

Supporting Information

Ene-hydrazide from Enol Triflate for the Regioselective Fischer Indole Synthesis

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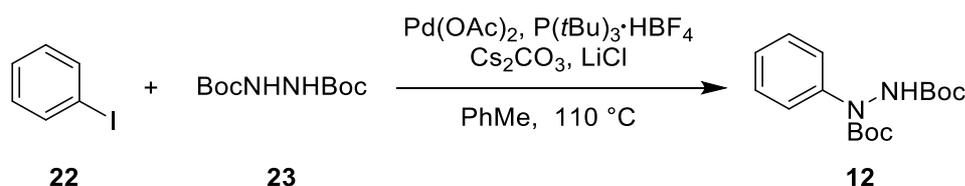
Experimental Section

General Methods

Reactions were carried out in oven or flame-dried glassware under argon atmosphere, unless otherwise noted. Tetrahydrofuran, dichloromethane, toluene, and ethyl ether were dried by passing through activated alumina columns (Grubbs column). Degassing was carried out by standard freeze-pumping-thaw cycle (FPT). All other commercially obtained reagents and solvent were used as received. Reactions were magnetically stirred and monitored by thin layer chromatography (TLC) using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm). The TLC spots were visualized under UV lamp or using staining solutions such as ceric ammonium molybdate solution, potassium permanganate solution, *p*-anisaldehyde solution and ninhydrine solution. Flash column chromatography was performed with indicated solvents using silica gel (GS60 – 40/75) purchased from Fuji Silysia Chemical. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise stated. ¹H NMR spectra were recorded at 400 MHz and ¹³C NMR spectra at 100 MHz using a Varian Mercury 400 instrument. Chemical shifts were reported relative to TMS (0 ppm) or internal chloroform ($\delta = 7.26$) for ¹H NMR and internal chloroform ($\delta = 77.16$) for ¹³C NMR. Infrared spectra were recorded on an ABB FTLA2000 FTIR spectrometer. CEM DISCOVER SYSTEM (MODEL NO: 908005) microwave oven was used.

I. Synthetic procedures for aryl hydrazides

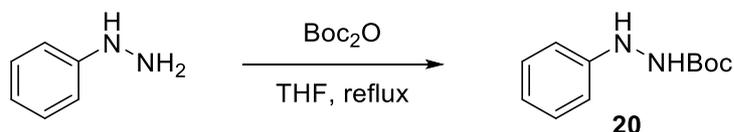
1) Preparation of di-*tert*-butyl 1-phenylhydrazine-1,2-dicarboxylate **12**



N,N'-bis-Boc-phenyl hydrazide **12** was prepared based on the method we reported previously after modification.¹ To a sealed tube were charged iodobenzene (0.06 mL, 0.5 mmol, 1.0 equiv), di-*tert*-butyl hydrazine-1,2-dicarboxylate **23** (93 mg, 0.4 mmol, 0.8 equiv), Pd(OAc)₂ (6 mg, 0.025 mmol, 5 mol%), P(*t*-Bu)₃·HBF₄ (15 mg, 0.05 mmol, 10 mol%), Cs₂CO₃ (228 mg, 0.7 mmol, 1.4 equiv.) and dry toluene (1.5 mL) at room temperature. The mixture solution was degassed, charged with Ar and heated at 110 °C until all di-*tert*-butyl hydrazine-1,2-dicarboxylate disappeared on TLC (~12 h). The reaction mixture was then cooled to rt and filtered through a plug of Celite with ethyl acetate. The organic solution was concentrated and purified by flash column chromatography on silica gel (hexane:ethyl acetate = 10:1) to afford hydrazide **12** (102 mg, 82% yield) as a pale yellow solid. ¹H

NMR (400 MHz, CDCl₃) δ 7.40 (m, 2H), 7.33 – 7.29 (m, 2H), 7.17 – 7.13 (m, 1H), 6.81/6.60 (rotamers, 2 x br s, 1H, NHBoc), 1.49 (s, 18H); ¹³C NMR (100 MHz, CDCl₃) δ 155.5, 153.7, 142.3, 128.5, 125.6, 123.8, 82.3, 81.6, 28.31, 28.26.

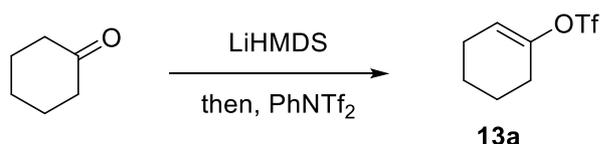
2) Preparation of *tert*-butyl 2-phenylhydrazine-1-carboxylate **20**



1-Boc-2-phenylhydrazine **20** was prepared according to the literature method.² A microwave vial was charged with di-*tert*-butyl dicarbonate (1.56 g, 9.6 mmol, 1.2 equiv) and anhydrous THF (0.5 mL). To the vial was added phenylhydrazine (0.8 mL, 8.0 mmol) dropwise at 0 °C. After sealing, the mixture was heated under microwave irradiation for 30 min (program; run time = 1 min, hold time = 30 min, power = 300 W, ramp to temperature = 75 °C, stirring = high). After the completion of the reaction, the reaction mixture was concentrated under reduced pressure to afford the crude product as a yellow solid, which was subsequently recrystallized from CH₂Cl₂/hexane to give hydrazide **20** (1.42 g, 85% yield) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.24 -7.20 (m, 2H), 6.89 – 6.86 (m, 1H), 6.82 – 6.80 (m, 2H), 6.43 (br s, 1H), 5.77 (br s, 1H), 1.46 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 156.4, 148.5, 129.1, 120.7, 113.0, 81.1, 28.3.

II. Synthetic procedures for enol triflates

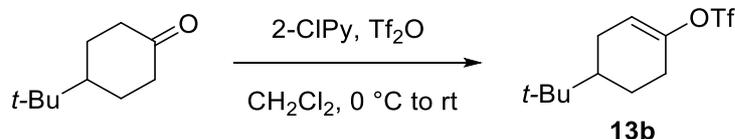
1) Preparation of cyclohex-1-en-1-yl trifluoromethanesulfonate **13a**



[Method A]³: To solution of cyclohexanone (0.07 mL, 0.65 mmol) in anhydrous THF (3 mL) was added LiHMDS (0.78 mL, 1.0 M in THF, 1.2 equiv) at -78 °C. After 1 h at -78 °C under an argon atmosphere, a solution of PhNTf₂ (325 mg, 0.91 mmol, 1.4 equiv) in anhydrous THF (3.5 mL) was slowly added to the mixture solution at -78 °C. The mixture solution was allowed to warm to rt over a period of 4 h and stirred for additional 12 h. After the completion of the reaction, the resulting solution was treated with saturated aqueous NH₄Cl (1.0 ml) and extracted with Et₂O (3 x 10 mL). The combined organic solution was washed with saturated aqueous Na₂CO₃ (1.0 mL), dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue oil was purified by flash column chromatography on silica gel (100% pentane) to afford the product **13a** (126 mg, 84% yield) as a pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 5.76 – 5.74 (m, 1H), 2.32 – 2.29 (m, 2H), 2.19 – 2.14 (m, 2H), 1.80 – 1.74 (m, 2H), 1.62 – 1.56 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 149.5,

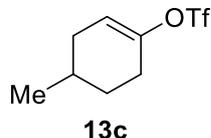
118.7 (q, $J_{19F-13C} = 318$ Hz), 118.6, 27.7, 24.0, 22.8, 21.1.

2) Preparation of 4-*tert*-butylcyclohex-1-en-1-yl trifluoromethanesulfonate **13b**



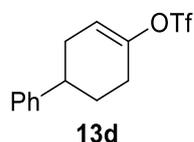
[Method B]⁴: To a solution of 4-*tert*-butylcyclohexanone (162 mg, 1.1 mmol) in anhydrous CH_2Cl_2 (5 mL) was added 2-chloropyridine (0.11 mL, 1.2 mmol, 1.1 equiv) at 0 °C. After 10 min at 0 °C, trifluoromethanesulfonic anhydride (0.21 mL, 1.3 mmol, 1.2 equiv) was added dropwise. The resulting solution was warmed to rt and stirred for 2 h under an argon atmosphere. After the completion of the reaction, the solution was concentrated and the resulting cherry-color oil was purified by flash column chromatography on silica gel (100% pentane) to afford the product **13b** (255 mg, 81% yield) as a pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.75 – 5.74 (m, 1H), 2.43 – 2.29 (m, 2H), 2.23 – 2.18 (m, 1H), 1.97 – 1.95 (m, 2H), 1.42 – 1.26 (m, 2H), 0.90 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.4, 118.7 (q, $J_{19F-13C} = 318$ Hz), 118.6, 43.0, 32.1, 28.7, 27.3, 25.4, 24.2.

3) Preparation of 4-methylcyclohex-1-en-1-yl trifluoromethanesulfonate **13c**



Method B, pale yellow oil, 78% yield. ^1H NMR (400 MHz, CDCl_3) δ 5.71 (s, 1H), 2.37 (s, 1H), 2.31 – 2.22 (m, 2H), 1.82 – 1.73 (m, 3H), 1.48 – 1.38 (m, 1H), 0.98 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.3, 118.7 (q, $J_{19F-13C} = 318$ Hz), 118.0, 32.0, 30.6, 28.0, 27.4, 20.6.

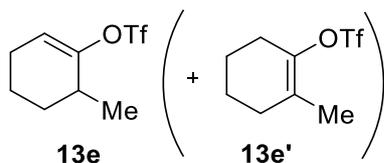
4) Preparation of 4-phenylcyclohex-1-en-1-yl trifluoromethanesulfonate **13d**



Method B, pale yellow oil, 82% yield. ^1H NMR (400 MHz, CDCl_3) δ 7.33 – 7.30 (m, 2H), 7.23 – 7.19 (m, 3H), 5.84 – 5.82 (m, 1H), 2.86 – 2.79 (m, 1H), 2.56 – 2.27 (m, 4H), 2.07 – 2.02 (m, 1H), 1.94 (dddd, $J = 13.1, 11.6, 10.5, 5.6$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.1, 144.7, 128.1, 126.9, 126.7, 118.7 (q, $J_{19F-13C} = 318$ Hz), 118.2, 38.8, 31.6, 29.7, 27.9.

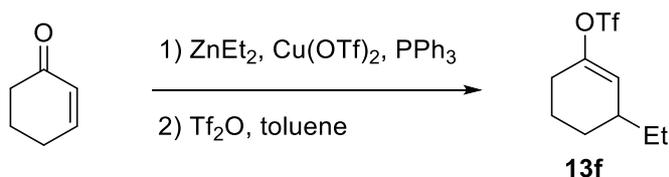
5) Preparation of 6-methylcyclohex-1-en-1-yl trifluoromethanesulfonate **13e** (contains small

amount of 2- methylcyclohex-1-en-1-yl trifluoromethanesulfonate **13e'**)



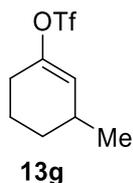
Method A, 89% total yield (**13e:13e'** = 30:1 by ^1H NMR), pale yellow oil. ^1H and ^{13}C NMR data of **13e** and **13e'** were deduced from the spectra of the mixture. **13e**. ^1H NMR (400 MHz, CDCl_3) δ 5.73 – 5.72 (m, 1H) 2.55 – 2.53 (m, 1H), 2.18 – 2.16 (m, 2H), 1.96 – 1.90 (m, 1H), 1.69 – 1.60 (m, 1H), 1.60 – 1.52 (m, 1H), 1.50 – 1.42 (m, 1H), 1.14 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.5, 118.7 ($J_{19\text{F}-13\text{C}} = 318$ Hz), 118.4, 32.5, 31.6, 24.6, 19.4, 17.9. **13e'**. ^1H NMR (400 MHz, CDCl_3) δ 2.30 (m, 2H), 2.21 (m, 2H), 1.75 (m, 4H), 1.55 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 30.8, 27.7, 23.4, 21.9, 16.8.

6) Preparation of 3-ethylcyclohex-1-en-1-yl trifluoromethanesulfonate **13f**



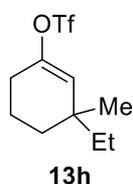
[Method C]⁵: To a sealed tube equipped with a septum and stirring bar were added $\text{Cu}(\text{OTf})_2$ (7.5 mg, 0.02 mmol, 2 mol%), PPh_3 (10.8 mg, 0.04 mmol, 4 mol%) and anhydrous toluene (5 mL). After stirring under an argon atmosphere for 0.5 h at rt, 2-cyclohexen-1-one **25f** (0.1 mL, 1.03 mmol) was added. The resulting solution was cooled to -30 °C and diethylzinc (1.21 mL of 15 wt. % solution in toluene, 1.34 mmol, 1.3 equiv) was added dropwise. After stirring under an argon atmosphere for 2 h, trifluoromethanesulfonic anhydride (0.35 mL, 2.06 mmol, 2.0 equiv) was added at 0 °C. The resulting solution was allowed to warm to rt and stirred for 12 h. After the completion of the reaction, the resulting solution was treated with saturated aqueous NaHCO_3 (2.0 mL), and extracted with Et_2O (3 x 10 mL). The combined organic solution was dried over anhydrous MgSO_4 , filtered and concentrated under reduced pressure. The residue oil was purified by flash column chromatography on silica gel (100% pentane) to afford the product **13g** (189 mg, 71% yield) as a pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.68 (s, 1H), 2.36 – 2.29 (m, 2H), 2.22 – 2.21 (m, 1H), 1.93 – 1.85 (m, 1H), 1.81 – 1.74 (m, 1H), 1.72 – 1.62 (m, 1H), 1.48 – 1.35 (m, 2H), 1.32 – 1.18 (m, 1H), 0.93 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.5, 122.8, 118.7 (q, $J_{19\text{F}-13\text{C}} = 319$ Hz), 36.2, 28.3, 27.9, 27.2, 21.6, 11.3.

7) Preparation of 3-methylcyclohex-1-en-1-yl trifluoromethanesulfonate **13g**



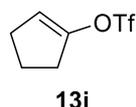
Method C, 69% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.62 (s, 1H), 2.42 – 2.37 (m, 1H), 2.32 – 2.24 (m, 2H), 1.93 – 1.85 (m, 1H), 1.81 – 1.72 (m, 1H), 1.71 – 1.63 (m, 1H), 1.23 – 1.16 (m, 1H), 1.05 (d, $J = 7.2$, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.3, 124.2, 118.7 (q, $J_{19\text{F}-13\text{C}} = 319$ Hz), 30.0, 29.8, 27.6, 21.5, 21.0.

8) Preparation of 3-ethyl-3-methylcyclohex-1-en-1-yl trifluoromethanesulfonate 13h



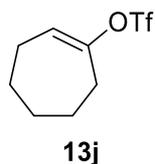
Method C, 72% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.51 (s, 1H), 2.29 -2.26 (m, 2H), 1.82 – 1.75 (m, 2H), 1.52 – 1.45 (m, 1H), 1.42 -1.31 (m, 3H), 1.01 (s, 3H), 0.86 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.7, 127.2, 118.7 (q, $J_{19\text{F}-13\text{C}} = 319$ Hz), 36.2, 34.7, 33.0, 27.8, 26.3, 19.6, 8.3.

9) Preparation of cyclopent-1-en-1-yl trifluoromethanesulfonate 13i



Method A, 67% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.63 (s, 1H), 2.59 – 2.56 (m, 2H), 2.44 – 2.40 (m, 2H), 2.07 – 2.00 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.7, 118.7 (q, $J_{19\text{F}-13\text{C}} = 319$ Hz), 117.9, 30.9, 28.1, 20.9.

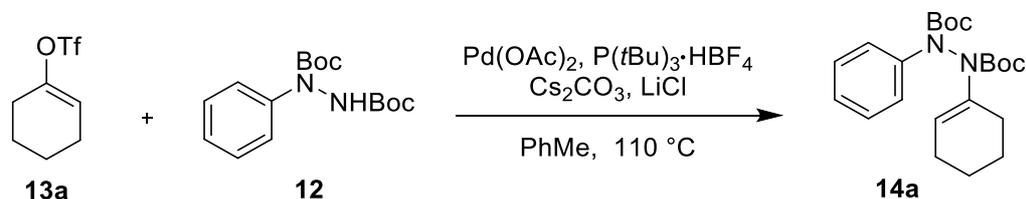
10) Preparation of cyclohept-1-en-1-yl trifluoromethanesulfonate 13j



Method A, 62% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.88 (t, $J = 6.5$ Hz, 1H), 2.53 – 2.51 (m, 2H), 2.18 – 2.14 (m, 2H), 1.74 1.67 (m, 4H) 1.66– 1.61 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.3, 123.2, 118.7 (q, $J_{19\text{F}-13\text{C}} = 318$ Hz), 33.3, 30.0, 26.4, 24.9, 24.8.

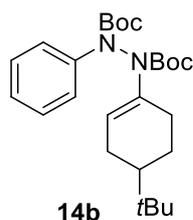
III. General synthetic procedure for ene-hydrazide

1) Synthesis of di-*tert*-butyl 1-(cyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate (**14a**)



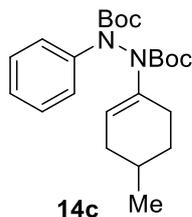
To a sealed tube were charged 1-cyclohexenyl trifluoromethanesulfonate **13a** (300 mg, 1.30 mmol), di-*tert*-butyl 1-phenylhydrazine-1,2-dicarboxylate **12** (482 mg, 1.56 mmol, 1.2 equiv), Pd(OAc)₂ (15 mg, 0.07 mmol, 5 mol%), P(*t*Bu)₃·HBF₄ (38 mg, 0.13 mmol, 10 mol%), Cs₂CO₃ (847 mg, 2.60 mmol, 2.0 equiv), LiCl (386 mg, 9.1 mmol, 7.0 equiv), and anhydrous toluene (1.5 mL) at rt. The reaction mixture was degassed, charged with Ar and heated at 110 °C for 9 h. After the completion of the reaction, the resulting solution was cooled to rt and filtered through a plug of Celite with ethyl acetate. The organic solution was concentrated and purified by flash column chromatography on silica gel (hexane:ethyl acetate = 20:1) to afford ene-hydrazide **14a** (490 mg, 97% yield) as a pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.36 (m, 2H), 7.33 – 7.29 (m, 2H), 7.17 – 7.13 (m, 2H), 5.63 (br s, 1H), 2.26 – 2.05 (m, 4H), 1.67 – 1.61 (m, 4H), 1.52 (s, 9H), 1.49/1.48 (rotamers, 2 x s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 153.3, 141.5, 137.4, 128.5, 125.5, 123.8/123.3, 121.5, 81.9, 81.5, 28.4, 28.3, 26.4, 24.6, 22.9, 21.8; FT-IR (CH₂Cl₂) 2978, 2934, 1722, 1369, 1339, 1159 cm⁻¹; HRMS (ESI) Calcd for C₂₂H₃₂N₂NaO₄ [M+Na]⁺ 411.2255, found 411.2254.

2) di-*tert*-Butyl 1-(4-(*tert*-butyl)cyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate **14b**



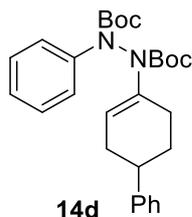
Prepared by the method used for **14a**. 87% yield, pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.36 (m, 2H), 7.33 – 7.30 (m, 2H), 7.17 – 7.13 (m, 1H), 5.67/5.61 (rotamers, 2 x br s, 1H), 2.37 – 2.02 (m, 3H), 1.83 – 1.64 (m, 2H), 1.52 (s, 9H), 1.50/1.48 (rotamers, 2 x br s, 9H), 0.85/0.84 (rotamers, 2 x s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 153.4, 141.4, 137.2, 128.47/128.44, 125.5, 123.1, 122.1, 81.90/81.86, 81.47, 43.5/43.3, 32.2, 28.33, 28.31, 28.29, 27.3/27.2, 26.09/26.04, 24.2; FT-IR (CH₂Cl₂) 2966, 1723, 1368, 1344, 1160 cm⁻¹; HRMS (ESI) Calcd for C₂₆H₄₀N₂NaO₄ [M+Na]⁺ 467.2881, found 467.2877.

3) di-tert-Butyl 1-(4-methylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate 14c



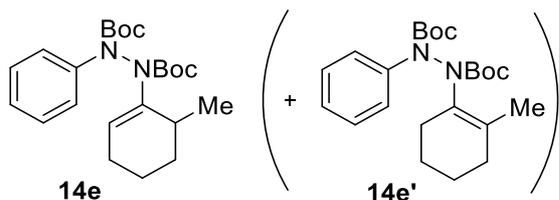
Prepared by the method used for **14a**. 79% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (m, 2H), 7.33 – 7.29 (m, 2H), 7.17 – 7.13 (m, 1H), 5.61 (m, 1H), 2.30 (m, 1H), 2.18 – 2.09 (m, 2H), 1.78 – 1.66 (m, 3H), 1.52 (s, 9H), 1.49/1.48 (rotamers, 2 x s, 9H), 0.93 (d, $J = 5.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.3, 141.4, 137.1, 128.5, 125.5, 123.1, 120.7, 81.91/81.88, 81.4, 33.0/ 32.9, 31.1/31.0, 28.34, 28.32/28.31, 27.9/27.7, 26.4/26.1, 21.4; FT-IR (CH_2Cl_2) 2977, 2929, 1722, 1369, 1343, 1156 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{34}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 425.2411, found 425.2405.

4) di-tert-Butyl 1-(4-phenylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate 14d



Prepared by the method used for **14a**. 84% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.40 (m, 2H), 7.34 – 7.31 (m, 2H), 7.28 – 7.24 (m, 2H), 7.18 – 7.17 (m, 4H), 5.75 (br s, 1H), 2.81 – 2.79 (m, 1H), 2.41- 2.32 (m, 3H), 2.26 – 2.16 (m, 1H), 2.01 – 1.96 (m, 1H), 1.83 – 1.81 (m, 1H), 1.52 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.2, 146.3, 141.3, 137.3, 128.44/128.38, 126.8, 126.2, 125.5, 122.9, 120.9, 120.2, 82.0/81.9, 81.6, 39.3/39.2, 32.5, 29.8, 28.2, 26.9/26.4; FT-IR (CH_2Cl_2) 2976, 2930, 1718, 1368, 1343, 1152 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{28}\text{H}_{36}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 487.2568, found 487.2558.

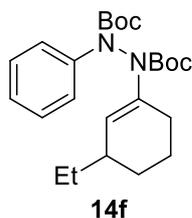
5) di-tert-Butyl 1-(6-methylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate 14e (contains small amount of di-tert-butyl 1-(2-methylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate **14e'**)



Prepared by the method used for **14a**. 86% total yield (**14e:14e'** = 30:1 estimated by ^1H NMR

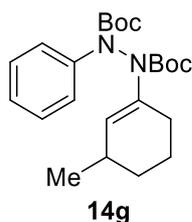
spectrum), pale yellow oil. ^1H and ^{13}C NMR data of **14e** were deduced from those of the mixture. ^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.37 (m, 1H), 7.30 – 7.27 (m, 3H), 7.20 – 7.13 (m, 1H), 5.77/5.53/5.44 (rotamers, 3 x br s, 1H), 2.66 – 2.51 (m, 1H), 2.25 – 2.02 (m, 2H), 1.67 – 1.21 (m, 22H), 0.91 (d, $J = 3.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.8, 141.0, 129.1, 128.4/128.3, 126.6, 125.9, 123.6, 123.0, 120.7, 113.4, 81.9, 81.4, 30.8, 29.7, 28.3, 25.0, 21.7, 19.4, 18.3, 17.9; FT-IR (CH_2Cl_2) 2977, 2931, 1723, 1368, 1345, 1160 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{34}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 425.2411, found 425.2413.

6) di-*tert*-Butyl 1-(3-ethylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate (**14f**)



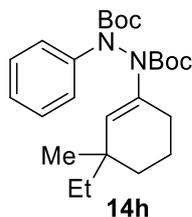
Prepared by the method used for **14a**. 92% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (m, 2H), 7.33 – 7.29 (m, 2H), 7.17 – 7.14 (m, 1H), 5.59/5.48 (rotamers, 2 x brs, 1H), 2.24 (m, 1H), 2.07 (m, 2H), 1.74 – 1.67 (m, 2H), 1.52 (s, 9H), 1.49/1.48 (rotamers, 2 x s, 9H), 1.39 – 1.22 (m, 3H), 1.19 – 1.11 (m, 1H), 0.88/0.80 (rotamers, 2 x t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.2, 141.4, 137.3, 128.4, 125.5, 123.4 (2 x C), 81.7, 81.27/81.22, 36.7/36.5, 28.8/28.7, 28.24, 28.19, 27.9/27.8, 26.7, 21.8, 11.4/11.2; FT-IR (CH_2Cl_2) 2976, 2932, 2859, 1723, 1368, 1342, 1322, 1159 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{24}\text{H}_{36}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 439.2568, found 439.2576.

7) di-*tert*-Butyl 1-(3-methylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate **14g**



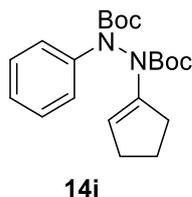
Prepared by the method used for **14a**. 87% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (m, 2H), 7.32- 7.29 (m, 2H), 7.17 – 7.13 (m, 1H), 5.51/5.44 (rotamers, 2 x br s, 1H), 2.26 – 2.00 (m, 4H), 1.74 – 1.72 (m, 2H), 1.51 (s, 9H), 1.48 (s, 9H), 1.35-1.28 (m, 1H), 1.13 – 1.08 (m, 1H), 0.97/0.91 (rotamers, 2 x d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.3, 141.5, 136.9, 128.5, 125.6, 123.2 (2 x C), 81.9, 81.5, 30.7/30.6, 30.2/30.1, 29.9, 28.4, 28.3, 26.5, 21.8/21.6; FT-IR (CH_2Cl_2) 2977, 2931, 2989, 1722, 1368, 1345, 1321, 1159 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{34}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 425.2411, found 425.2419.

8) di-*tert*-Butyl 1-(3-ethyl-3-methylcyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate **14h**



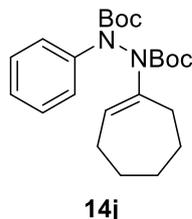
Prepared by the method used for **14a**. 81% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (m, 2H), 7.32 – 7.28 (m, 2H), 7.16 – 7.13 (m, 1H), 5.44/5.33/5.28 (rotamers, 3 x br s, 1H), 2.23 – 2.04 (m, 2H), 1.74 – 1.61 (m, 3H), 1.51 (s, 9H), 1.49/1.47 (rotamers, 2 x s, 9H), 1.32- 1.22 (m, 3H), 0.98 – 0.88 (m, 3H), 0.80/0.70 (rotamers, 2 x t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.3, 141.5, 136.4/136.1, 128.5, 125.6, 123.3 (2 x C), 81.8, 81.3, 35.1, 33.62/33.60, 30.7/30.6/30.1/29.9, 28.38, 28.32, 26.9, 21.8/21.6, 19.8, 8.6/8.4; FT-IR (CH_2Cl_2) 2974, 2933, 1723, 1368, 1344, 1322, 1162 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{25}\text{H}_{38}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 453.2724, found 453.2723.

