Supporting Information

Nickel-Catalyzed Alkylation of Amide Derivatives

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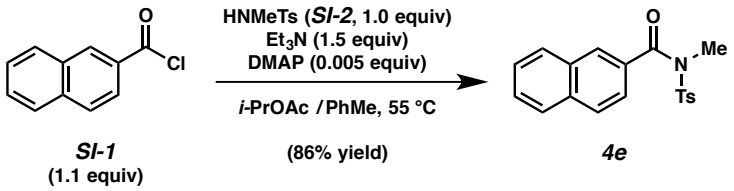
Supporting Information – Table of Contents

Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen and commercially obtained reagents were used as received. Non-commercially available substrates were synthesized following protocols specified in Section A in the Experimental Procedures. Prior to use, tetrahydrofuran was purified by distillation and taken through five freeze-pump-thaw cycles. Iodine was obtained from Spectrum Chemical. Benzyl bromide, 1-bromopropane, 1-iodo-2,2-dimethylpropane, 2-bromopropane, 2-bromobutane, iodocyclohexane, bromocyclopentane, acid chlorides **SI-1**, **SI-5**, and carboxylic acid **SI-3** were obtained from Sigma–Aldrich and used as received. *N*,4-Dimethylbenzenesulfonamide (**SI-2**) and carboxylic acid **SI-7** were obtained from Combi-Blocks. $Ni(cod)_2$, SIPr, and Zn powder (325 mesh, 99.9%) were obtained from Strem Chemicals and stored in a glove box. Anhydrous lithium chloride (99%) was obtained from Alfa Aesar and stored in a glove box. Chlorotrimethylsilane and 1,2-dibromoethane were obtained from Alfa Aesar and Sigma–Aldrich, respectively, and distilled before use. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm for analytical chromatography and 0.50 mm for preparative chromatography) and visualized using a combination of UV, anisaldehyde, and potassium permanganate staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Bruker spectrometers (at 300, 400 and 500 MHz) and are reported relative to residual solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (at 75 and 125 MHz). ^{19}F NMR spectra were recorded on Bruker spectrometers (at 282 MHz) and reported in terms of chemical shift (δ ppm). IR spectra were recorded on a Perkin-Elmer UATR Two FT-IR spectrometer and are reported in terms of frequency absorption (cm^{-1}). High-resolution mass spectra were obtained on Thermo Scientific™ Exactive Mass Spectrometer with DART ID-CUBE.

Experimental Procedures

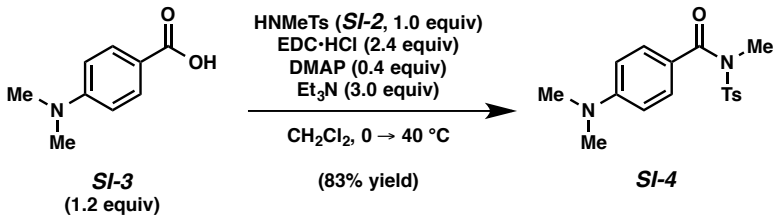
A. Syntheses of Amide Substrates

Representative Procedure A for the synthesis of amide substrates from Scheme 1, Figures 2 and 3 (synthesis of amide 4e is used as an example).



To a solution of sulfonamide **SI-2** (3.00 g, 16.2 mmol, 1.0 equiv), DMAP (9.9 mg, 0.081 mmol, 0.005 equiv), triethylamine (3.40 mL, 24.3 mmol, 1.5 equiv), and *i*-PrOAc (35.2 mL) at 55 °C was added dropwise a solution of acid chloride **SI-1** (3.41 g, 17.8 mmol, 1.1 equiv) in toluene (10.0 mL, 0.46 M in total) over 1 min. The reaction mixture was stirred at 55 °C for 1 h. After cooling the reaction mixture to room temperature, the reaction was quenched by the addition of 1.0 M aqueous HCl (10 mL). The resulting biphasic mixture was transferred to a separatory funnel with EtOAc (30 mL) and extracted with EtOAc (3 x 30 mL). The organic layers were combined, dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by flash chromatography (24:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) to yield amide **4e** (5.2 g, 86% yield) as a white solid. Amide **4e**: mp: 96–98 ºC; R_f 0.50 (7:3 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07–8.06 (s, 1H), 7.87–7.84 (m, 5H), 7.62–7.53 (m, 3H), 7.34–7.32 (m, 2H), 3.34 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.9, 145.2, 135.6, 135.1, 132.5, 132.0, 130.0, 129.9, 129.3, 128.8, 128.5, 128.5, 128.2, 127.3, 124.9, 36.0, 22.0; IR (film): 3060, 2954, 2922, 1682, 1356 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{18}NO_3S$, 340.10074; found 340.09984.

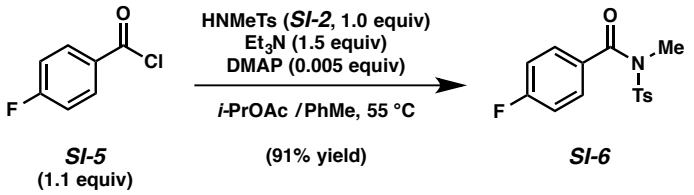
Representative Procedure B for the synthesis of amide substrates from Scheme 1, Figures 2 and 3 (synthesis of amide SI-4 is used as an example).



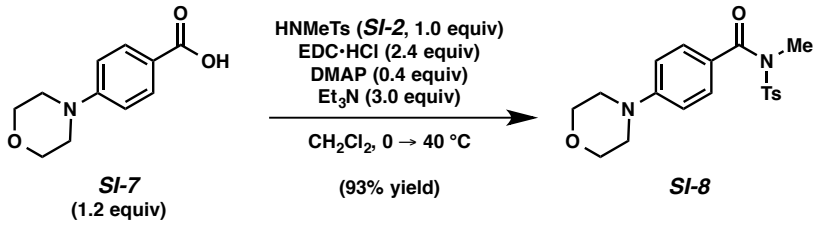
To a solution of sulfonamide **SI-2** (1.00 g, 5.40 mmol, 1.0 equiv), EDC•HCl (2.48 g, 13.0 mmol, 2.4 equiv), DMAP (263 mg, 2.16 mmol, 0.4 equiv), triethylamine (2.30 mL, 16.2 mmol, 3.0 equiv), and CH_2Cl_2 (15.4 mL, 0.35 M) at 0 °C was added carboxylic acid **SI-3** (1.07 g, 6.48 mmol, 1.2 equiv) as a solid in one portion. The reaction mixture was allowed to come to room temperature and then stirred at 40 °C for 16 h. After cooling to room temperature, the reaction mixture was transferred to a separatory funnel with EtOAc (30 mL) and washed with 1.0 M aqueous HCl (2 x 10 mL), followed by 1.0 M aqueous NaOH (2 x 10 mL), and deionized water (10 mL). The organic layer was dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by flash chromatography (20:1 Benzene:Et_2O) to yield amide **SI-4** (1.48 g, 83% yield) as an off-white solid. Amide **SI-4**: mp: 104–106 ºC; R_f 0.52 (3:2 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.85 (d, J = 8.2, 2H), 7.65 (d, J = 9.0, 2H), 7.31 (d, J = 8.2, 2H), 6.63 (d, J = 8.9, 2H), 3.20 (s, 3H), 3.05 (s, 6H), 2.43 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.0, 153.7, 144.7, 135.5, 132.3, 129.8, 128.8, 120.5, 110.8, 40.4, 36.4, 21.9; IR (film): 3060, 2917, 2823, 1672, 1600 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{17}H_{21}N_2O_3S$, 333.12729; found 333.12611

Note: Supporting information for the syntheses of some amides shown in Scheme 1, Figures 2 and 3, and Scheme 2 have previously been reported: **4a**,[1a] **4b**,[1b] **4c**,[1c] **4d**,[1d] **SI-14**,[1e] **SI-15**,[1f] **SI-16**,[1d] and **19**.[1d] Syntheses for the remaining substrates shown in Scheme 1, Figures 2 and 3 are as follows:

Any modifications of the conditions shown in the representative procedures above are specified in the following schemes.

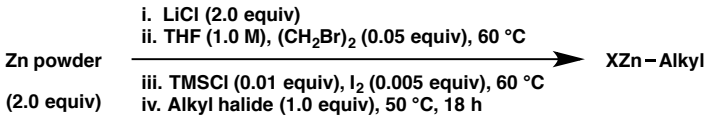


Amide SI-6. Followed representative procedure A. Purification by flash chromatography (9:1 Hexanes:EtOAc) generated amide **SI-6** (91% yield) as a white solid. Amide **SI-6**: mp: 80–83 °C; R_f 0.54 (7:3 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, J = 8.3, 2H), 7.62–7.59 (m, 2H), 7.33 (d, J = 8.0, 2H), 7.12–7.08 (m, 2H), 3.24 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.0, 165.3 (d, $J_{C–F}$ = 253.9), 145.4, 135.3, 131.7 (d, $J_{C–F}$ = 9.1) 131.1 (d, $J_{C–F}$ = 3.3), 130.1, 128.6, 115.8 (d, $J_{C–F}$ = 22.2), 35.8, 22.0; ^{19}F NMR (282 MHz, $CDCl_3$): δ 106.1; IR (film): 3074, 2954, 2924, 1683, 1596 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{15}H_{15}FNO_3S$, 308.07567; found 308.07463.



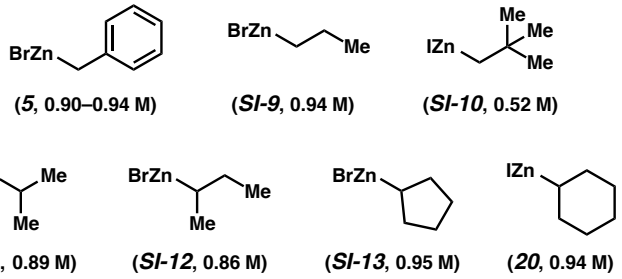
Amide SI-8. Followed representative procedure B. Purification by flash chromatography (1:1 Hexanes:EtOAc) generated amide **SI-8** (93% yield) as a white solid. Amide **SI-8**: mp: 141–143 ºC; R_f 0.60 (3:7 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83 (d, J = 8.3, 2H), 7.63 (d, J = 8.8, 2H), 7.32 (d, J = 8.3, 2H), 6.84 (d, J = 8.8, 2H), 3.85 (t, J = 4.8, 4H), 3.29 (t, J = 4.8, 4H), 3.21 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.7, 154.3, 144.9, 135.4, 131.8, 129.9, 128.8, 124.1, 113.5, 66.9, 47.9, 36.3, 22.0; IR (film): 3049, 2964, 2854, 1673, 1601 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{19}H_{23}N_2O_4S$, 375.13785; found 375.13717.

B. Preparation of Organozinc Halides

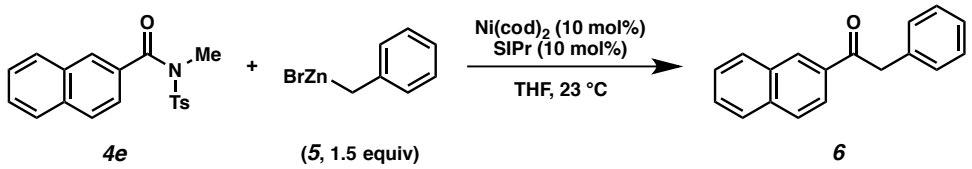


Following a modification of the procedure reported by Knochel,[2] a flame-dried 25 mL round bottom flask equipped with a magnetic stir bar and rubber septum was brought into a glove box where Zn powder (650 mg, 10.0 mmol, 2.0 equiv, Strem 325 mesh) and anhydrous LiCl (420 mg, 10.0 mmol, 2.0 equiv) were added. The flask was then removed from the glove box and heated with a heat gun for 10 min under high vacuum, cooled to room temperature, and then backfilled with N_2. Freshly distilled THF (5.0 mL) and 1,2-dibromoethane (22 μL, 0.25 mmol, 0.05 equiv) were added via syringe and the reaction mixture was heated at 60 °C for 20 min. After cooling to room temperature, freshly distilled TMSCl (6 μL, 0.05 mmol, 0.01 equiv) followed by a solution of I_2 (6.4 mg, 0.025 mmol, 0.005 equiv) in THF (25 μL, 1.0 M) were added via syringe and the reaction mixture was heated again at 60 °C for 20 min. After cooling to room temperature, the alkyl halide (5.0 mmol, 1.0 equiv) was added dropwise via syringe over 1 min. A flame-dried air condenser was attached to the flask under N_2 and the reaction vessel was heated at 50 °C for 18 h. The reaction mixture was cooled to room temperature and allowed to stand for 1 h before the supernatant fluid was transferred to a flame-dried schlenk flask via syringe. The concentration of the organozinc halide was determined by iodometric titration using Knochel's procedure.[3]

Note: The use of organozinc reagents with lower titers led to lower yields in the subsequent coupling reactions.



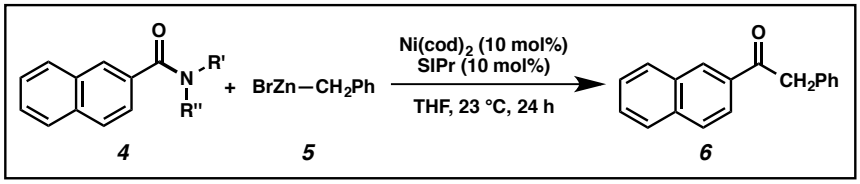
C. Initial Survey of Naphthamide Substrates with Benzylzinc Bromide (5)



Representative Procedure for alkylation reactions of naphthamides from Tables S1 and S2 (coupling of amide 4e and benzylzinc bromide (5) is used as an example). A 1-dram vial was charged with a magnetic stir bar and flame-dried under reduced pressure, and then allowed to cool under N_2. Amide substrate **4e** (67.8 mg, 0.200 mmol, 1.0 equiv) and hexamethylbenzene (3.2 mg, 0.020 mmol, 0.1 equiv) were added, and the vial was flushed with N_2. The vial was taken into a glove box and charged with $Ni(cod)_2$ (5.5 mg, 0.020 mmol, 10 mol%) and SIPr (7.8 mg, 0.020 mmol, 10 mol%). Subsequently, THF (0.20 mL, 1.0 M) was added, and the vial was removed from the glove box and the reaction was allowed to stir at 23 °C for 1 h. Concurrently, the benzylzinc bromide solution (**5**) was heated in a water bath at 50 °C for 1 h. A portion of the preheated solution of **5** (333 μL, 0.300 mmol, 1.5 equiv, 0.90 M in THF) was then added to the reaction mixture dropwise via syringe over 3 sec. The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was allowed to stir at 23 °C for 24 h. The reaction was quenched by the addition of a saturated aqueous solution of NH_4Cl (0.5 mL), and the resulting aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organics were filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard.

