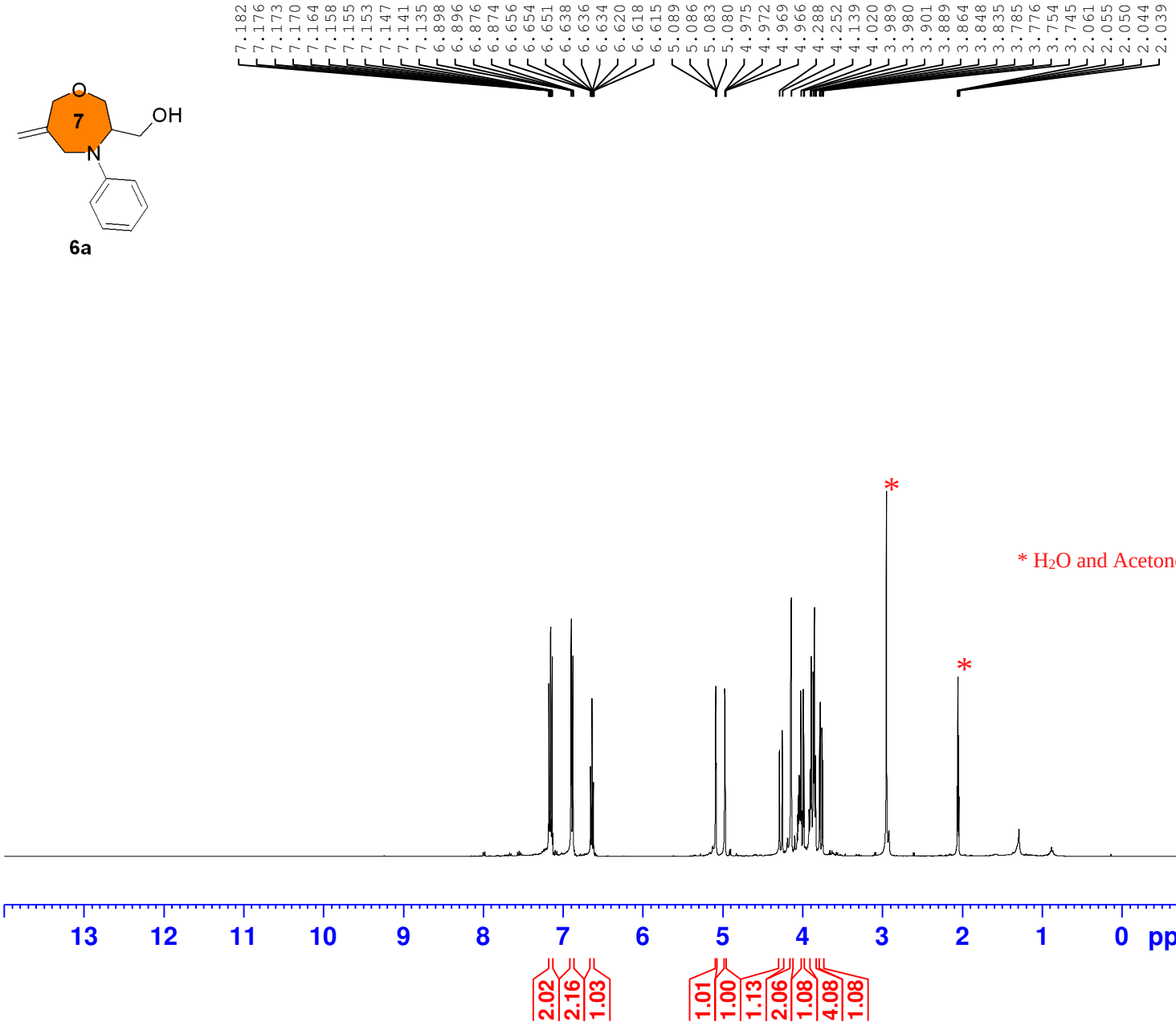
F2 - Acquisition Parameters
Date_           20230531
Time                7.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                   400
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                193.13
DW                20.800 usec
DE                 6.50 usec
TE                294.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6277767 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

ljx-2-45



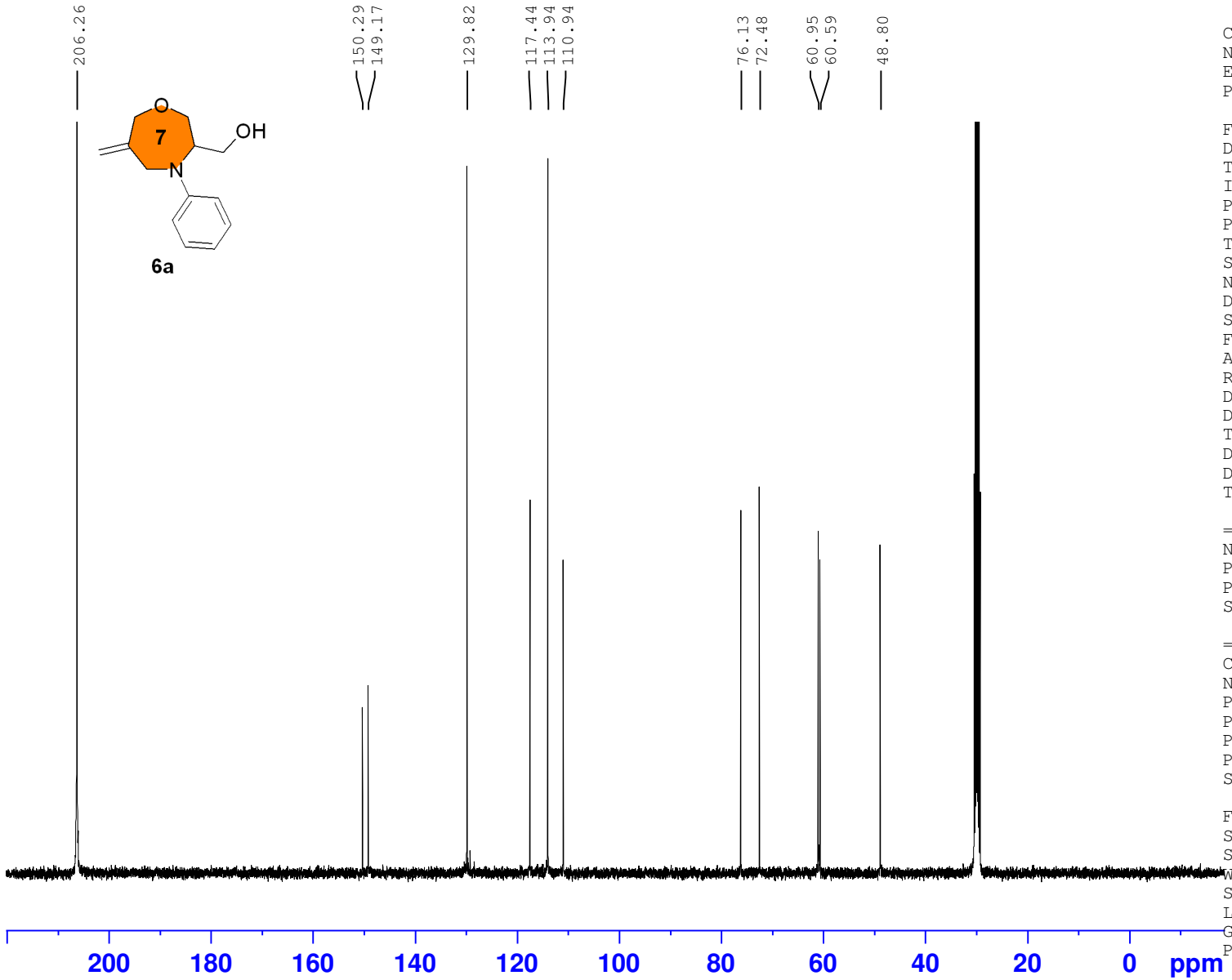

```
Current Data Parameters
NAME        20230610-400
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230609
Time               23.04
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 61.19
DW                60.800 usec
DE                 6.50 usec
TE                291.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900111 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

ljx-2-4-5




```
Current Data Parameters
NAME      20230614-400M
EXPNO                47
PROCNO                1

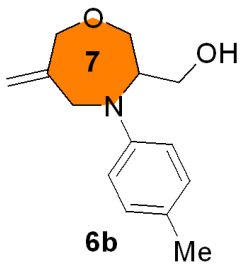
F2 - Acquisition Parameters
Date_          20230614
Time               6.57
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 44.2
DW               20.800 usec
DE                6.50 usec
TE               293.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

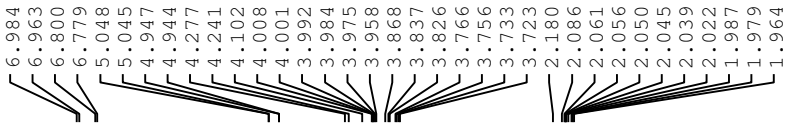
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.80 usec
PLW1        47.40000153 W
SFO1       100.6379178 MHz

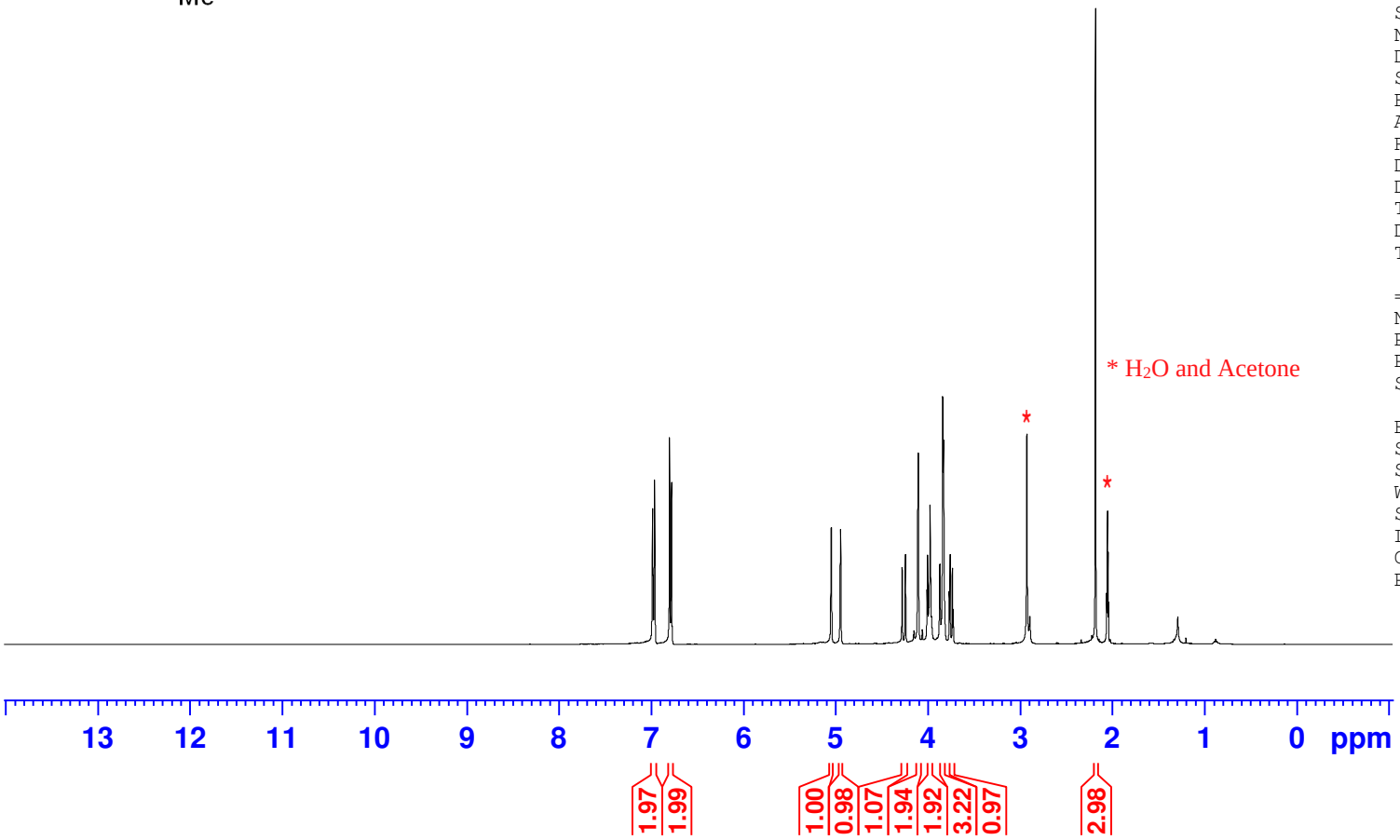
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        23.00000000 W
PLW12        0.30712000 W
PLW13        0.24877000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6277780 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-5-3