9) di-*tert*-Butyl 1-(cyclopent-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate **14i**



Prepared by the method used for **14a**. 83% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.40 (m, 2H), 7.33 – 7.29 (m, 2H), 7.14 – 7.11 (m, 1H), 5.44 (br s, 1H), 2.64 (m, 2H), 2.35 (m, 2H), 1.88 (quintet, $J = 7.2$ Hz, 2H), 1.51 (s, 9H), 1.45 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 152.4, 141.6/141.3, 128.4, 124.9, 122.9, 121.3, 112.1, 81.9, 32.5, 30.2, 28.2, 28.1, 22.4; FT-IR (CH_2Cl_2) 2977, 2853, 1726, 1369, 1341, 1320, 1155 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{21}\text{H}_{30}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 397.2098, found 397.2097.

10) di-*tert*-Butyl 1-(cyclohept-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate (**14j**)

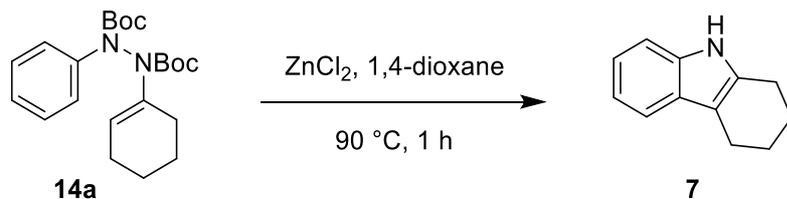


Prepared by the method used for **14a** except the amount of enol triflate **13f** (3.8 equiv), reaction temperature (100 $^\circ\text{C}$) and time (24 h), 45% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.34 – 7.29 (m, 4H), 7.17 – 7.16 (m, 1H), 5.75 (s, 1H), 2.35 (m, 2H), 2.06 – 2.04 (m, 2H), 1.69 – 1.68 (m,

2H), 1.51 (s, 18H), 1.41 – 1.25 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.5, 144.1, 128.6, 126.2, 125.7, 125.0, 123.8, 82.0, 81.6, 32.1, 30.3, 28.5, 28.4, 26.9, 26.7, 26.2; FT-IR (CH_2Cl_2) 2924, 2854, 1729, 1368, 1163 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{34}\text{N}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 425.2411, found 425.2411.

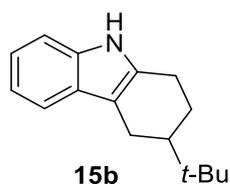
IV. General synthetic procedure for indoles

1) Synthesis of 2,3,4,9-tetrahydro-1H-carbazole 7



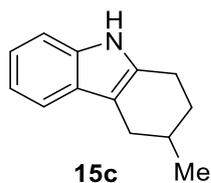
To a sealed tube were charged di-*tert*-butyl 1-(cyclohex-1-en-1-yl)-2-phenylhydrazine-1,2-dicarboxylate **14a** (100 mg, 0.26 mmol), ZnCl_2 (77 mg, 0.57 mmol, 2.2 equiv) and anhydrous 1,4-dioxane (2 mL) at rt. The reaction was heated at $90\text{ }^\circ\text{C}$ for 1 h. After the completion of the reaction, the reaction mixture was cooled to rt, quenched by adding saturated aqueous Na_2CO_3 (2.0 ml) and saturated aqueous NH_4Cl (1.0 mL), and extracted with CH_2Cl_2 (3 x 10 mL). The combined organic solution was dried over anhydrous MgSO_4 , filtered and concentrated under reduced pressure. The residue oil was purified by flash column chromatography on silica gel (CH_2Cl_2 :hexane = 1:4) to afford indole **7** (42 mg, 94% yield) as a white solid.

2) Synthesis of 3-(*tert*-butyl)-2,3,4,9-tetrahydro-1H-carbazole 15b



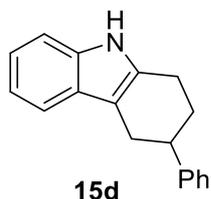
Prepared by the method used for **7**. 95% yield, white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (br s, 1H), 7.47 (d, $J = 7.6$ Hz), 7.23 (d, $J = 7.6$ Hz), 7.12 – 7.05 (m, 2H), 2.83-2.79 (m, 1H), 2.75 – 2.66 (m, 2H), 2.39 (t, $J = 11.8$ Hz, 1H), 2.10 – 2.07 (m, 1H), 1.53 – 1.51 (m, 2H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.1, 134.3, 128.1, 121.0, 119.2, 117.7, 110.6, 110.5, 45.5, 32.8, 27.7, 24.8, 24.2, 22.4.

3) Synthesis of 3-methyl-2,3,4,9-tetrahydro-1H-carbazole 15c



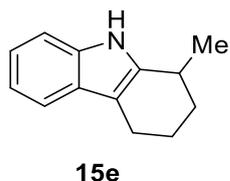
Prepared by the method used for 7. 92% yield, pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (br s, 1H), 7.44 (d, $J = 7.2$ Hz, 1H), 7.22 (d, $J = 7.6$ Hz), 7.12 – 7.04 (m, 2H), 2.83 (dd, $J = 15.4, 4.6$ Hz, 1H), 2.78 – 2.66 (m, 2H), 2.29 – 2.23 (m, 1H), 1.95 – 1.92 (m, 2H), 1.60 – 1.50 (m, 1H), 1.12 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.0, 134.0, 127.8, 121.0, 119.2, 117.8, 110.5, 110.2, 31.5, 29.7, 29.5, 23.0, 21.9.

4) Synthesis of 3-phenyl-2,3,4,9-tetrahydro-1H-carbazole 15d



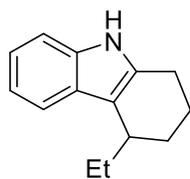
Prepared by the method used for 7. 96% yield, white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.74 (br s, 1H), 7.44 (d, $J = 7.6$ Hz, 1H), 7.35 – 7.29 (m, 4H), 7.26 – 7.25 (m, 2H), 7.15 – 7.06 (m, 2H), 3.10 – 3.06 (m, 2H), 2.97- 2.78 (m, 3H), 2.23 – 2.05 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 146.8, 136.0, 133.7, 128.6, 127.6, 127.2, 126.3, 121.2, 119.3, 117.8, 110.6, 110.1, 41.2, 30.4, 29.4, 23.4.

5) Synthesis of 1-methyl-2,3,4,9-tetrahydro-1H-carbazole 15e



Prepared by the method used for 7. 96% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.53 (br s, 1H), 7.45 (d, $J = 7.2$ Hz, 1H), 7.21 (d, $J = 7.2$ Hz, 1H), 7.12 – 7.05 (m, 2H), 2.93 – 2.85 (m, 1H), 2.73 – 2.60 (m, 2H), 2.04 – 1.92 (m, 2H), 1.78 – 1.68 (m, 1H), 1.53 – 1.45 (m, 1H), 1.23 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.7, 135.7, 127.8, 121.1, 119.2, 118.1, 110.6, 109.8, 32.4, 28.7, 22.0, 21.3, 20.3.

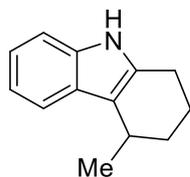
6) Synthesis of 4-ethyl-2,3,4,9-tetrahydro-1H-carbazole 15f



15f

Prepared by the method used for **7**. 95% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (s, 1H), 7.54 (d, $J = 7.2$ Hz, 1H), 7.24 (d, $J = 7.2$ Hz, 1H), 7.11 – 7.03 (m, 2H), 2.91 – 2.88 (m, 1H), 2.68 – 2.65 (m, 2H), 2.06 – 1.92 (m, 2H), 1.90 – 1.83 (m, 1H), 1.81 – 1.70 (m, 2H), 1.60 – 1.45 (m, 1H), 1.01 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 135.9, 134.2, 127.7, 120.9, 119.1, 118.8, 114.4, 110.6, 33.9, 27.9, 27.8, 23.6, 20.2, 12.2; FT-IR (CH_2Cl_2) 3405, 2932, 2859, 1680, 1620, 1461, 1326, 742 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{14}\text{H}_{17}\text{N}$ [M^+] 199.1361, found 199.1362.

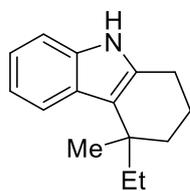
7) Synthesis of 4-methyl-2,3,4,9-tetrahydro-1H-carbazole **15g**



15g

Prepared by the method used for **7**. 91% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.56 – 7.54 (m, 2H), 7.23 (d, $J = 7.6$ Hz, 1H), 7.11 – 7.04 (m, 2H), 3.12 – 3.07 (m, 1H), 2.70 – 2.65 (m, 2H), 2.02 – 1.92 (m, 2H), 1.83 – 1.76 (m, 1H), 1.59 – 1.52 (m, 1H), 1.35 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 135.9, 134.0, 127.5, 120.9, 119.1, 118.8, 115.1, 110.6, 32.2, 27.2, 23.6, 21.4, 20.5.

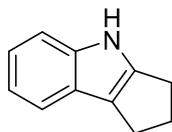
8) Synthesis of 4-ethyl-4-methyl-2,3,4,9-tetrahydro-1H-carbazole **15h**



15h

Prepared by the method used for **7**. 92% yield, pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.63 – 7.61 (m, 2H), 7.27 – 7.25 (m, 1H), 7.10 – 7.01 (m, 2H), 2.68 – 2.65 (m, 2H), 1.98 – 1.76 (m, 5H), 1.52 – 1.47 (m, 1H), 1.36 (s, 3H), 0.80 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.0, 134.0, 126.9, 120.6, 120.1, 118.9, 117.6, 110.6, 35.8, 35.3, 34.0, 27.3, 23.9, 19.8, 9.3; FT-IR (CH_2Cl_2) 3402, 2929, 1621, 1457, 1326, 1017, 741 cm^{-1} ; HRMS (EI) Calcd for $\text{C}_{15}\text{H}_{19}\text{N}$ [M^+] 213.1517, found 213.1514.

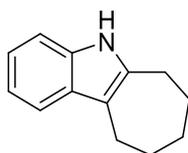
9) Synthesis of 1,2,3,4-tetrahydrocyclopenta[*b*]indole 15i



15i

Prepared by the method used for 7. 95% yield, white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.71 (s, 1H), 7.44 – 7.42 (m, 1H), 7.26 – 7.22 (m, 1H), 7.10 – 7.05 (m, 2H), 2.84 – 2.80 (m, 4H), 2.56 – 2.49 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 143.8 141.1 124.8 120.6 119.9, 119.6, 118.6, 111.4, 28.8, 26.0, 24.5.

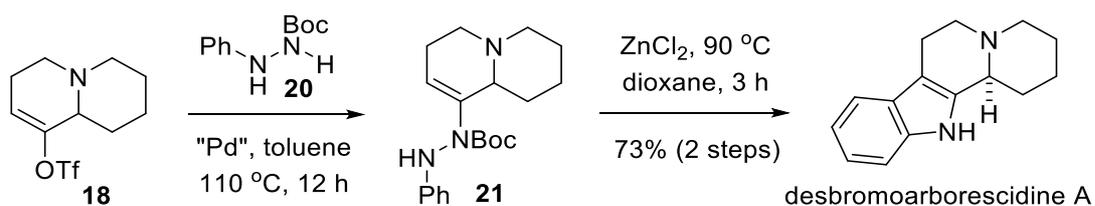
10) Synthesis of 5,6,7,8,9,10-hexahydrocyclohepta[*b*]indole 15j



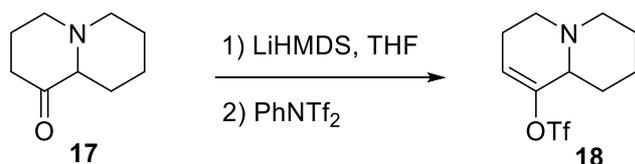
15j

Prepared by the method used for 7. 97% yield, pale yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.52 (s, 1H), 7.47 – 7.45 (m, 1H), 7.21 – 7.19 (m, 1H), 7.10 – 7.05 (m, 2H), 2.81 – 2.75 (m, 4H), 1.88 – 1.85 (m, 2H), 1.78 -1.75 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.5, 134.3, 129.3, 120.7, 119.1, 117.7, 113.8, 110.3, 31.9, 29.6, 28.8, 27.6, 24.8.

V. Procedure for the synthesis of desbromoarborescidine A



1) Preparation of 1,3,4,6,7,9a-hexahydro-2H-quinolizin-9-yl trifluoromethanesulfonate 18



To a solution of hexahydro-2H-quinolizin-1(6H)-one **17**⁶ (100 mg, 0.65 mmol) in anhydrous THF (3.0 mL) was added LiHMDS (1.43 mL, 1.0 M in THF, 1.43 mmol, 2.2 equiv) at $-78\text{ }^\circ\text{C}$. After 1 h at $-78\text{ }^\circ\text{C}$, a solution of PhNTf₂ (325 mg, 0.91 mmol, 1.4 equiv) in anhydrous THF (3.5 mL) was slowly

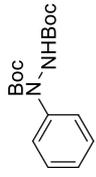
added to the mixture solution. The reaction mixture was then allowed to warm to rt over a period of 4 h and stirred for additional 12 h. After the completion of the reaction, the resulting solution was quenched by adding saturated aqueous NH_4Cl (3.0 mL) and extracted with ethyl acetate (3 x 20 mL). The combined organic solution was dried over anhydrous MgSO_4 , filtered and concentrated under reduced pressure. The residue oil was purified by flash column chromatography on silica gel (100% CH_2Cl_2 to hexane:ethyl acetate = 3:1) to afford enol triflate **18** (161 mg, 87% yield) as a pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 5.80 (t, J = 2.8 Hz, 1H), 2.91 – 2.89 (m, 1H), 2.80 – 2.76 (m, 2H), 2.56 – 2.46 (m, 1H), 2.41 (td, J = 10.8, 3.2 Hz, 1H), 2.26 (dt, J = 11.2, 7.6 Hz, 1H), 2.13 (dt, J = 16, 2.8 Hz, 1H), 1.98 – 1.96 (m, 1H), 1.88 – 1.87 (m, 1H), 1.66 – 1.60 (m, 2H), 1.37 – 1.31 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.5, 118.6 (q, $J_{19\text{F}-13\text{C}}$ = 318 Hz), 116.6, 62.4, 55.6, 51.1, 27.7, 25.5, 24.2; FT-IR (CH_2Cl_2) 2941, 2860, 2806, 2759, 1687, 1420, 1248, 1216, 1143, 1004, 973, 896, 860, 627, 603 cm^{-1} ; HRMS (ESI) Calcd for $\text{C}_{10}\text{H}_{15}\text{F}_3\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$ 286.0720, found 286.0724.

2) Synthesis of desbromoarborescidine A

A sealed tube was charged with enol triflate **18** (248 mg, 0.87 mmol), 1-*tert*-butoxycarbonyl-2-phenylhydrazine **20** (249 mg, 1.22 mmol, 1.4 equiv), $\text{Pd}(\text{OAc})_2$ (9.8 mg, 0.04 mmol, 5 mol%), $\text{P}(\text{tBu})_3\cdot\text{HBF}_4$ (25 mg, 0.09 mmol, 10 mol%), Cs_2CO_3 (567 mg, 1.74 mmol, 2.0 equiv), LiCl (258 mg, 6.09 mmol, 7.0 equiv), and anhydrous toluene (1.5 mL) at rt. The reaction was degassed, charged with Ar and heated to 110 °C for 9 h. After the completion of the reaction, the resulting solution was cooled to rt and filtered through a plug of Celite with ethyl acetate. The filtered solution was concentrated and diluted by adding anhydrous 1,4-dioxane (3 mL). To this solution was added ZnCl_2 (261 mg, 1.91 mmol, 2.2 equiv). The reaction mixture was heated at 90 °C for 3 h. After the completion of the reaction, the reaction mixture was cooled to rt, quenched by adding saturated aqueous Na_2CO_3 (2.0 ml) and saturated aqueous NH_4Cl (1.0 mL), and extracted with CH_2Cl_2 (3 x 10 mL). The combined organic solution was dried over anhydrous MgSO_4 , filtered and concentrated under reduced pressure. The residue oil was purified by flash column chromatography on silica gel (CH_2Cl_2 :MeOH: NH_4OH = 94:5:1) to afford desbromoarborescidine A (144 mg, 73% overall yield from enol triflate **18**) as a yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.71 (s, 1H), 7.47 (d, J = 8.0 Hz, 1H), 7.3 (d, J = 8.0 Hz, 1H), 7.13 (t, J = 8.0 Hz, 1H), 7.08 (t, J = 8.0 Hz, 1H), 3.24 (d, J = 10.4 Hz, 1H), 3.08 (t, J = 4.8 Hz, 1H), 3.05 – 2.97 (m, 2H), 2.75 – 2.59 (m, 2H), 2.39 (td, J = 11.2, 3.6 Hz, 1H), 2.06 (dd, J = 12.4, 2.4 Hz, 1H), 1.91 (d, J = 12.0 Hz, 1H), 1.83 – 1.73 (m, 2H), 1.61 (qd, J = 12.4, 3.6 Hz, 1H), 1.49 (qt, J = 12.4, 3.6 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 136.1, 135.3, 127.7, 121.4, 119.5, 118.3, 110.8, 108.3, 60.4, 55.9, 53.7, 30.2, 25.9, 24.5, 21.7.

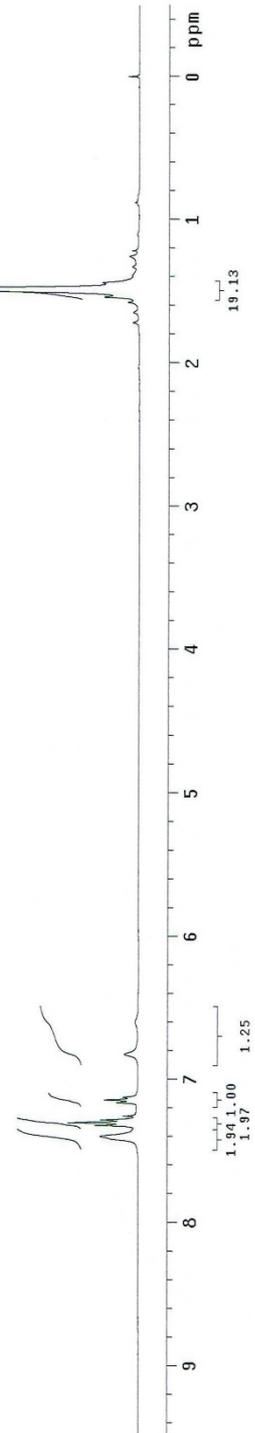
References

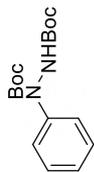
- 1) (a) Kim, K.-Y.; Shin, J.-T.; Lee, K.-S.; Cho, C.-G. *Tetrahedron Lett.* **2004**, *45*, 117; b) Arterburn, J. B.; Rao, K. V.; Ramdas, R.; Dible, B. R. *Org. Lett.* **2001**, *3*, 1351.
- 2) Wolter, M.; Klapars, A.; Buchwald, S. L. *Org. Lett.* **2001**, *3*, 2803.
- 3) Stanislawski, P. C.; Willis, A. C.; Banwell, M. G. *Org. Lett.* **2006**, *8*, 2143.
- 4) Magedov, I. V.; Evdokimov, N. M.; Karki, M.; Peretti, A. S.; Lima, D. T.; Frolova, L. V.; Reisenauer, M. R.; Romero, A. E.; Tongwa, P.; Fonari, A.; Altig, J.; Rogelj, S.; Antipin, M. Y.; Shusterb, C. B.; Kornienko, A. *Chem. Commun.* **2012**, *48*, 10416.
- 5) Suárez, R. M.; Peña, D.; Minnaard, A. J.; Feringa, B. L. *Org. Biomol. Chem.* **2005**, *3*, 729.
- 6) For the preparation of quinolizidinone **17**, see: Tong, S. T.; Barker, D. *Tetrahedron Lett.* **2006**, *47*, 5017.



12

OTf-1a-1H_CDC13_131211_LBY
 File: home/vmmr7/vmmrSYS/data/13_05-14.03/Lm/OTf/1/OTf-1a-1H_CDC13_131211_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition Temperature
 Operator: vmmr7
 File: OTf-1a-1H_CDC13_131211_LBY
 Mercury-40088 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6396.0 Hz
 L1 offset 0.000 Hz
 OBSERVE 1H
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





12

OTf-13C-1H_CDCl3_131211_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: dm

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

168 repetitions

OBSERVE C13, 100.5311265 MHz

DECOUPLE H1, 399.8073989 MHz

Power level

continuously on

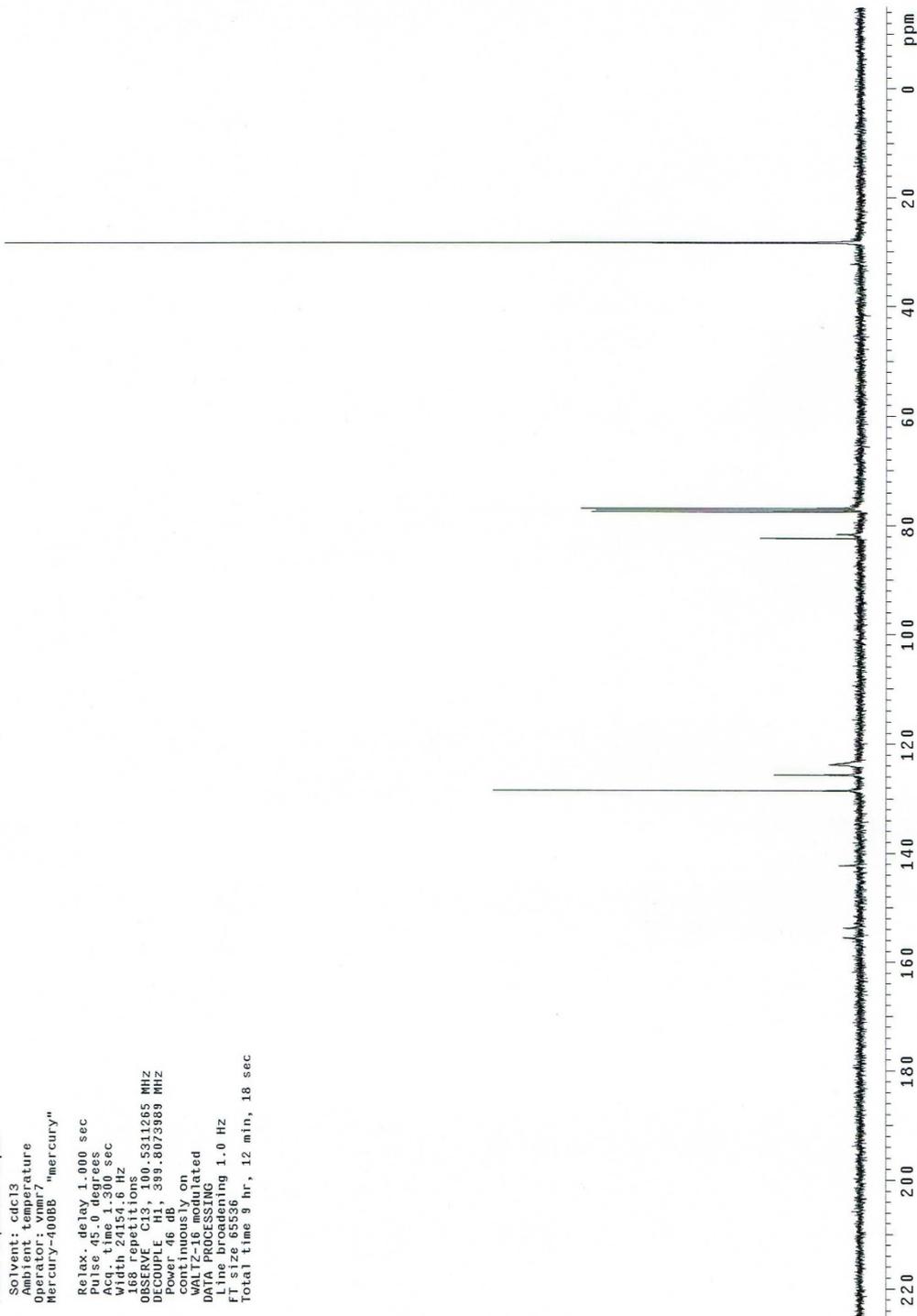
WALTZ-16 modulated

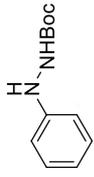
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





20

PHNHoc_1H_CDCl3_130324_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Observed frequency