Any modifications of the conditions shown in the representative procedure above are specified below in Tables S1 and S2.

Table S1. Initial Survey of Naphthamide Substrates with Benzylzinc Bromide (**5**)[a]

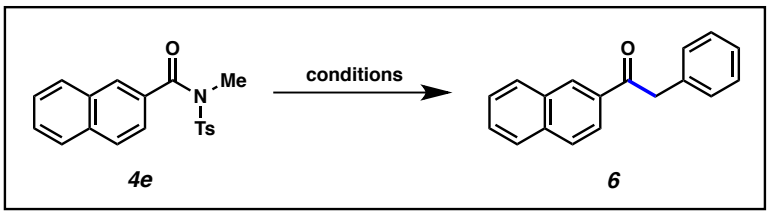


Entry			*Recovered 4*	*Yield of Ketone 6*
1		*4a*	*100%*	*0%*
2		*4b*	*51%*	*0%*
3		*4c*	*100%*	*0%*
4		*4d*	*40%*	*60%*
5		*4e*	*17%*	*81%*

[a] Yields were determined by ^{1}H NMR analysis using hexamethylbenzene as an internal standard.

D. Relevant Control Experiments in the Alkylation of Amide 4e

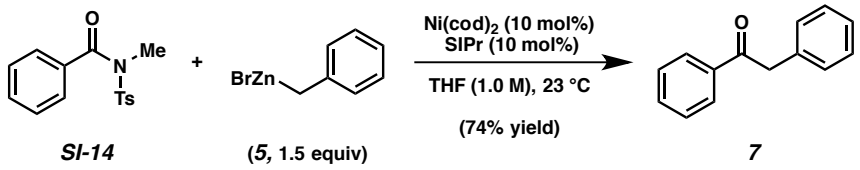
Table S2. Relevant Control Experiments in the Alkylation of Amide **4e**[a]



Reaction Conditions	*Experimental Results* 4e	6
BnZnBr (1.5 equiv), $Ni(cod)_2$ (10 mol%), SIPr (10 mol%) THF (1.0 M), 23 °C, 24 h	17%	81%
Control Experiments:		
BnZnBr (1.5 equiv) THF (1.0 M), 23 °C, 24 h	100%	0%
BnZnBr (1.5 equiv), SIPr (10 mol%) THF (1.0 M), 23 °C, 24 h	100%	0%
BnZnBr (1.5 equiv), $Ni(cod)_2$ (10 mol%) THF (1.0 M), 23 °C, 24 h	33%	35%[b]

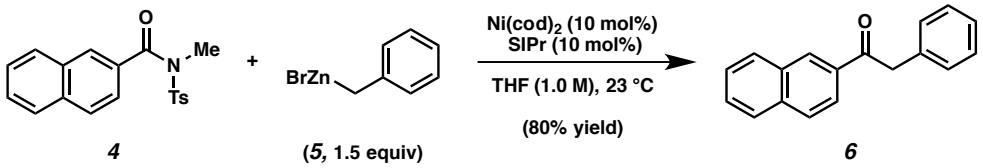
[a] Yields were determined by ^1H NMR analysis using hexamethylbenzene as an internal standard.
[b] Some conversion to the ketone was observed in the absence of SIPr, but in greatly diminished yield relative to the experiment run with both $Ni(cod)_2$ and SIPr. Additionally, use of these conditions with other substrates was even less successful.

E. Scope of Methodology

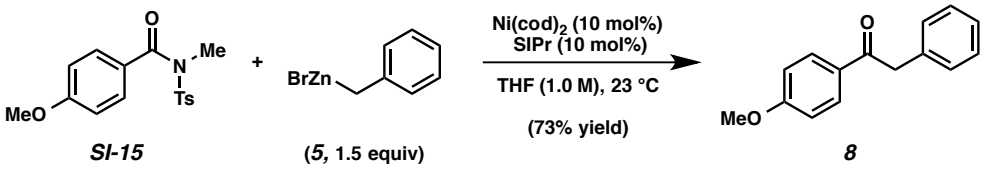


Representative Procedure (coupling of amide SI-14 and benzylzinc bromide (5) is used as an example). Ketone 7. A 1-dram vial was charged with a magnetic stir bar and flame-dried under reduced pressure, and then allowed to cool under N_2. Amide substrate **SI-14** (57.8 mg, 0.200 mmol, 1.0 equiv) was added, and the vial was flushed with N_2. The vial was taken into a glove box and charged with $Ni(cod)_2$ (5.5 mg, 0.020 mmol, 10 mol%) and SIPr (7.8 mg, 0.020 mmol, 10 mol%). Subsequently, THF (0.20 mL, 1.0 M) was added, and the vial was removed from the glove box and the reaction was allowed to stir at 23 °C for 1 h. Concurrently, the benzylzinc bromide solution (**5**) was heated in a water bath at 50 °C for 1 h. A portion of the preheated solution of **5** (319 μL, 0.300 mmol, 1.5 equiv, 0.94 M in THF) was then added to the reaction mixture dropwise via syringe over 3 sec. The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was allowed to stir at 23 °C for 24 h. The reaction was quenched by the addition of a saturated aqueous solution of NH_4Cl (0.5 mL), and the resulting aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organics were filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the crude residue was purified by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) to yield ketone product **7** (74% yield, average of two experiments) as a white solid. Ketone **7**: R_f 0.54 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[4]

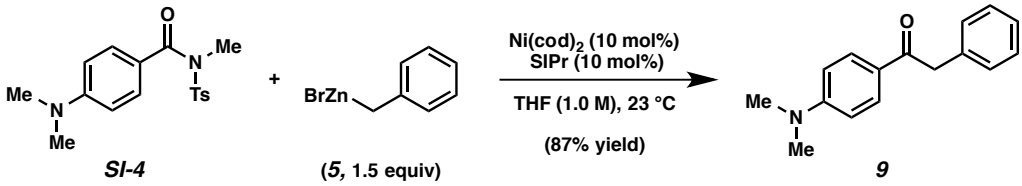
Any modifications of the conditions shown in the representative procedure above are specified in the following schemes, which depict all of the results shown in Figures 2 and 3.



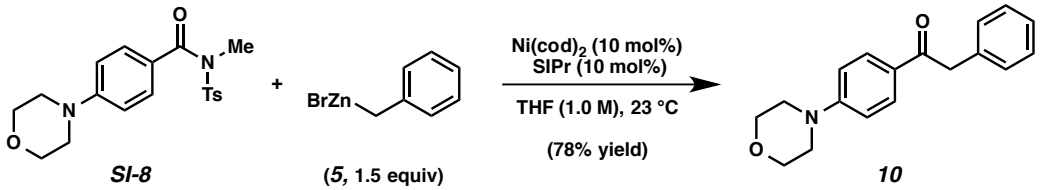
Ketone 6. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **6** (80% yield, average of two experiments) as a white solid. Ketone **6**: R_f 0.53 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[4]



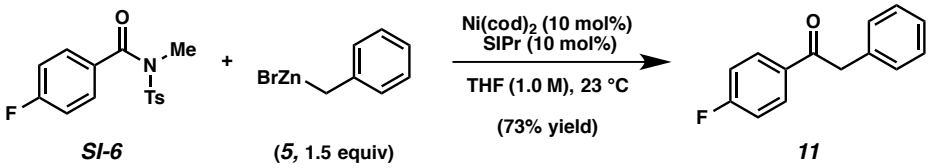
Ketone 8. Purification by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) generated ketone **8** (73% yield, average of two experiments) as a white solid. Ketone **8**: R_f 0.46 (4:1 Hexanes:EtOAc). Spectral data match those previously reported.[5]



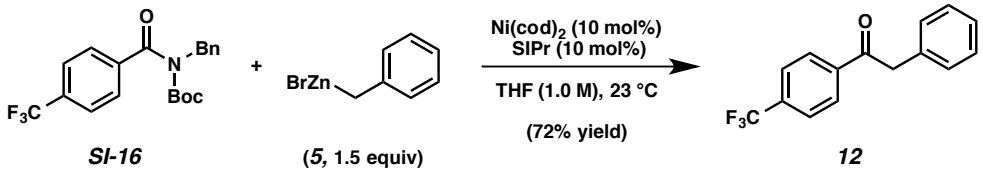
Ketone 9. Purification by flash chromatography (8:1:1 PhH:Et_2O:CH_2Cl_2) generated ketone **9** (87% yield, average of two experiments) as a white solid. Ketone **9**: R_f 0.46 (8:1:1 PhH:Et_2O:CH_2Cl_2). Spectral data match those previously reported.[6]



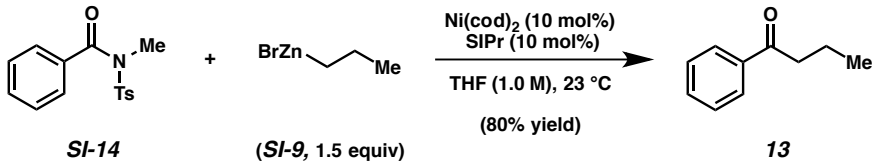
Ketone 10. Purification by flash chromatography (10:5:1 $CHCl_3$:Hexanes:CH_3CN) followed by preparative thin-layer chromatography (10:2:1 $CHCl_3$:Hexanes:CH_3CN) generated ketone **10** (78% yield, average of two experiments) as a white solid. Ketone **10**: mp: 138–139 °C; R_f 0.64 (3:2 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CD_3CN): δ 7.94–7.88 (m, 2H), 7.33–7.19 (m, 5H), 6.95–6.89 (m, 2H), 4.21 (s, 2H), 3.77–3.73 (m, 4H), 3.29–3.24 (m, 4H); ^{13}C NMR (125 MHz, CD_3CN): δ 196.8, 155.5, 137.1, 131.4, 130.6, 129.4, 127.9, 127.4, 114.1, 67.1, 48.1, 45.5; IR (film): 3042, 2957, 2857, 2840, 1675, 1595 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{11}H_8O_2$, 282.14940; found 282.14800.



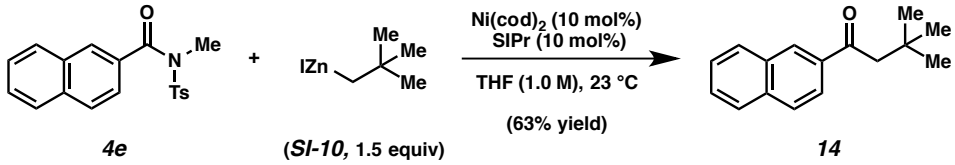
Ketone 11. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **11** (73% yield, average of two experiments) as a white solid. Ketone **11**: R_f 0.50 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[4]



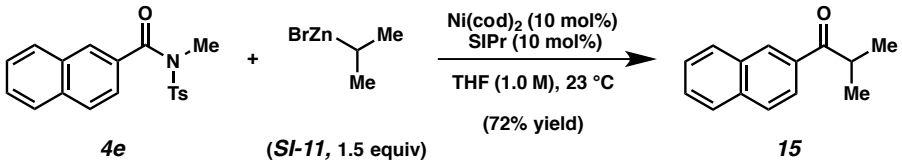
Ketone 12. Purification by flash chromatography (100% PhH) generated ketone **12** (72% yield, average of two experiments) as a white solid. Ketone **12**: R_f 0.68 (100% PhH). Spectral data match those previously reported.[7]



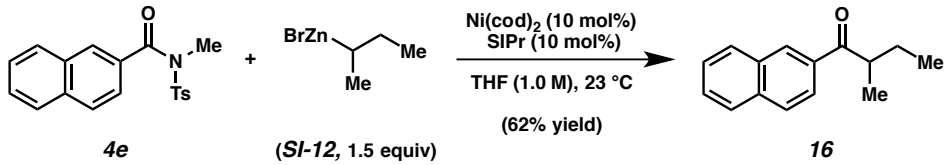
Ketone 13. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **13** (80% yield, average of two experiments) as a colorless oil. Ketone **13**: R_f 0.57 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[4]



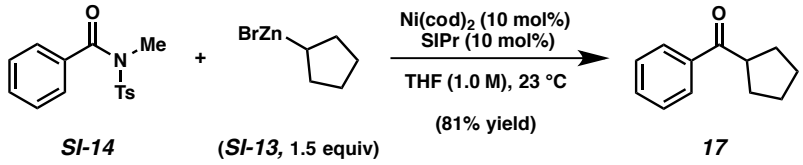
Ketone 14. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **14** (63% yield, average of two experiments) as a colorless oil. Ketone **14**: R_f 0.63 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[8]



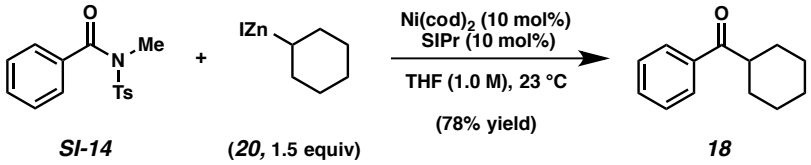
Ketone 15. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **15** (72% yield, average of two experiments) as a colorless oil. Ketone **15**: R_f 0.64 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[9]



Ketone 16. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **16** (62% yield, average of two experiments) as a colorless oil. Ketone **16**: R_f 0.44 (10:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, C_6D_6): δ 8.35 (br s, 1H), 8.13 (dd, J = 8.6, 1.6, 1H), 7.63 (d, J = 7.9, 1H), 7.56 (d, J = 8.6, 1H), 7.53 (d, J = 7.9, 1H) 7.27–7.19 (m, 1H), 3.26–3.18 (m, 1H), 1.93–1.83 (m, 1H), 1.48–1.38 (m, 1H), 1.14 (d, J = 6.9, 3H), 0.83 (t, J = 7.5, 3H);^{13}C NMR (125 MHz, CD_3CN): δ 205.3, 136.4, 135.2, 133.7, 130.7, 130.5, 129.5, 129.4, 128.6, 127.8, 125.0, 42.7, 27.6, 17.2, 12.0; IR (film): 3060, 2965, 2932, 2875, 1677, 1625 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{11}H_8O_2$, 213.12794; found 213.12691.