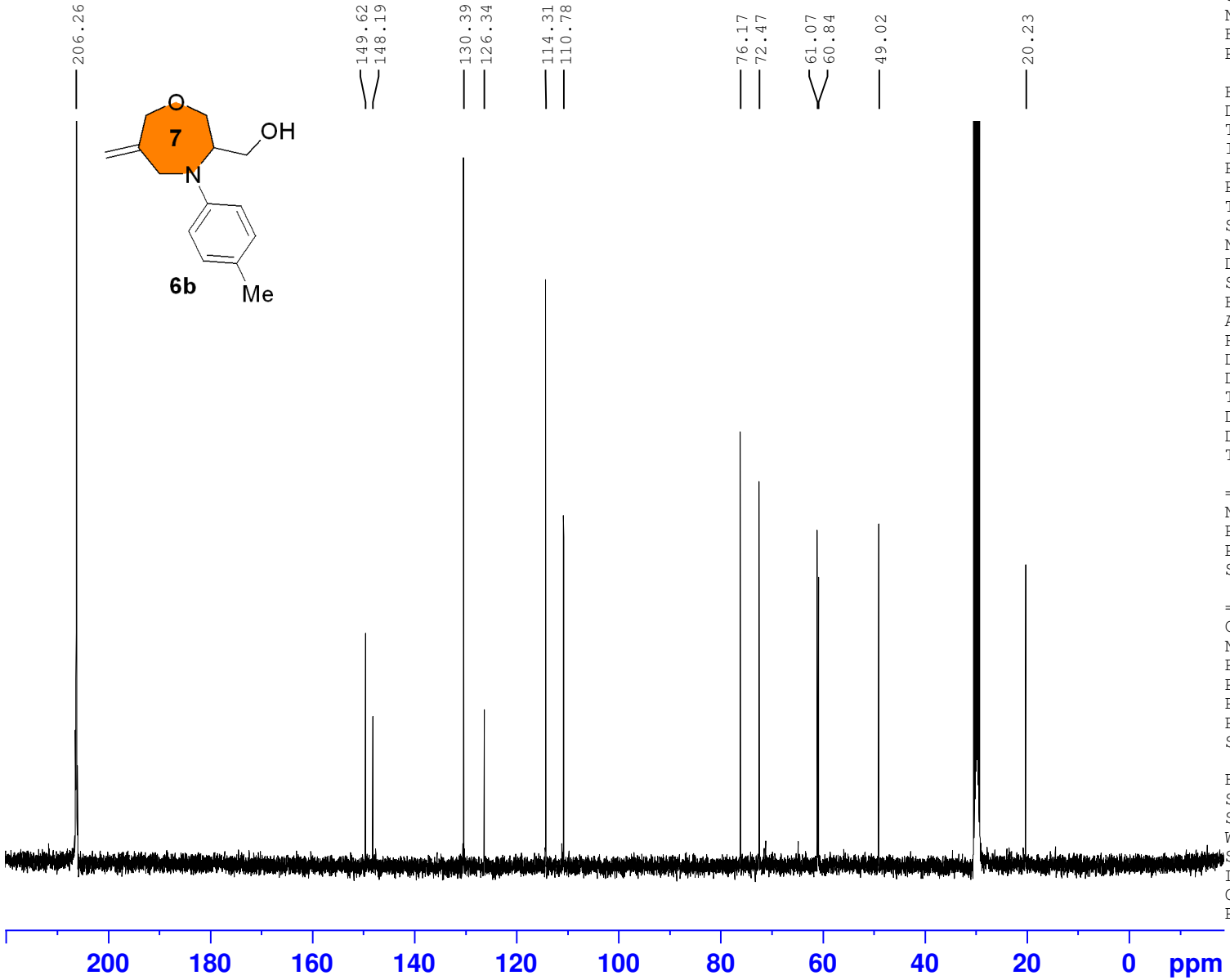
Current Data Parameters
NAME 2023-9-25-400
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230924
Time 22.24
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 10
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 75.43
DW 60.800 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LJX-3-54




```
Current Data Parameters
NAME                  72-1
EXPNO                   72
PROCNO                   1

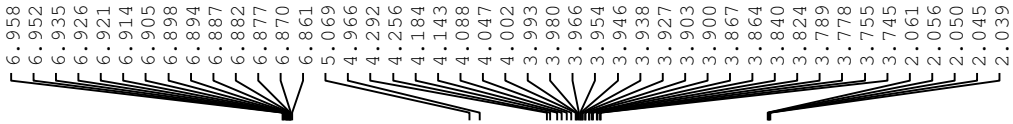
F2 - Acquisition Parameters
Date_            20230409
Time                 6.15
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT           Acetone
NS                   1000
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                 193.13
DW                20.800 usec
DE                 6.50 usec
TE                 289.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

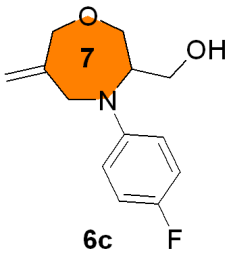
======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                   32768
SF          100.6277706 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                   1.40
```

ljx-3-63



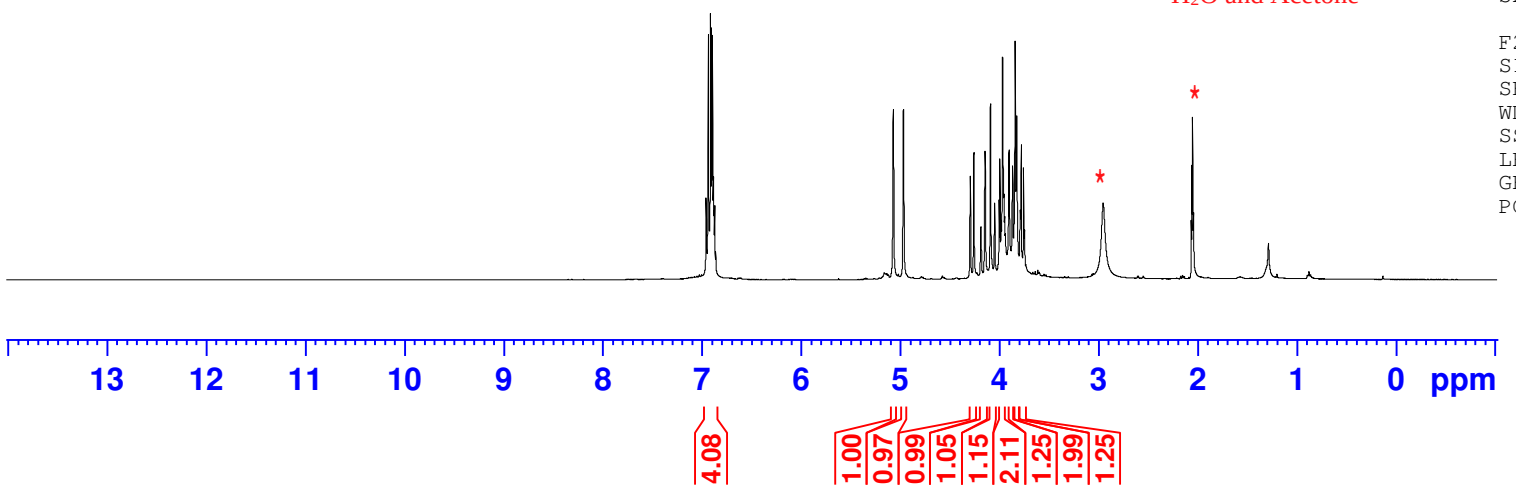


Current Data Parameters
NAME 2023-9-27-400
EXPNO 7
PROCNO 1

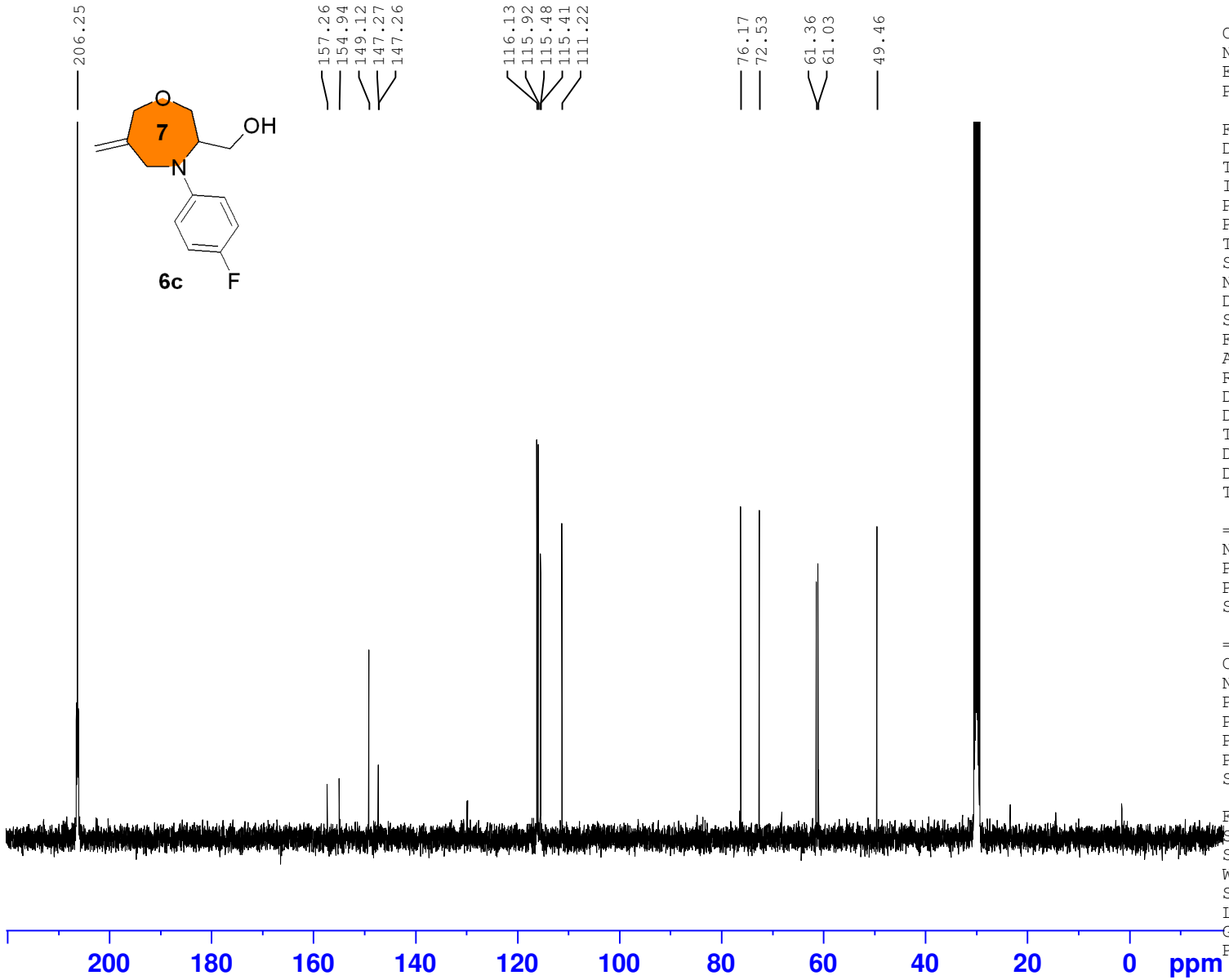
F2 - Acquisition Parameters
Date_ 20230926
Time 23.21
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 75.43
DW 60.800 usec
DE 6.50 usec
TE 291.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