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.988 sec

Width 6398.0 Hz

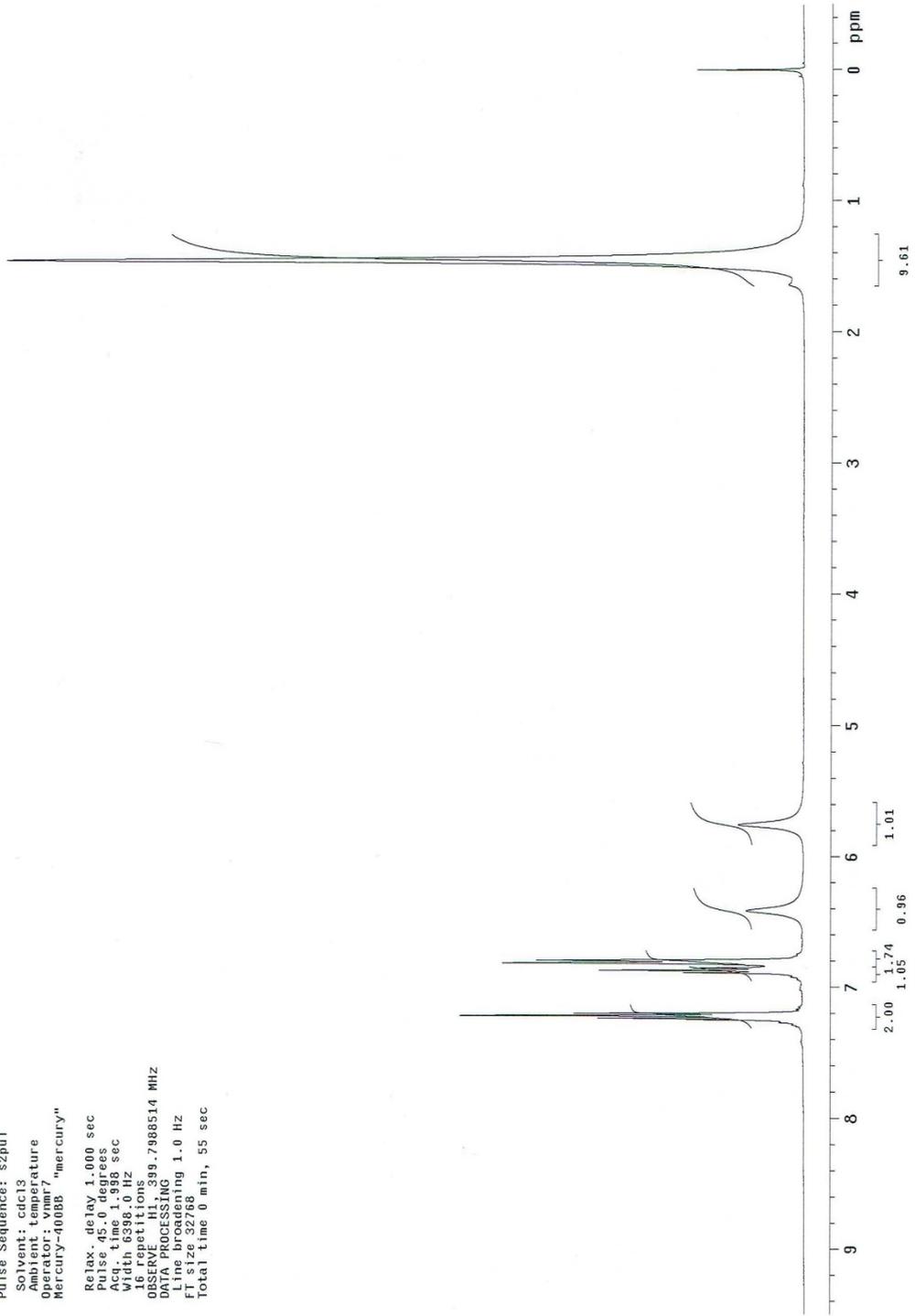
16 repetitions

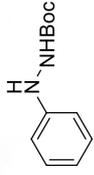
OBSERVE F1 399.7988514 MHz

DATA PROCESSING

FT size 32768

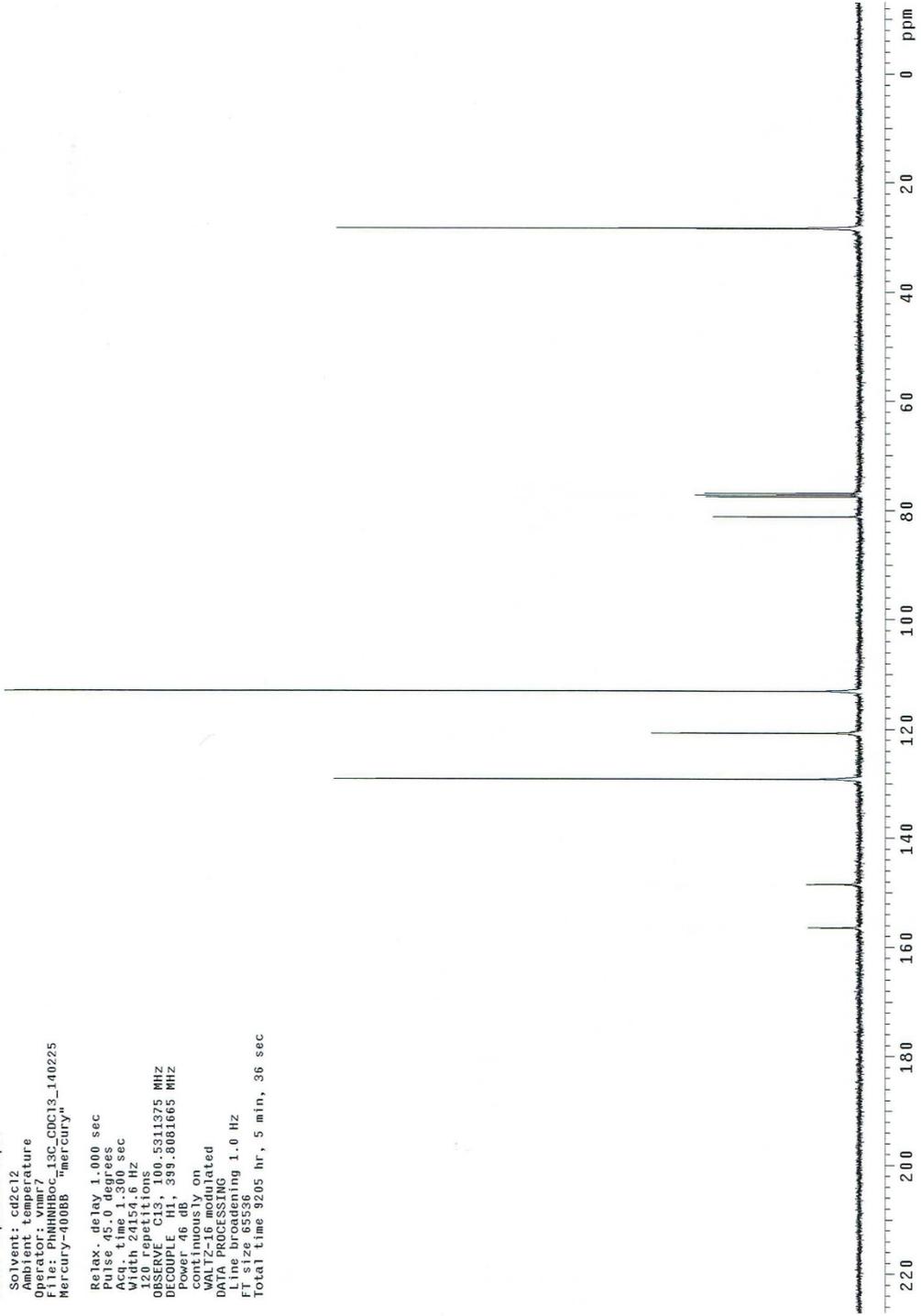
Total time 0 min, 55 sec

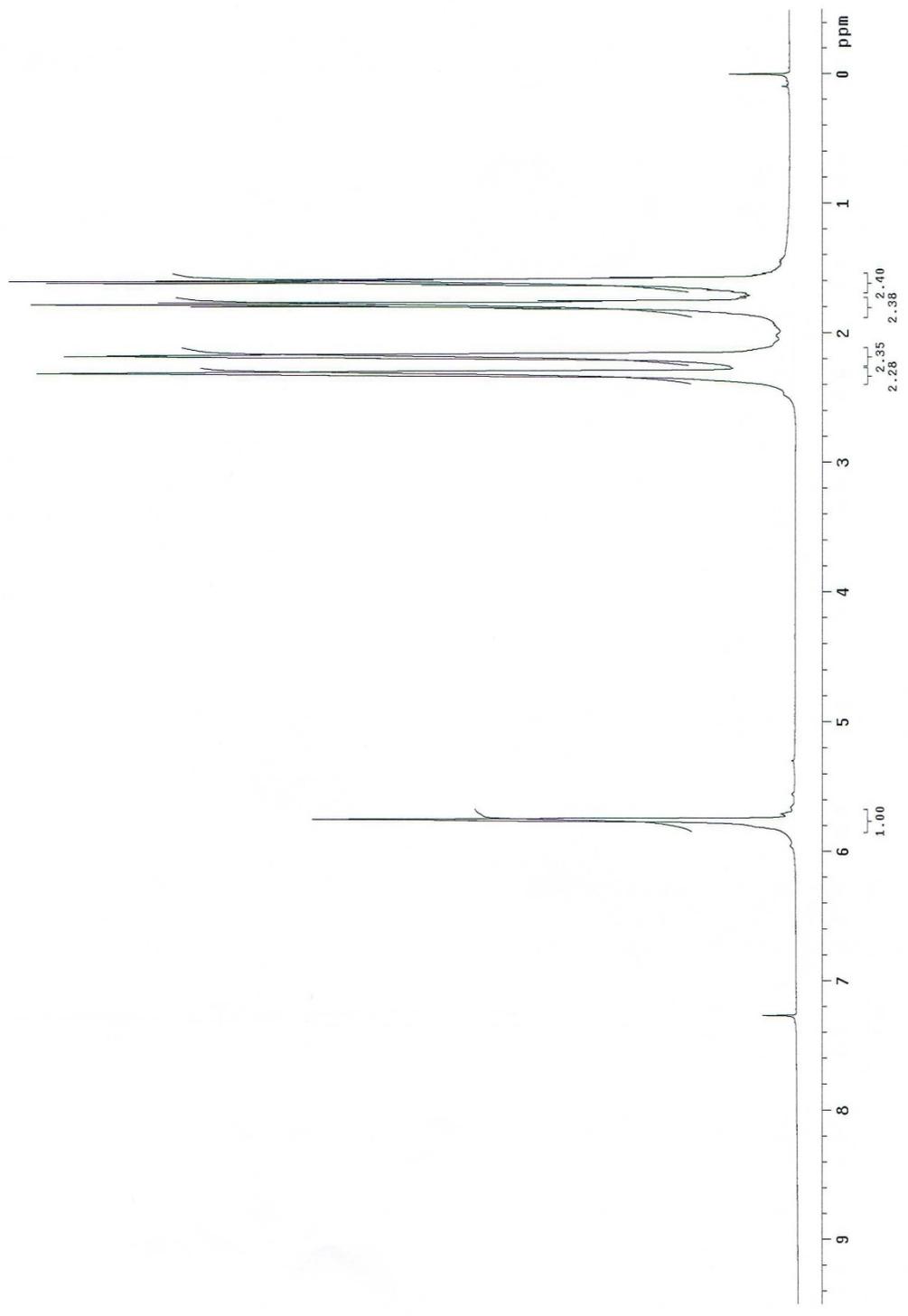
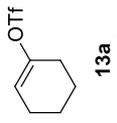


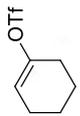


20

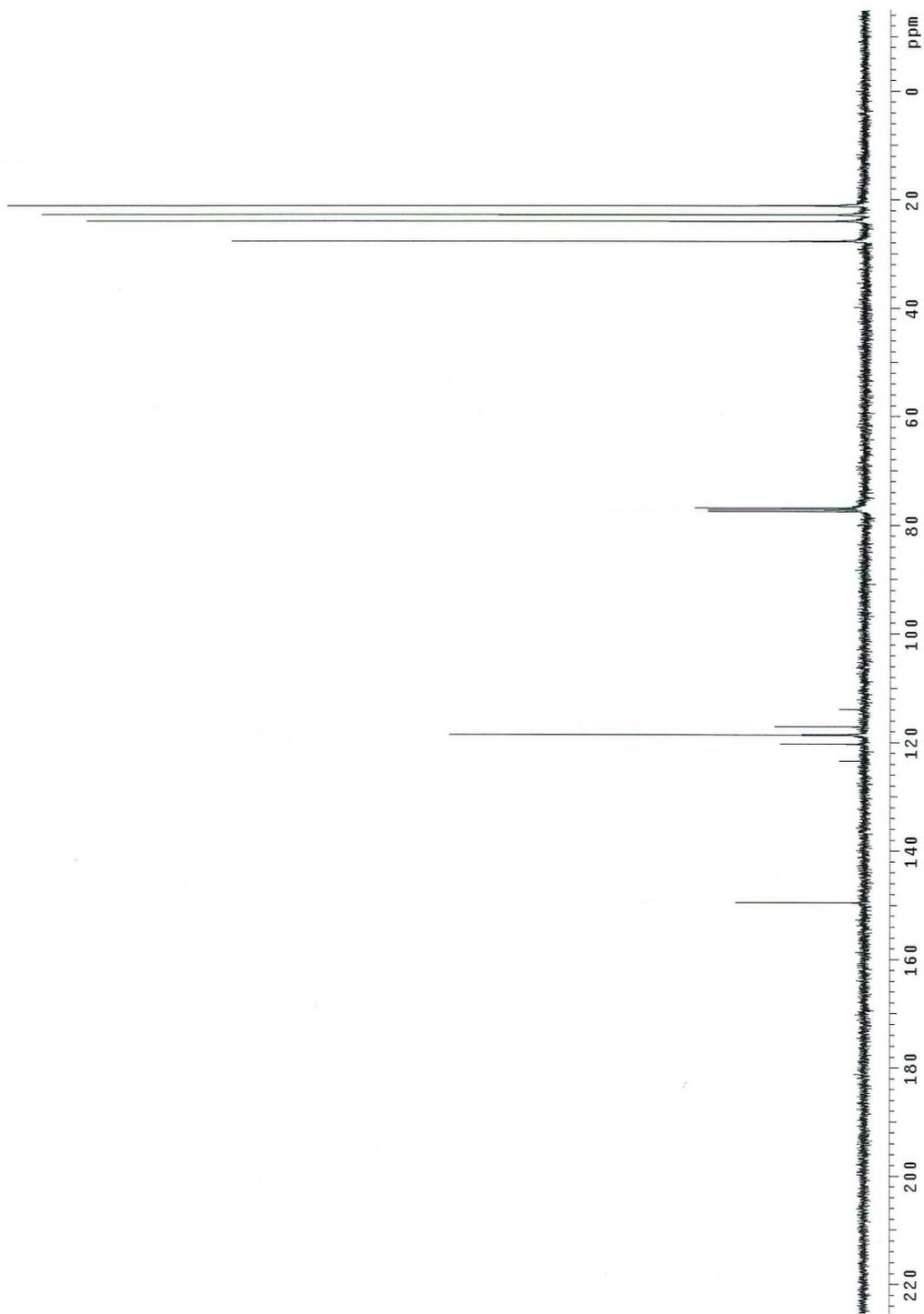
PHNHHBoc_13C_CDCl3_140225
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 Pulse Sequence: s2pul
 Solvent: cdcl2
 Ambient temperature
 Operator: vnmr7
 Mercury-13C_CDCl3_140225
 Mercury-100BB "mercury"
 Relax delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 120 repetitions
 OBSERVE C13, 100.5311375 MHz
 DECOUPLE H1, 399.5061665 MHz
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 32.05 hr, 5 min, 36 sec

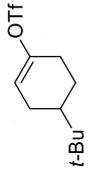






13a





OTf-2c-1H_CDCl3_130803_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Operator: vmmr7

Mercury-4000BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 0.98 sec

Width 6298.0 Hz

16 repetitions

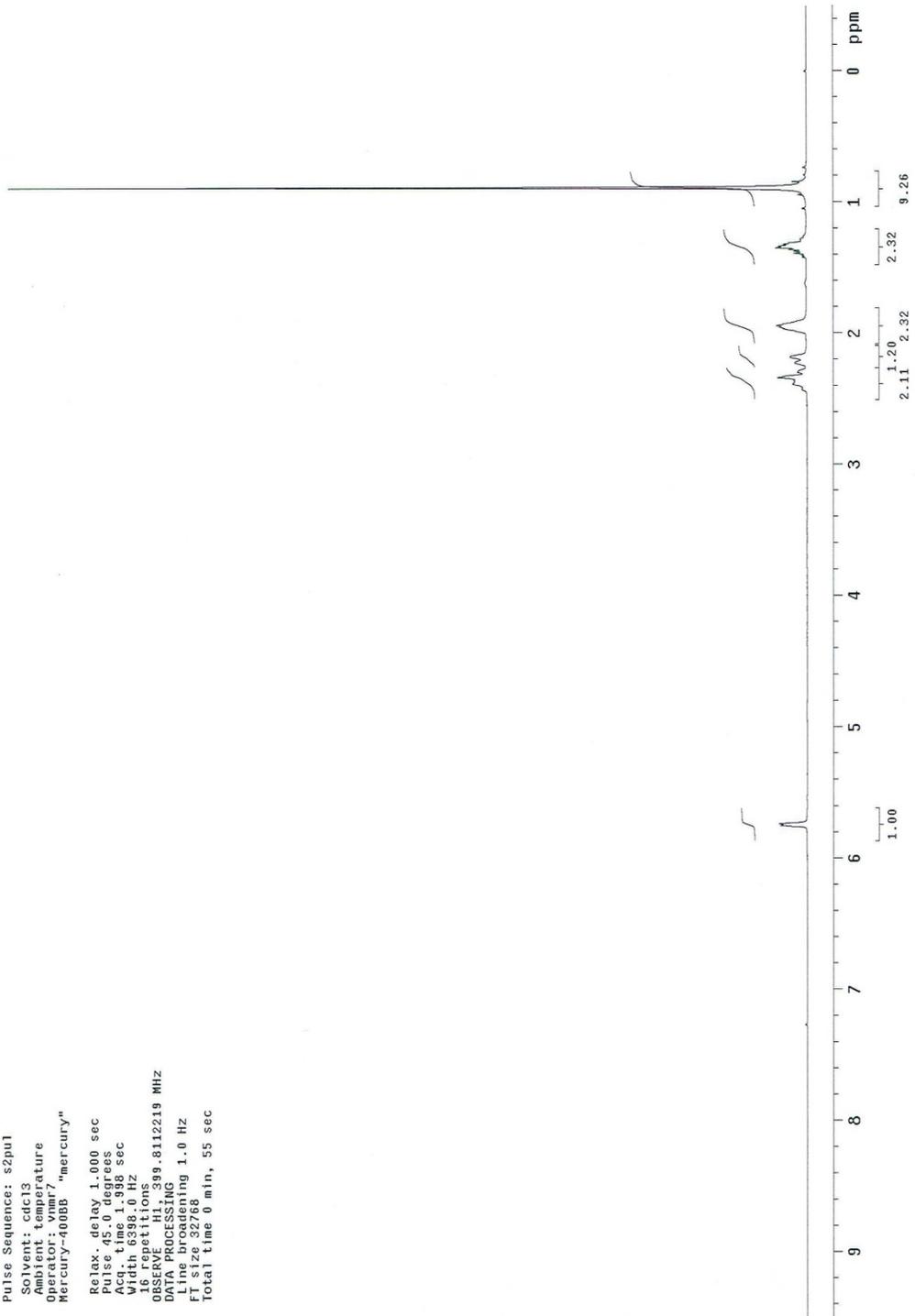
OBSERVE H1, 399.811219 MHz

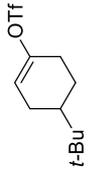
DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





OTf-2c-13C_CDCl3_130803_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vmmr7

Mercury-400BB "mercury"

Relax. delay 4.000 sec

Pulse 45.0 degrees

Acq. time 17.000 sec

Width 20154.8 Hz

60 repetitions

OBSERVE C13, 100.5325964 MHZ

DECOUPLE H1, 399.8132388 MHZ

Power 46 dB

continuously on

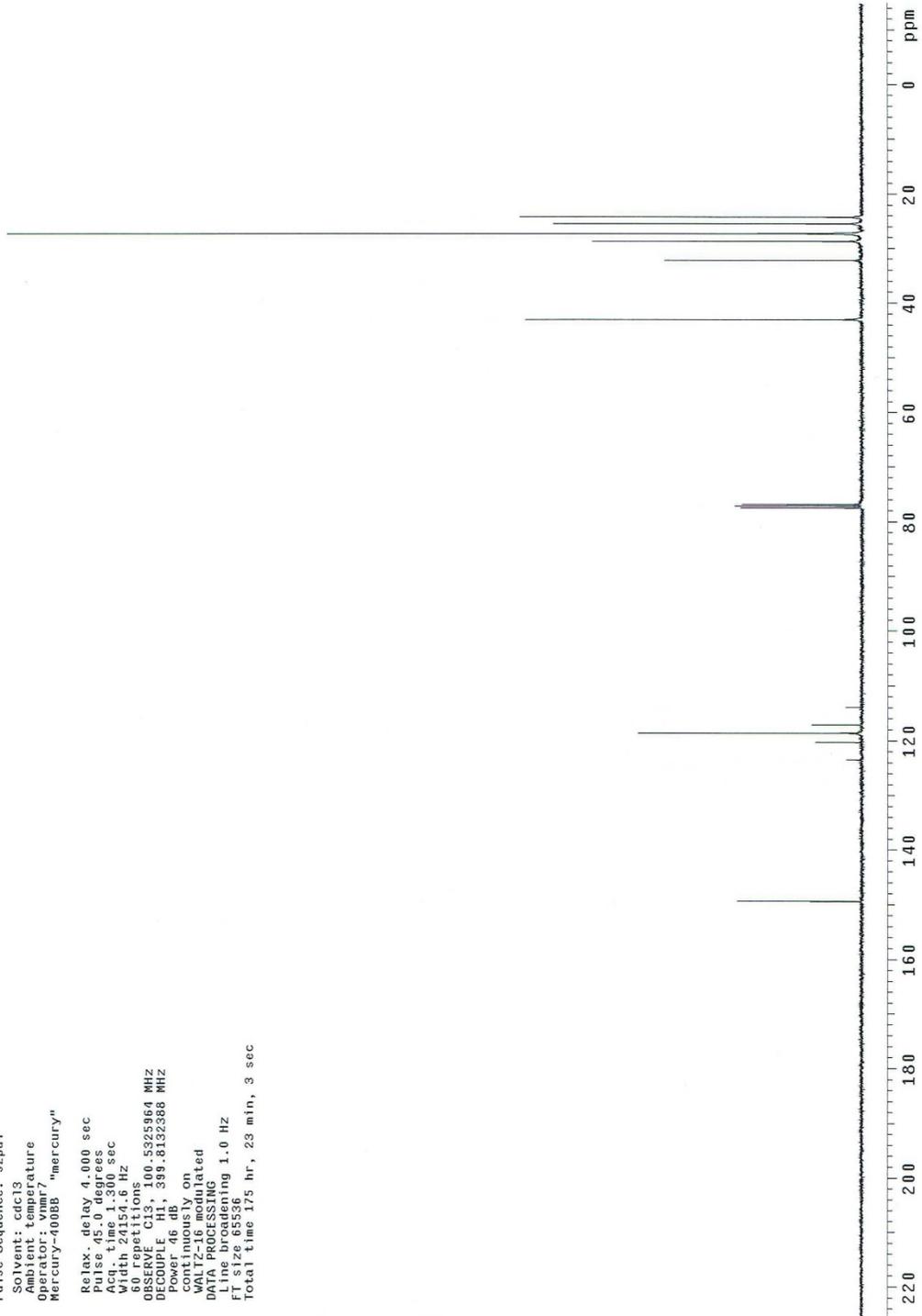
Watergate gated

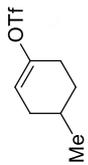
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 175 hr, 23 min, 3 sec





0Tf-2_4-Me-6R-1H_CDCl3_131101_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: jco

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

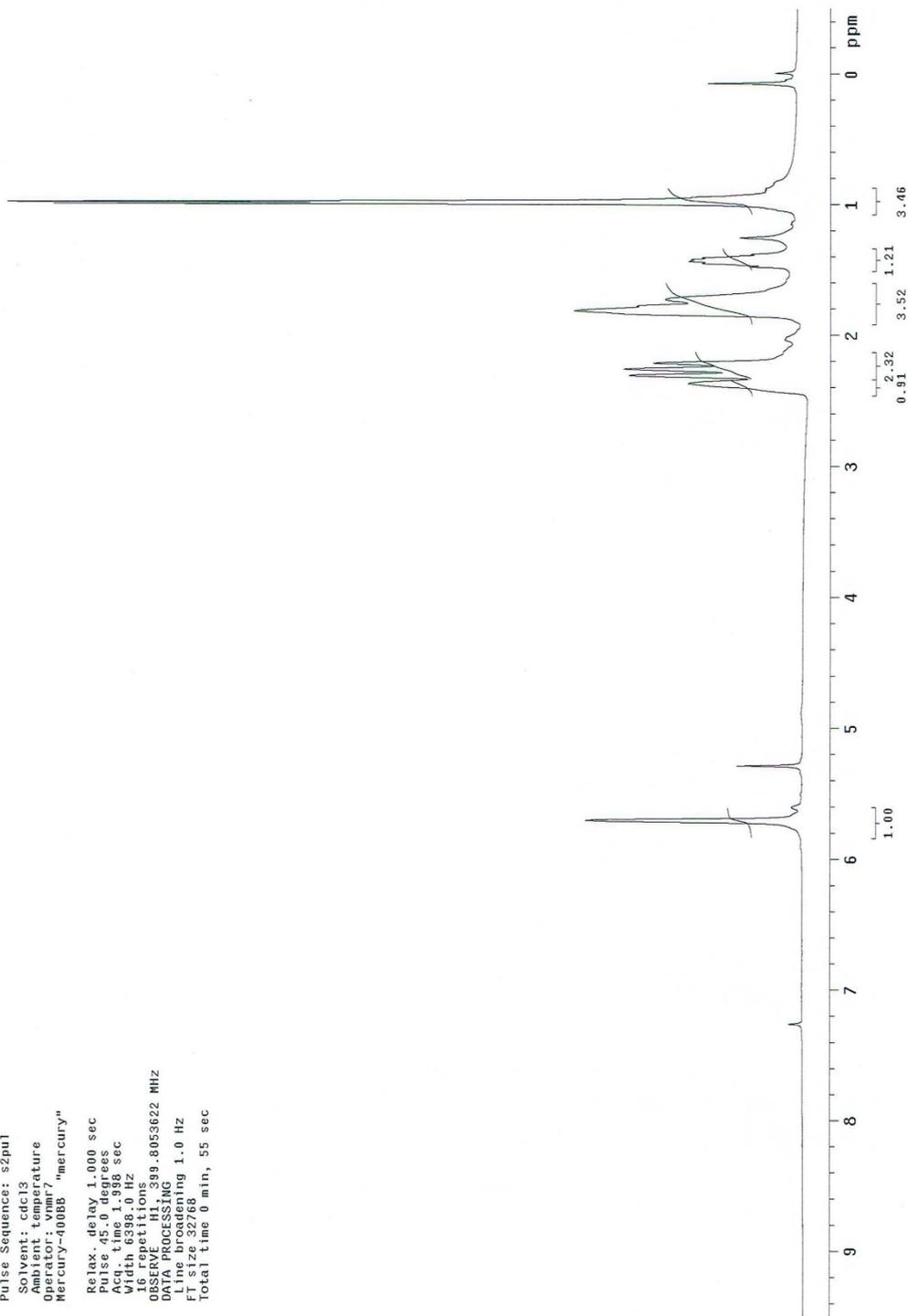
OBSERVE H1, 399.8053622 MHz

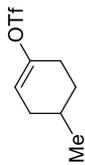
DATA PROCESSING

Acquiring 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





0Tf-2_4-Me-6R-13C_CDCl3_131101_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: jsm