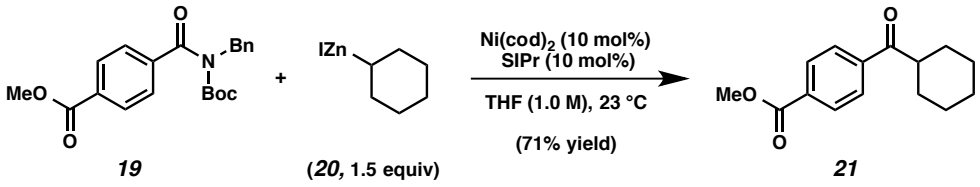


Ketone 17. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **17** (81% yield, average of two experiments) as a colorless oil. Ketone **17**: R_f 0.64 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[10]



Ketone 18. Purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **18** (78% yield, average of two experiments) as a colorless oil. Ketone **18**: R_f 0.54 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[11]

F. Gram-Scale Alkylation to Form Ketone 21



Ketone 21. A 20 mL scintillation vial was charged with a magnetic stir bar and flame-dried under reduced pressure, and then allowed to cool under N_2. Amide substrate **19** (1.00 g, 2.71 mmol, 1.0 equiv) was added, and the vial was flushed with N_2. The vial was taken into a glove box and charged with $Ni(cod)_2$ (74.5 mg, 0.270 mmol, 10 mol%) and SIPr (106 mg, 0.270 mmol, 10 mol%). Subsequently, THF (2.7 mL, 1.0 M) was added, and the vial was removed from the glove box and the reaction was allowed to stir at 23 °C for 1 h. Concurrently, the cyclohexylzinc iodide solution (**20**) was heated in a water bath at 50 °C for 1 h. A portion of the preheated solution of **20** (4.32 mL, 4.07 mmol, 1.5 equiv, 0.94 M in THF) was then added to the reaction mixture dropwise via syringe over 5 sec. The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was allowed to stir at 23 °C for 24 h. The reaction was quenched by the addition of a saturated aqueous solution of NH_4Cl (3 mL), and the resulting aqueous layer was extracted with EtOAc (3 x 5 mL). The combined organics were filtered over a plug of silica gel (50 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography (24:1 Hexanes:EtOAc) to yield ketone product **21** (472 mg, 71% yield) as a pale yellow solid. Ketone **21**: R_f 0.50 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.

References

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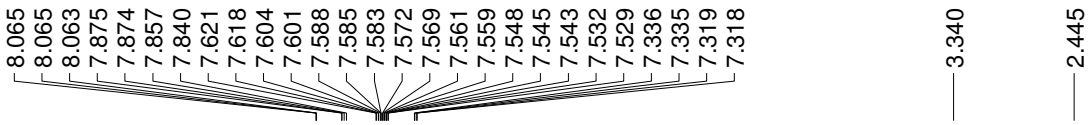
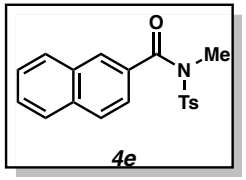
^{1}H NMR Spectra





4e

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TD0 1

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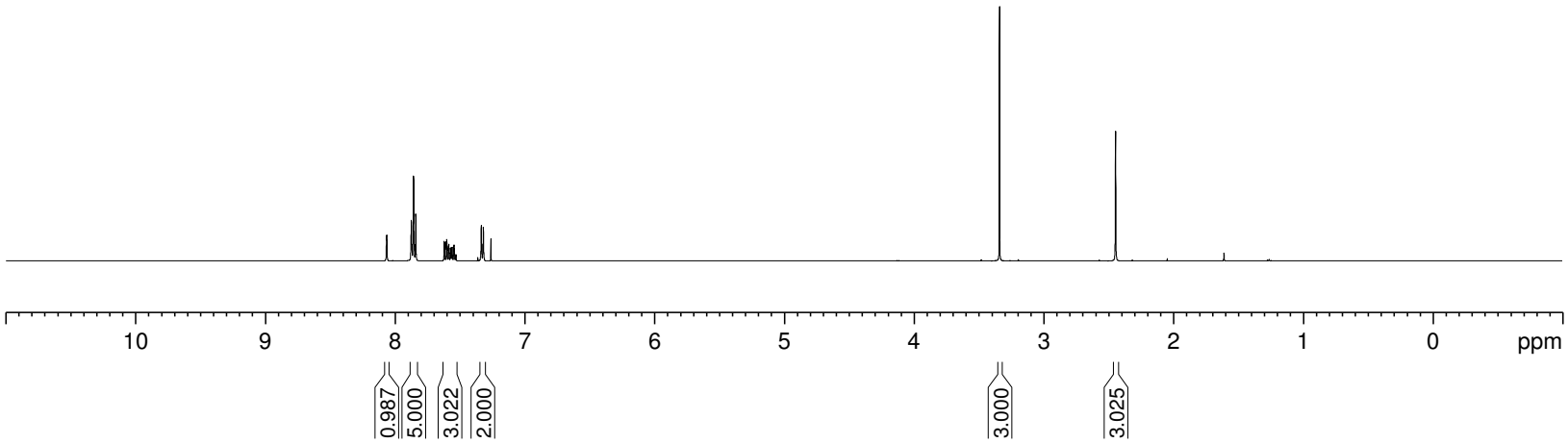

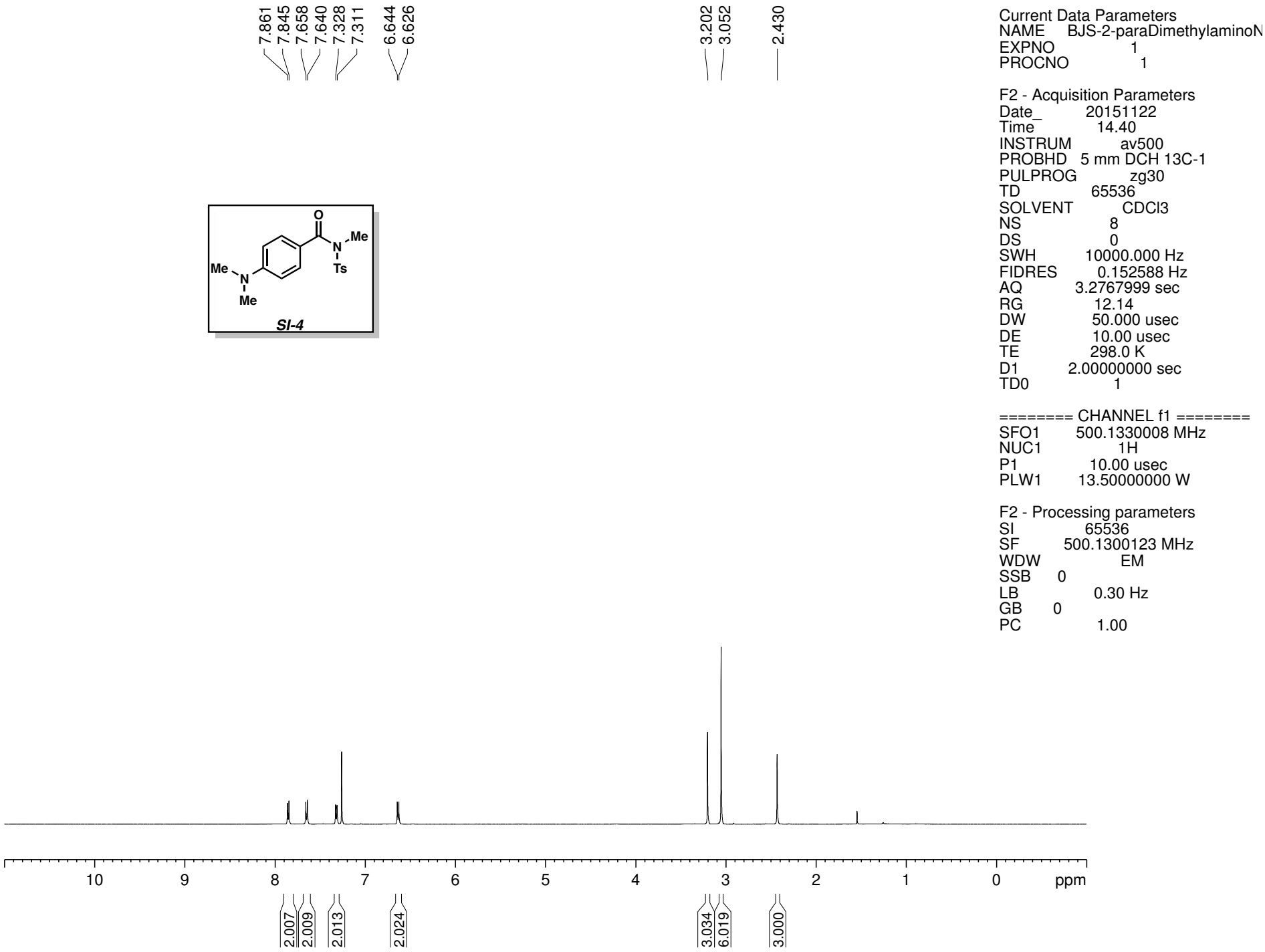
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7.311
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6.626
3.202
3.052
2.430
Me
N
Me
Ts
Me
N
Me
O
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PROCNO 1
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FIDRES 0.152588 Hz
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RG 12.14
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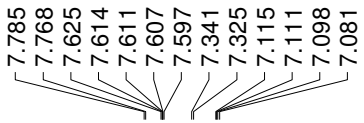

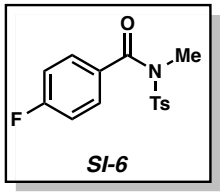

SI-6

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PROCNO 1

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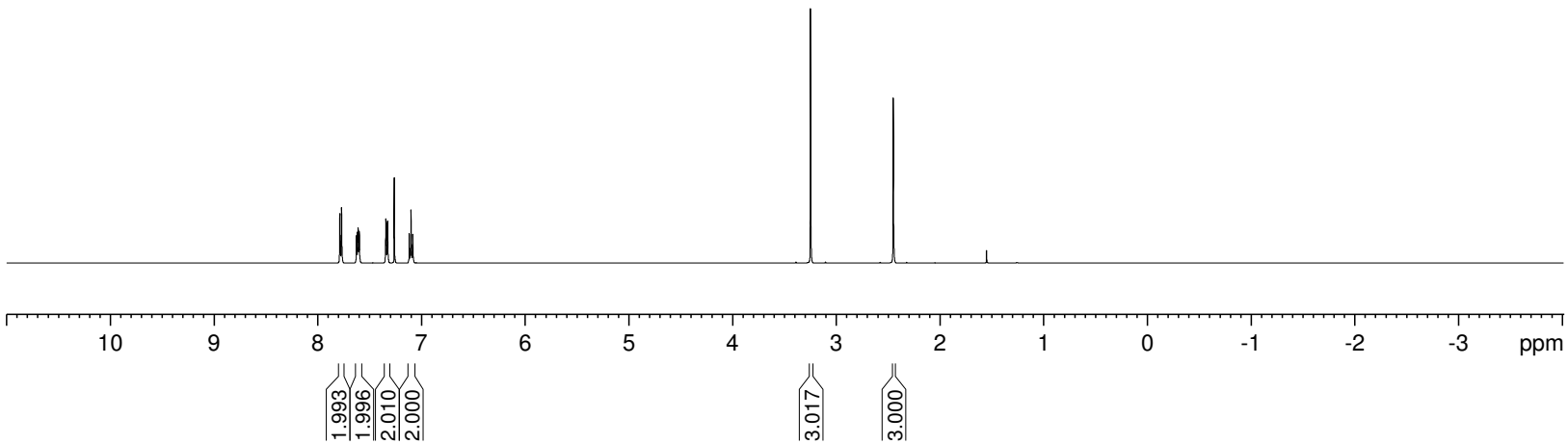

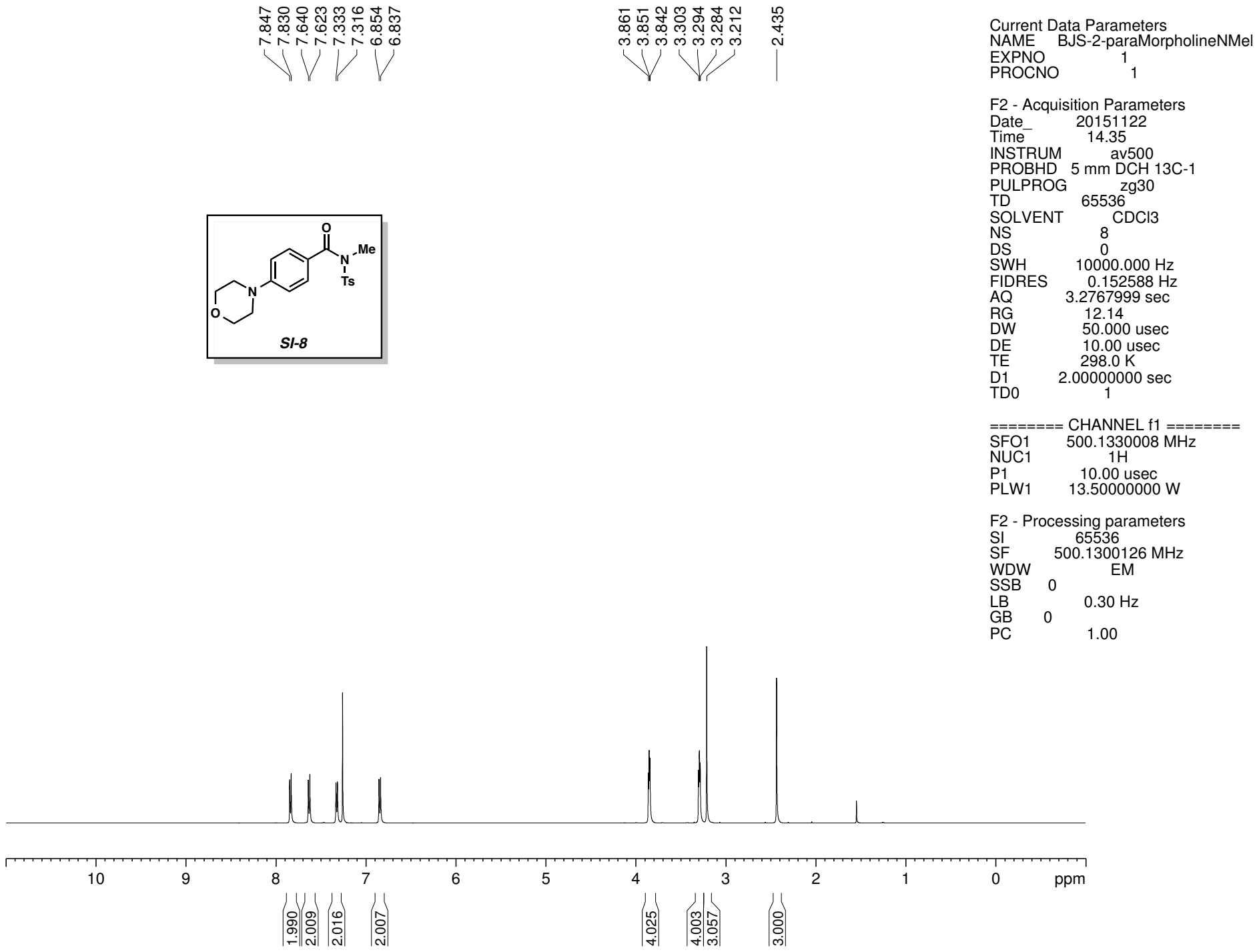

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RG 12.14
DW 50.000 usec
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TE 298.0 K
D1 2.00000000 sec
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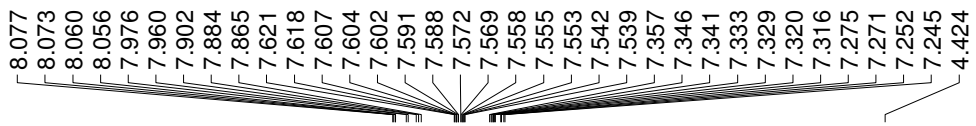