LJX-3-63




```
Current Data Parameters
NAME                 ljxNMR
EXPNO                    53
PROCNO                    1

F2 - Acquisition Parameters
Date_              20230422
Time                   6.18
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                      900
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                   193.13
DW                  20.800 usec
DE                   6.50 usec
TE                   290.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

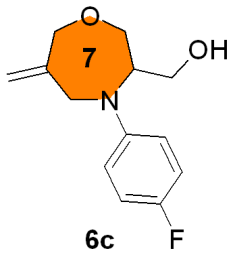
======== CHANNEL f1 ========
NUC1                    13C
P1                   12.00 usec
PLW1           53.00000000 W
SFO1          100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           14.00000000 W
PLW12           0.37246999 W
PLW13           0.30170000 W
SFO2          400.1916008 MHz

F2 - Processing parameters
SI                    32768
SF           100.6277688 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

^{19}F NMR

LJX-3-63



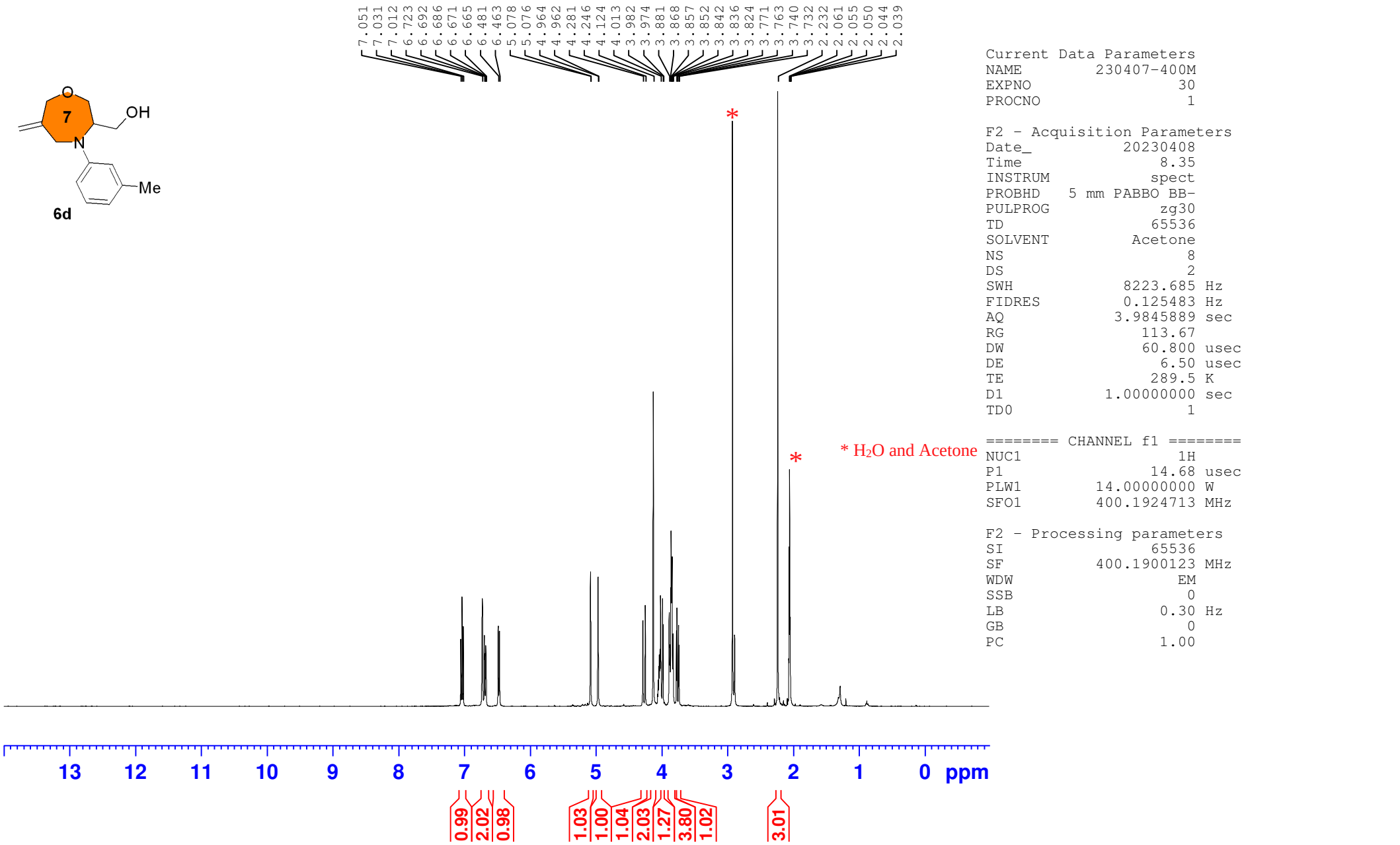
6c


```
Current Data Parameters
NAME                  0927HH
EXPNO                      6
PROCNO                     1

F2 - Acquisition Parameters
Date_              20230927
Time                  16.32 h
INSTRUM              Avance
PROBHD    Z116098_0833 (
PULPROG                zgig
TD                   131072
SOLVENT             Acetone
NS                        16
DS                         4
SWH              90909.094 Hz
FIDRES            1.387163 Hz
AQ               0.7208960 sec
RG                       101
DW                   5.500 usec
DE                   6.50 usec
TE                   294.5 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                        1
SFO1           376.4607164 MHz
NUC1                    19F
P1                   18.00 usec
PLW1           16.73100090 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           20.73200035 W
PLW12           0.25595000 W

F2 - Processing parameters
SI                     65536
SF             376.4983662 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

LJX-3-53



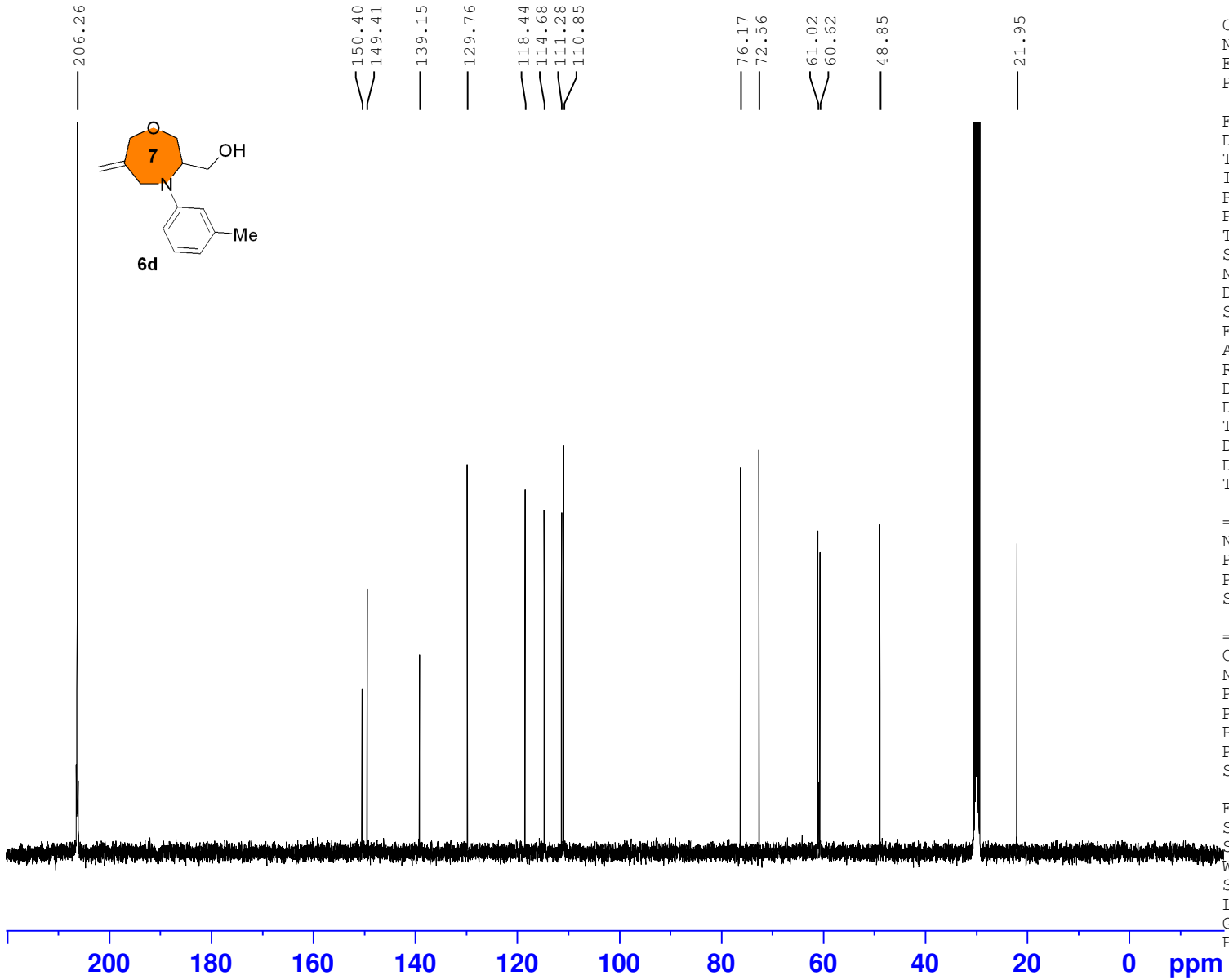

```
Current Data Parameters
NAME         230407-400M
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230408
Time                8.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                113.67
DW                60.800 usec
DE                 6.50 usec
TE                289.5 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900123 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

LJX-3-53




```
Current Data Parameters
NAME         230407-400M
EXPNO                 31
PROCNO                 1

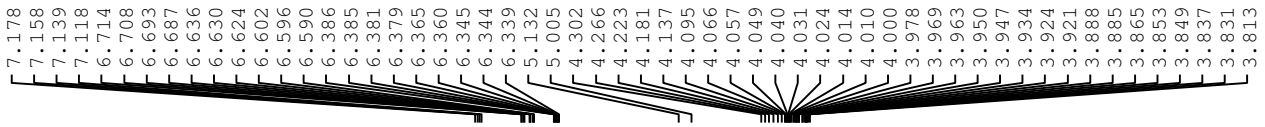
F2 - Acquisition Parameters
Date_           20230408
Time                9.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               290.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

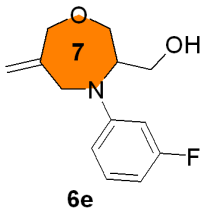
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1        53.00000000 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.37246999 W
PLW13        0.30170000 W
SFO2       400.1916008 MHz

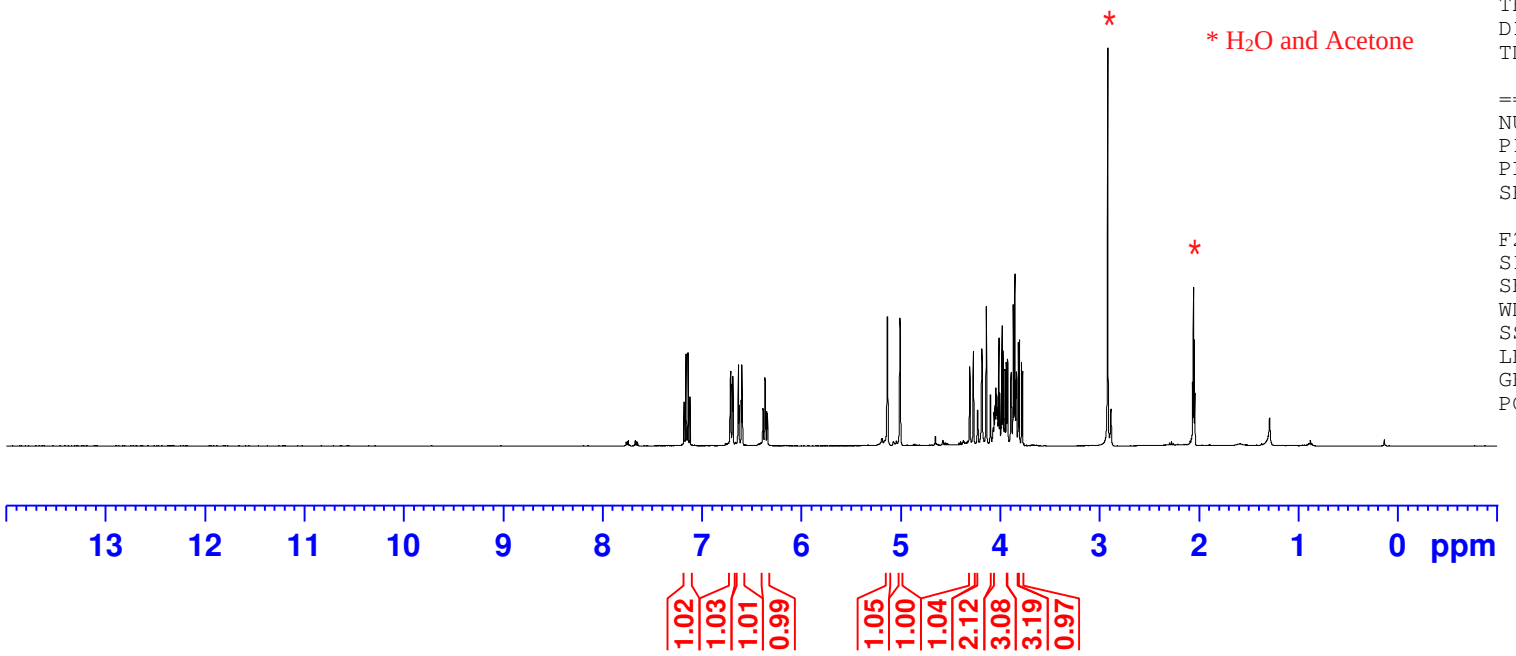
F2 - Processing parameters
SI                32768
SF         100.6277717 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