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

100 repetitions

OBSERVE C13, 100.531214 MHz

DECOUPLE H1, 399.8073969 MHz

continuously on

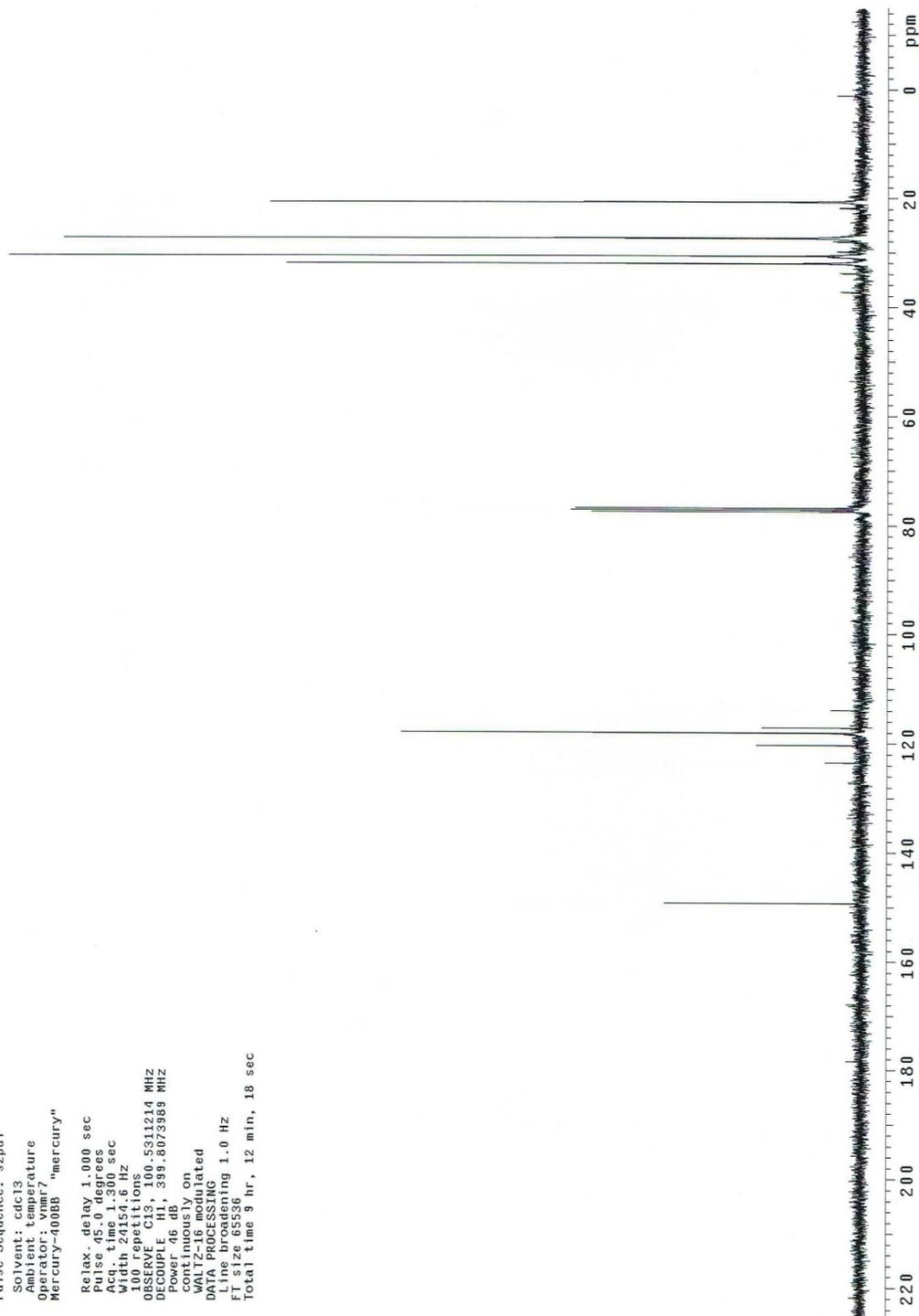
WALTZ-16 modulated

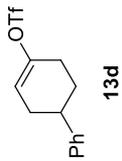
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec

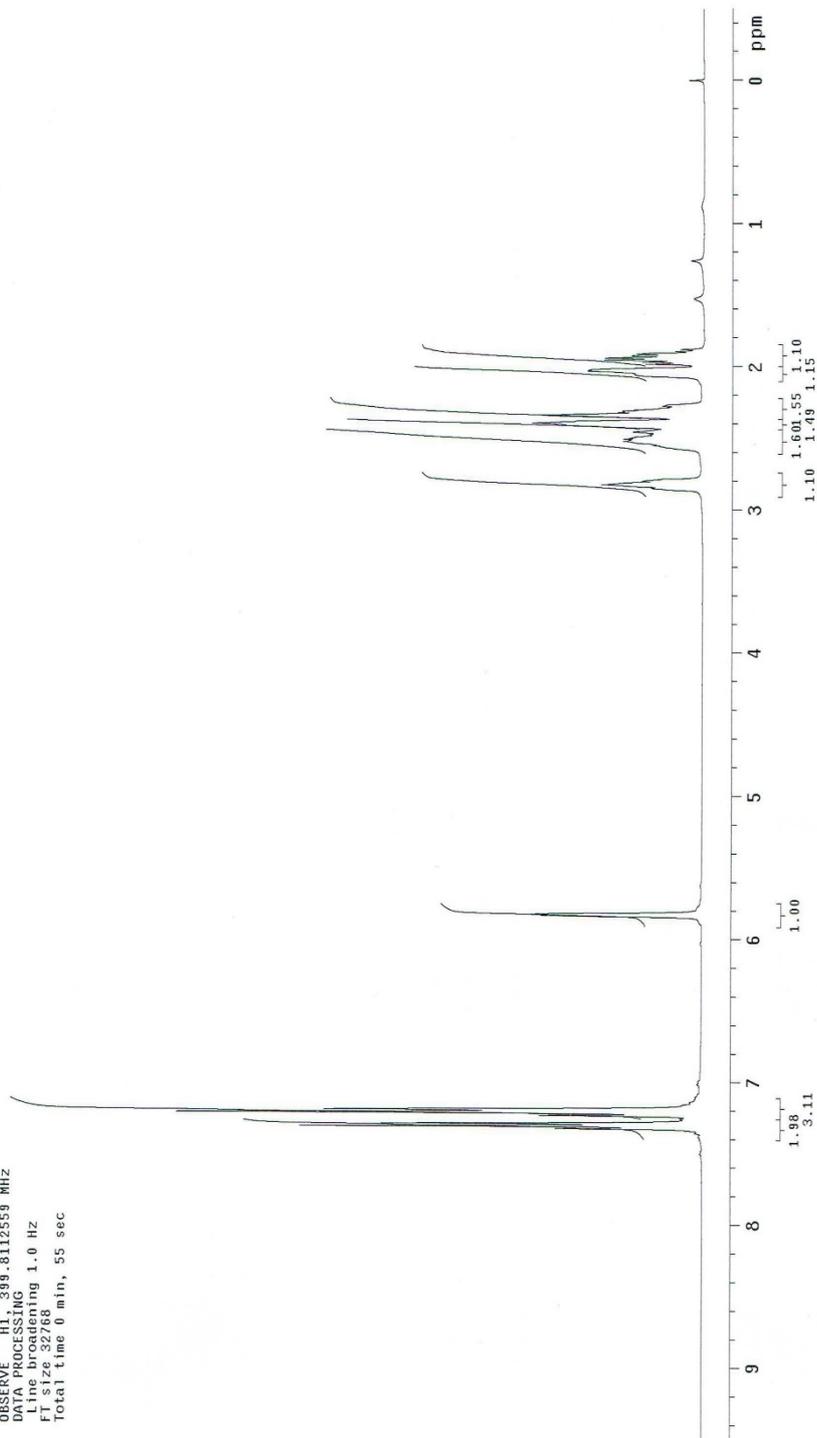


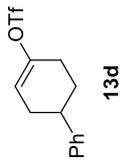


OTf-2_4-Ph-6R-1H_CDC13_130829_LBY
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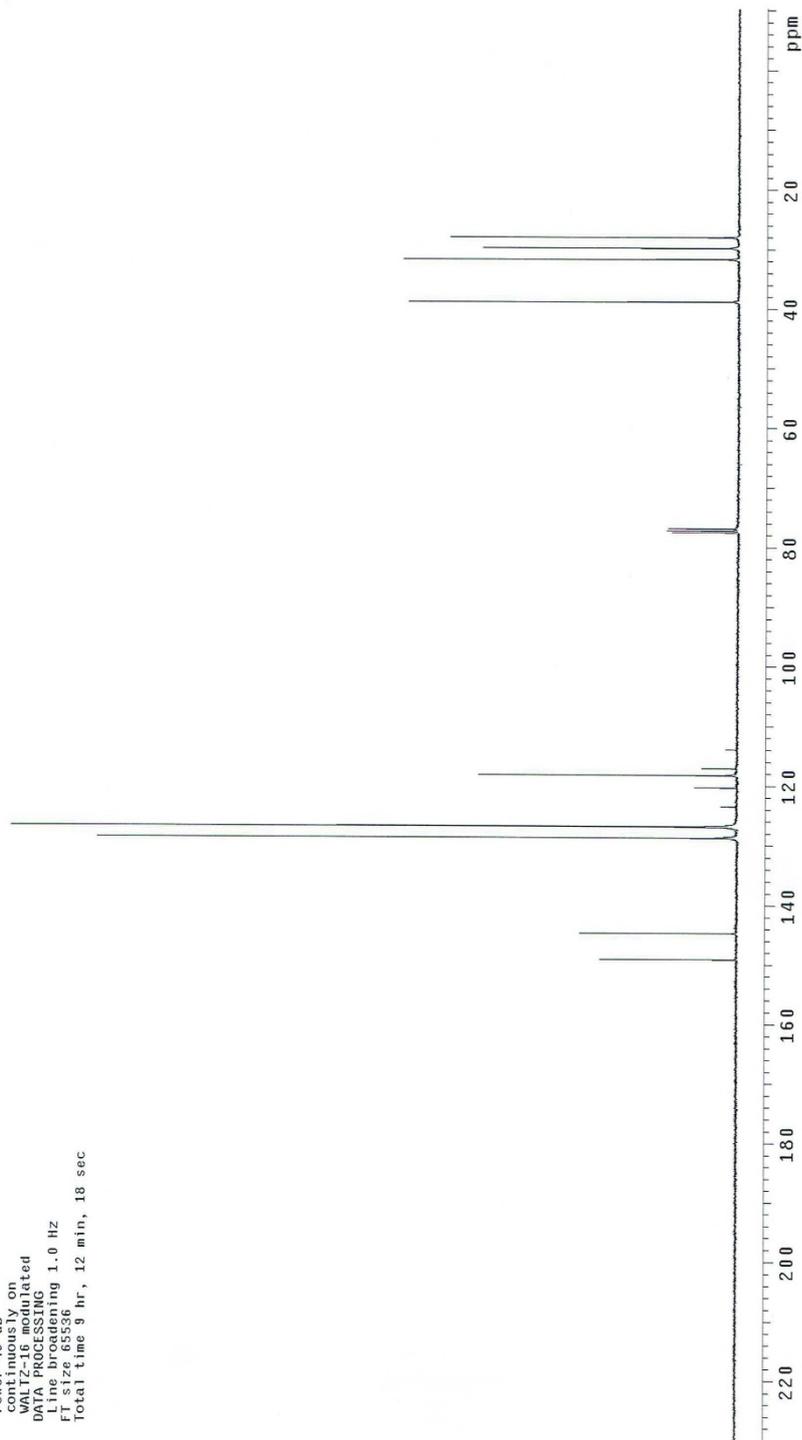
Pulse Sequence: s2pul
 Solvent: cdc13
 Temp: 30.0 C
 Omega: 276.1 K
 File: OTf-2_4-Ph-6R-1H_CDC13_130829_LBY
 Mercury-400BB "mercury"

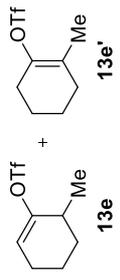
Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6398.0 Hz
 16 repetitions
 Omega 99.8112559 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





OTf-2_4-Ph-6R-13C_CDCl3_130829_LBY
 File: home/vnmr7/vnmrSYS/data/13.05-14.03/Lm/OTf/2/OTf-2_4-Ph-6R-13C_CDCl3_130829_LBY.fid
 Pulse Sequence: s2pul
 Solvent: dms0
 Ambient temperature
 Operator: vnmr7
 File: OTf-2_4-Ph-6R-13C_CDCl3_130829_LBY
 Mercury-40068 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 140 repetitions
 OBSERVE C13, 100.526074 MHz
 DECOUPLE H1, 399.8151379 MHz
 Preset: zgpg30
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 hr, 12 min, 18 sec





OTf-2-Me-6R-1H_CDCl3_131011_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: jh

Mercury-400BB "mercury"

Relax delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 Repetitions

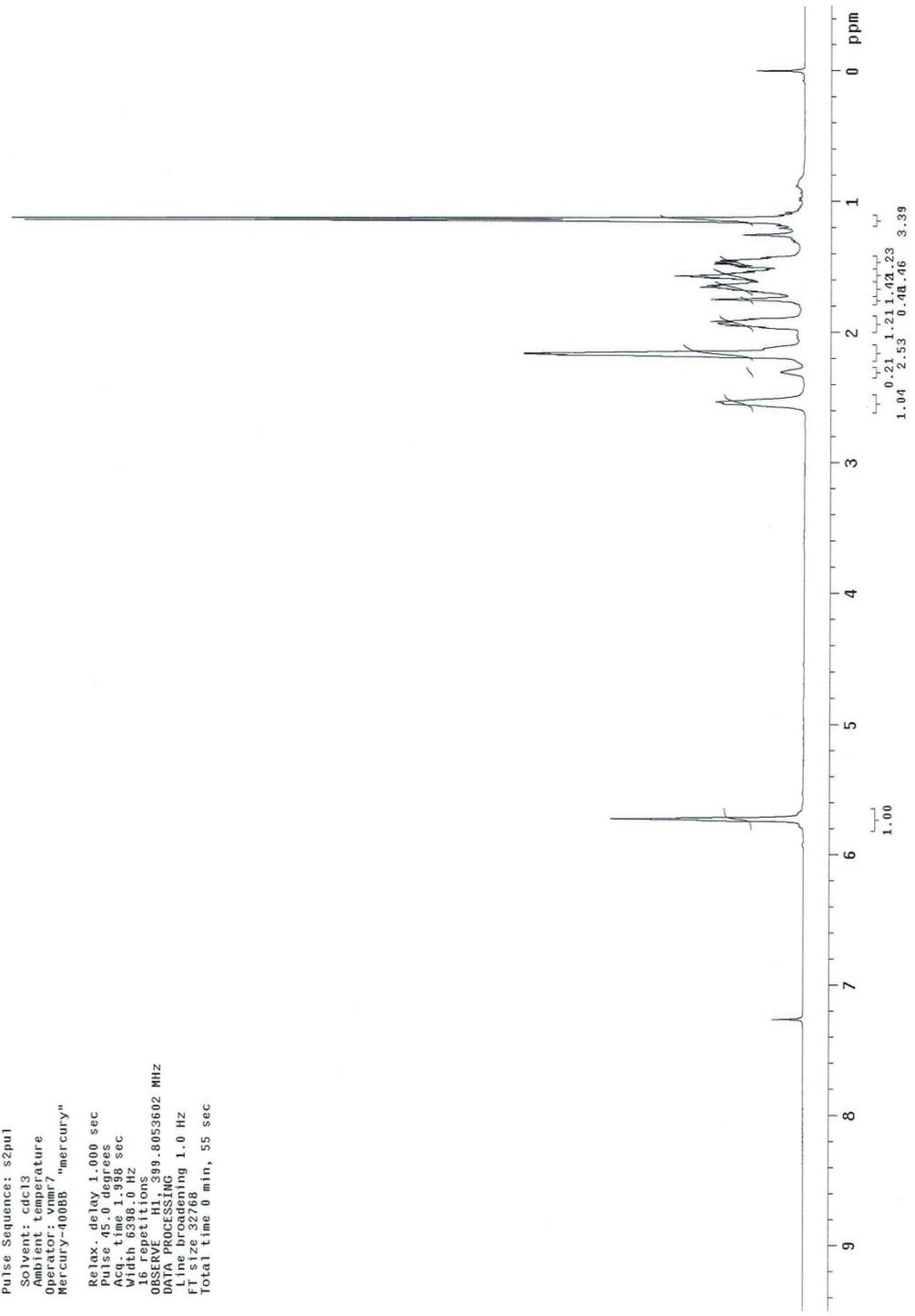
OBSERVE H1, 399.8053602 MHz

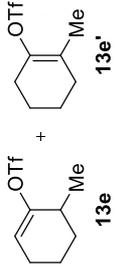
DATA PROCESSING

File Processing 1.0 Hz

FT size 32768

Total time 0 min, 55 sec



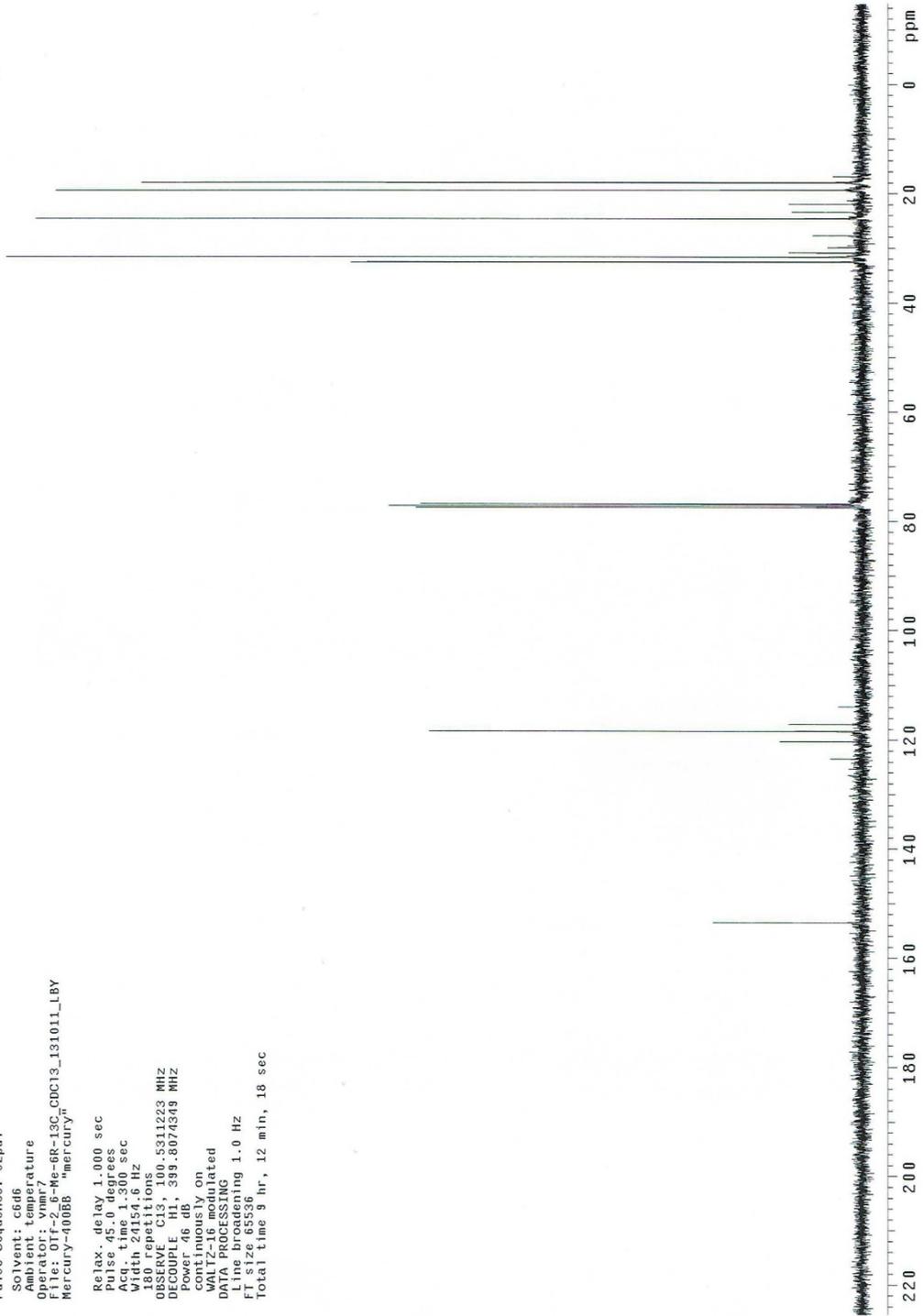


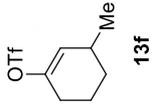
OTF-2_6-Me-6R-13C_CDCl3_131011_LBY
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Pulse Sequence: s2pul

Solvent: c6d6
 Ambient temperature
 Observed nucleus: ¹³C
 File: OTF-2_6-Me-6R-13C_CDCl3_131011_LBY
 Mercury-400BB "mercury"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 180 repetitions
 OBSERVE C13, 100.531223 MHz
 PULSE PRG, 339.6074543 MHz
 Power 46 dB
 continuous IV on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 hr, 12 min, 18 sec



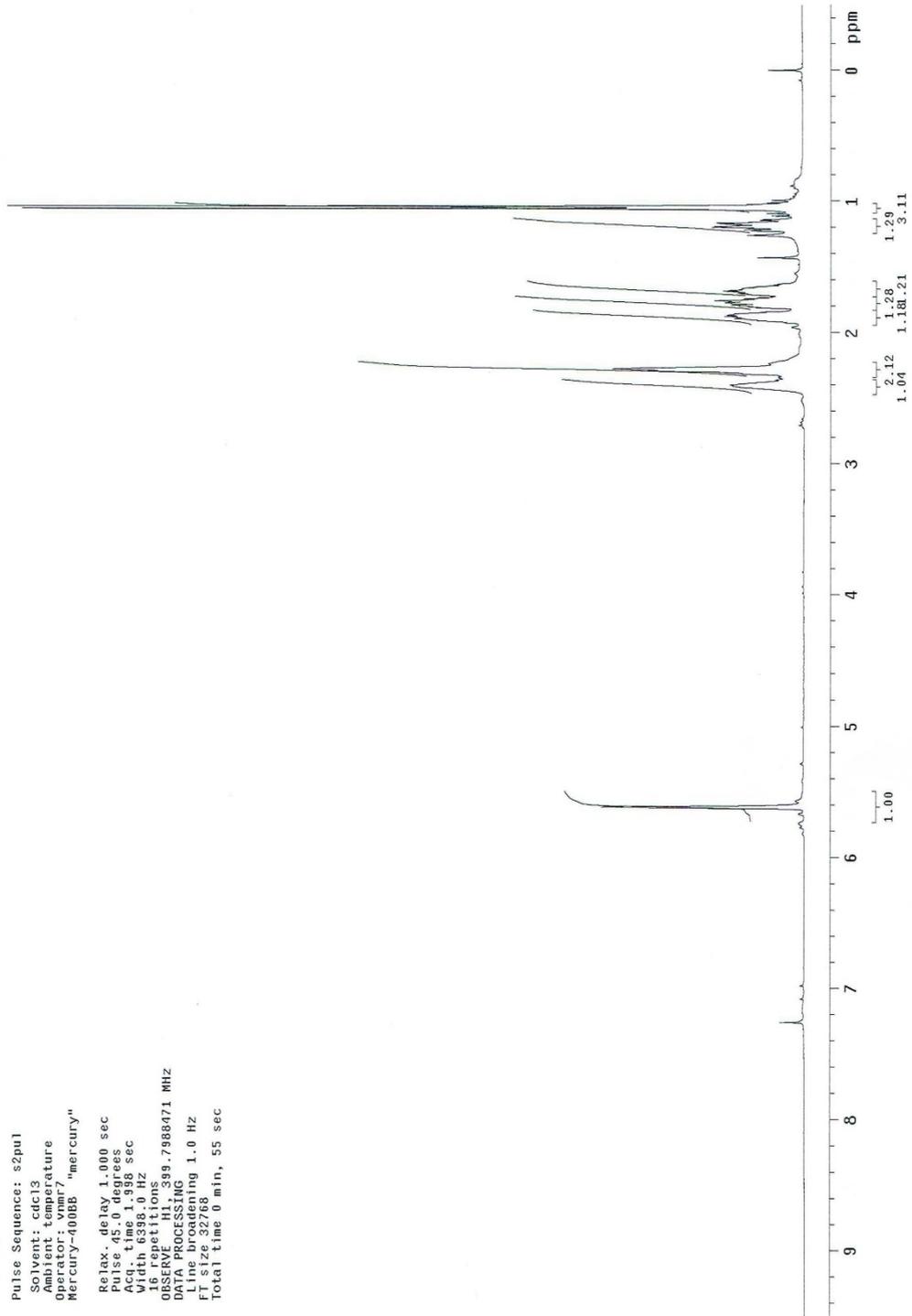


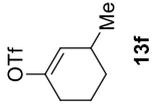
OTf-2_3-Me-6R-1H_CDCl3_140423_LBY

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Operator: ymm
 Mercury-400BB "mercury"

Relax. delay 1.000 sec
 Puls. 45.000 sec
 Acq. time 1.998 sec
 Width 6398.0 Hz
 16 repetitions
 OBSERVE H1, 399.7988471 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 Frequency offset 0.000 MHz
 Total time 0 min, 55 sec





OTf-2_3-Me-6R-13C_CDCl3_140423_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Operator: Vmmr7 "mercury"

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse delay 0.000 sec

Acq. time 1.300 sec

Width 24154.6 Hz

240 repetitions

OBSERVE C13, 100.5294818 MHz

DECOUPLE H1, 399.8008135 MHz

Power 46.00 dB

Anti-coupling on

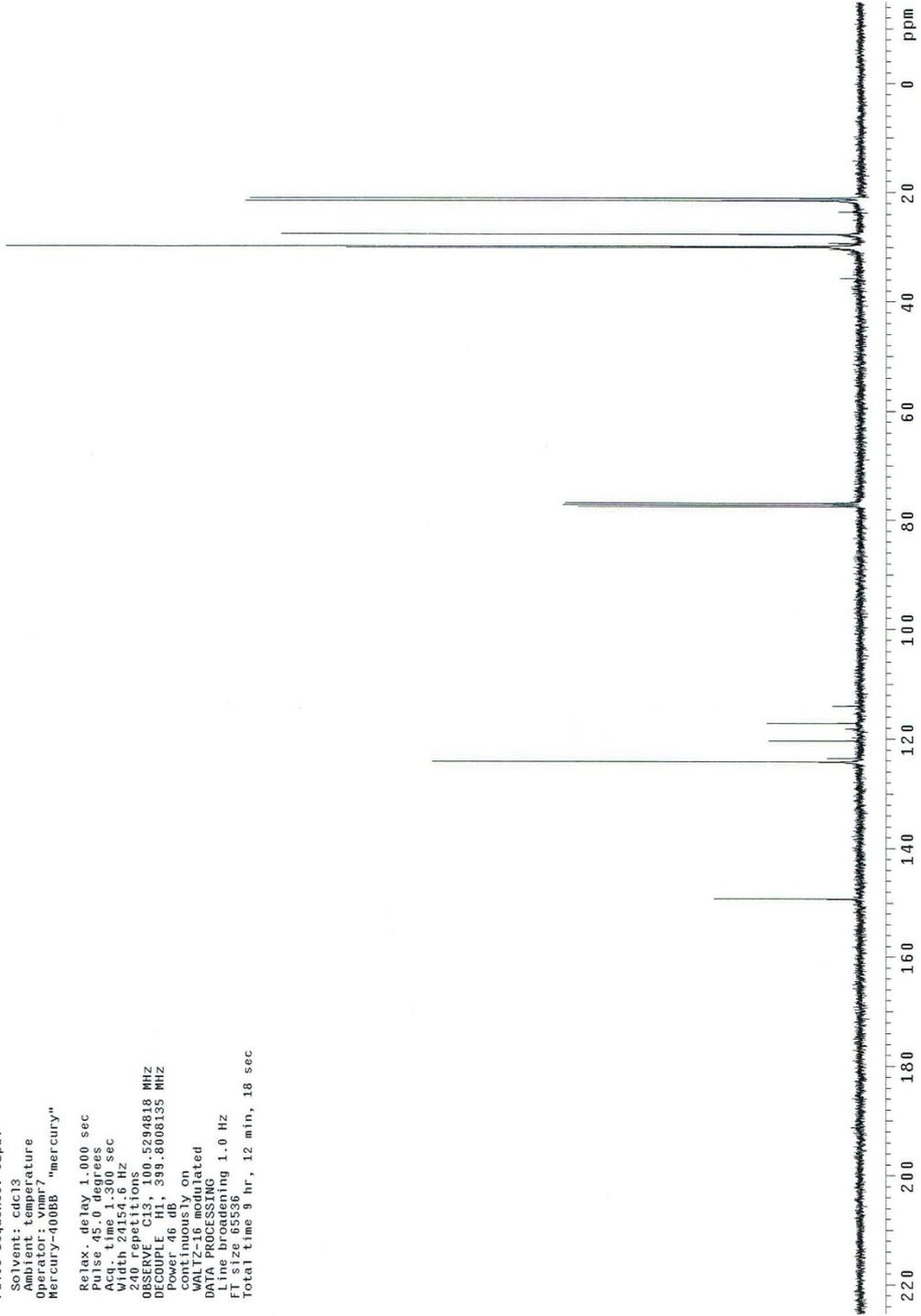
WALTZ-16 modulated

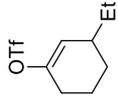
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