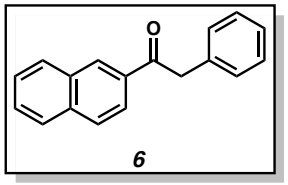

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P1 10.00 usec
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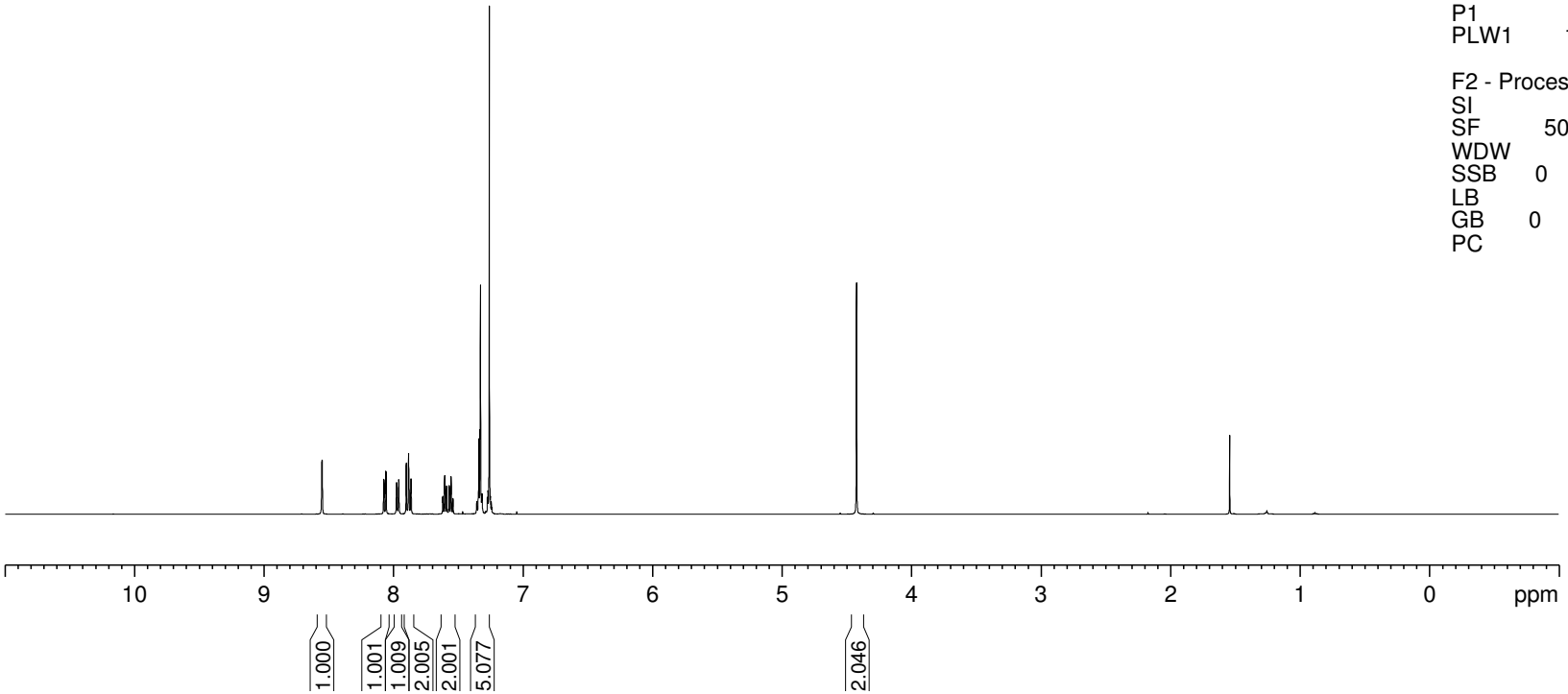

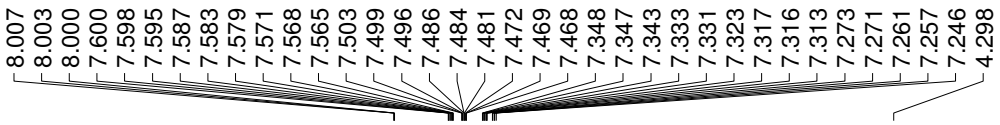

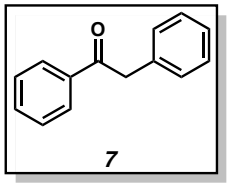

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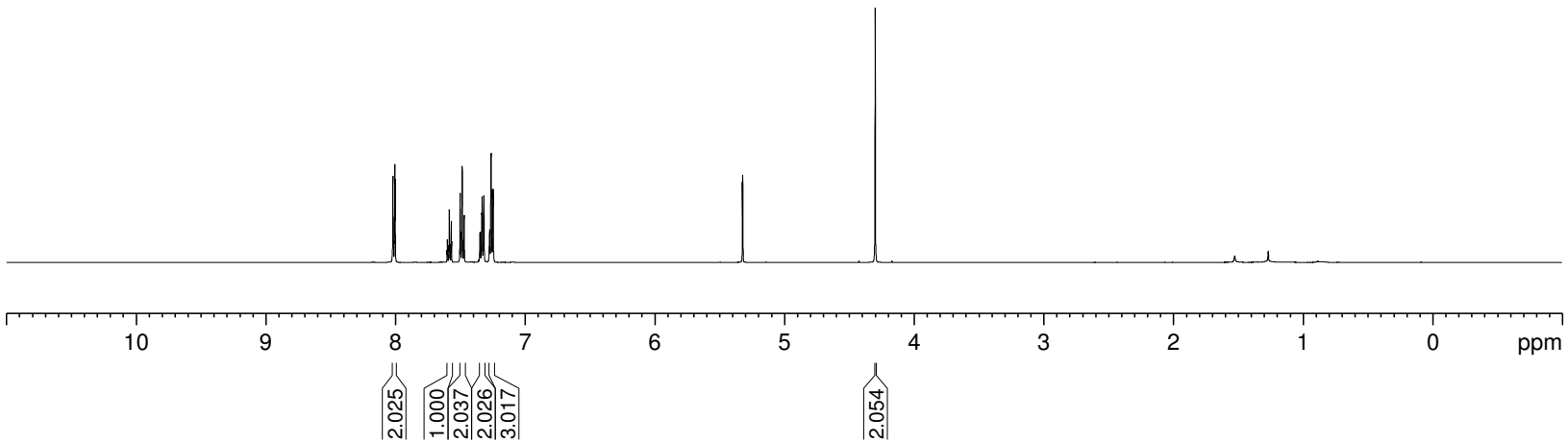

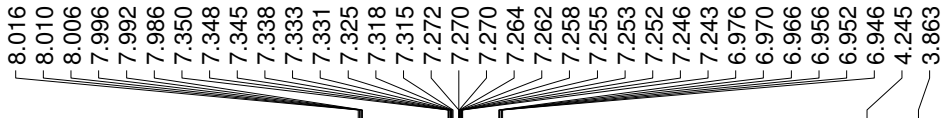

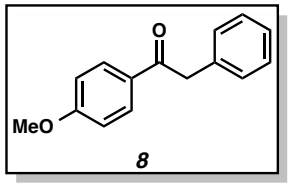

8

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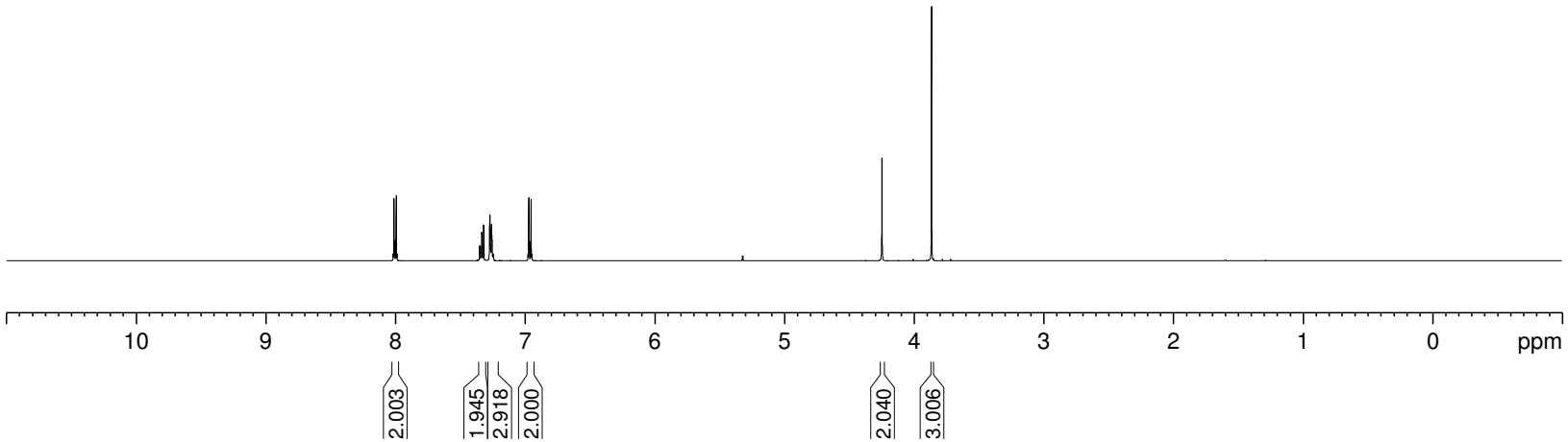

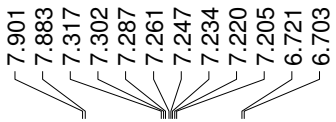

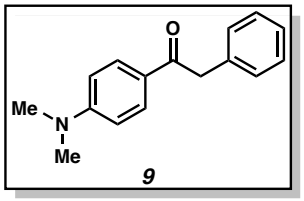

9

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RG 161.3
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DE 6.00 usec
TE 296.9 K
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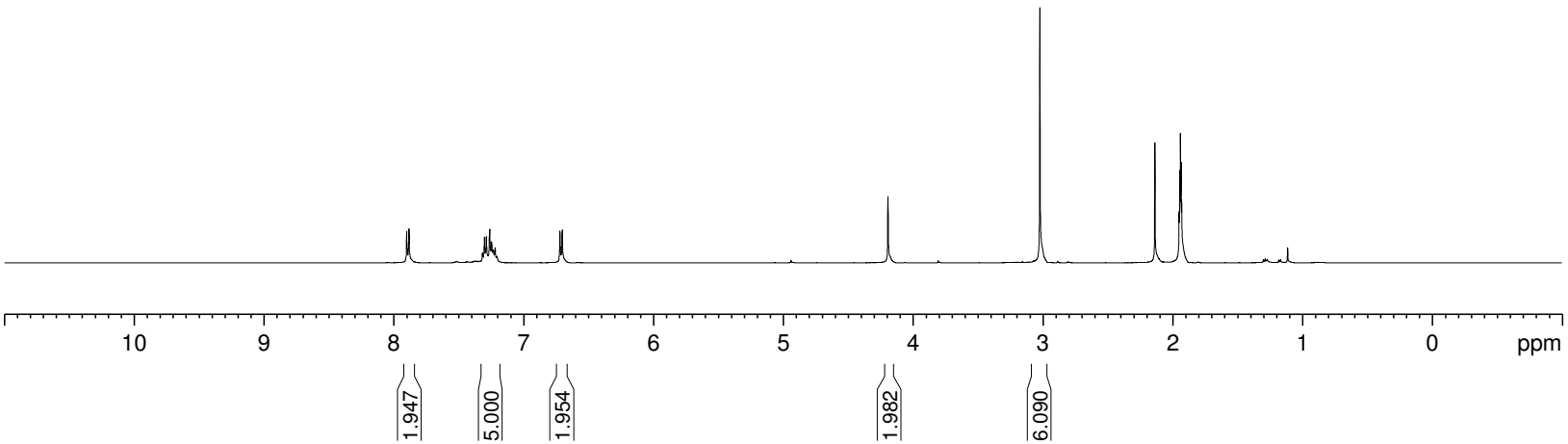