ljx-5-5





6e



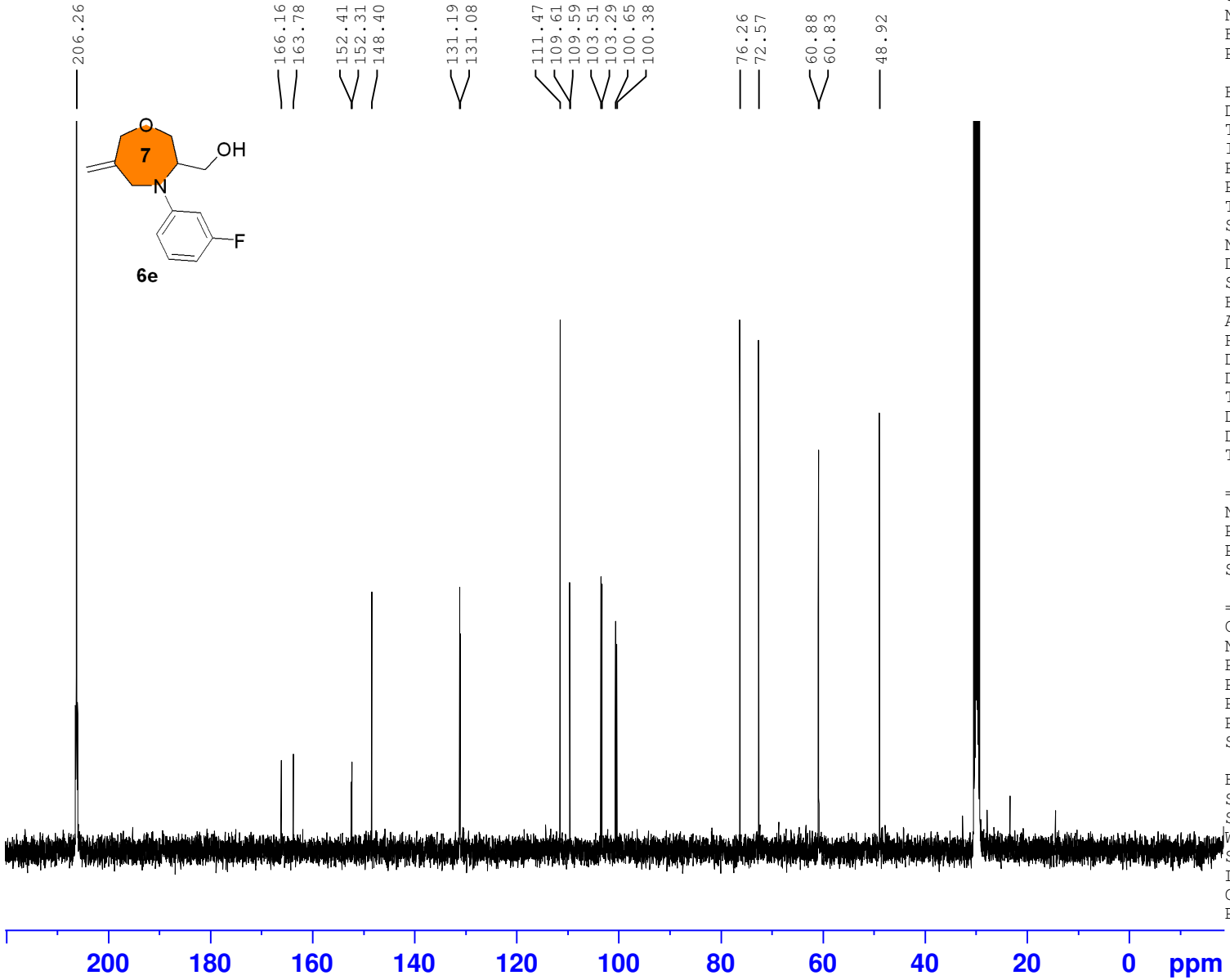
Current Data Parameters
NAME 20231028-400M
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20231027
Time 21.55
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 75.43
DW 60.800 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LJX-3-65




```
Current Data Parameters
NAME                 ljxNMR
EXPNO                    57
PROCNO                    1

F2 - Acquisition Parameters
Date_              20230422
Time                   8.12
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                      900
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                   193.13
DW                  20.800 usec
DE                   6.50 usec
TE                  290.6 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

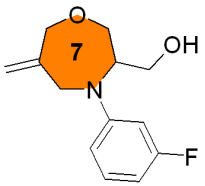
======== CHANNEL f1 ========
NUC1                    13C
P1                   12.00 usec
PLW1           53.00000000 W
SFO1          100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           14.00000000 W
PLW12           0.37246999 W
PLW13           0.30170000 W
SFO2          400.1916008 MHz

F2 - Processing parameters
SI                    32768
SF             100.6277688 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

^{19}F NMR

LJX-3-65



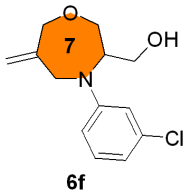
6e


```
Current Data Parameters
NAME                  0927HH
EXPNO                      7
PROCNO                     1

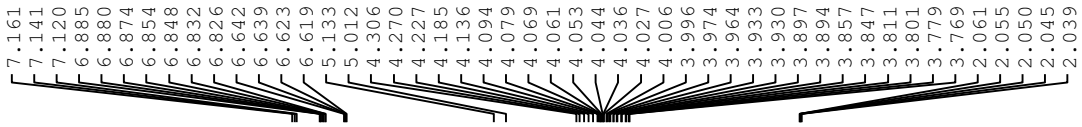
F2 - Acquisition Parameters
Date_               20230927
Time                   16.36 h
INSTRUM               Avance
PROBHD    Z116098_0833 (
PULPROG                 zgig
TD                    131072
SOLVENT              Acetone
NS                         16
DS                          4
SWH                90909.094 Hz
FIDRES              1.387163 Hz
AQ                 0.7208960 sec
RG                       101
DW                     5.500 usec
DE                     6.50 usec
TE                     294.4 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                        1
SFO1             376.4607164 MHz
NUC1                     19F
P1                    18.00 usec
PLW1             16.73100090 W
SFO2             400.1316005 MHz
NUC2                     1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2             20.73200035 W
PLW12             0.25595000 W

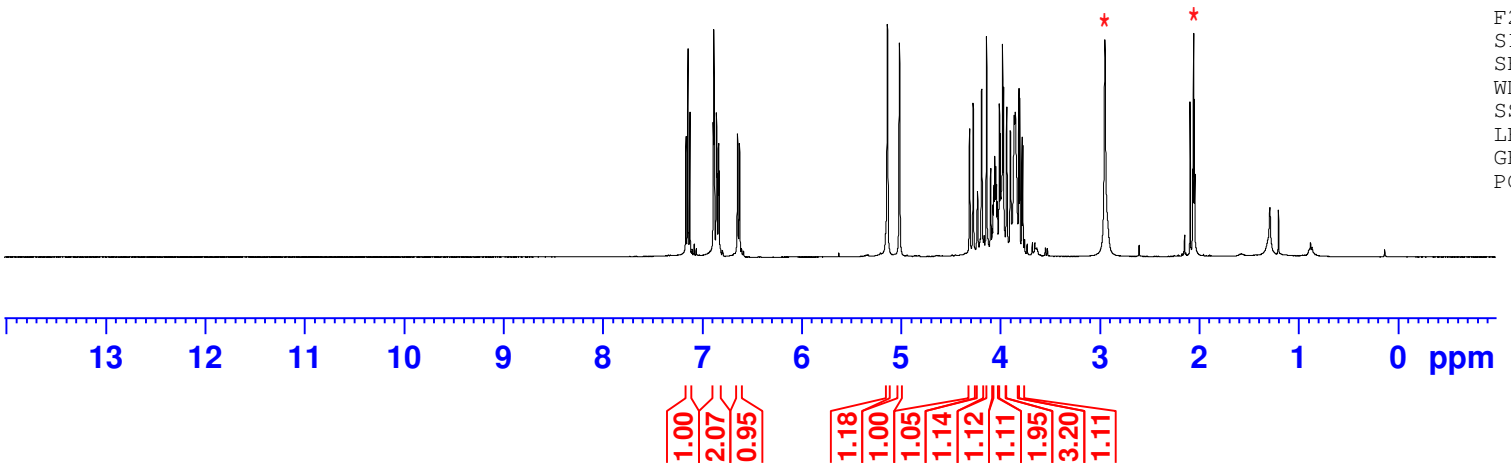
F2 - Processing parameters
SI                       65536
SF               376.4983662 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```

ljx-3-37



6f





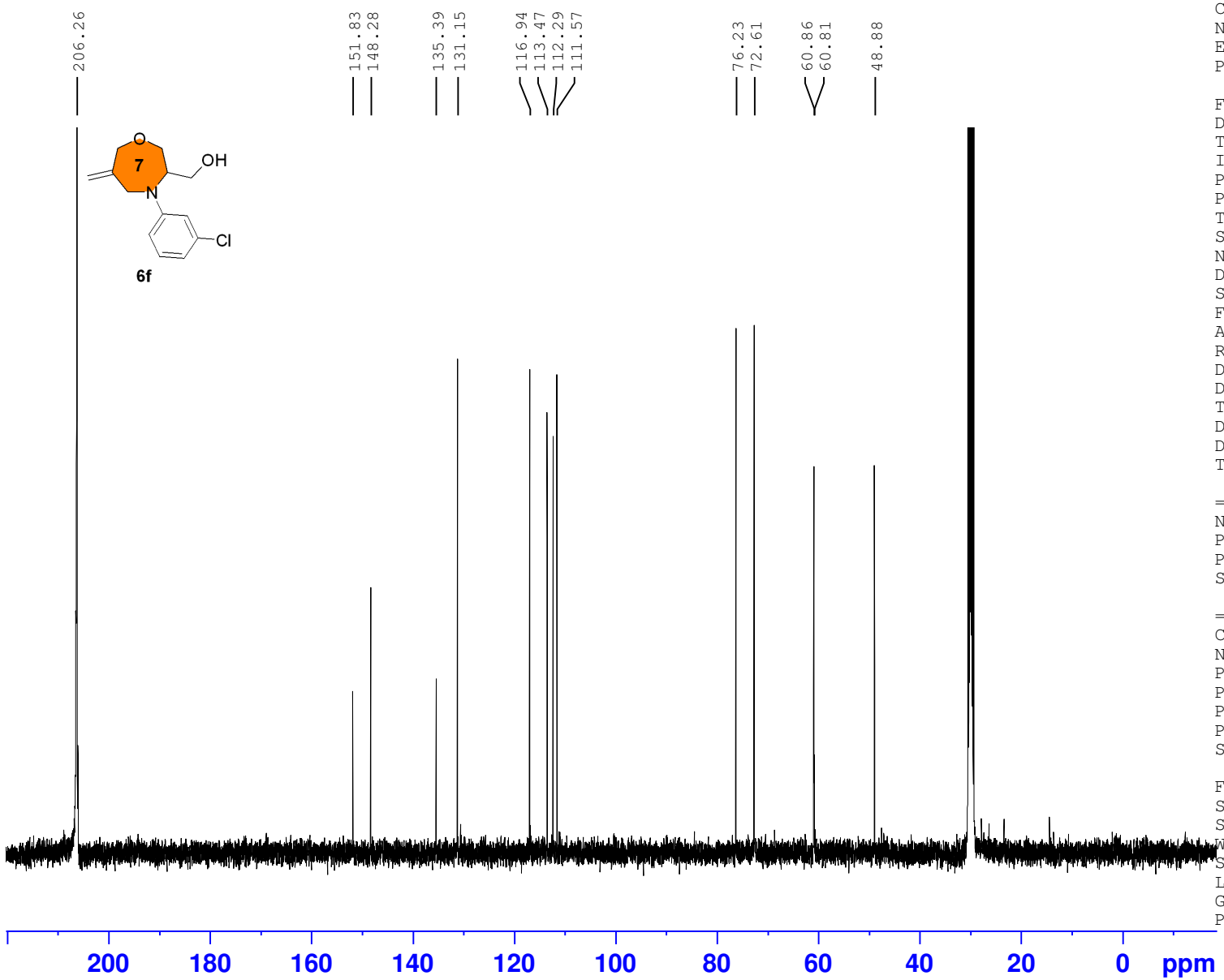
Current Data Parameters
NAME 2023-9-27-400
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230926
Time 23.07
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 75.43
DW 60.800 usec
DE 6.50 usec
TE 291.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LJX-3-37




```
Current Data Parameters
NAME                  2023.0507
EXPNO                        10
PROCNO                        1

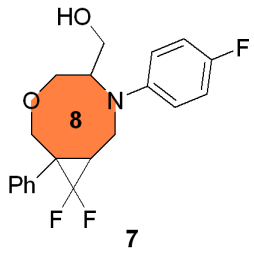
F2 - Acquisition Parameters
Date_                 20230507
Time                     22.07
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                Acetone
NS                          1000
DS                             4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       193.13
DW                       20.800 usec
DE                        6.50 usec
TE                       291.9 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                       12.00 usec
PLW1                53.00000000 W
SFO1               100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PLW2                14.00000000 W
PLW12                0.37246999 W
PLW13                0.30170000 W
SFO2               400.1916008 MHz

F2 - Processing parameters
SI                          32768
SF                 100.6277690 MHz
WDW                            EM
SSB                            0
LB                        1.00 Hz
GB                             0
PC                        1.40
```