139

OTF-2-3-Et-6R-1H_CDCl3_130910_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Temperature:

Operator: vnm7

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

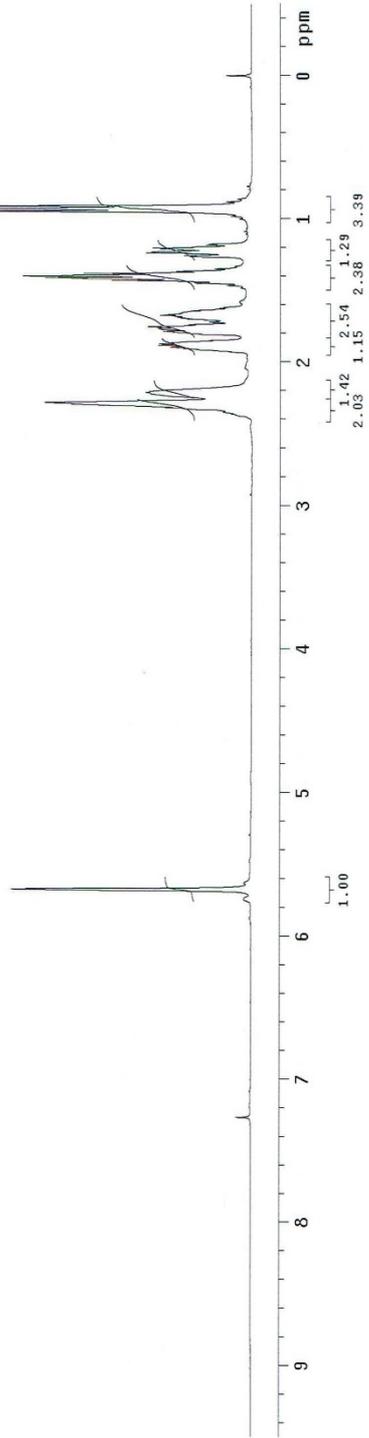
Q2FREQ 99.811223 MHz

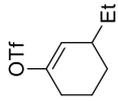
DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





139

OTF-2-Et-6R-13C_CDCl3_130910_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cd3od

Ambient Temperature

Operator: jmr

Mercury-4000B "mercury"

Relax delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

200 repetitions

OBSERVE C13, 100.5325952 MHz

Power 46 dB, 399.616140 MHz

continuously on

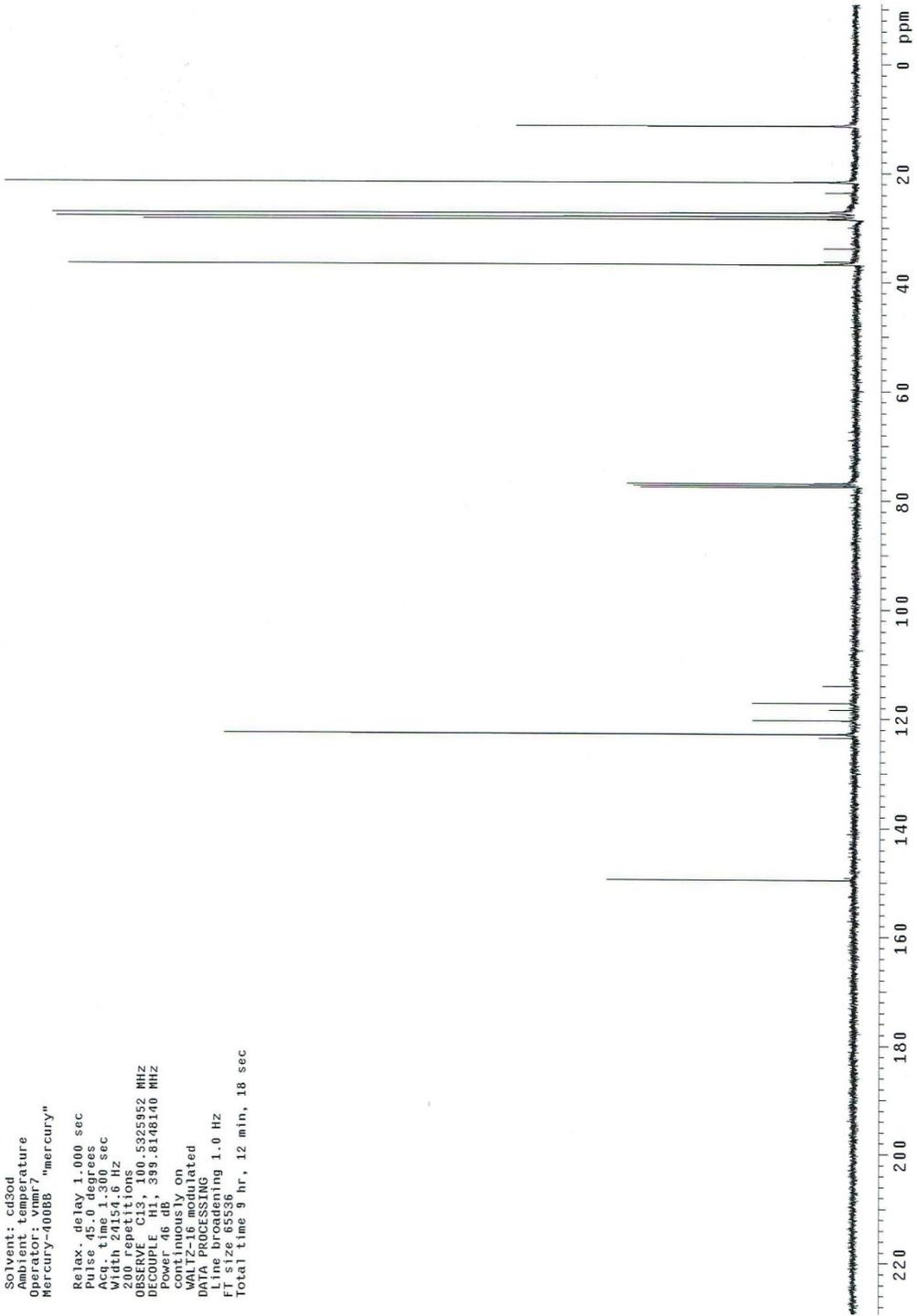
WALTZ-16 modulated

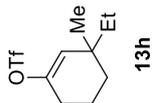
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





OTf-2_3-EtMe-6R-1H_CDCl3_140422_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: jay

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

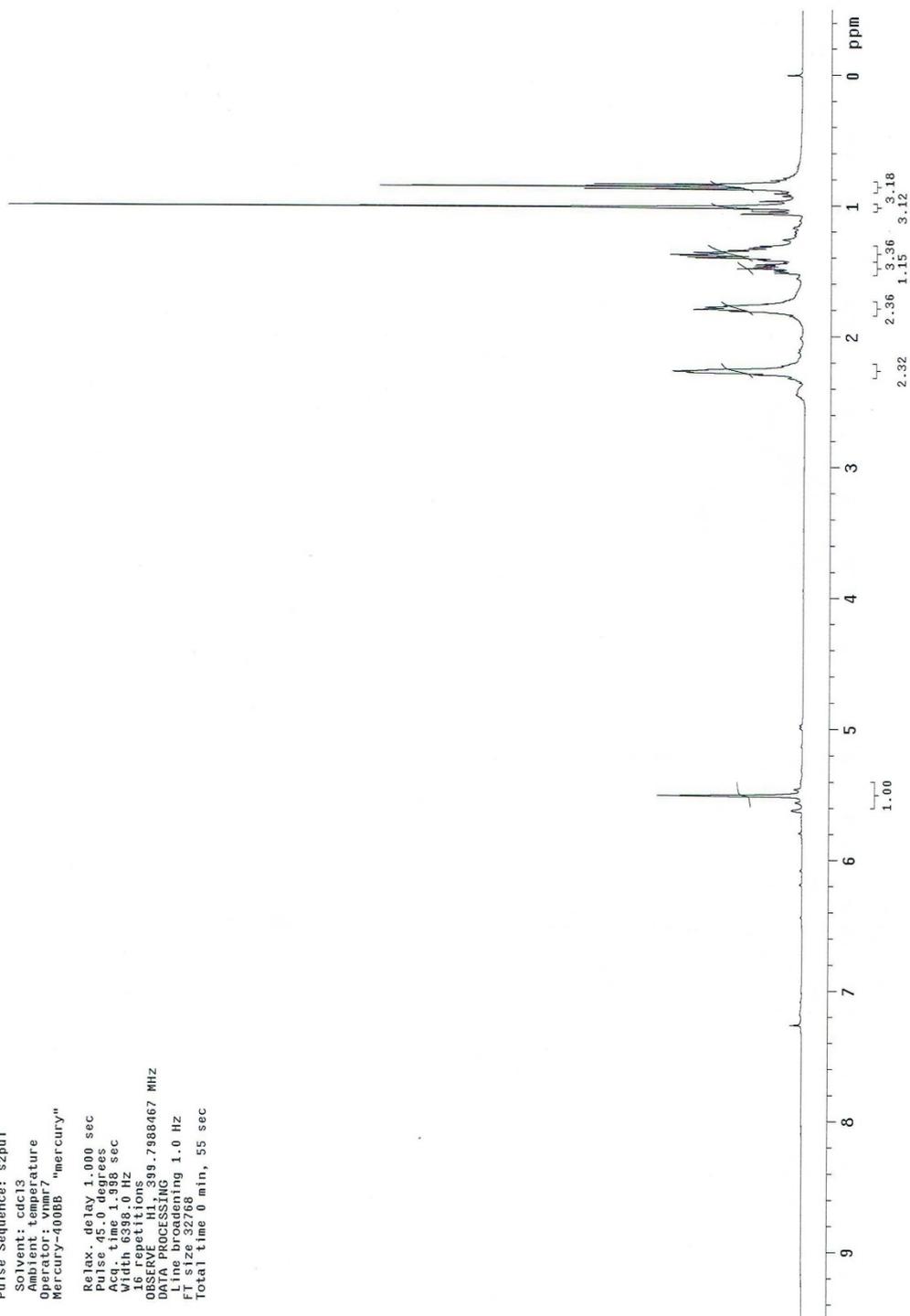
OBSERVE HI, 399.7888467 MHz

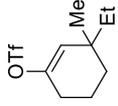
DATA ACQUISITION

Time: 1.0 Hz

FT size 32768

Total time 0 min, 55 sec

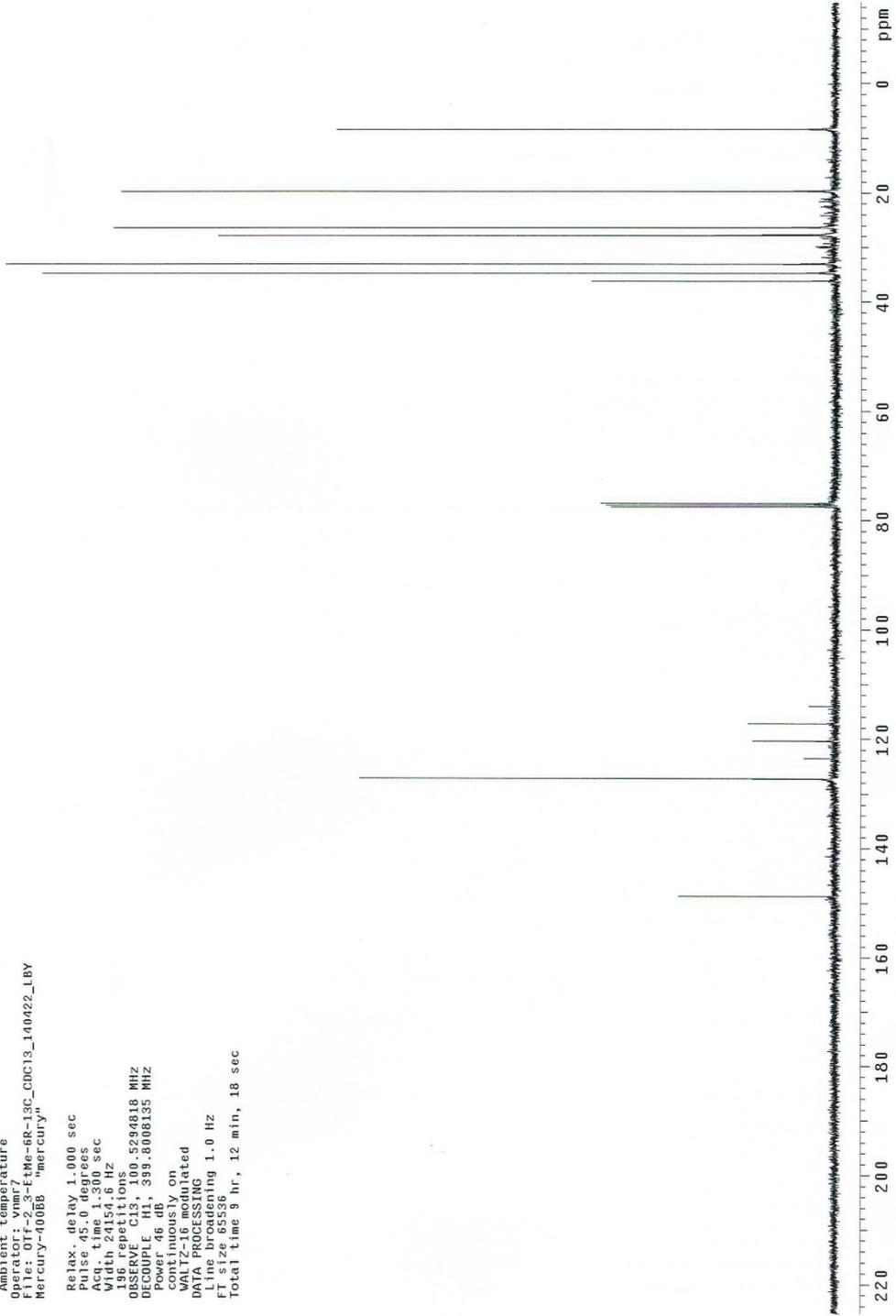


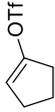


OTf-2_3-EtMe-6R-13C_CDC13_140422_LBY
 File: home/vnmr7/vnmrSYS/data/14_04/Lm/NewFolder/OTf-2_3-EtMe-6R-13C_CDC13_140422_LBY.fid

Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 Operator: vnmr7
 File: OTf-2_3-EtMe-6R-13C_CDC13_140422_LBY
 Mercury-40055 "mercury"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.380 sec
 Width 24154.6 Hz
 196 repetitions
 OBSERVE C13, 100.5294818 MHz
 DECOUPLE H1, 399.8008135 MHz
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 hr, 12 min, 18 sec





131

OTf-2a-1H_CDC13_131108_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Observer: jmm

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

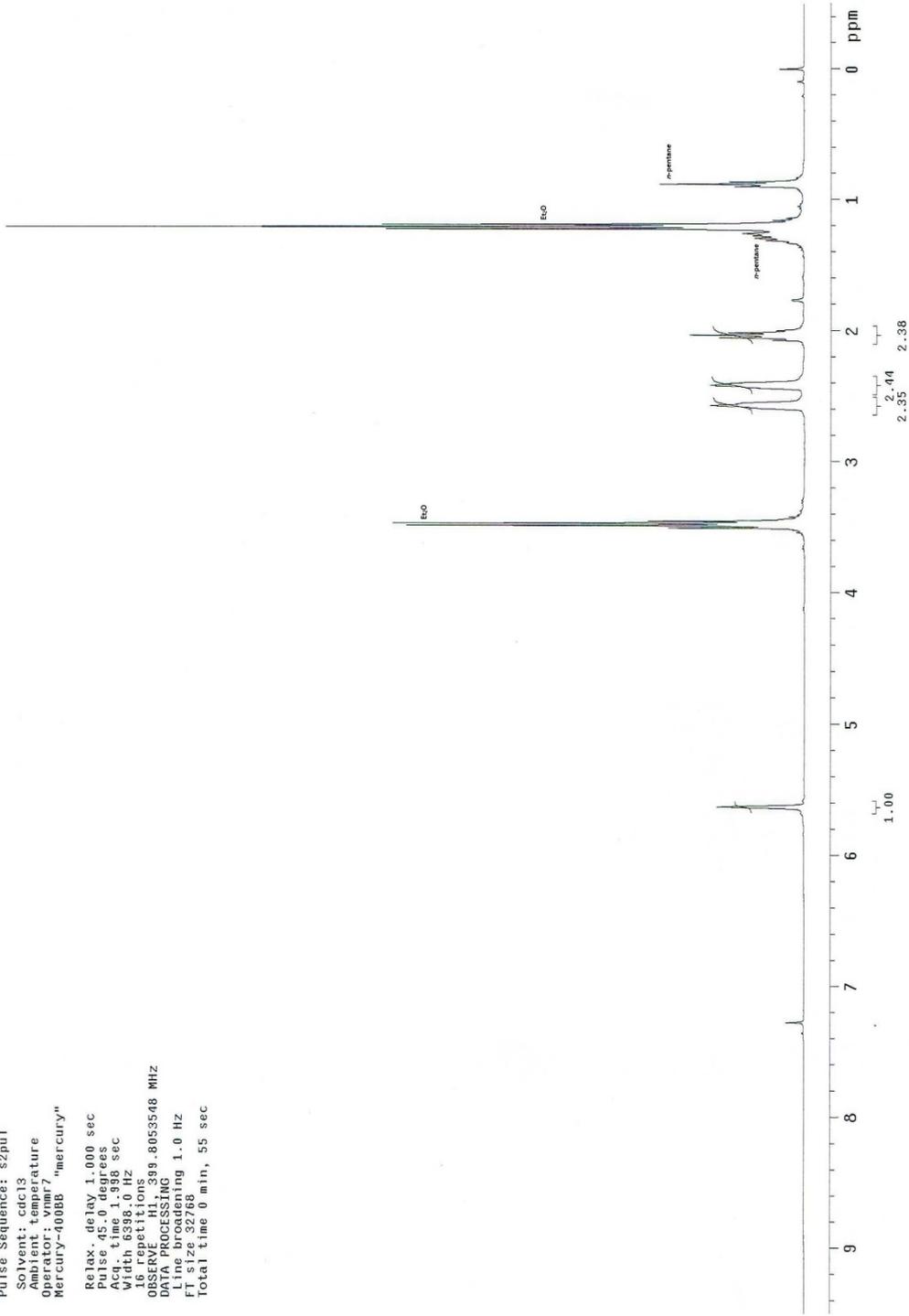
16 repetitions

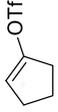
OBSERVE H1, 399.8053548 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 55 sec





13i

0Tf-2a-13C_CDCl3_131108_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Reference: 0ppm

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

604 repetitions

OBSERVE C13, 100.5311221 MHz

DECOUPLE H1, 399.8073969 MHz

coupling

continuously on

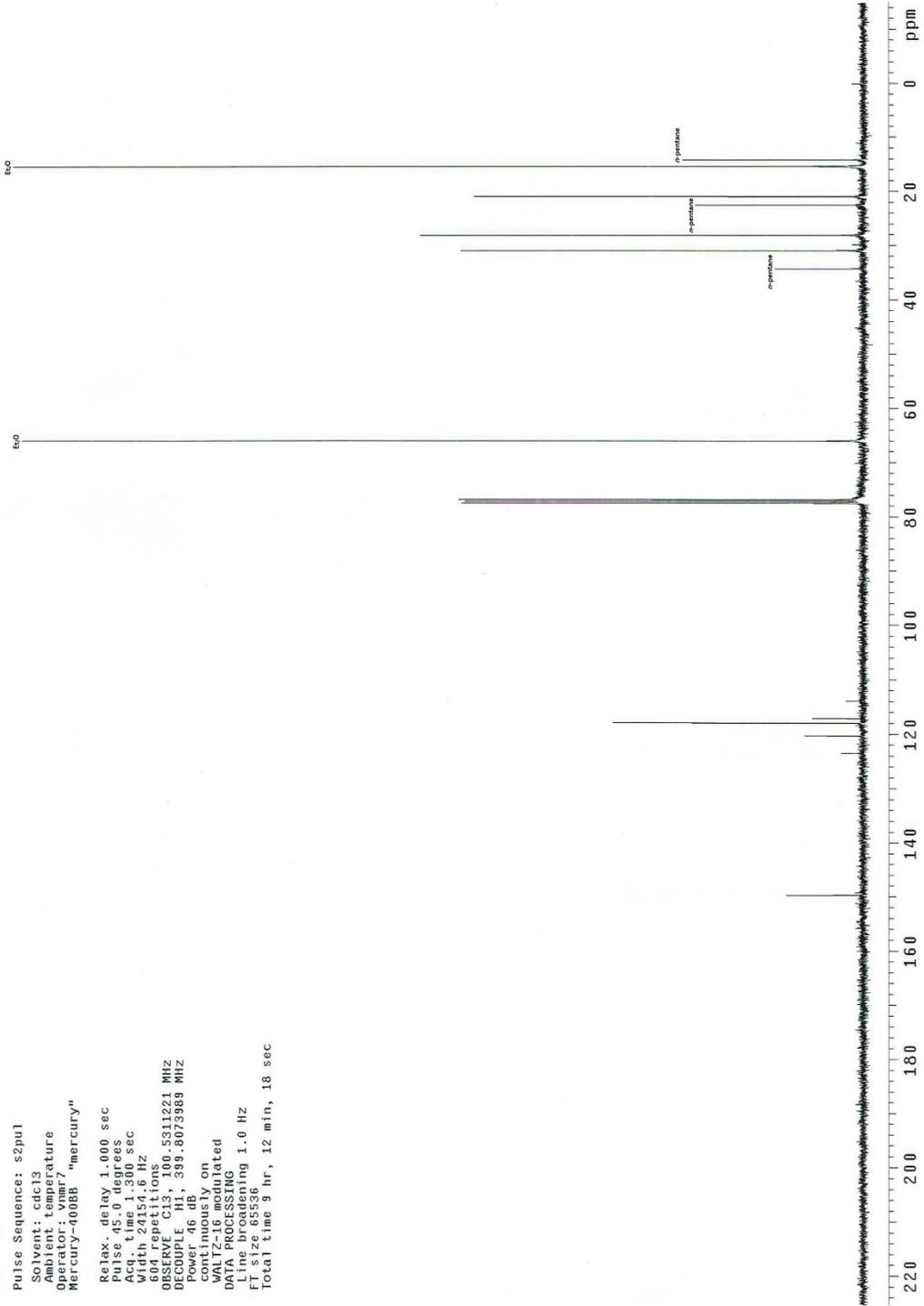
WALTZ-16 modulated

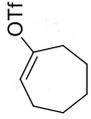
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





13j

OTf-7R-1H_CDCl3_131203_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vnmrj

Mercury-400BBB "mercury"

Relax. delay 1.000 sec

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

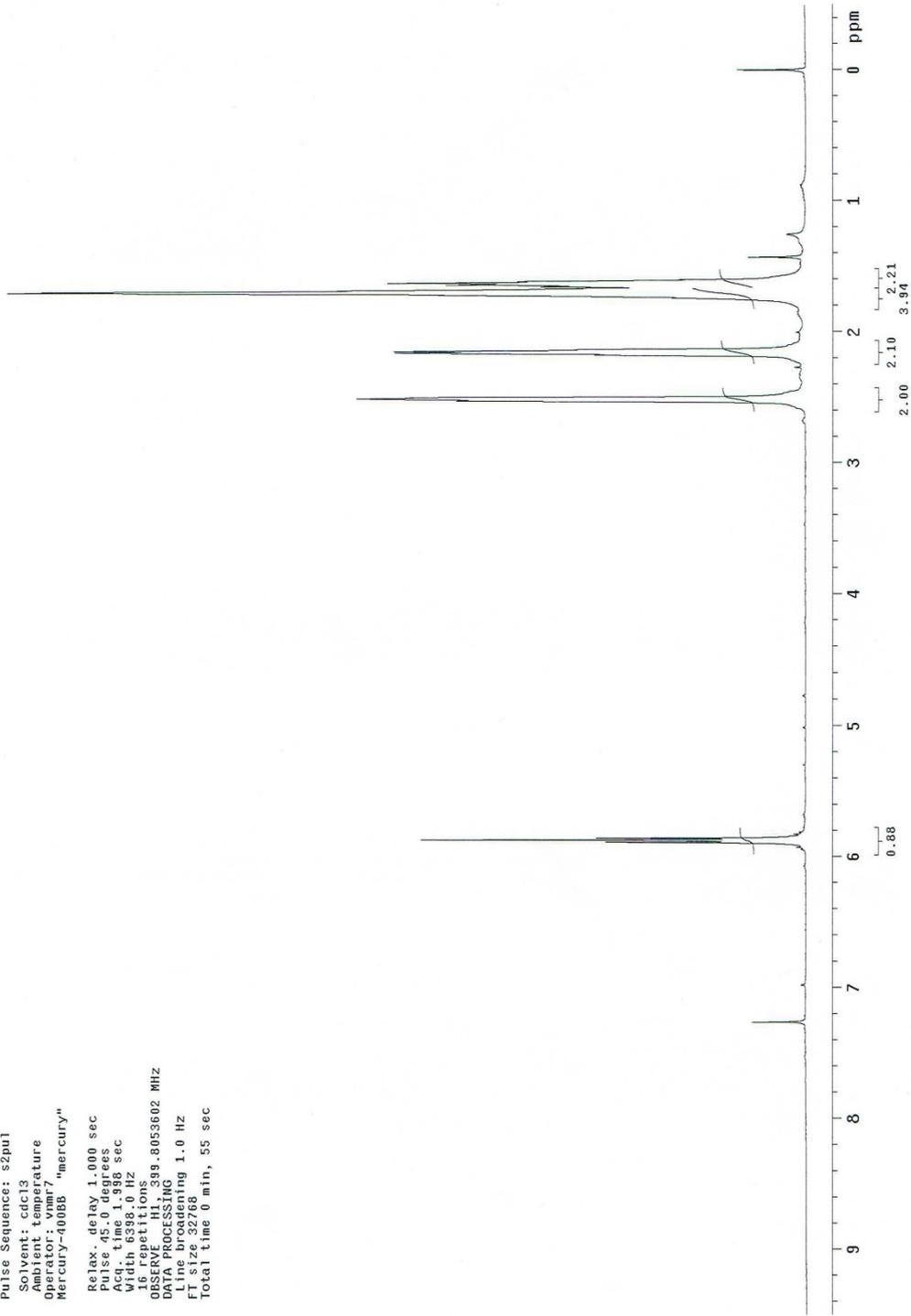
OBSERVE H1, 399.8053602 MHz

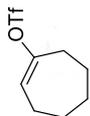
DATA PROCESSING

Line broadening 1.0 Hz

File: 131203

Total time 0 min, 55 sec





13j

0Tf-7R-13C_CDCl3_131202_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vnmr7