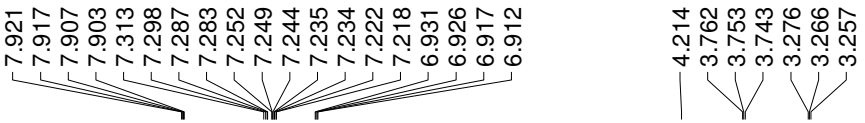

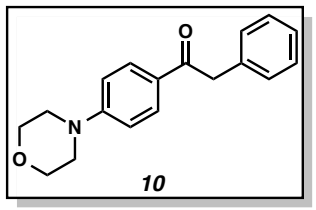

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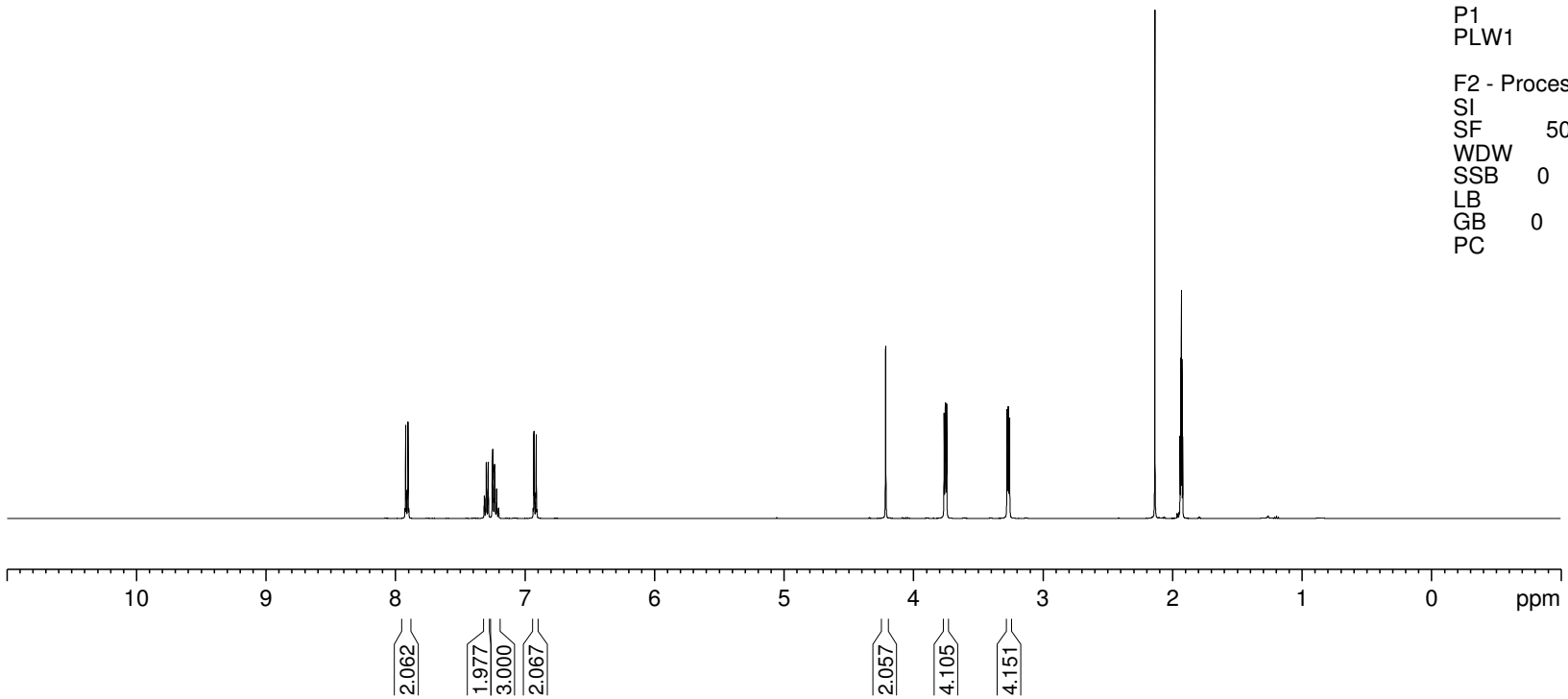
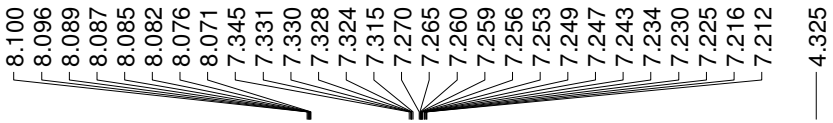
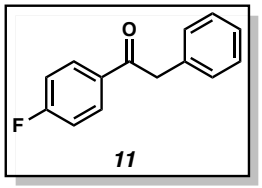






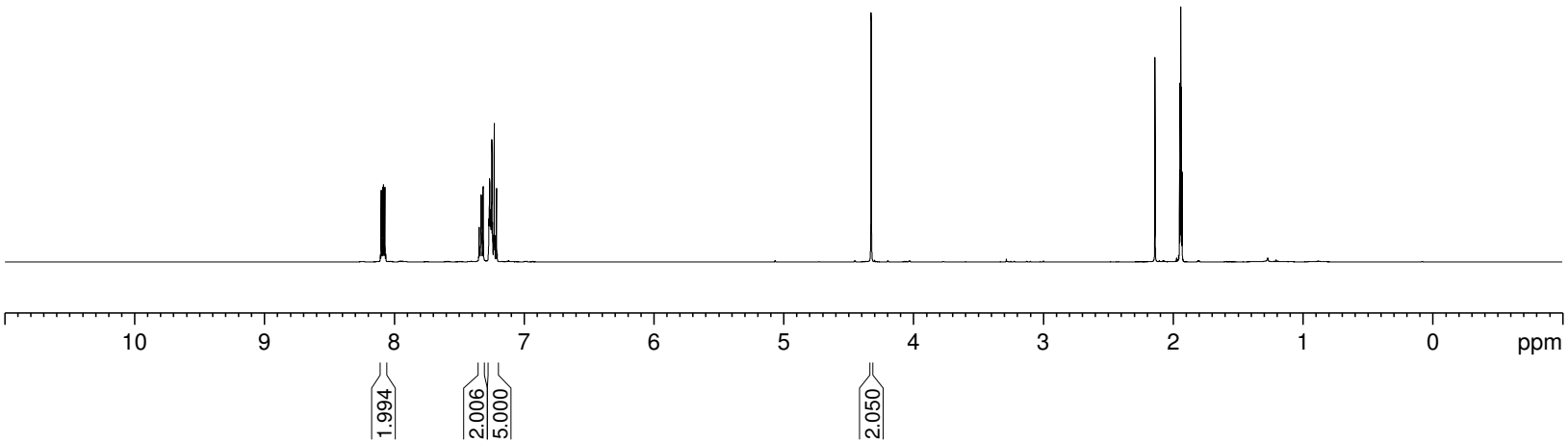
11

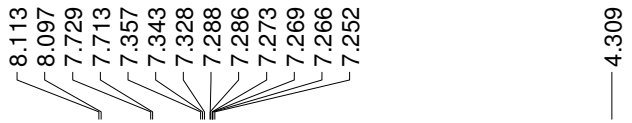
Current Data Parameters
NAME BJS-2-225-CHLORO3
EXPNO 1
PROCNO 1

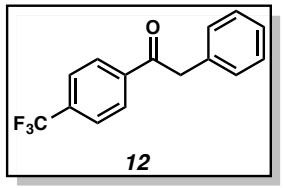
F2 - Acquisition Parameters
Date_ 20160108
Time 13.17
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00







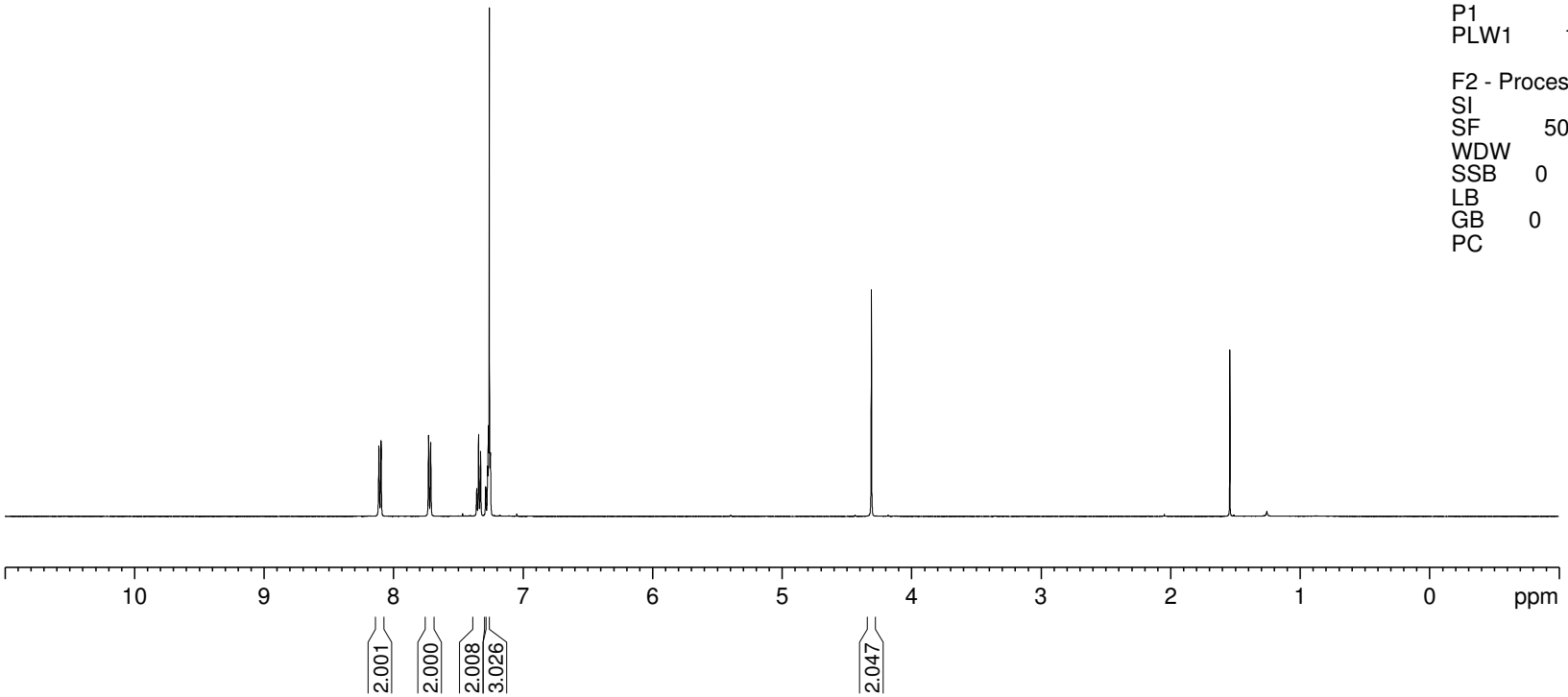
12

Current Data Parameters
NAME NAW-4-225p
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160115
Time 10.16
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

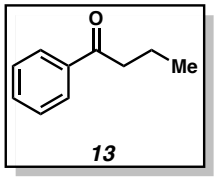
======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7.971
7.956
7.568
7.553
7.538
7.475
7.459
7.444

2.967
2.952
2.938
1.814
1.799
1.784
1.770
1.755
1.740
1.024
1.009
0.995



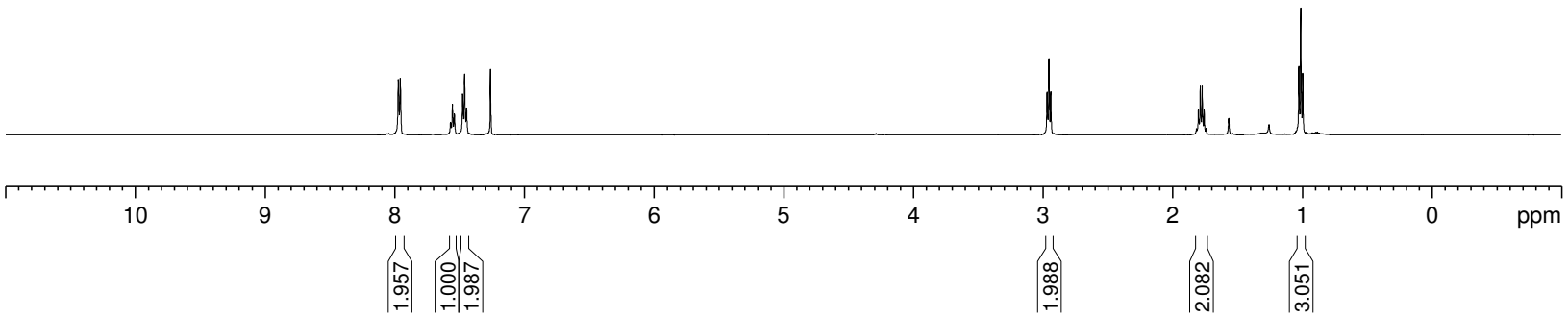
13

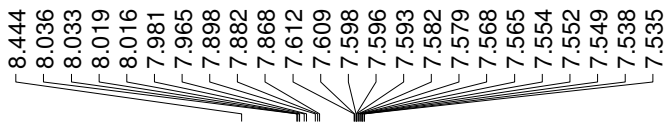
Current Data Parameters
NAME BJS-2-194-PREP2
EXPNO 1
PROCNO 1

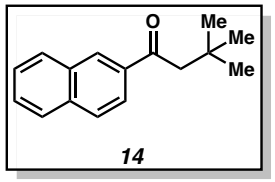
F2 - Acquisition Parameters
Date_ 20151201
Time 17.48
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 161.3
DW 50.000 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300221 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00







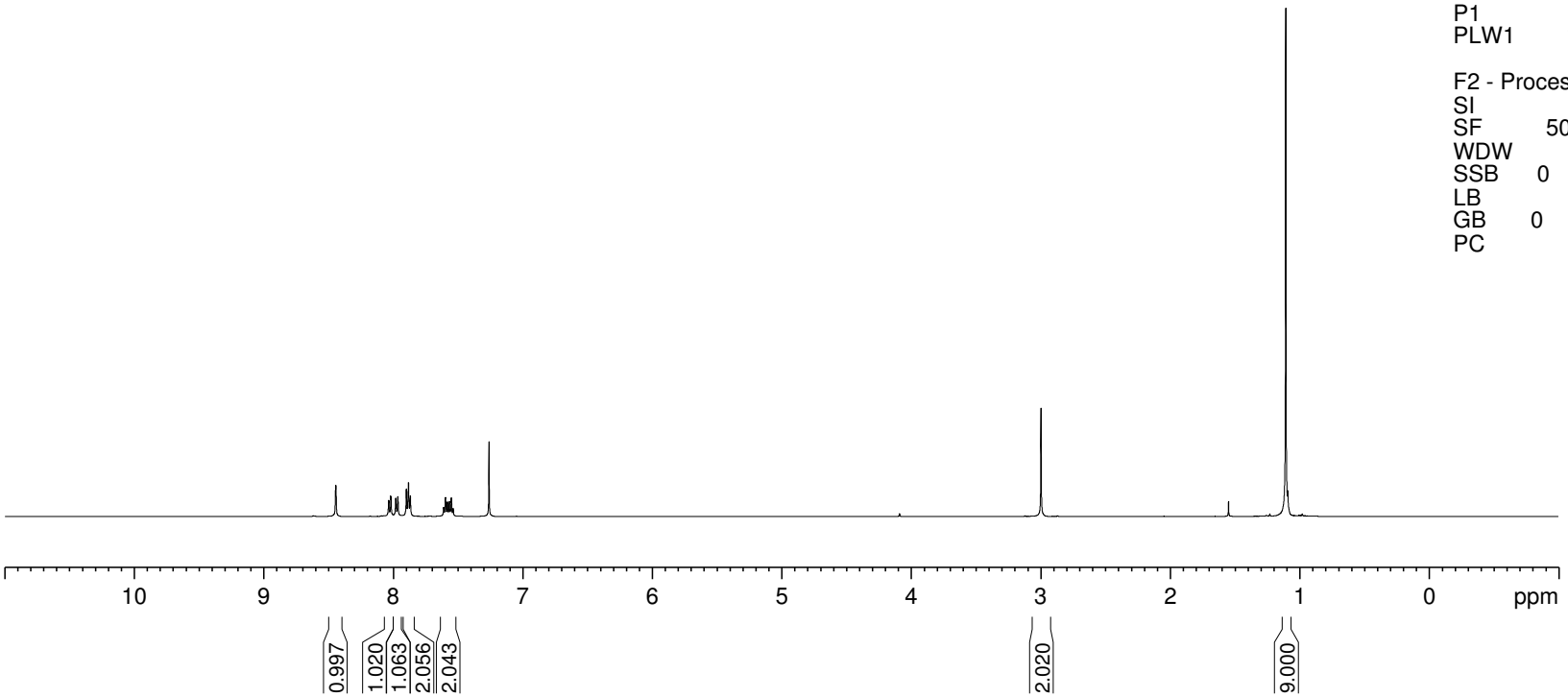
14

Current Data Parameters
NAME NAW-4-210p
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151130
Time 10.52
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