ljx-3-100



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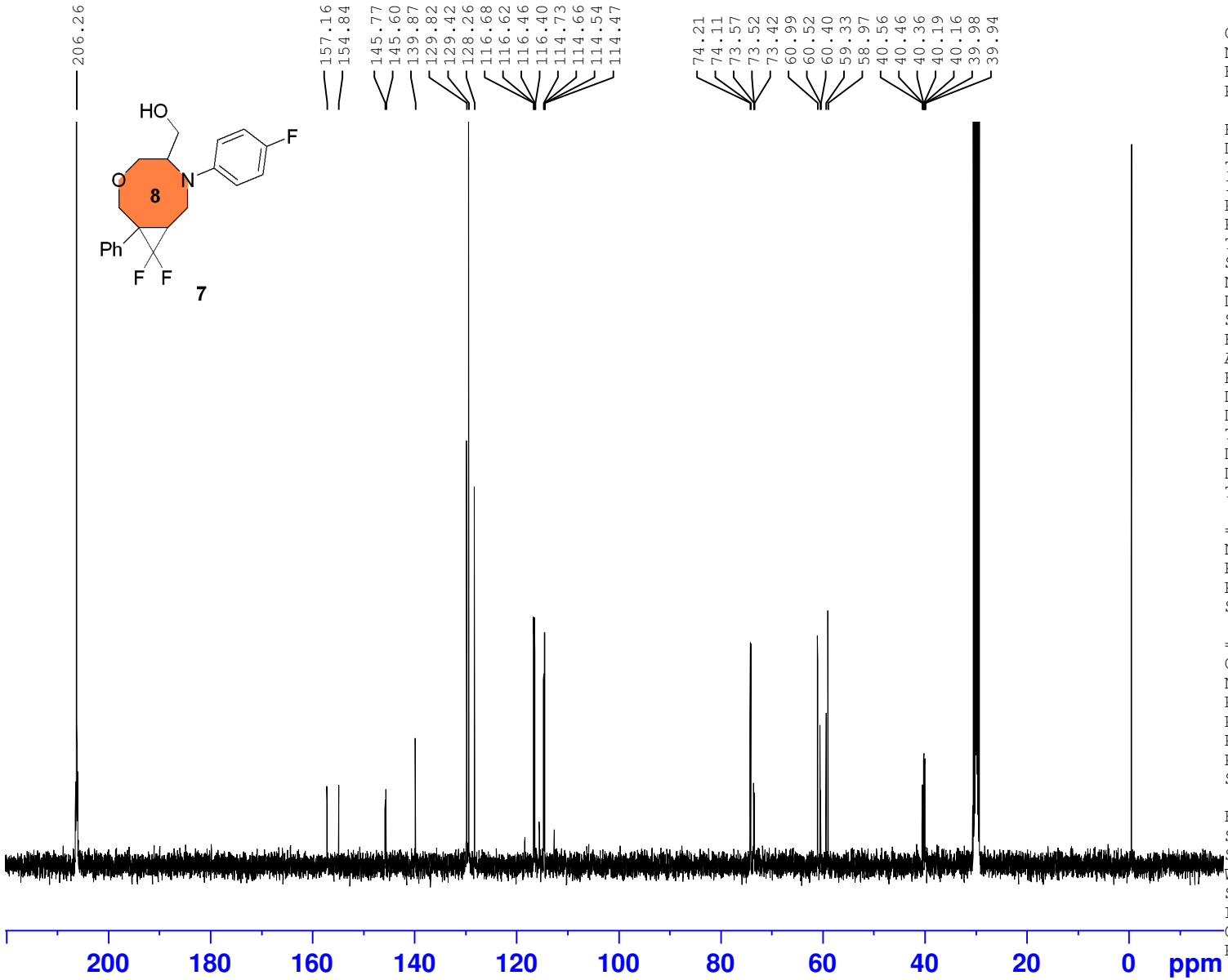
```
Current Data Parameters
NAME       2023-5-27-400
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230527
Time                2.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                292.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1         14.00000000 W
SFO1         400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF           400.1900128 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-3-100




```
Current Data Parameters
NAME      2023-5-27-400
EXPNO                31
PROCNO                1

F2 - Acquisition Parameters
Date_          20230527
Time               2.39
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               293.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

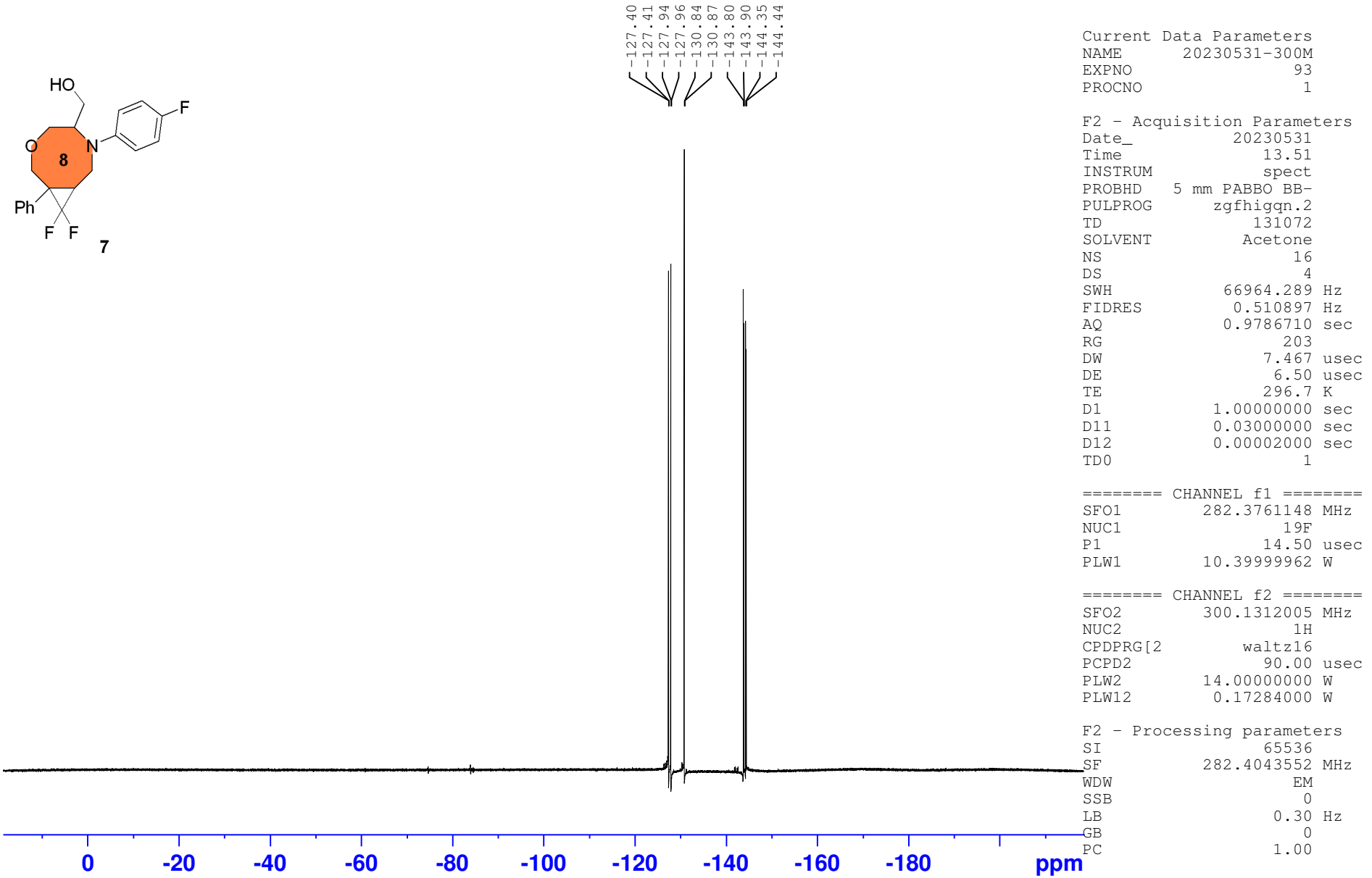
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                32768
SF          100.6277670 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR

ljx-3-100




```
Current Data Parameters
NAME      20230531-300M
EXPNO                93
PROCNO                1

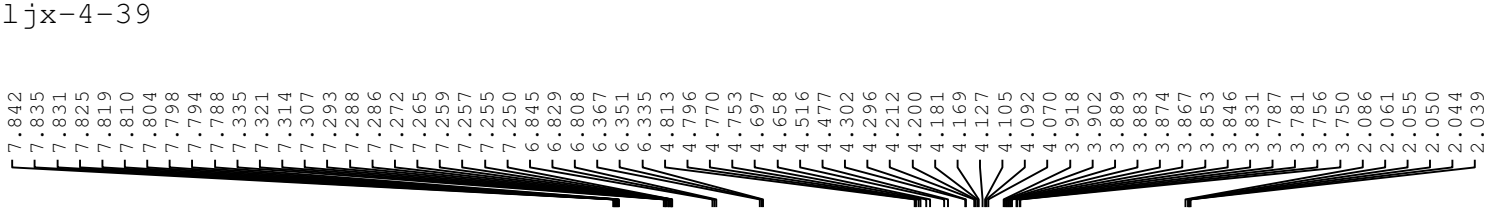
F2 - Acquisition Parameters
Date_          20230531
Time               13.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG      zgfhigqn.2
TD              131072
SOLVENT         Acetone
NS                   16
DS                    4
SWH           66964.289 Hz
FIDRES         0.510897 Hz
AQ            0.9786710 sec
RG                  203
DW                7.467 usec
DE                6.50 usec
TE                296.7 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                   1

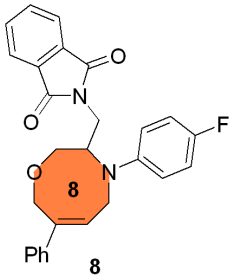
======== CHANNEL f1 ========
SFO1        282.3761148 MHz
NUC1                19F
P1                14.50 usec
PLW1        10.39999962 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.17284000 W

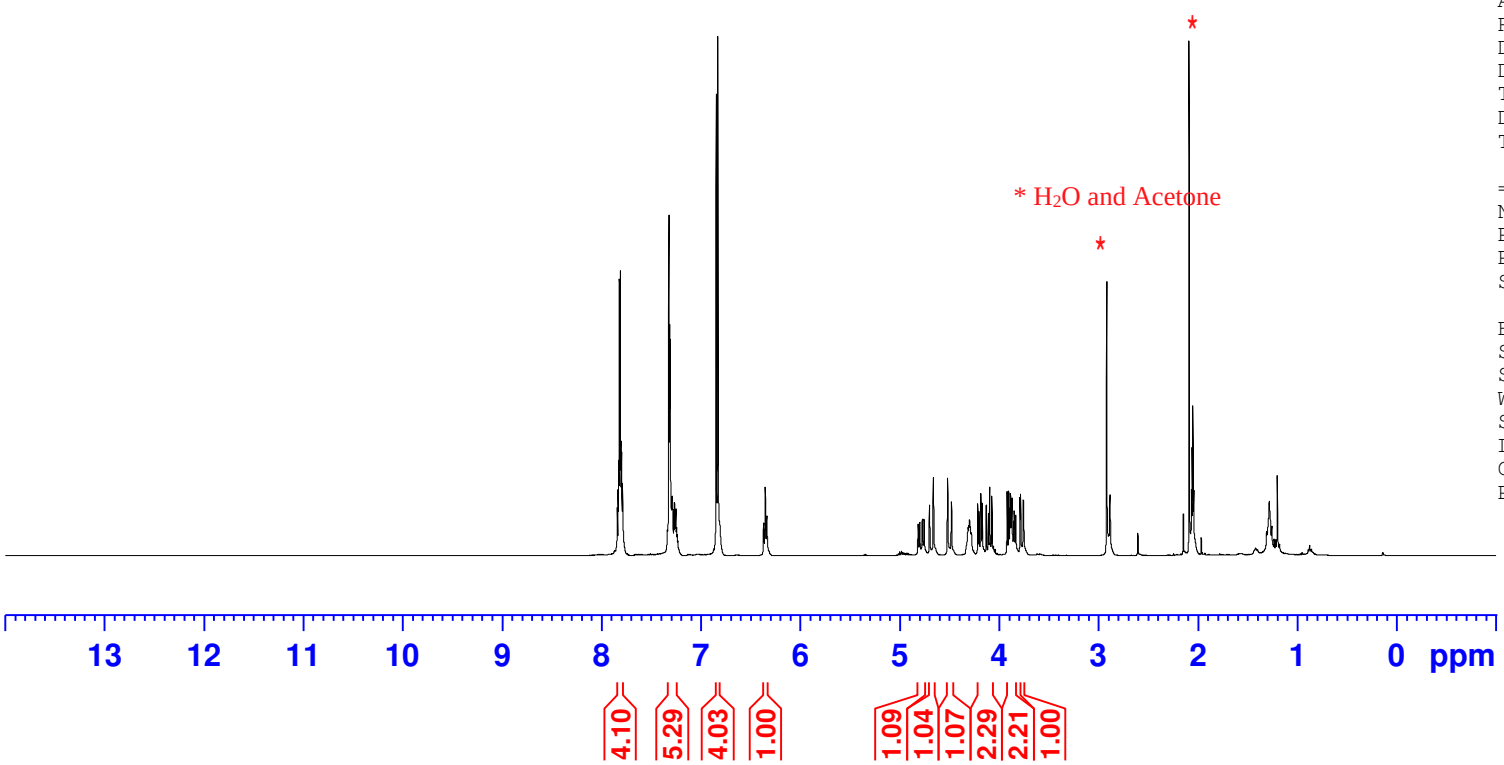
F2 - Processing parameters
SI                 65536
SF          282.4043552 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

ljx-4-39





8



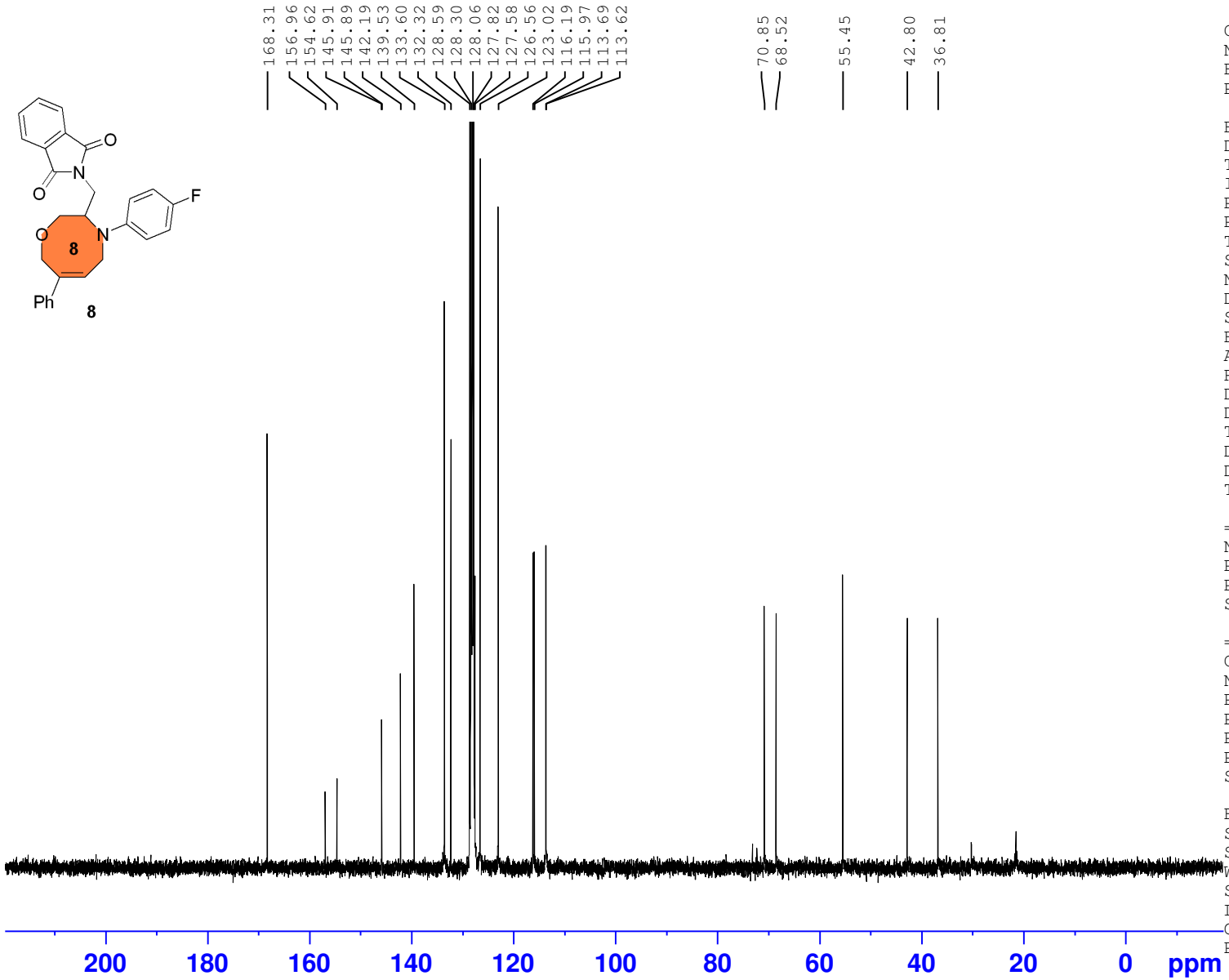
Current Data Parameters
NAME 2023-9-19-400
EXPNO 27
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230918
Time 23.26
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 68.24
DW 60.800 usec
DE 6.50 usec
TE 289.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ljx-4-39




```
Current Data Parameters
NAME      20230701-400m
EXPNO                49
PROCNO                1

F2 - Acquisition Parameters
Date_          20230701
Time               2.58
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT             C6D6
NS                   500
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                37.77
DW               20.800 usec
DE                6.50 usec
TE               292.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

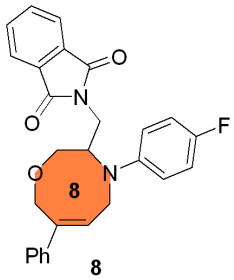
======== CHANNEL f1 ========
NUC1                 13C
P1                  9.80 usec
PLW1        47.40000153 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              90.00 usec
PLW2        23.00000000 W
PLW12        0.30712000 W
PLW13        0.24877000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6278215 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR

LJX-4-39



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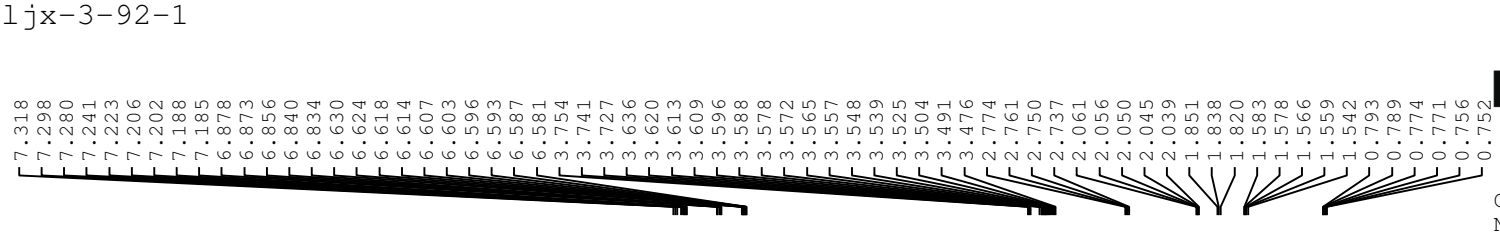
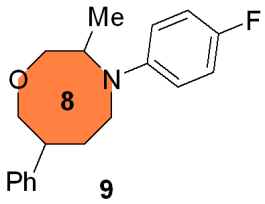
Current Data Parameters
NAME 0927HH
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230927
Time 15.59 h
INSTRUM Avance
PROBHD Z116098_0833 (
PULPROG zgig
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 90909.094 Hz
FIDRES 1.387163 Hz
AQ 0.7208960 sec
RG 101
DW 5.500 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 18.00 usec
PLW1 16.73100090 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 20.73200035 W
PLW12 0.25595000 W

F2 - Processing parameters
SI 65536
SF 376.4983662 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ljx-3-92-1