Mercury-0008B "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 18.000 sec

Width 24154.6 Hz

72 repetitions

OBSERVE C13, 100.5311214 MHZ

DECOUPLE H1, 399.8073989 MHZ

Power 46 dB

continuously on

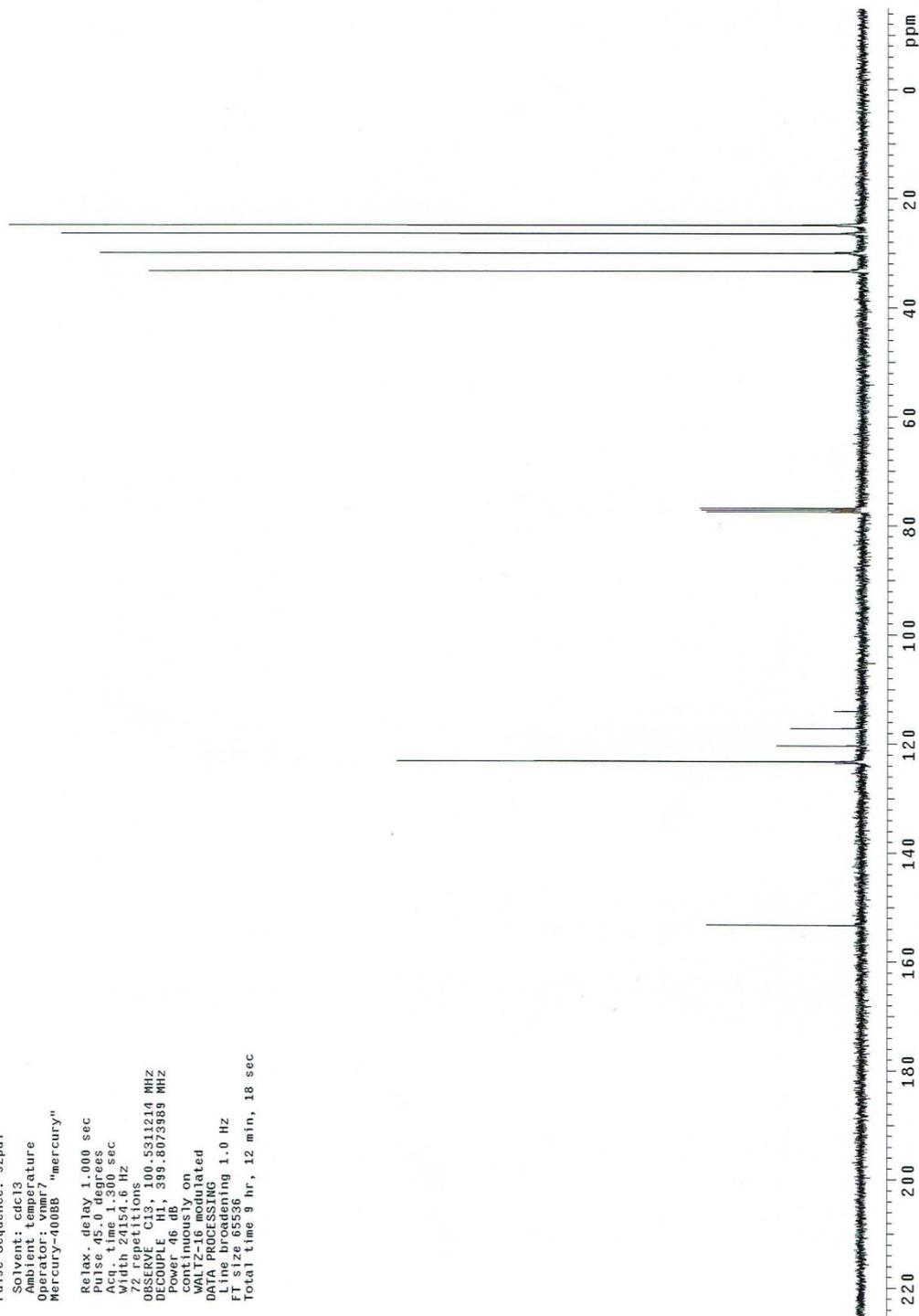
with 100% duty

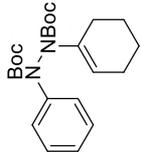
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

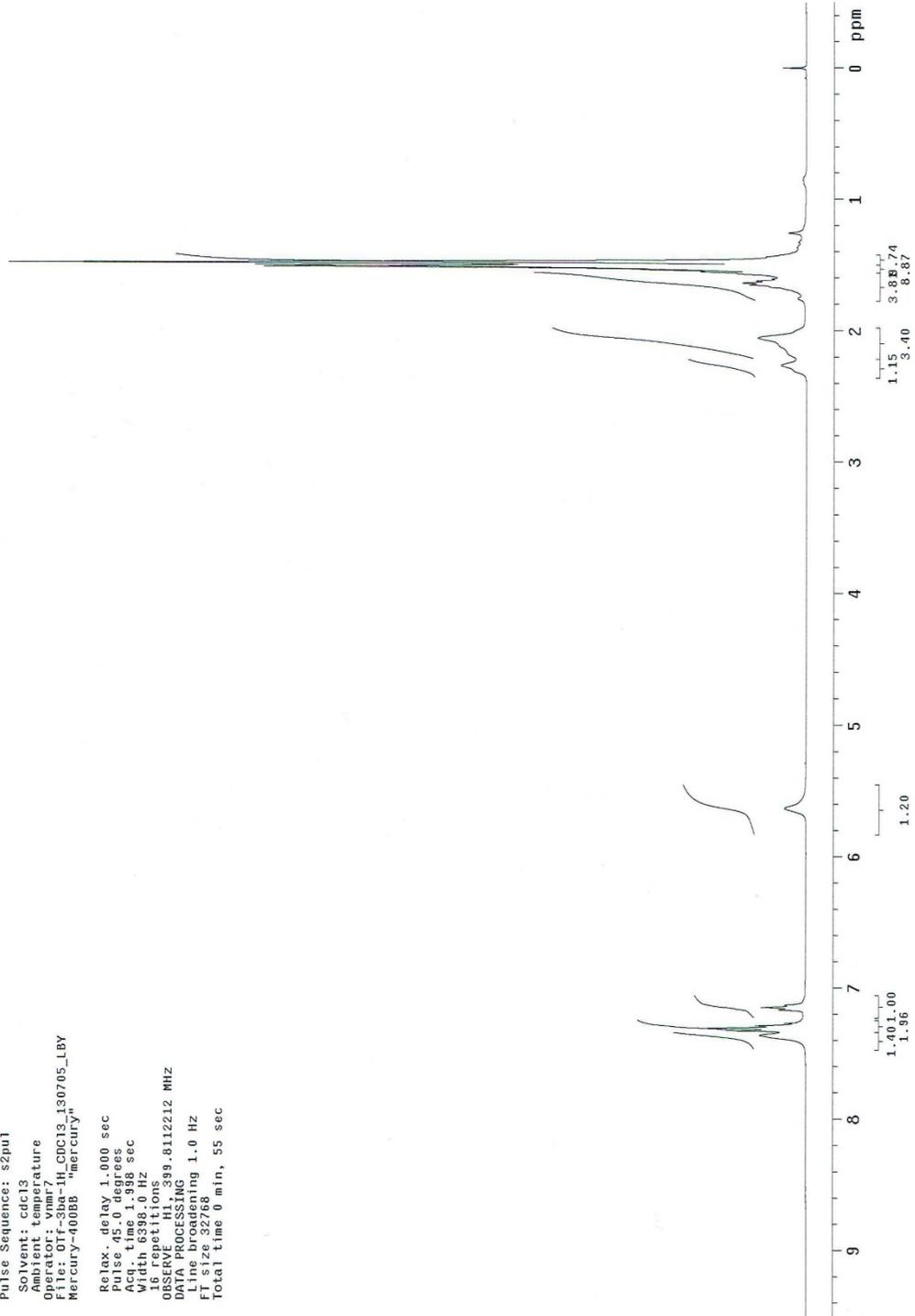
Total time 9 hr, 12 min, 18 sec

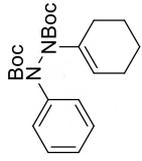




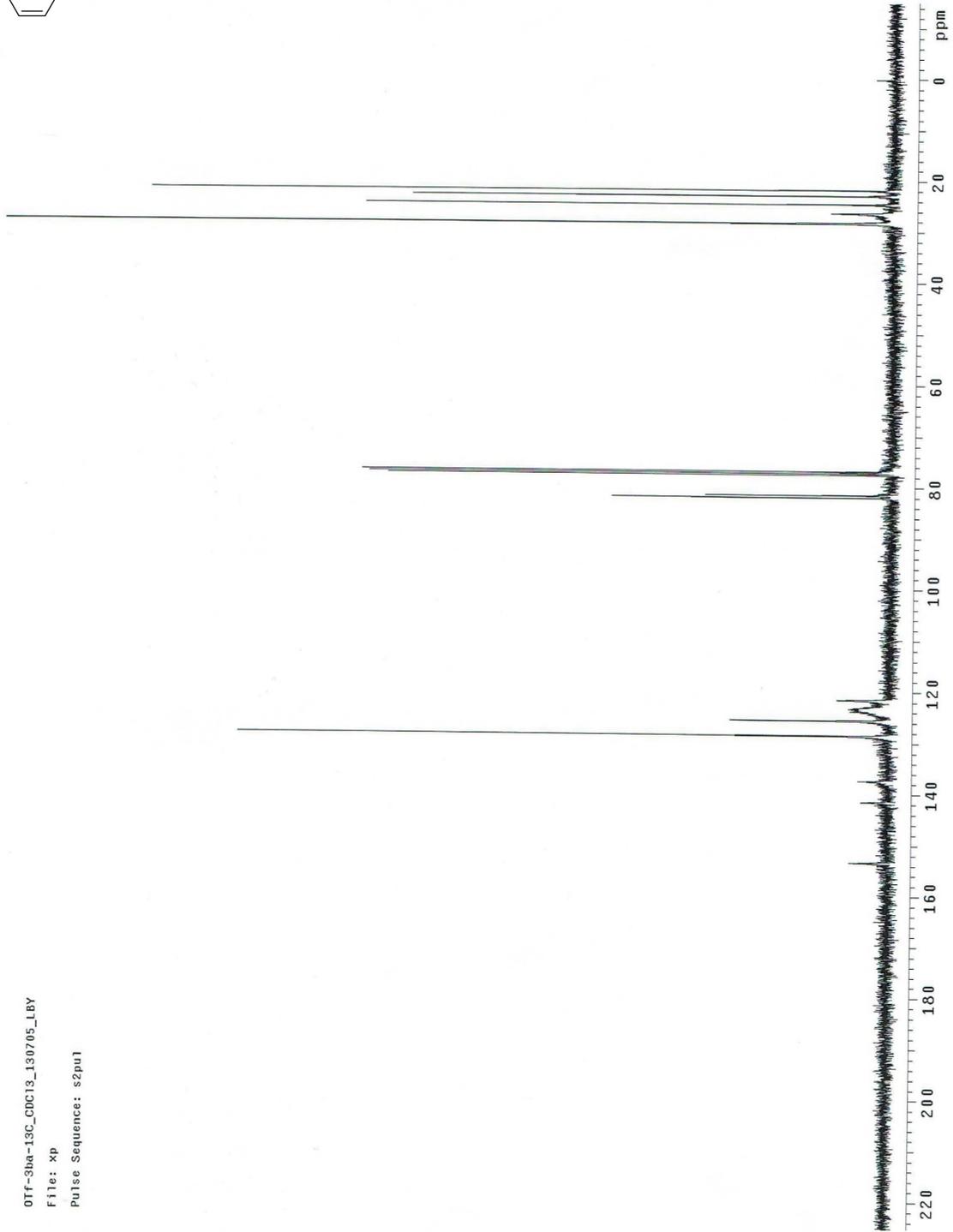
14a

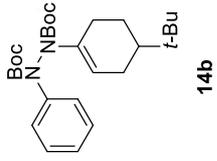
OTf-3ba-1H_CDC13_130705_LBY.fid
 File: home/vnmr7/vnmrSYS/data/13.05-14.03/Lm/OTf/3/OTf-3ba-1H_CDC13_130705_LBY.fid
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 Date_ Acquired: 13.05.14
 File: OTf-3ba-1H_CDC13_130705_LBY
 Mercury-40088B "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.998 sec
 Width 6398.0 Hz
 18 repetitions
 OBSERVED F1 F2
 0.00000000 99.8112212 MHz
 0.00000000 130.8251913 MHz
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec



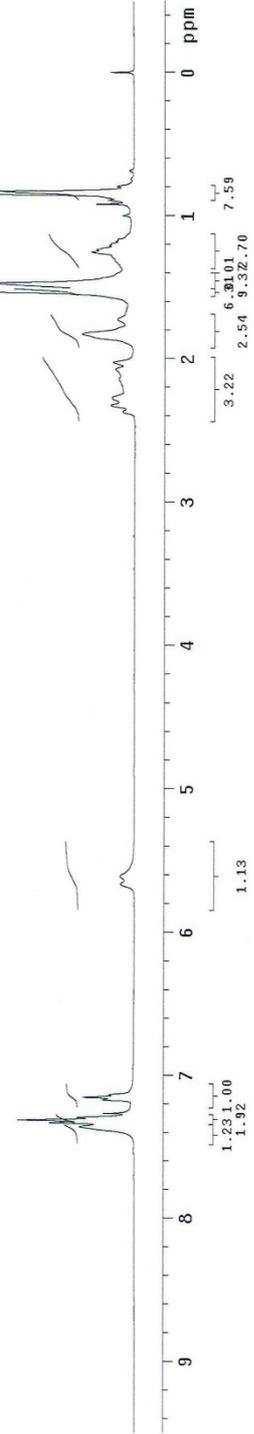


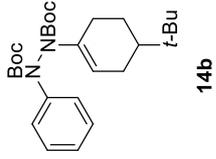
OTf-3ba-13C_CDCl3_130705_LBY
File: xp
Pulse Sequence: s2pu1



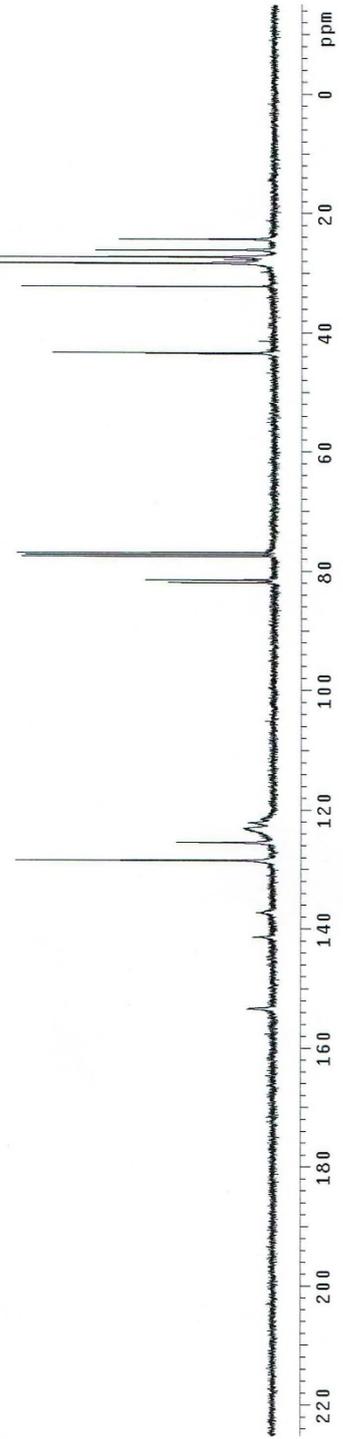


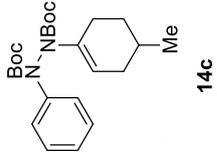
OTF-3c-1H_CDC13_130808_LBY
 File: home/vnmr7/vnmrSYS/data/13_05-14.03/Lim/OTF/3/OTF-3c-1H_CDC13_130808_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition Temperature
 Operator: vnmr7
 File: OTF-3c-1H_CDC13_130808_LBY
 Mercury-40088 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6398.0 Hz
 Observed F1 F2 F3 399.8112227 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec



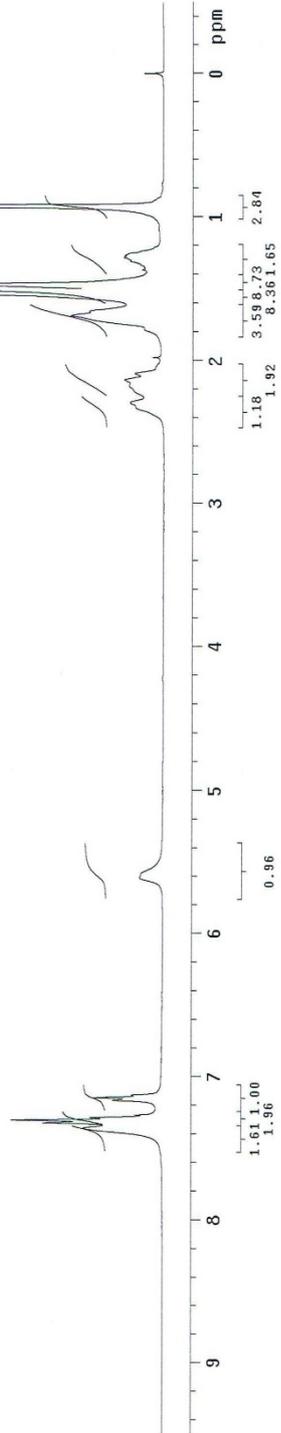


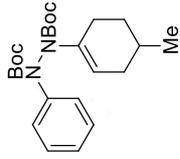
OTf-3c-13C_CDC13_130812_LBY
 File: home/vmm7/vmmrSYS/data/13-05-14.03/Lm/OTf/3/OTf-3c-13C_CDC13_130812_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 On-resonance
 File: OTf-3c-13C_CDC13_130812_LBY
 Mercury-40088 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 200 repetitions
 0.5326060 MHz
 DECOUPLE CH1, 599.8132388 MHz
 Power 46 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 F1 size 63536
 Total time 32 hr, 3 min, 3 sec





OTf-3_4-Me-6R-1H_CDC13_130823_LBY
 File: home/vnmr7/vnmrSYS/data/13-05-14_03/Lm/OTf/3/OTf-3_4-Me-6R-1H_CDC13_130823_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition Temperature
 Operator: vnmr
 File: OTf-3_4-Me-6R-1H_CDC13_130823_LBY
 Mercury-400BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6398.0 Hz
 Observations 39.8112208 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





OTf-3_4-Me-6R-13C_CDCl3_130823_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Operator: tom

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.360 sec

Width 24154.6 Hz

400 repetitions

OBSERVE C13, 100.5326045 MHz

PCOUPLE H1, 399.8132388 MHz

continuously on

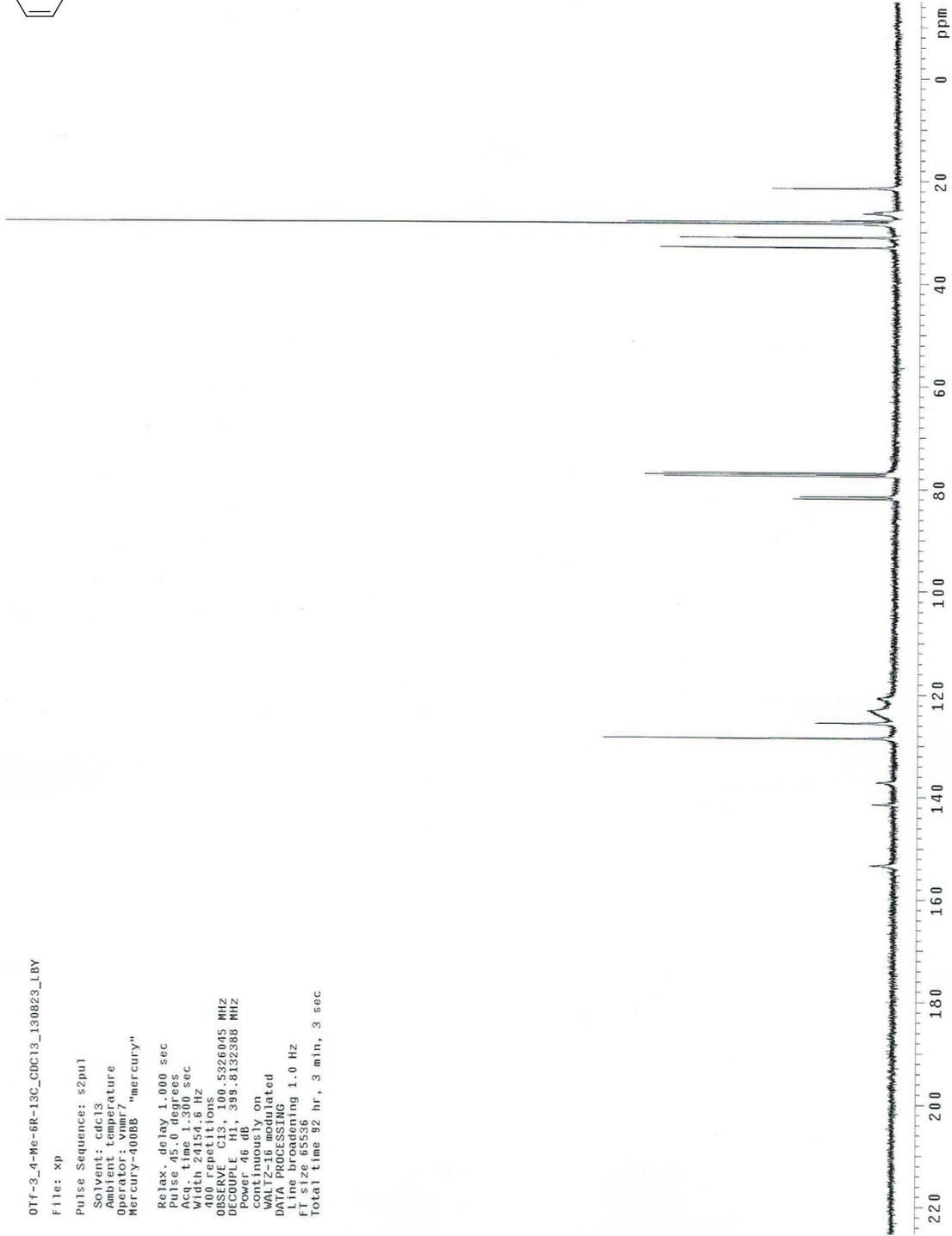
WALTZ-16 modulated

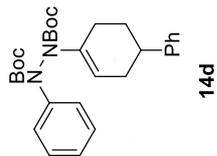
DATA PROCESSING

Line broadening 1.0 Hz

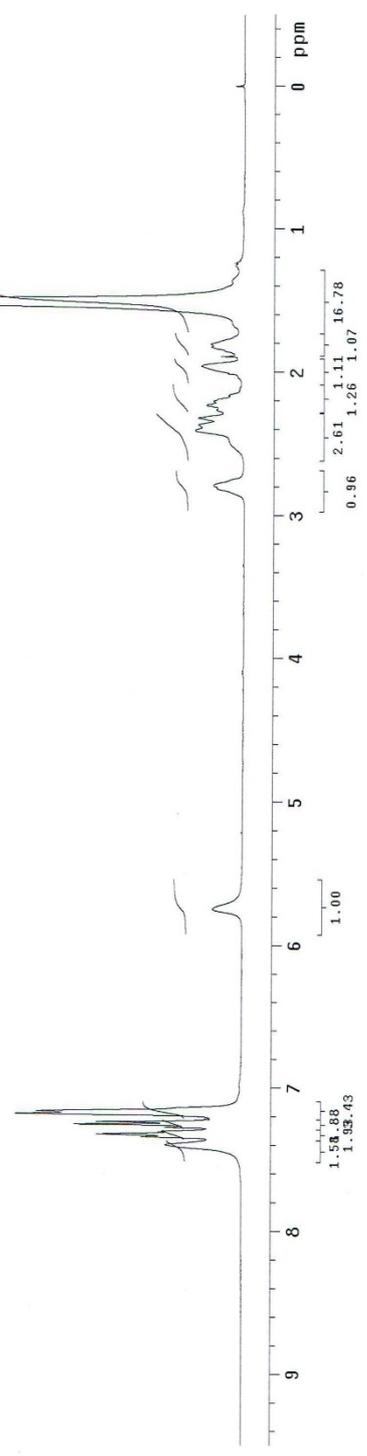
FI size 65536

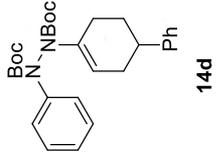
Total time 92 hr, 3 min, 3 sec



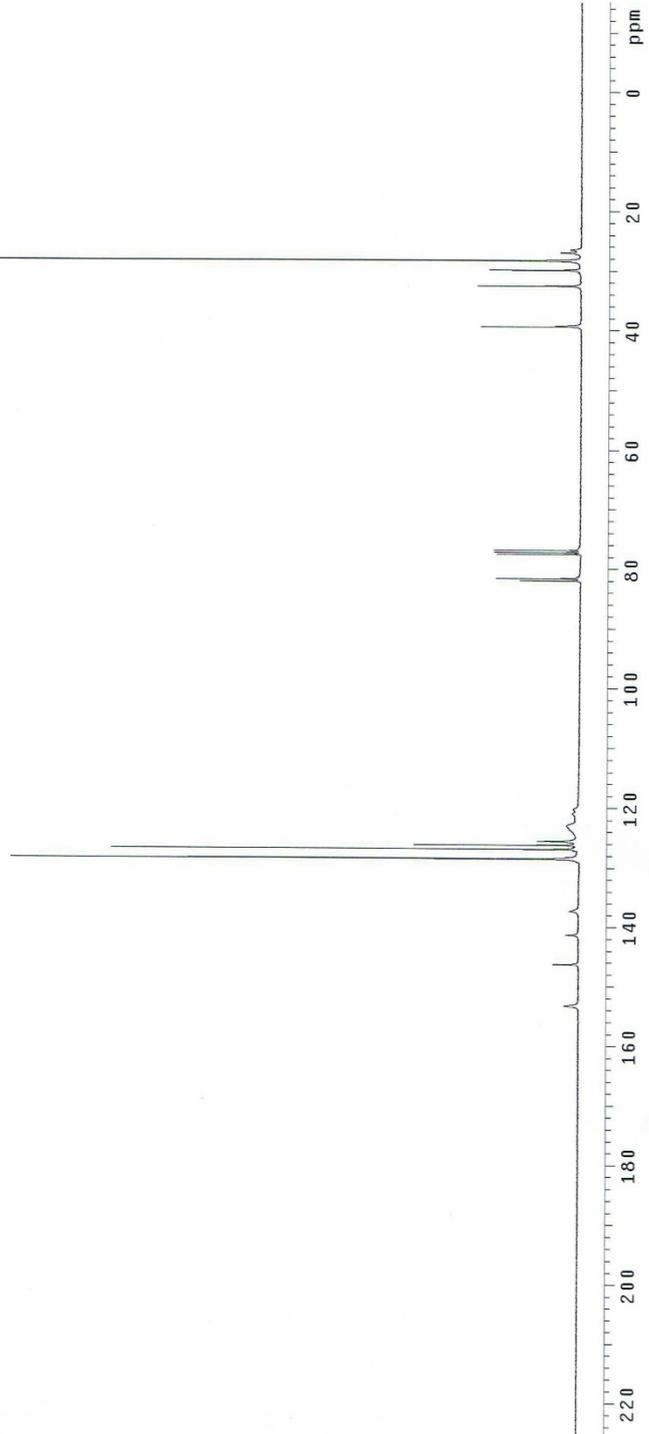


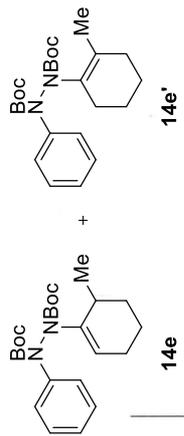
OTF-3-4-Ph-6R-1H_CDC13_131002_LBY
 File: home/vmmr7/vmmrSYS\data/13-05-14.03/Lm/OTF/3/OTF-3-4-Ph-6R-1H_CDC13_131002_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition temperature
 Operator: vmmr7
 File: OTF-3-4-Ph-6R-1H_CDC13_131002_LBY
 Mercury-40088 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6398.0 Hz
 Spectrometer 399.8053794 MHz
 OBSERVE H1
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec



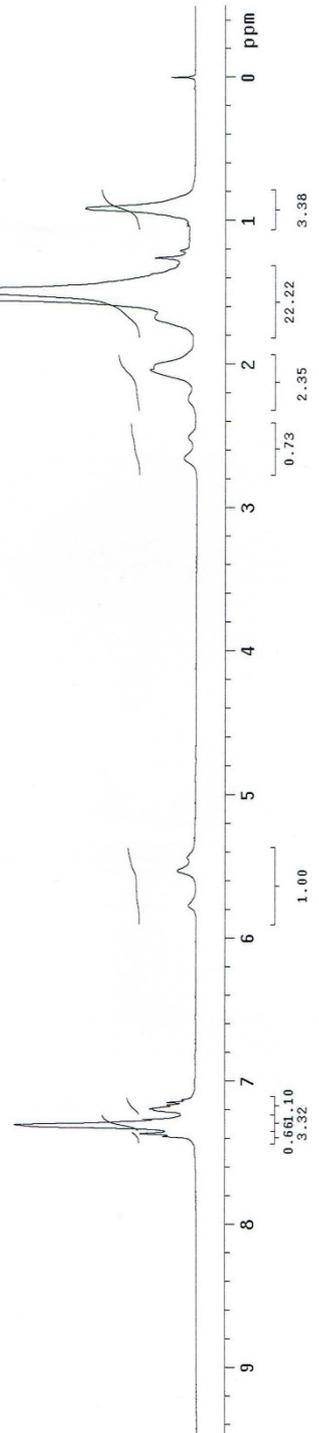


OTF-3_4-Ph-6R-13C_CDCl3_131002_LBY
 File: home/vmmr7/vmmrSYS/data/LBY/OTF/3/L1m.fid
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 File: C:\vmmr7
 Mercury-400BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 1260 repetitions
 RECEIVE C13, 100.5311435 MHz
 DECODE H1, 399.8973969 MHz
 Power 66 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 65536
 Total time 9 hr, 12 min, 18 sec





OTf-3_6-Me-6R-1H_CDC13_140520_LBY
 File: home/vnmr7/vnmrsys/data/14_05/1im/OTf-3_6-Me-6R-1H_CDC13_140520_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition Temperature: 300.2 K
 Operator: vnmr7
 File: OTf-3_6-Me-6R-1H_CDC13_140520_LBY
 Mercury-400BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6398.0 Hz
 Spectroscopy Observations 99.7988467 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec



OTf-3,6-Me-6R-13C_CDC13_140514_LBY

File: home/vnmr7/Lim140513.f1d

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

0 deg Celsius

File: Lim140513

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

12272 repetitions

0.2272 sec

DECODE CH, 399.808135 MHz

Power 46 dB

continuously on

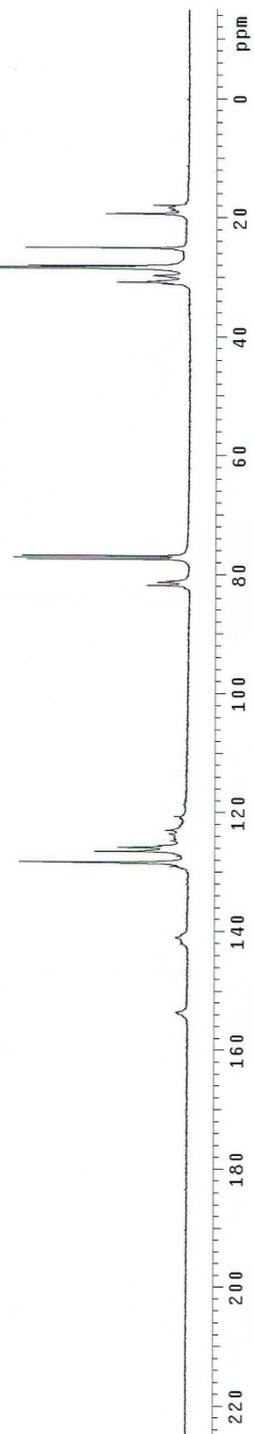
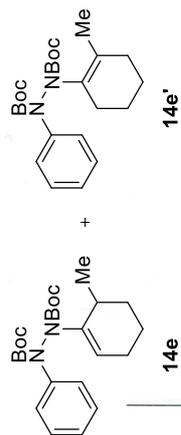
WALTZ-16 modulated

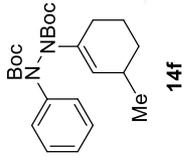
DATA PROCESSING

Line broadening 1.0 Hz

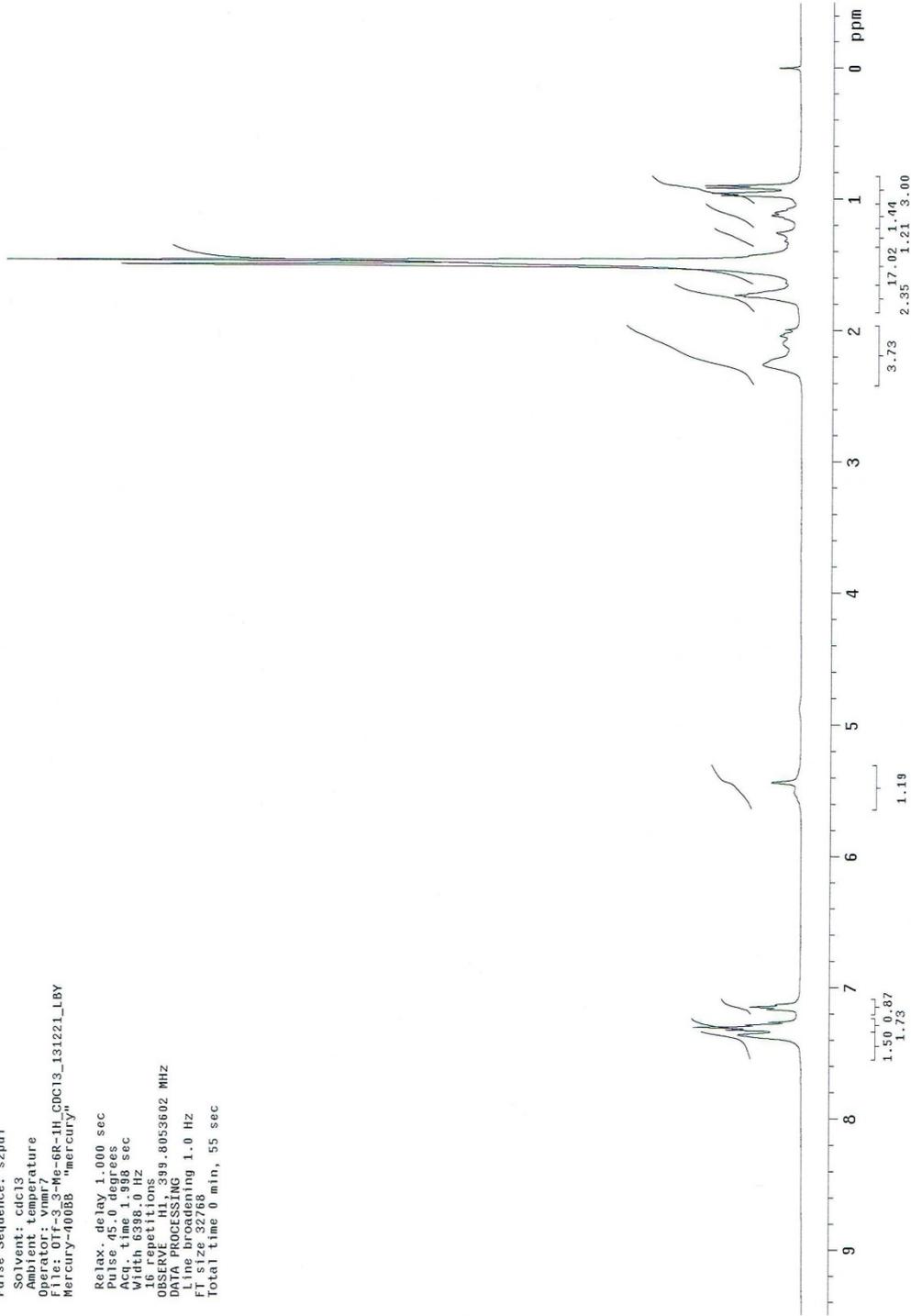
FT size 65536

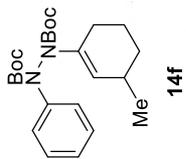
Total time 32050 hr, 56 min, 0 sec





OTf-3_3-Me-6R-1H_CDC13_131221_LBY
 File: home/vmmr7/vmmrSYS/data/13_05-14.03/Lm/OTf/3/OTf-3_3-Me-6R-1H_CDC13_131221_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition Temperature
 Operator: vmmr
 File: OTf-3_3-Me-6R-1H_CDC13_131221_LBY
 Mercury-40088 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6398.0 Hz
 Line positions 99.8053602 MHz
 OBSERVE: 1H
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





01f-3_3-Me-6R-13C_CDCl3_140102_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Amplitude: 10.0000

Operator: vmm7

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

836 repetitions

NUC1 13, 100.626130 MHz

DECOUPLE 1H, 399.3073689 MHz

Power 46 dB

continuously on

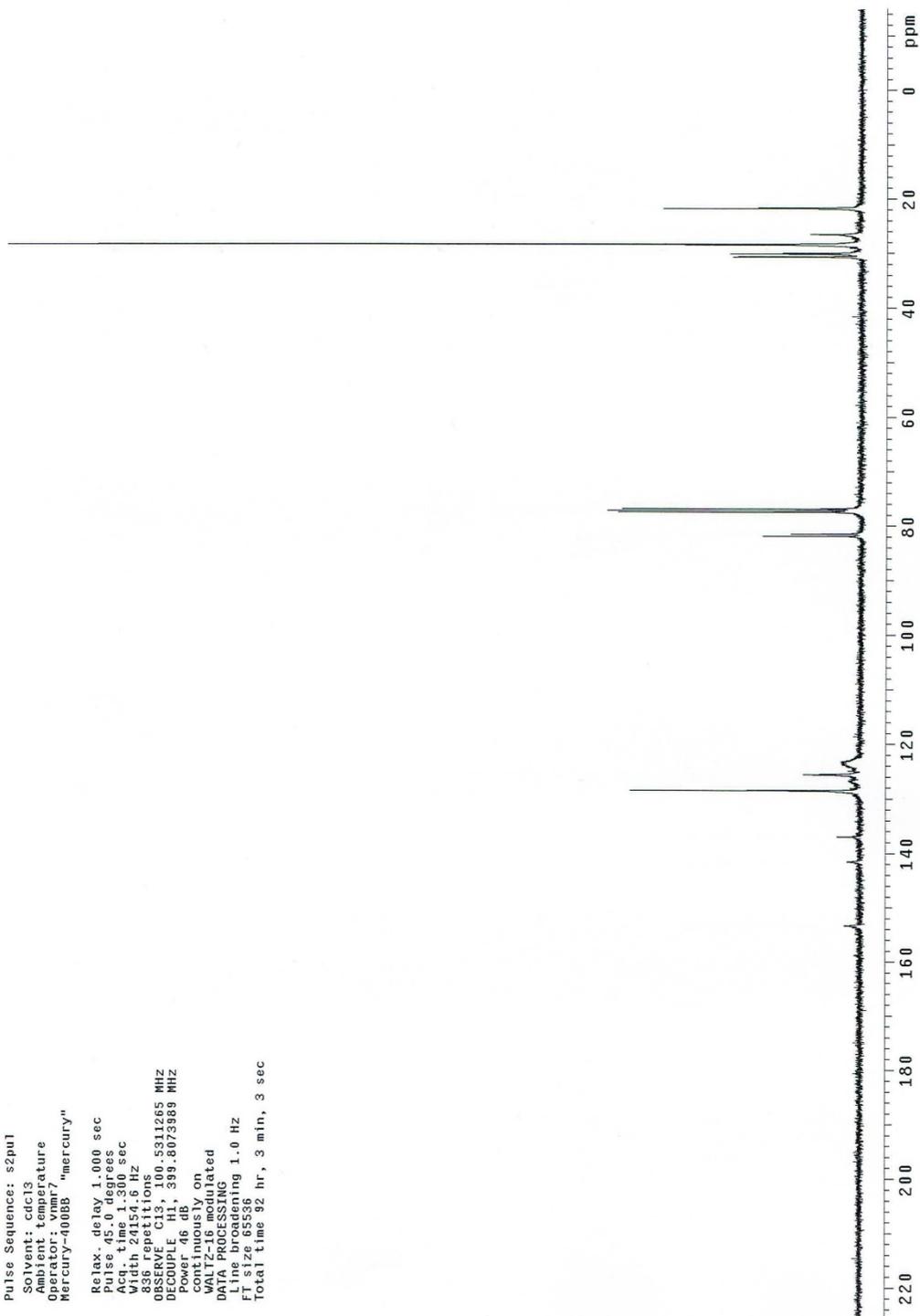
WALTZ-16 modulated

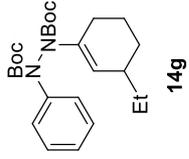
DATA PROCESSING

Line broadening 1.0 Hz

F1 size 65536

Total time 32 hr, 3 min, 3 sec





0Tf-3-Et-6R-1H_C0C13_131105_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Shimadzu temperature

Operator: tsm

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

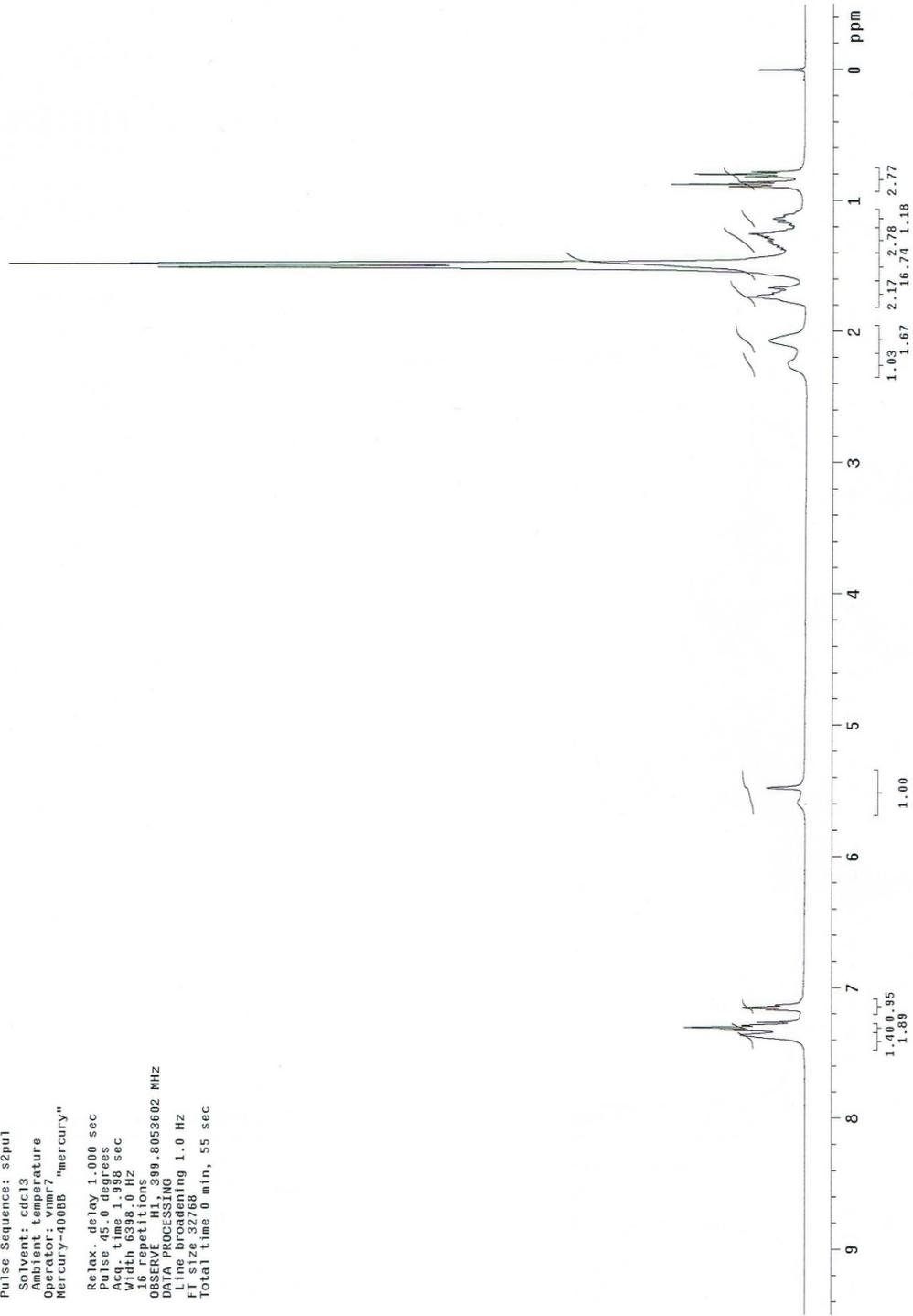
OBSERVED F1 399.8053602 MHz

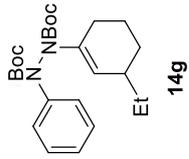
DATE_000511

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





0Tf-3_3-Et-6R-13C_CDCl3_131105_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

124 repetitions

DESCRIBE C13, 100.5511376 MHz

Power 46 dB, 399.8073989 MHz

continuously on

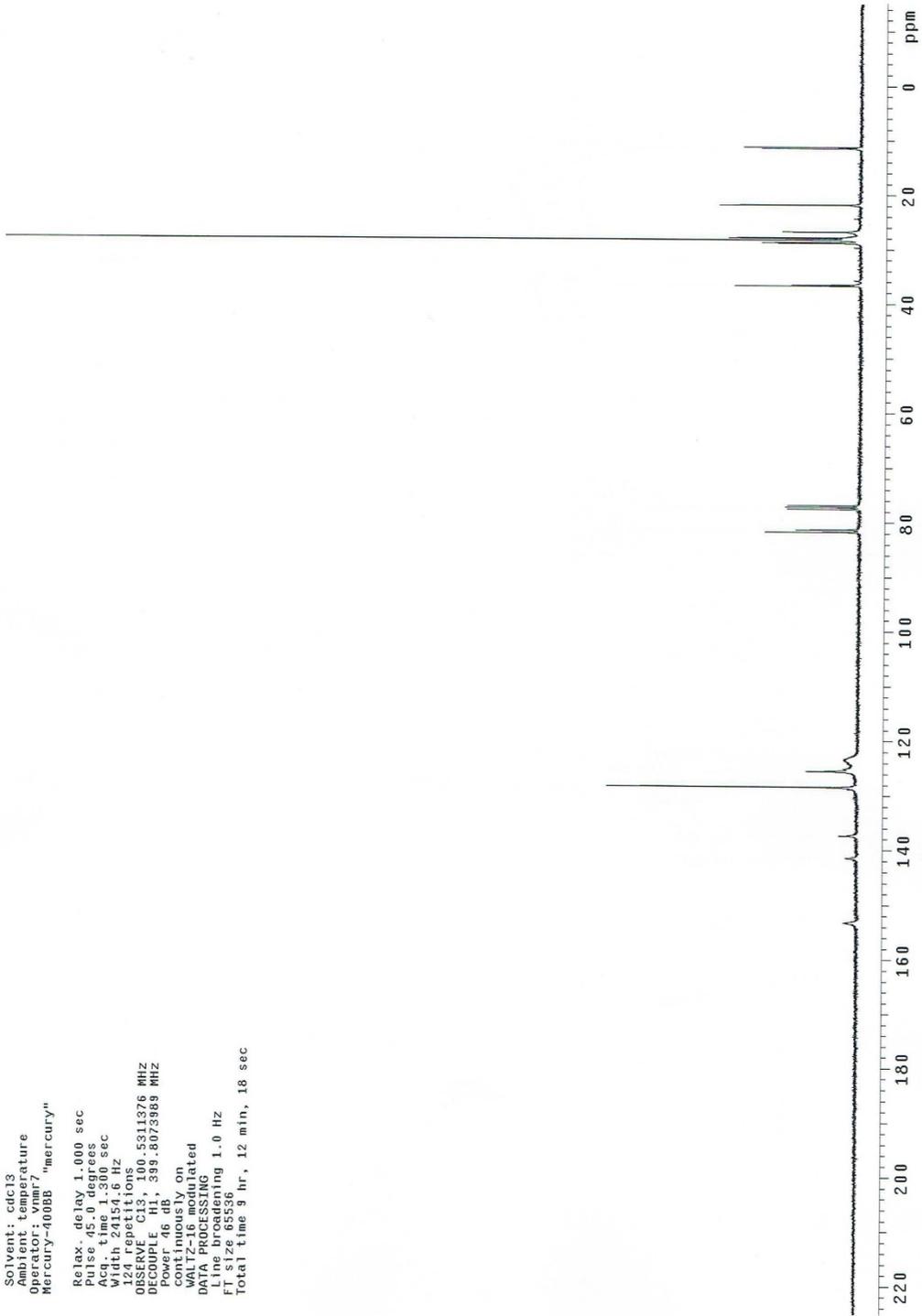
WALTZ-16 modulated

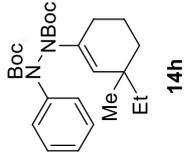
DATA PROCESSING

Line broadening 1.0 Hz

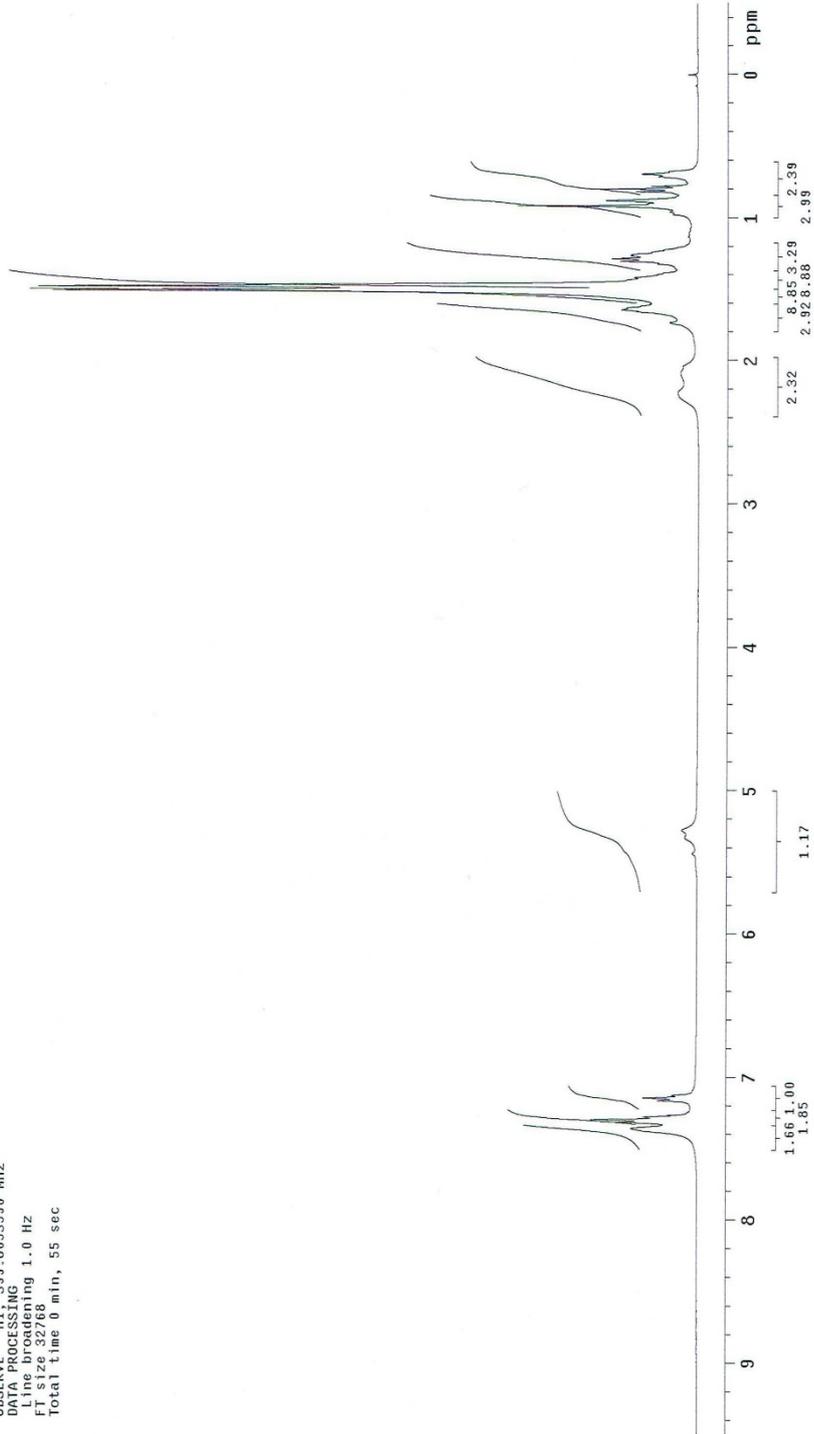
FI size 65536

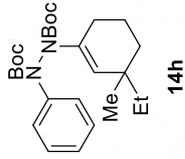
Total time 9 hr, 12 min, 18 sec





OTF-3_3-Et-Me-6R-1H_CDC13_131209_LBY
 File: home/vnmr7/vnmrSYS/data/13.05-14.03/Lim/OTF/3/OTF-3_3-Et-Me-6R-1H_CDC13_131209_LBY.fid
 Pulse Sequence: s2pul1
 Solvent: cdcl3
 Ambient temperature
 File: OTF-3_3-Et-Me-6R-1H_CDC13_131209_LBY
 Mercury-4000BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.998 sec
 Width 6398.0 Hz
 18 repetitions
 OBSERVED F1 F2 99.8053590 MHZ
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