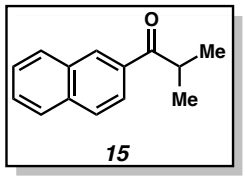
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



8.477
8.045
8.042
8.028
8.025
7.979
7.963
7.916
7.898
7.890
7.874
7.616
7.614
7.600
7.586
7.584
7.570
7.568
7.554
7.540
7.538

3.778
3.765
3.751
3.737
3.724
3.710
3.696

1.292
1.278



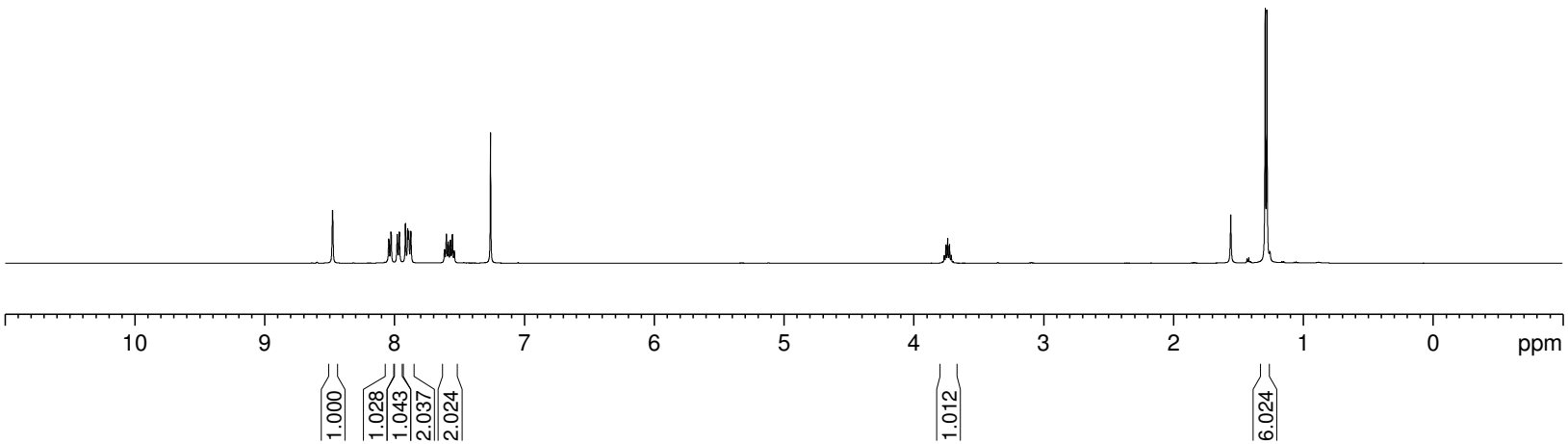
15

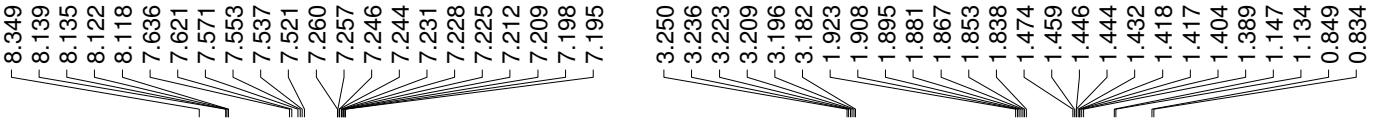
Current Data Parameters
NAME NAW-4-203p
EXPNO 5
PROCNO 1

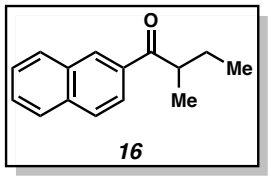
F2 - Acquisition Parameters
Date_ 20151125
Time 13.02
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00







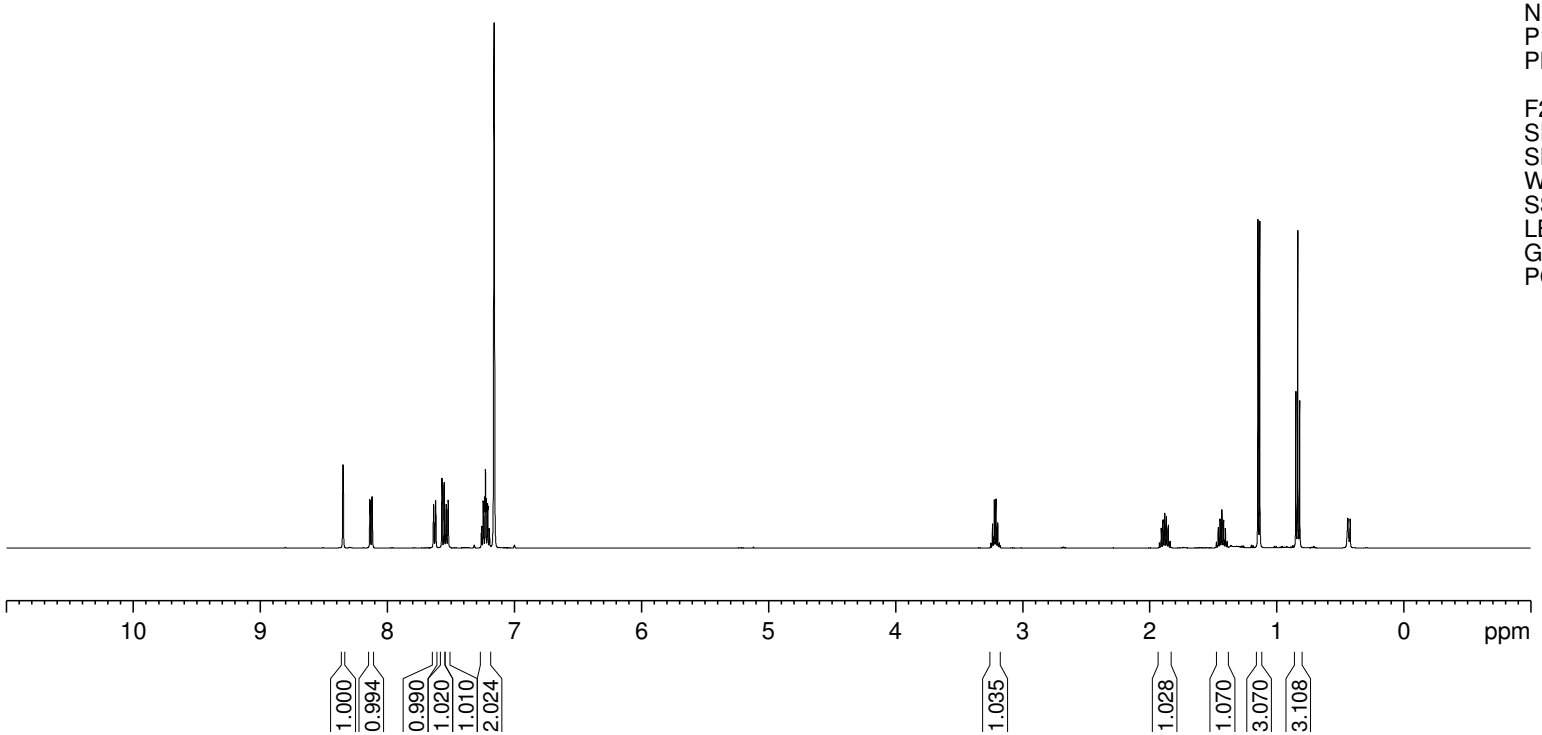
16

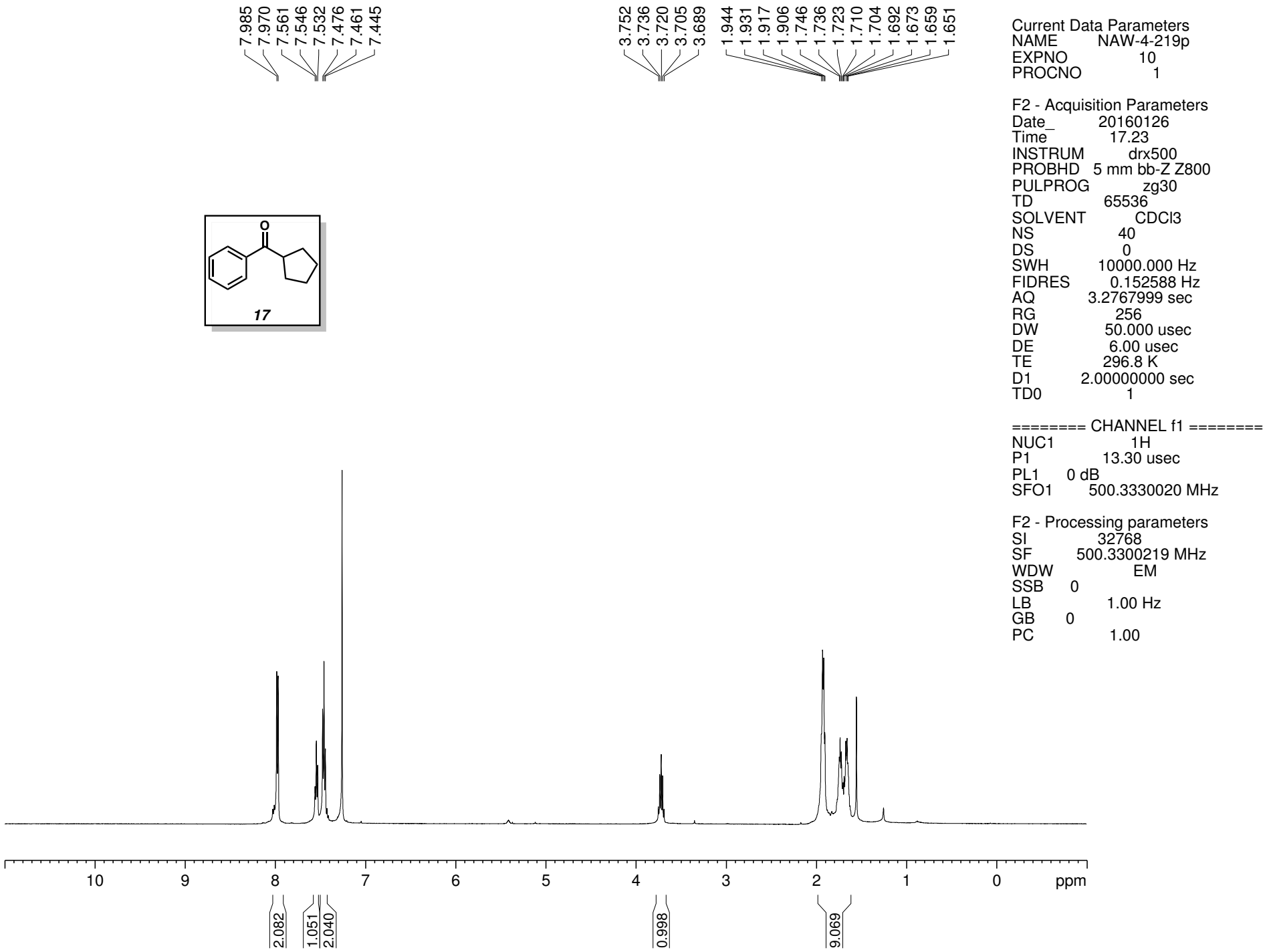
Current Data Parameters
NAME NAW-4-206-4-1-2
EXPNO 1
PROCNO 1

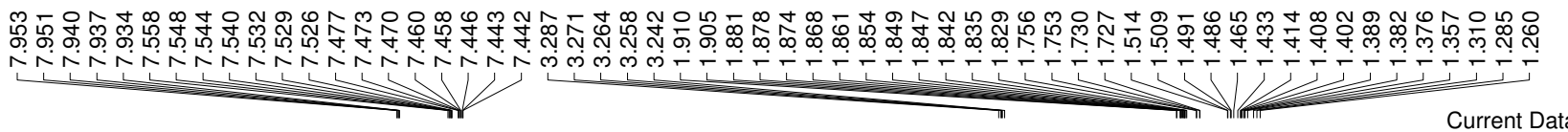
F2 - Acquisition Parameters
Date_ 20160114
Time 14.22
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

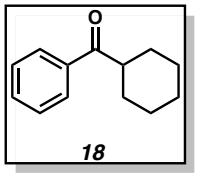
======== CHANNEL f1 =====
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1299958 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




7.985
7.970
7.561
7.546
7.532
7.476
7.461
7.445
3.752
3.736
3.720
3.705
3.689
1.944
1.931
1.917
1.906
1.746
1.736
1.723
1.710
1.704
1.692
1.673
1.659
1.651
O
17
Current Data Parameters
NAME NAW-4-219p
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160126
Time 17.23
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 40
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 256
DW 50.000 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300219 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.082
1.051
2.040
0.998
9.069