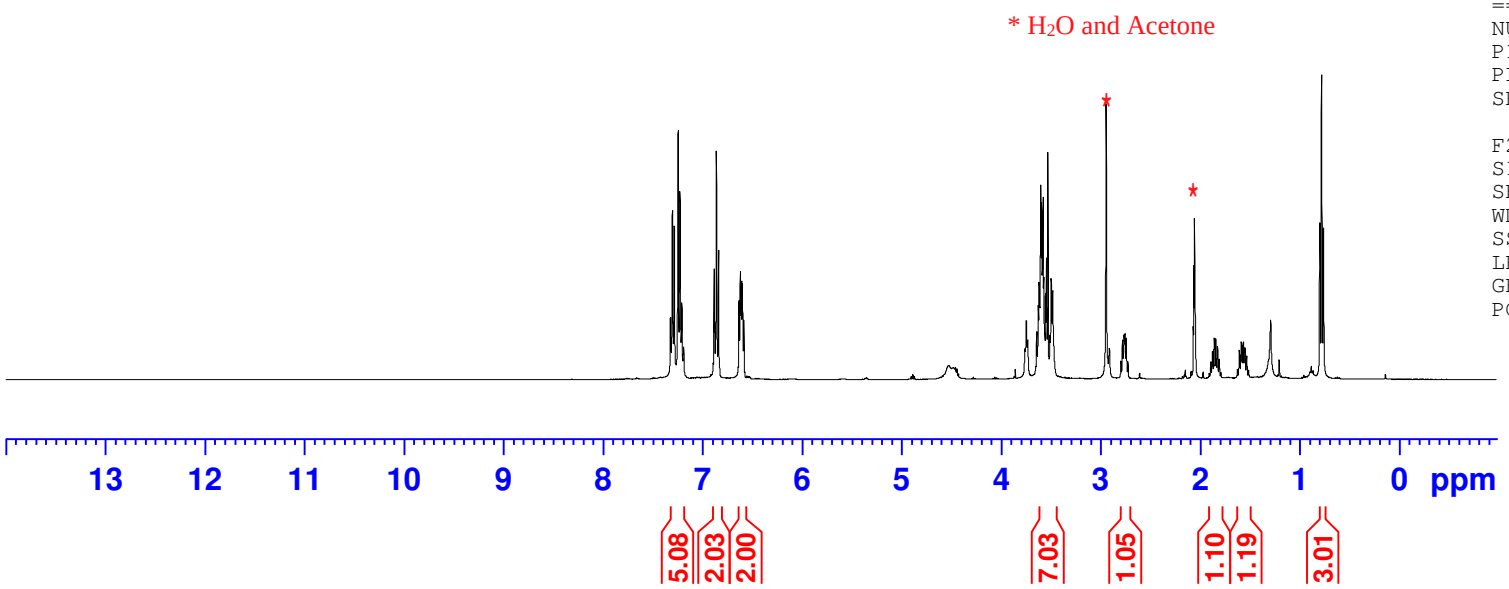


Current Data Parameters
NAME 2023-9-19-400
EXPNO 25
PROCNO 1

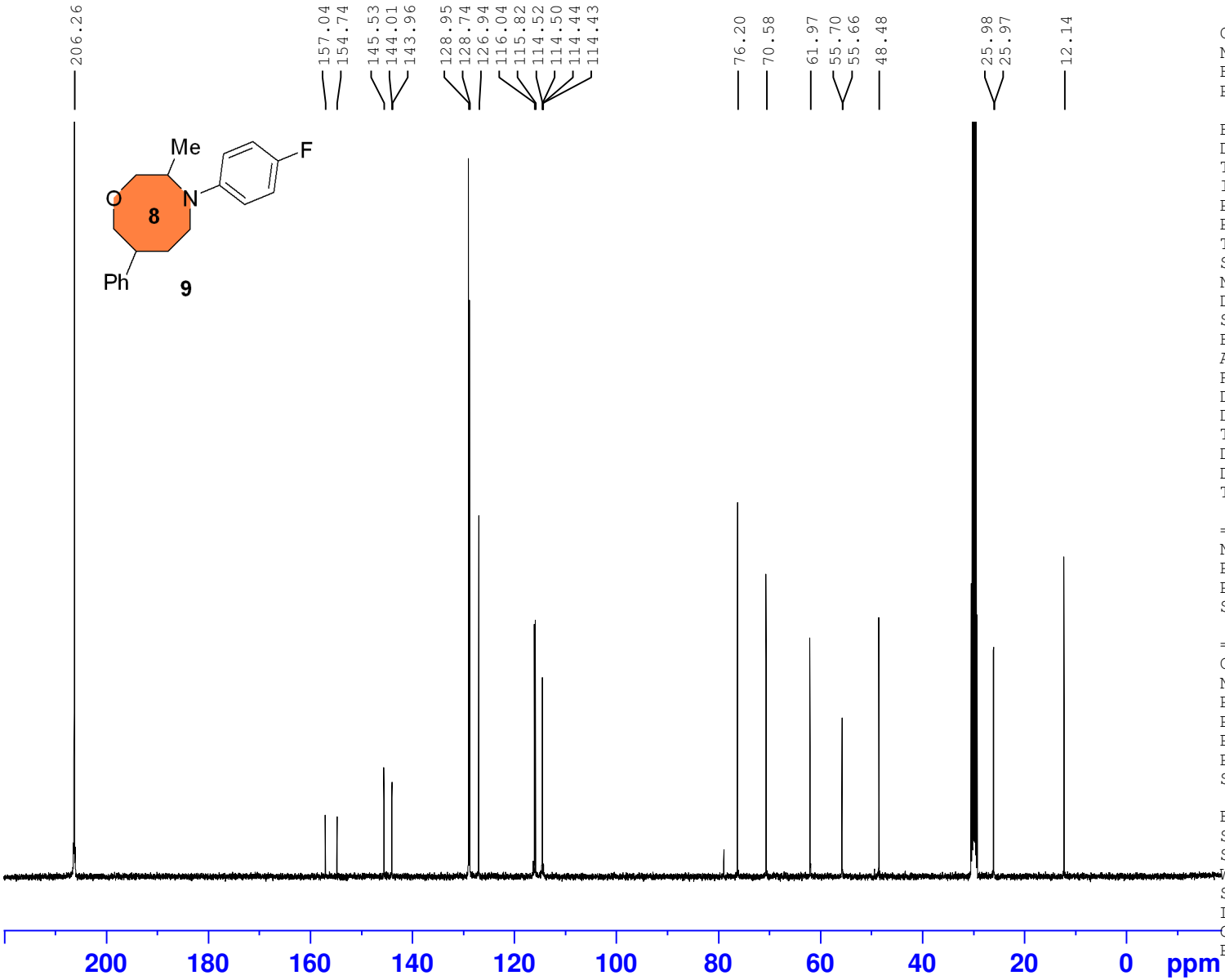
F2 - Acquisition Parameters
Date_ 20230918
Time 23.18
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 61.19
DW 60.800 usec
DE 6.50 usec
TE 289.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.90 usec
PLW1 23.00000000 W
SFO1 400.1924713 MHz

F2 - Processing parameters
SI 65536
SF 400.1900110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



LJX-3-92-1C




```
Current Data Parameters
NAME                  92-93
EXPNO                    92
PROCNO                    1

F2 - Acquisition Parameters
Date_              20230522
Time                   0.34
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                      800
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631488 sec
RG                   193.13
DW                   20.800 usec
DE                    6.50 usec
TE                   289.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

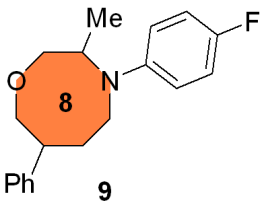
======== CHANNEL f1 ========
NUC1                    13C
P1                   12.00 usec
PLW1           53.00000000 W
SFO1          100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           14.00000000 W
PLW12           0.37246999 W
PLW13           0.30170000 W
SFO2          400.1916008 MHz

F2 - Processing parameters
SI                    32768
SF            100.6277826 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

^{19}F NMR

LJX-3-92-1



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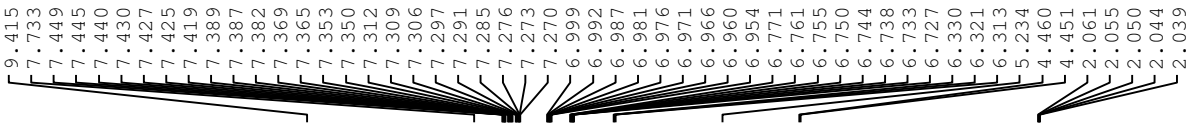
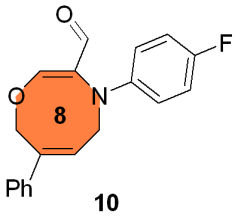
```
Current Data Parameters
NAME                  0927HH
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_              20230927
Time                  16.05 h
INSTRUM              Avance
PROBHD    Z116098_0833 (
PULPROG                zgig
TD                   131072
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              90909.094 Hz
FIDRES            1.387163 Hz
AQ               0.7208960 sec
RG                      101
DW                    5.500 usec
DE                    6.50 usec
TE                    294.5 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           376.4607164 MHz
NUC1                   19F
P1                   18.00 usec
PLW1           16.73100090 W
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           20.73200035 W
PLW12           0.25595000 W

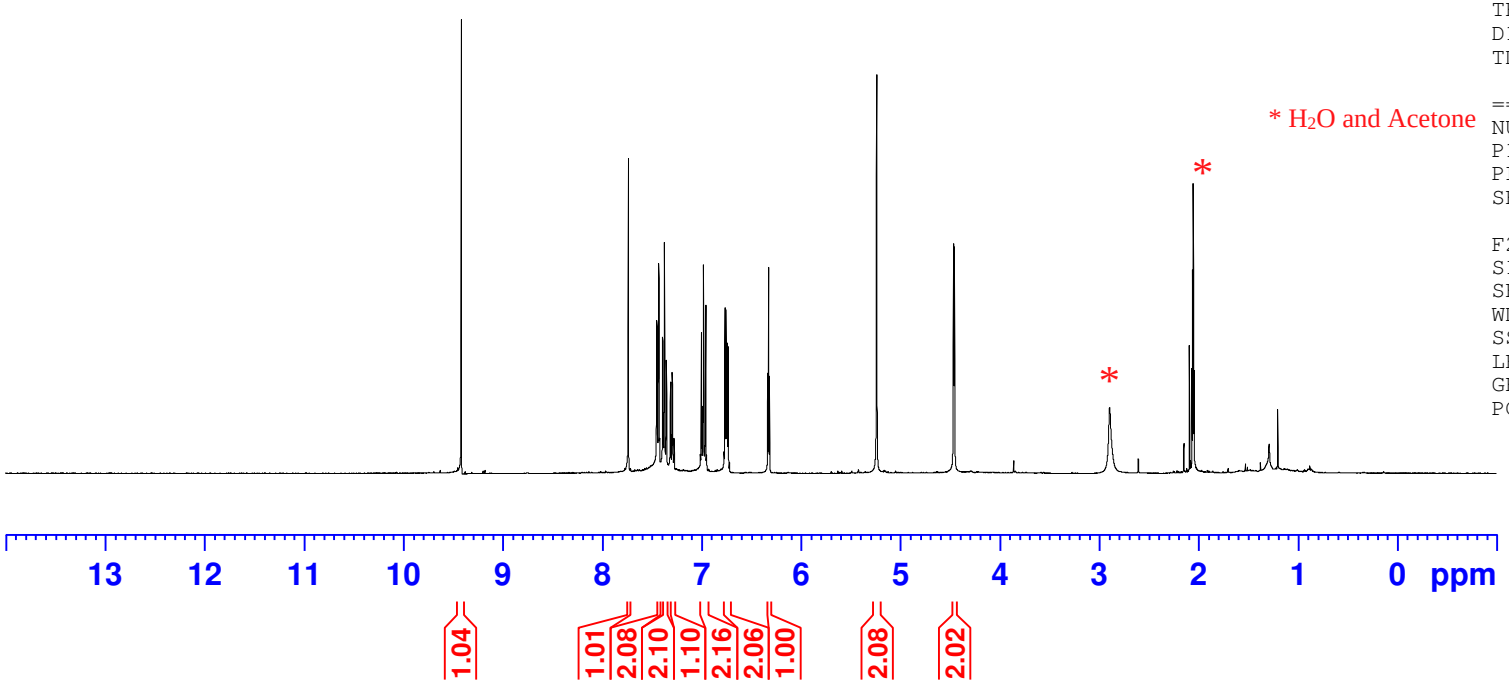
F2 - Processing parameters
SI                    65536
SF             376.4983662 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

ljx-4-18





10



* H_2O and Acetone

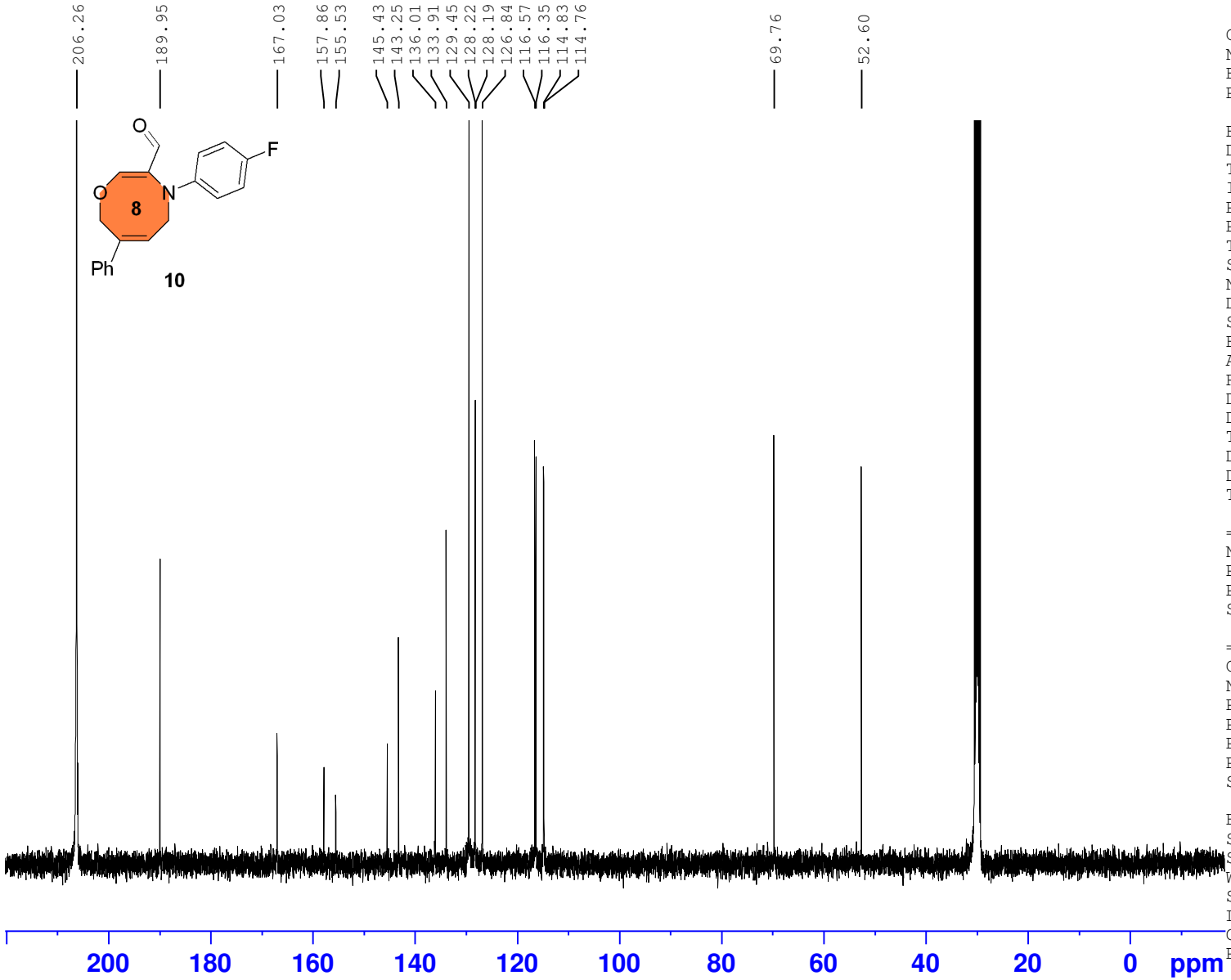
```
Current Data Parameters
NAME        20230610-400
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230609
Time               23.07
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     6
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                100.49
DW                60.800 usec
DE                 6.50 usec
TE                291.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.90 usec
PLW1        23.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900111 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-4-18