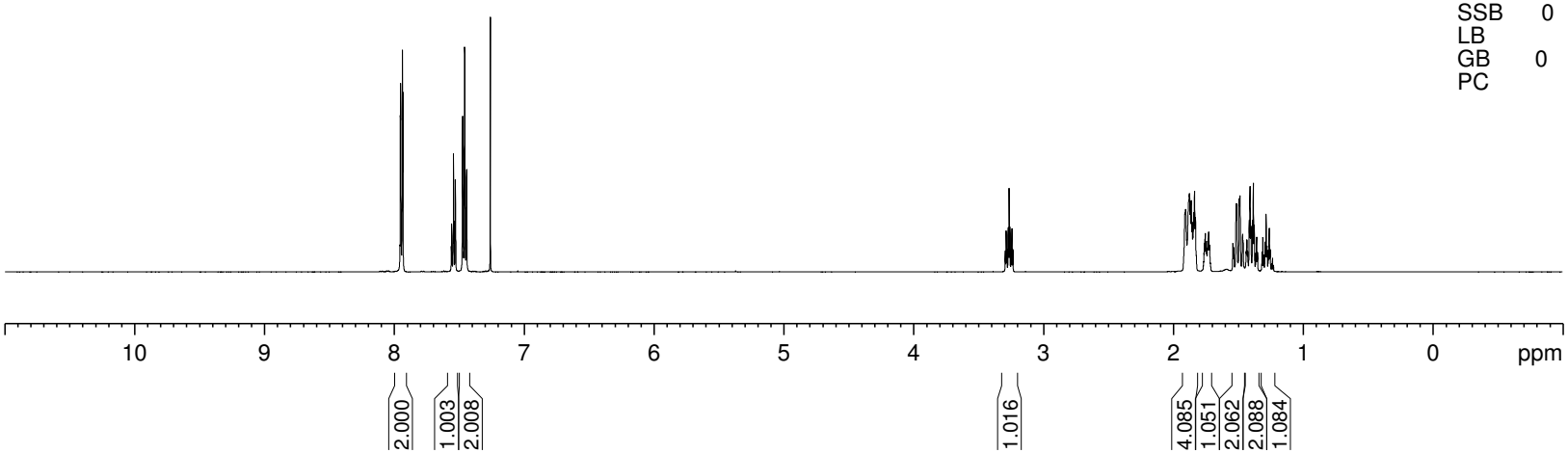


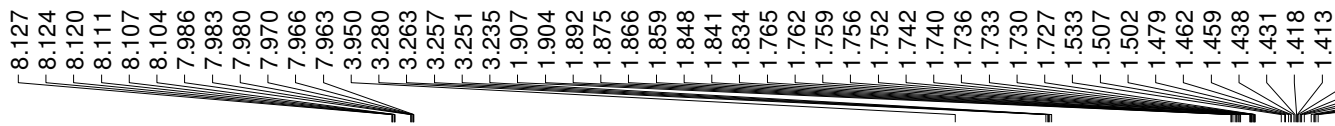
Current Data Parameters
NAME JED-1-144p
EXPNO 1
PROCNO 1

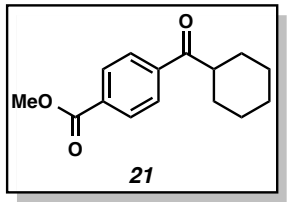
F2 - Acquisition Parameters
Date_ 20151129
Time 10.33
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





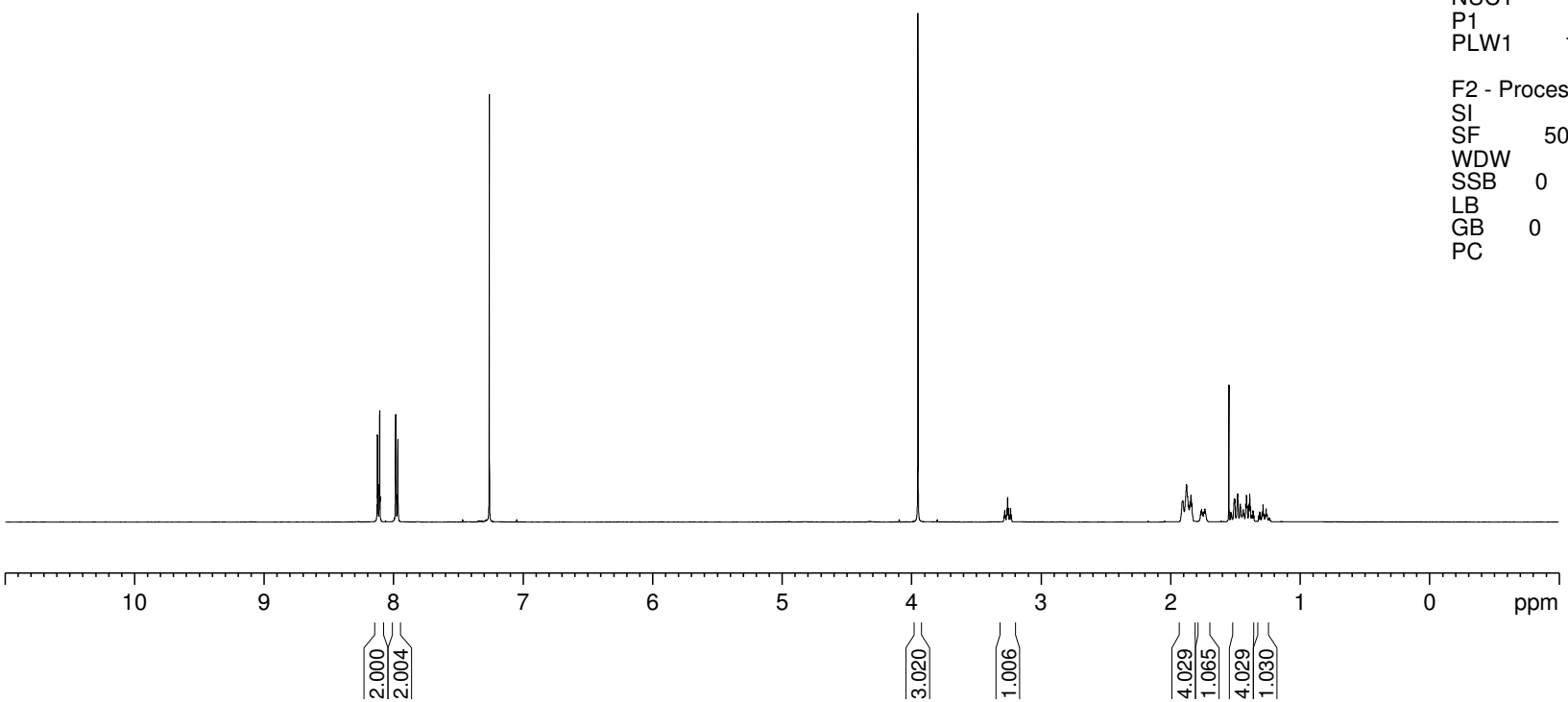


Current Data Parameters
NAME NAW-4-223p
EXPNO 1
PROCNO 1

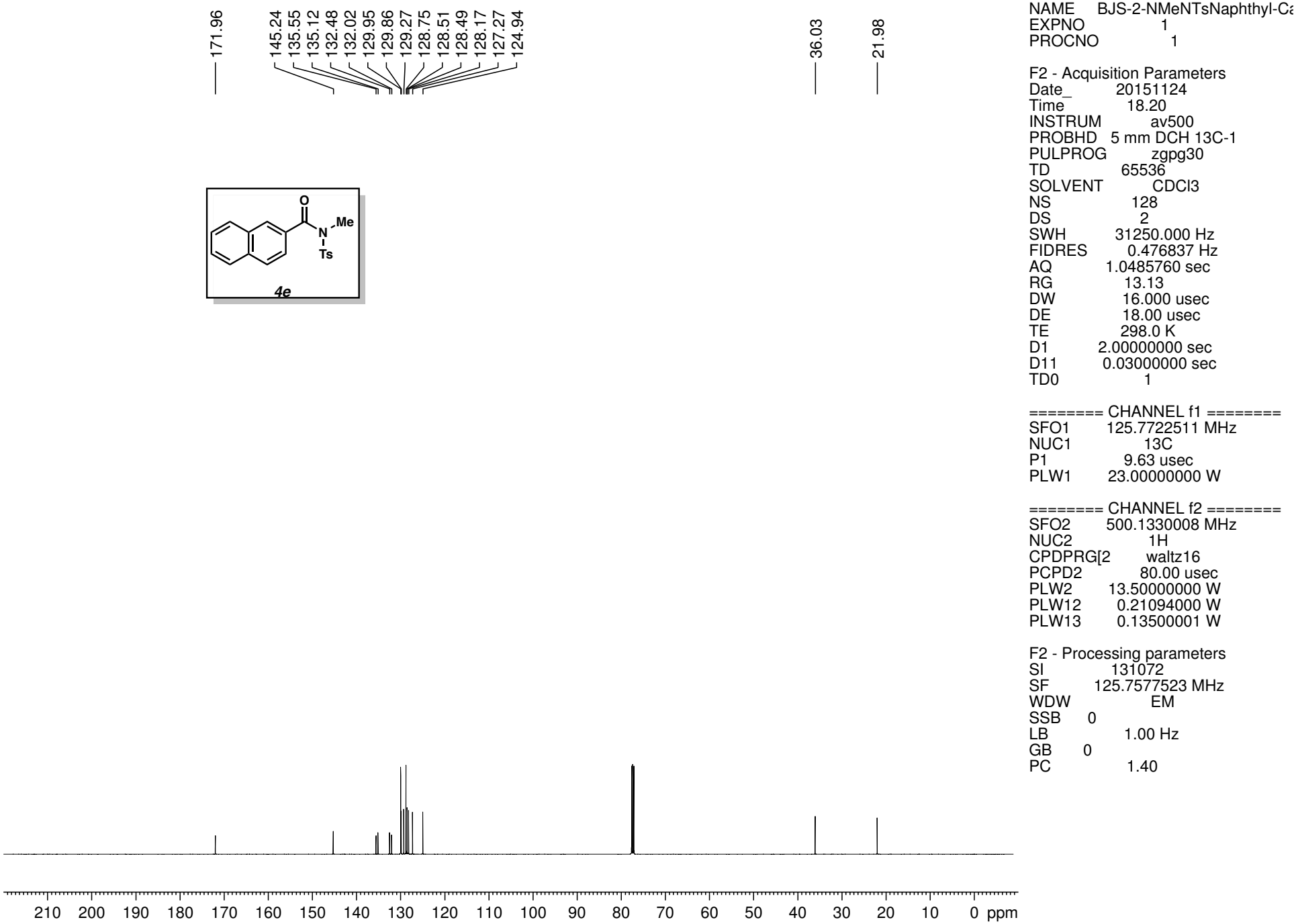
F2 - Acquisition Parameters
Date_ 20151209
Time 14.41
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



^{13}C NMR Spectra



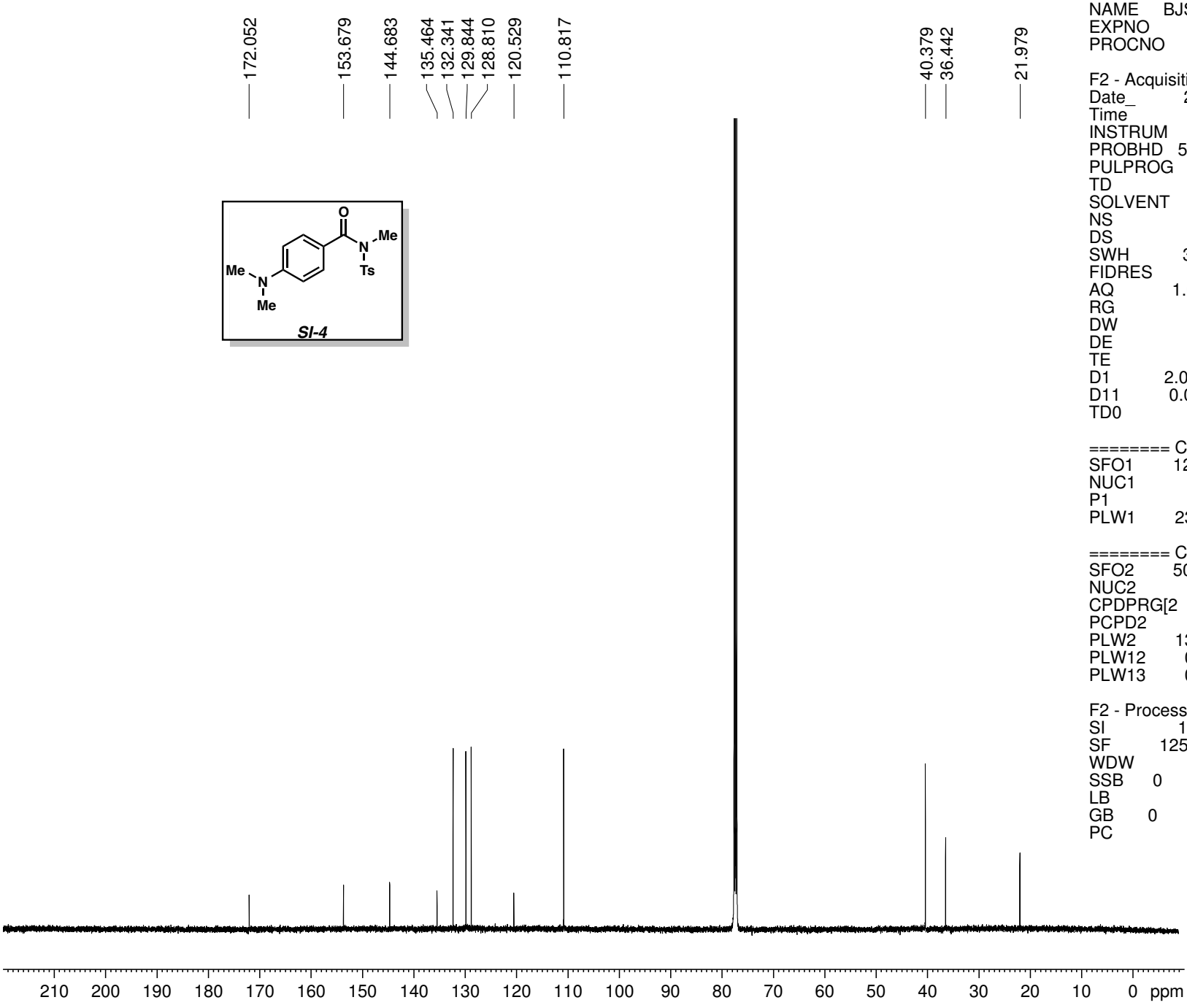
Current Data Parameters
NAME BJS-2-NMeNTsNaphthyl-Ca
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151124
Time 18.20
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577523 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



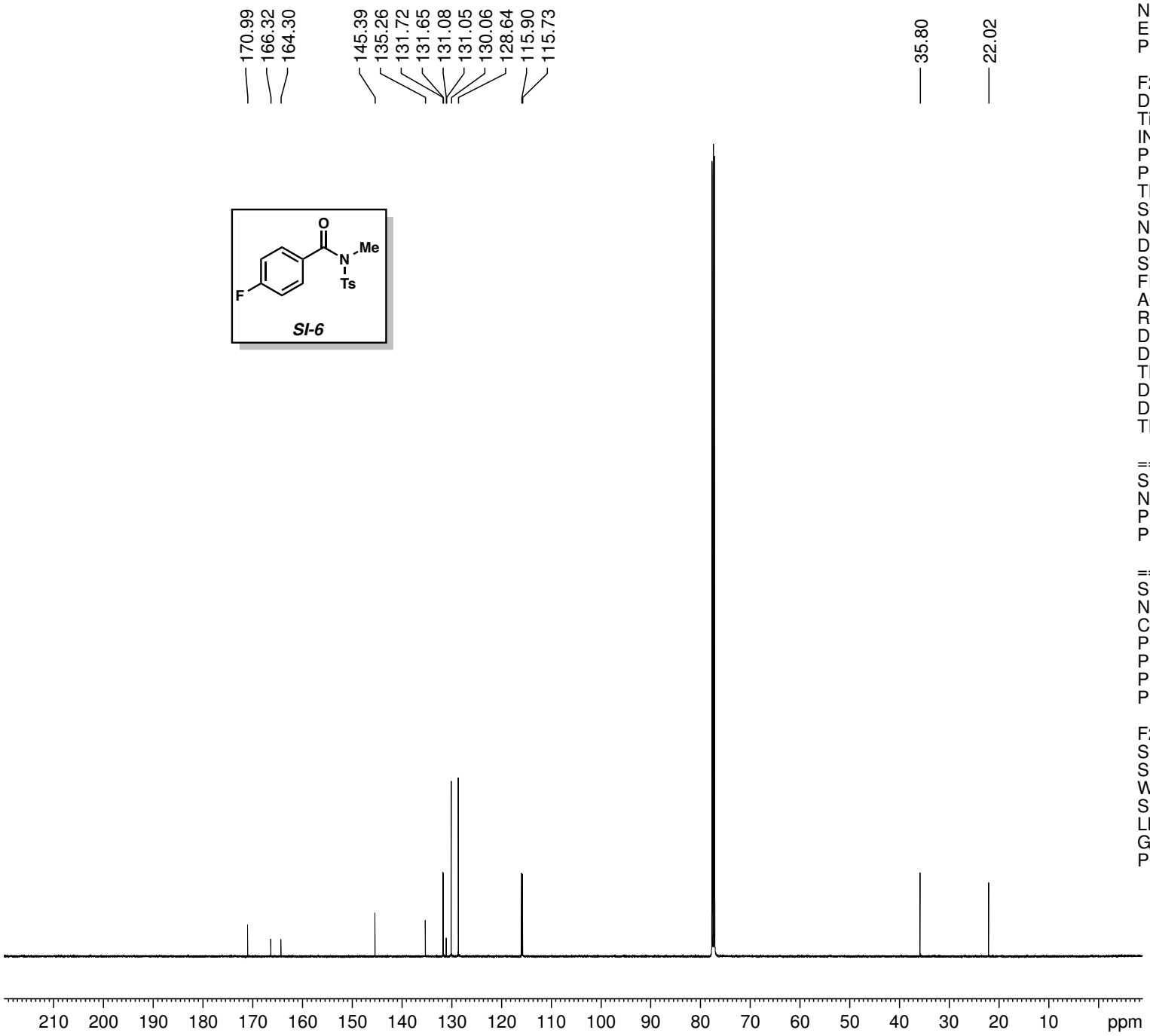
Current Data Parameters
NAME BJS-2-paraDimethylaminoN
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151122
Time 14.51
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 12.14
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577467 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



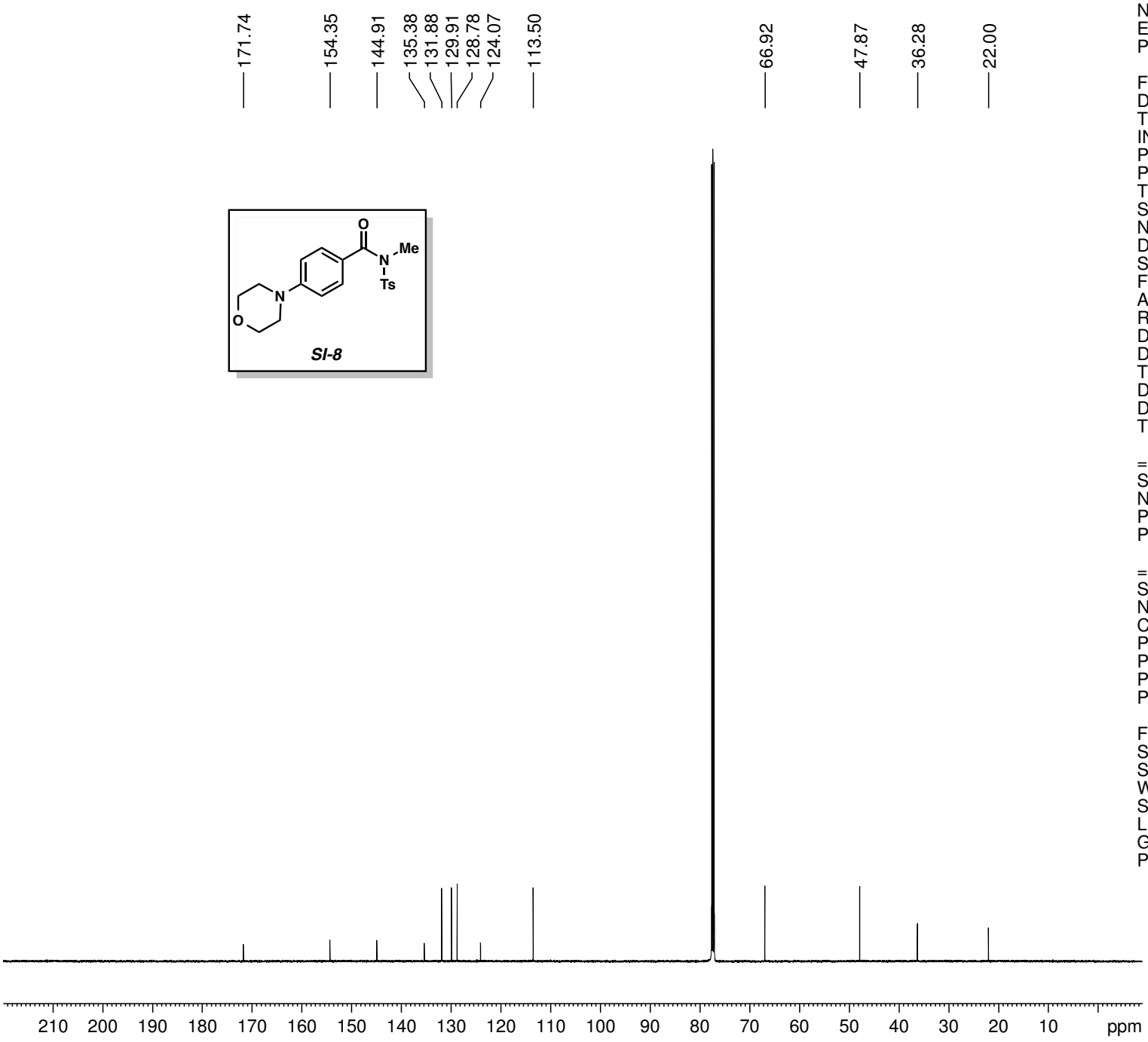
Current Data Parameters
NAME BJS-2-paraFluoroNMeNTsE
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151123
Time 9.11
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 17.16
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577471 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



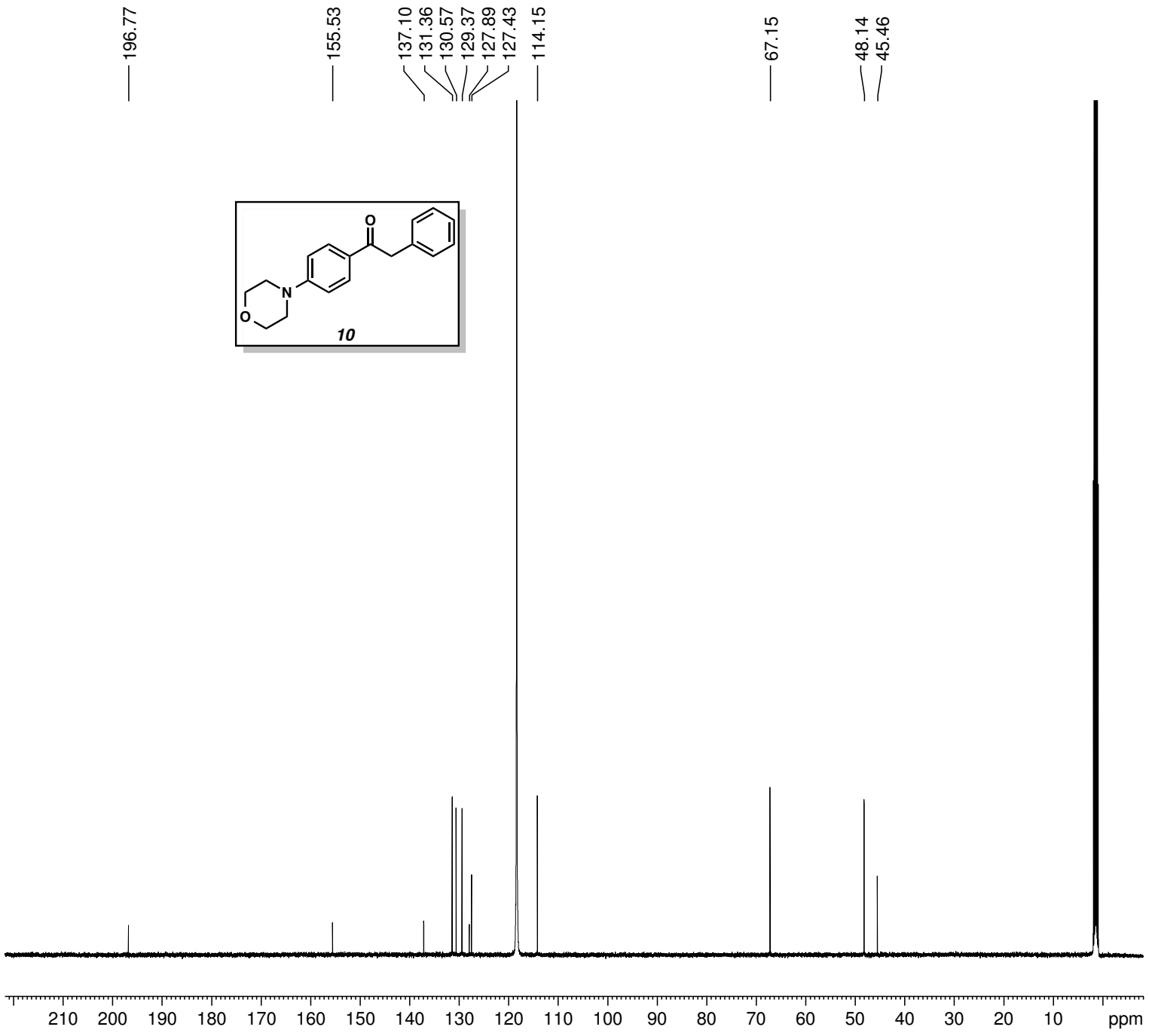
Current Data Parameters
NAME BJS-2-paraMorpholineNMel
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151123
Time 9.22
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 17.16
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7577469 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



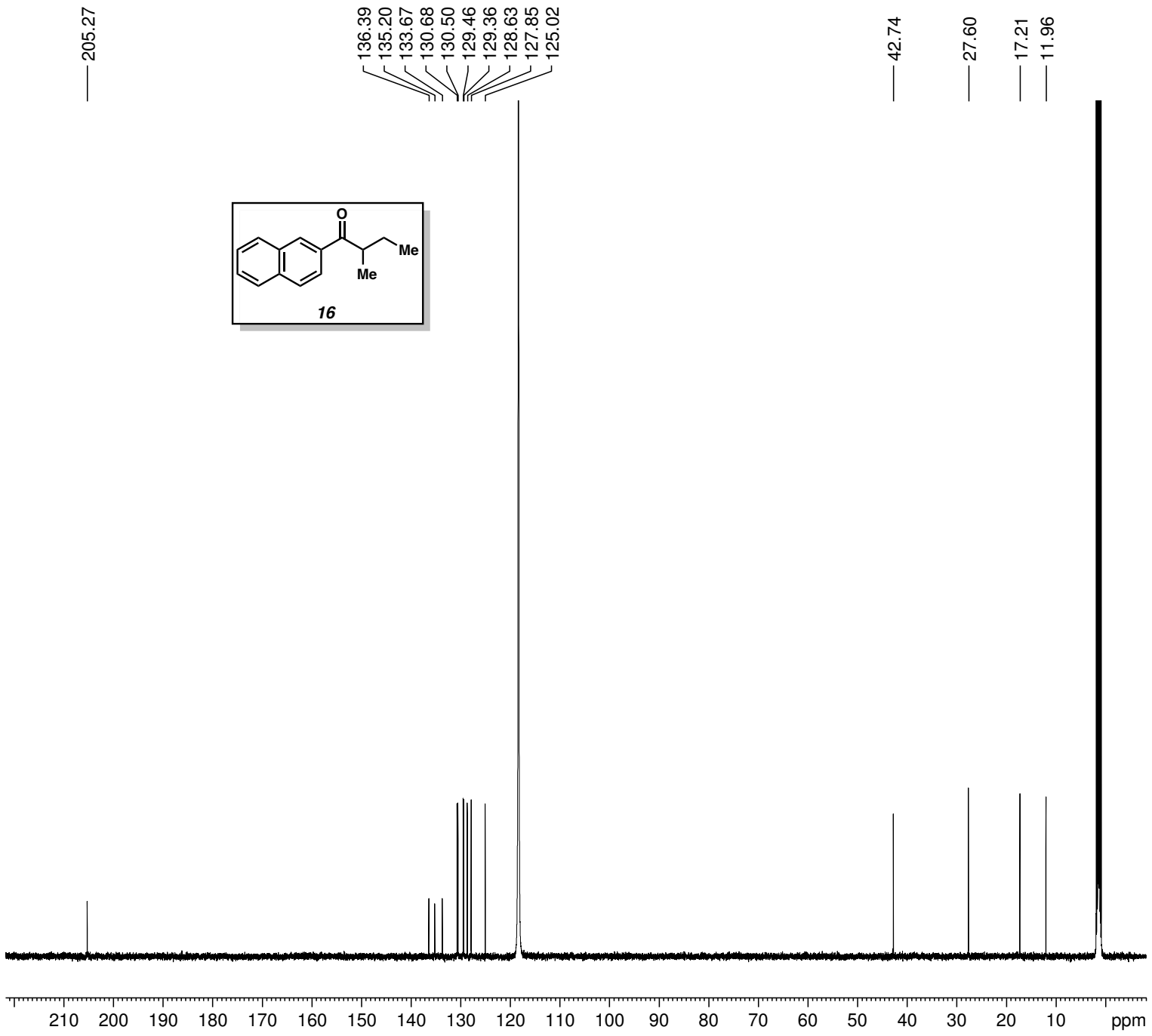
Current Data Parameters
NAME JED-1-164-CHLORO-CARE
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160106
Time 16.27
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 32
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 14.67
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7576653 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME NAW-4-206-4-1-CARBON2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160115
Time 10.00
INSTRUM av500
PROBHD 5 mm DCH 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W

======== CHANNEL f2 ========
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W

F2 - Processing parameters
SI 131072
SF 125.7576650 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40