```
Current Data Parameters
NAME      20230614-400M
EXPNO                46
PROCNO                1

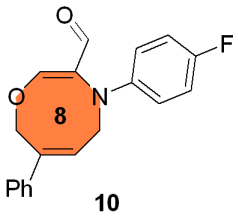
F2 - Acquisition Parameters
Date_          20230614
Time               6.24
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 44.2
DW               20.800 usec
DE                6.50 usec
TE               294.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.80 usec
PLW1        47.40000153 W
SFO1       100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        23.00000000 W
PLW12        0.30712000 W
PLW13        0.24877000 W
SFO2       400.1916008 MHz

F2 - Processing parameters
SI                32768
SF         100.6277664 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR



10

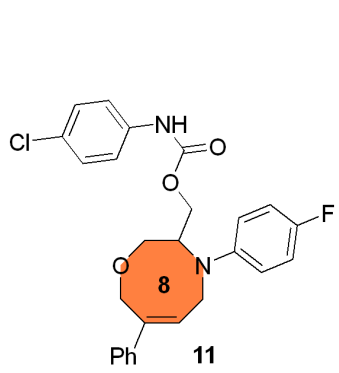
BRUKER

```
Current Data Parameters
NAME                  3-11
EXPNO                    1
PROCNO                   1

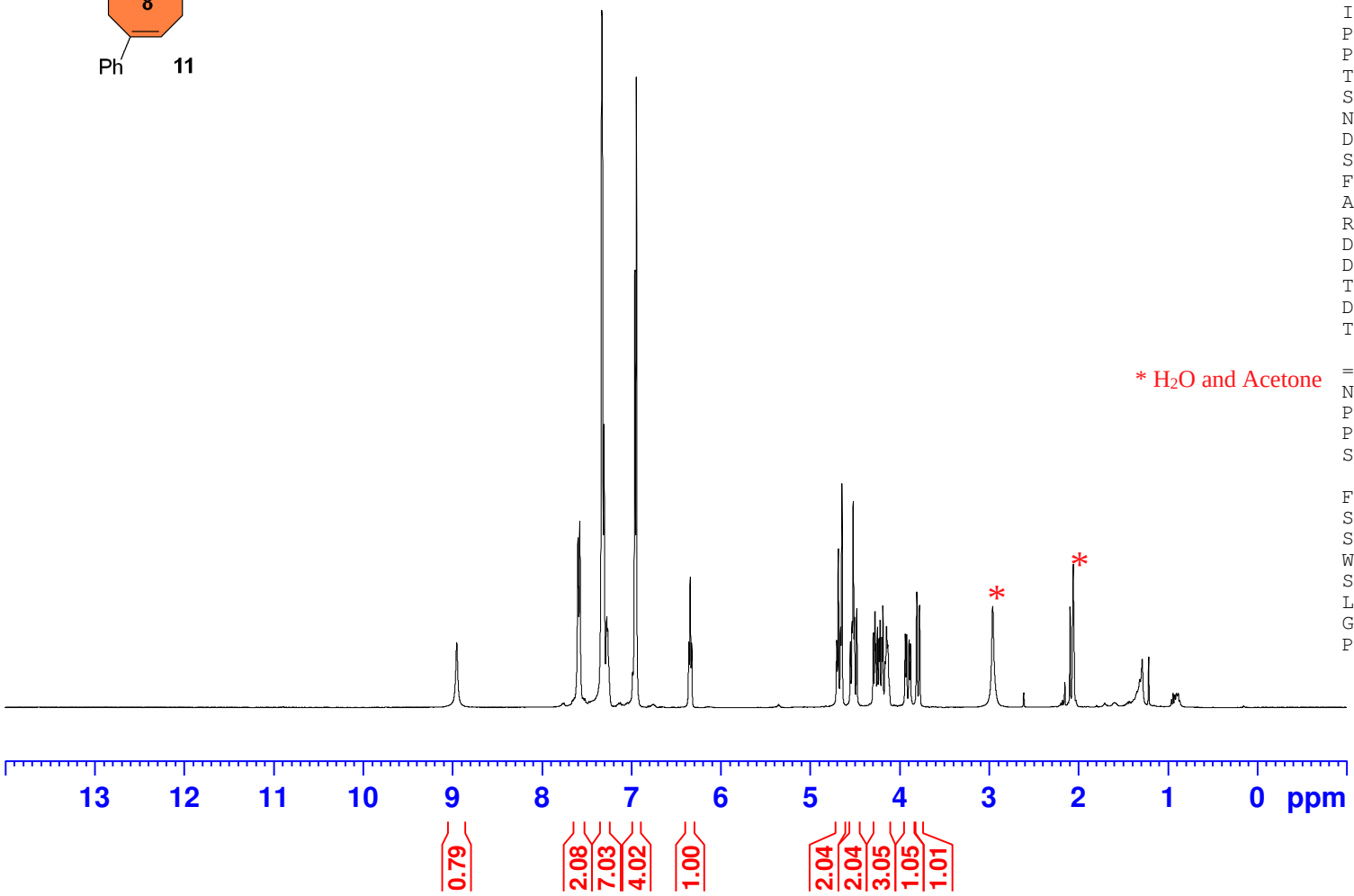
F2 - Acquisition Parameters
Date_              20230927
Time                 20.37 h
INSTRUM              Avance
PROBHD   Z116098_0833 (
PULPROG               zgig
TD                  131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH              90909.094 Hz
FIDRES            1.387163 Hz
AQ               0.7208960 sec
RG                     101
DW                   5.500 usec
DE                   6.50 usec
TE                   294.5 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           376.4607164 MHz
NUC1                   19F
P1                   18.00 usec
PLW1           16.73100090 W
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           20.73200035 W
PLW12           0.25595000 W

F2 - Processing parameters
SI                   65536
SF             376.4983662 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

ljx-4-5



8.948
7.592
7.571
7.326
7.320
7.299
7.276
7.270
7.265
7.260
6.955
6.938
6.351
6.335
6.320
4.697
4.679
4.654
4.639
4.543
4.525
4.513
4.498
4.473
4.287
4.268
4.259
4.241
4.223
4.211
4.192
4.180
4.155
4.140
4.125
3.930
3.915
3.887
3.872
3.802
3.771
2.054
2.049



* H_2O and Acetone

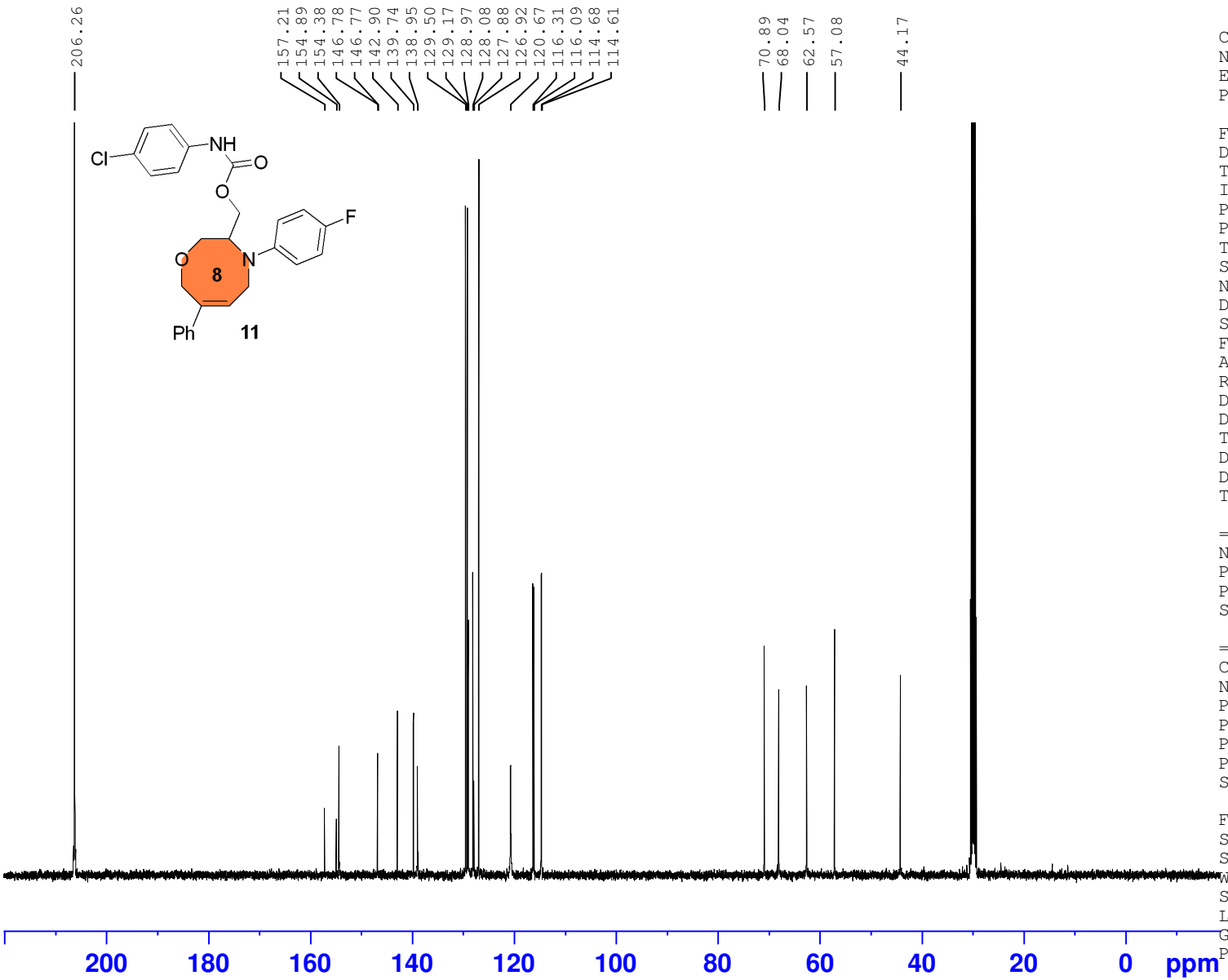
```
Current Data Parameters
NAME       20230531-400M
EXPNO                203
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230531
Time                6.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                     8
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                 61.19
DW                60.800 usec
DE                 6.50 usec
TE                293.6 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.68 usec
PLW1        14.00000000 W
SFO1        400.1924713 MHz

F2 - Processing parameters
SI                 65536
SF          400.1900129 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ljx-4-5




```
Current Data Parameters
NAME      20230531-400M
EXPNO               204
PROCNO                1

F2 - Acquisition Parameters
Date_          20230531
Time               6.30
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                  400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               193.13
DW               20.800 usec
DE                6.50 usec
TE               294.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

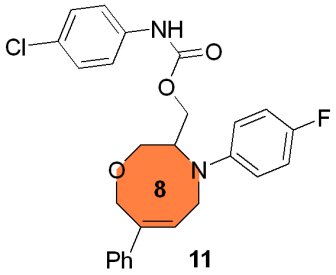
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PLW1         53.00000000 W
SFO1        100.6379178 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.37246999 W
PLW13         0.30170000 W
SFO2        400.1916008 MHz

F2 - Processing parameters
SI                32768
SF          100.6277761 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

^{19}F NMR

ljx-4-5



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```
Current Data Parameters
NAME      20230725-300M
EXPNO               407
PROCNO                1

F2 - Acquisition Parameters
Date_          20230725
Time              12.01
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG      zgfhigqn.2
TD              131072
SOLVENT           CDCl3
NS                   20
DS                    4
SWH           66964.289 Hz
FIDRES         0.510897 Hz
AQ            0.9786710 sec
RG                  203
DW                7.467 usec
DE                6.50 usec
TE                298.3 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        282.3761148 MHz
NUC1                19F
P1                14.50 usec
PLW1        10.39999962 W

======== CHANNEL f2 ========
SFO2        300.1312005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        14.00000000 W
PLW12        0.17284000 W

F2 - Processing parameters
SI                  65536
SF          282.4043552 MHz
WDW                    EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```