0Tf-3-Et-Me-6R-13C_CDCl3_131209_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vmm7

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

NUC1 13C

244 repetitions

OBSERVE C13, 100.5311273 MHz

DECOUPLE H1, 399.8073989 MHz

Power 46 dB

continuously on

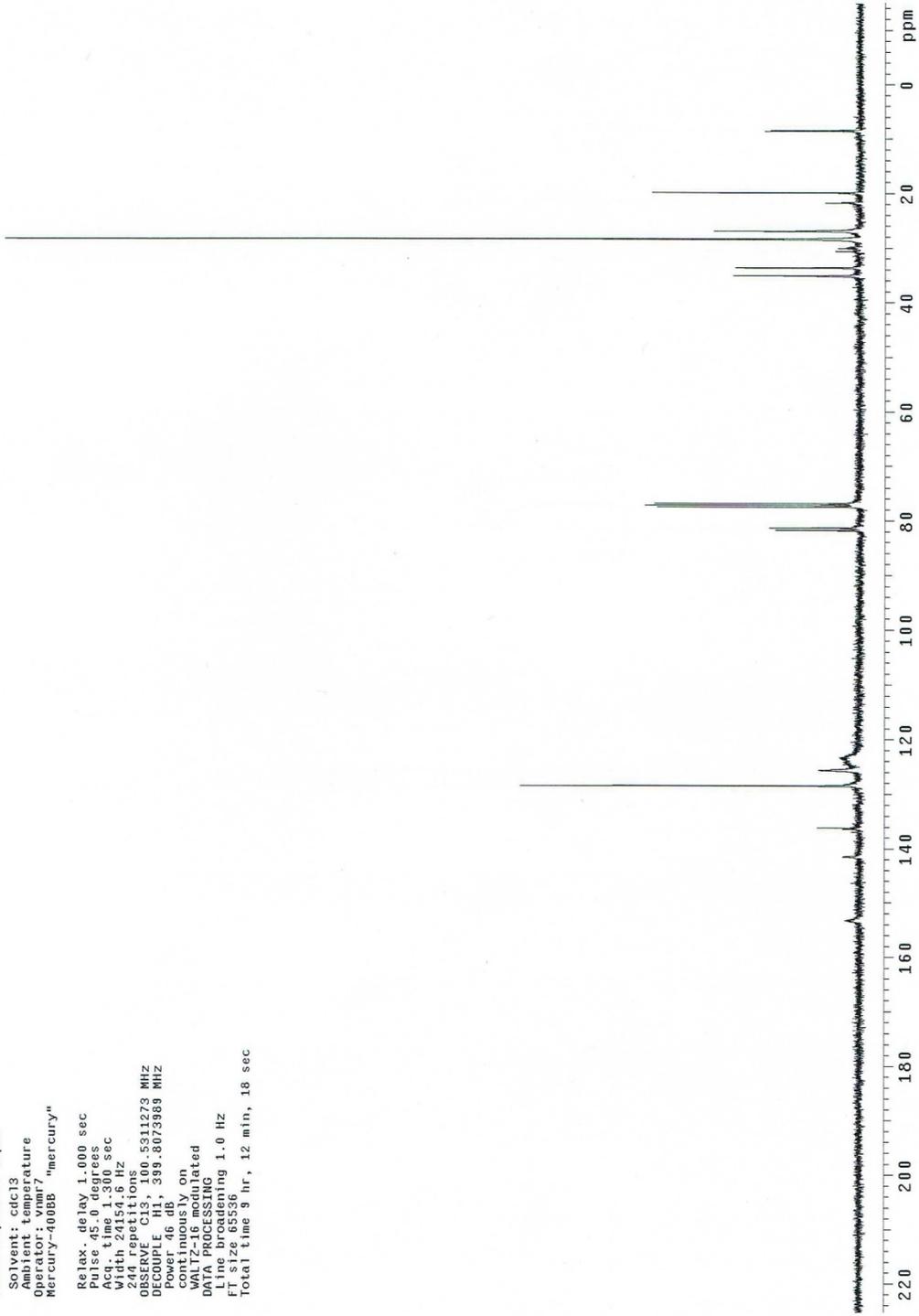
WALTZ16 modulated

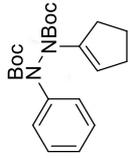
DRPG on

LINE PROTECTING 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





OTF-3_5R-1H_CDCl3_140516_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vmm7

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.988 sec

Width 6398.0 Hz

IS repetitions

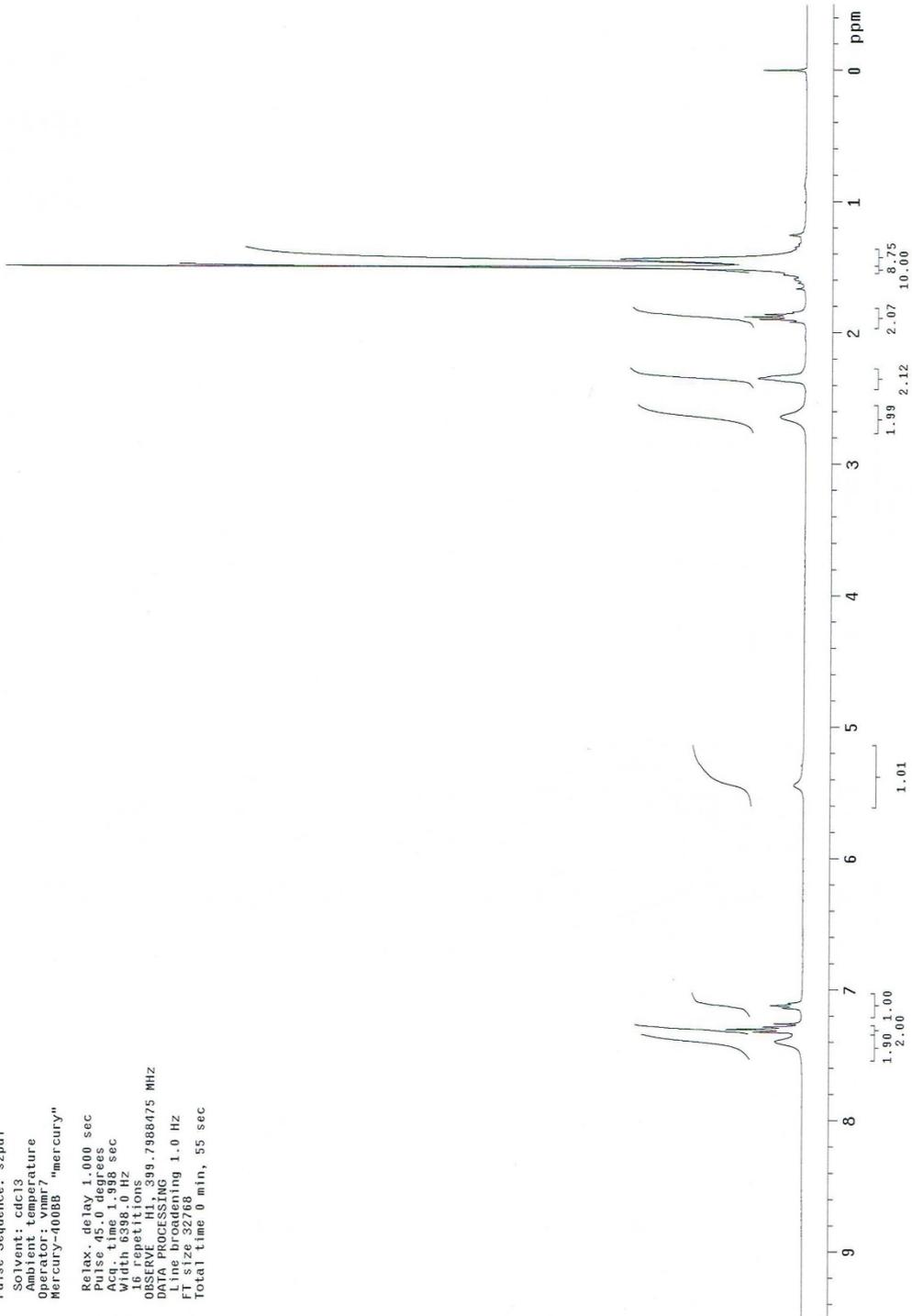
OPERATION 99.7988475 MHz

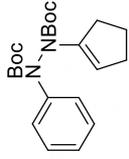
DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

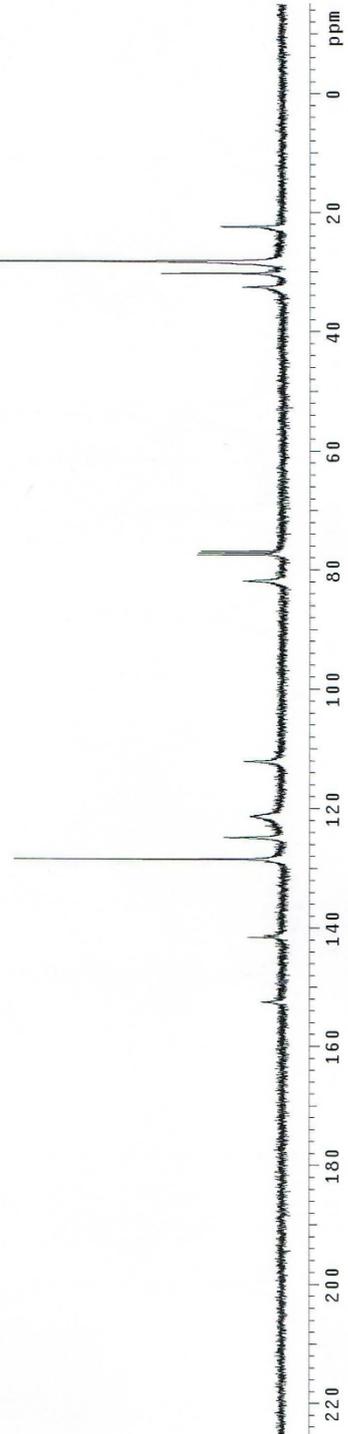
Total time 0 min, 55 sec

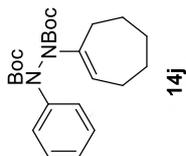




OTf-3_5R-13C_CDC13_140515_LBY
 File: home/vnmr7/vnmrSYS/data/14.05/Lim/OTf-3_5R-13C_CDC13_140515_LBY.fid

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Operator: vnmr7_13C_CDC13_140515_LBY
 Mercury-40088 "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 124 repetitions
 OBSERVE C13, 100.5294958 MHz
 DECOUPLE H1, 399.8006135 MHz
 Post-acquisition processing
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 920 hr, 30 min, 34 sec

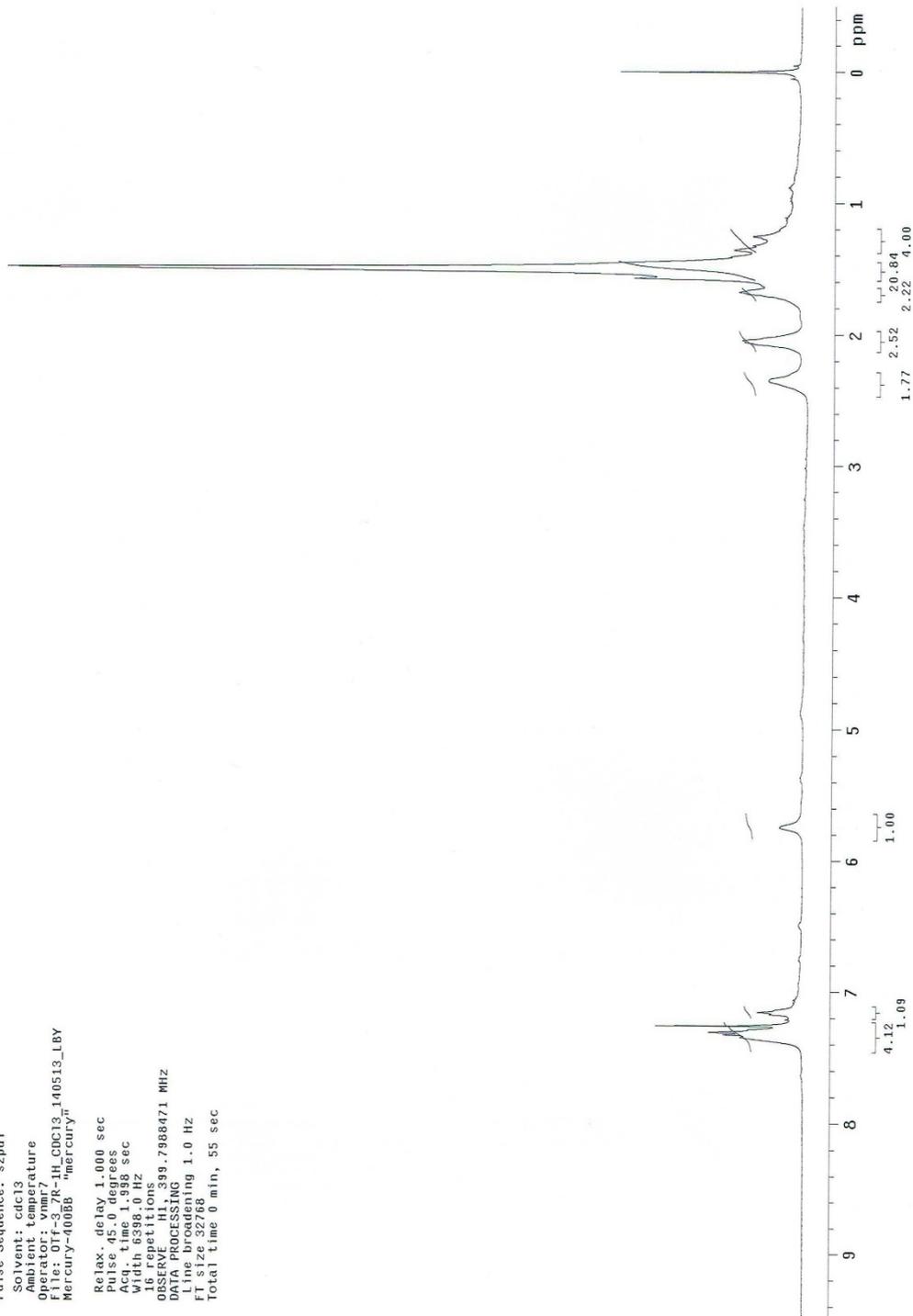


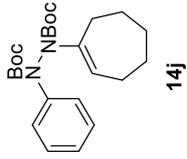


OTf-3_7R-1H_CDC13_140513_LBY
 File: home/vnmr7/vnmrSYS/data/14.05/Lm/OTf-3_7R-1H_CDC13_140513_LBY.fid

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Operator: vnmr7
 File: OTf-3_7R-1H_CDC13_140513_LBY
 Mercury-400BB "mercury"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6396.0 Hz
 Spectrometer 399.7968471 MHz
 OBSERVE H1
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





OTf-3_7R-13C_CDCl3_140513_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: cmr

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.900 sec

Width 24154.6 Hz

13328 repetitions

OBSERVE C13, 100.5294848 MHz

DECOUPLE H1, 399.8006135 MHz

Power level

continually on

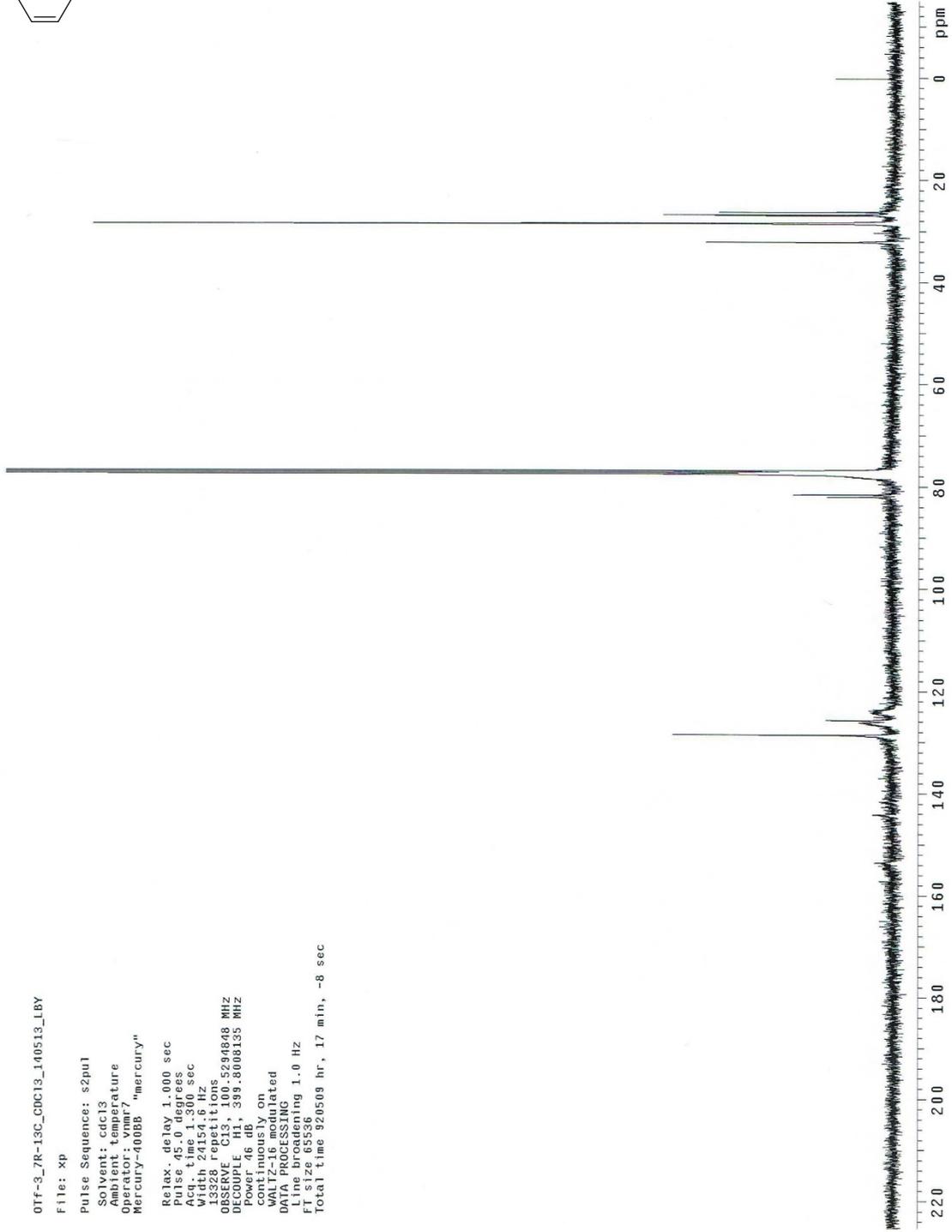
WALTZ-16 modulated

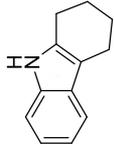
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 920509 hr, 17 min, -8 sec





7

0Tf-4ba-1H_CDC13_140529_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdc13

Ambient temperature

Operator: vmmr

Mercury-100BB "mercury"

Relax. delay 1.000 sec

Pulse delay 0.000 sec

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

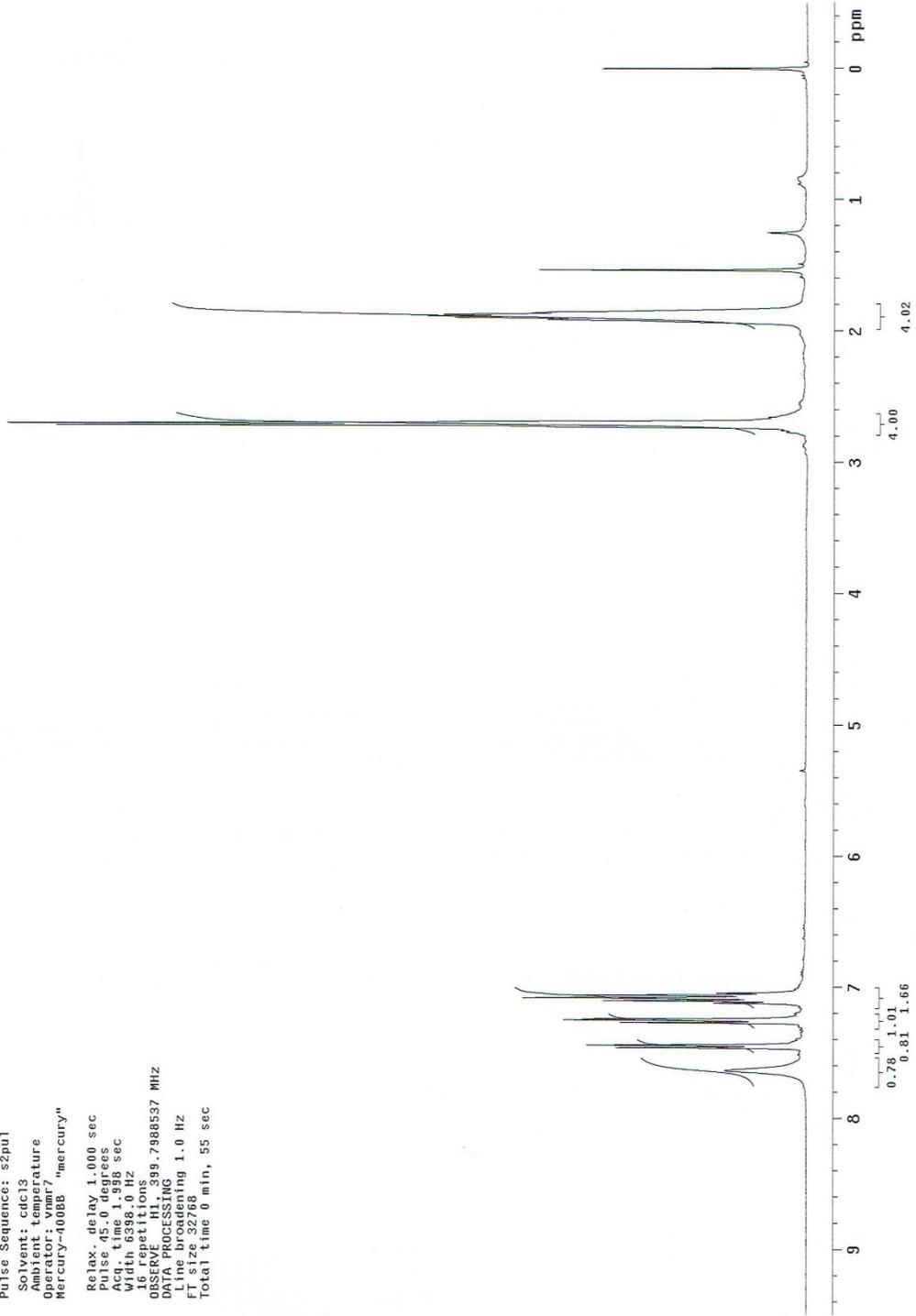
OBSERVE H1, 399.7986537 MHz

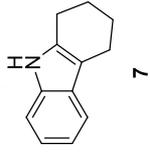
DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





OTf-4ba-13C_CDCl3_130808_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vmm

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Puls. 45 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

160 repetitions

OBSERVE C13, 100.5326030 MHz

DECOUPLE H1, 399.8132388 MHz

Power level

Continuously on

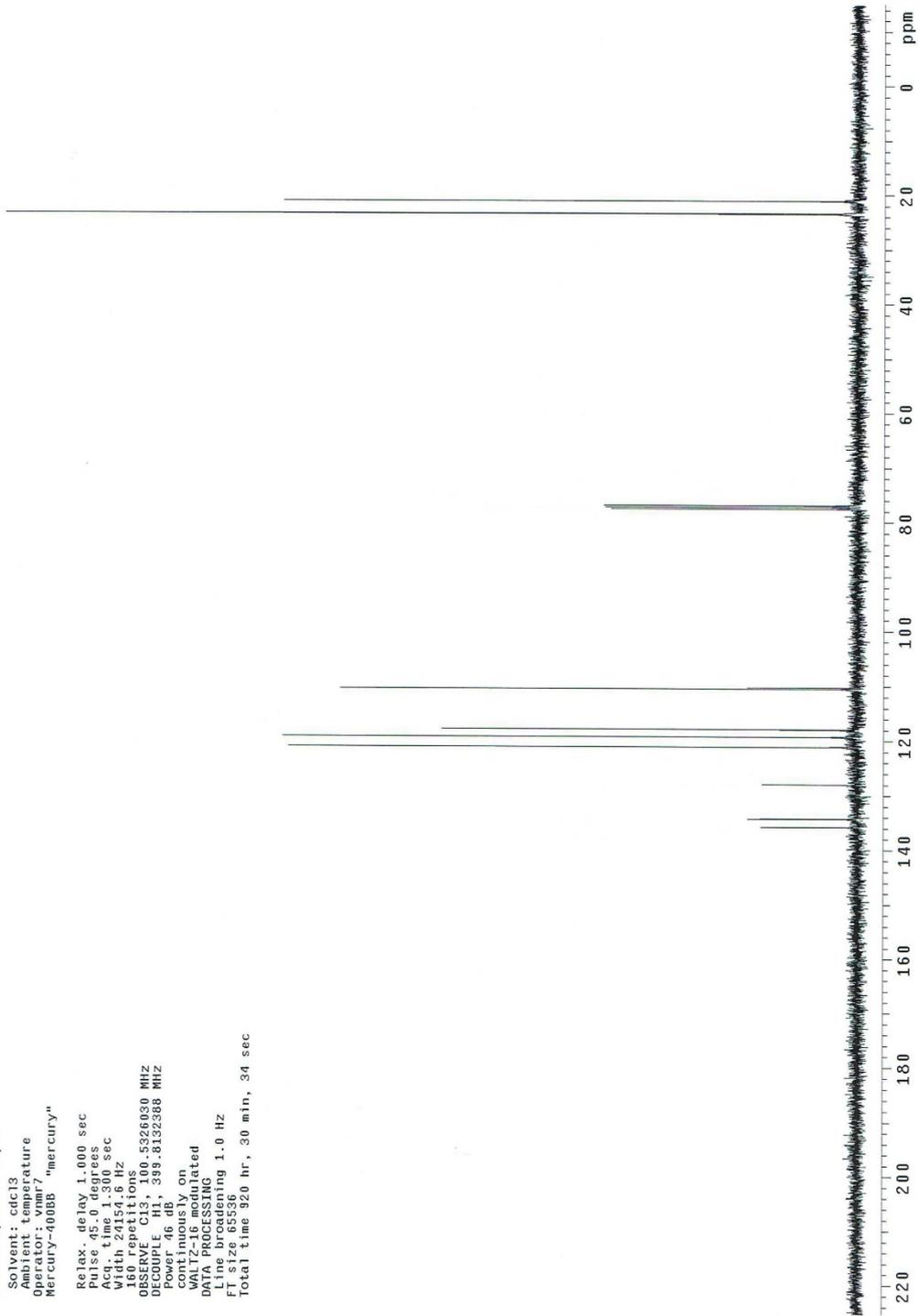
WALTZ-16 modulated

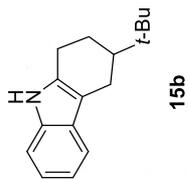
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 920 hr., 30 min, 34 sec





0Tf-4c-1H_CDCl3_130810_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: ymm

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Puls. 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

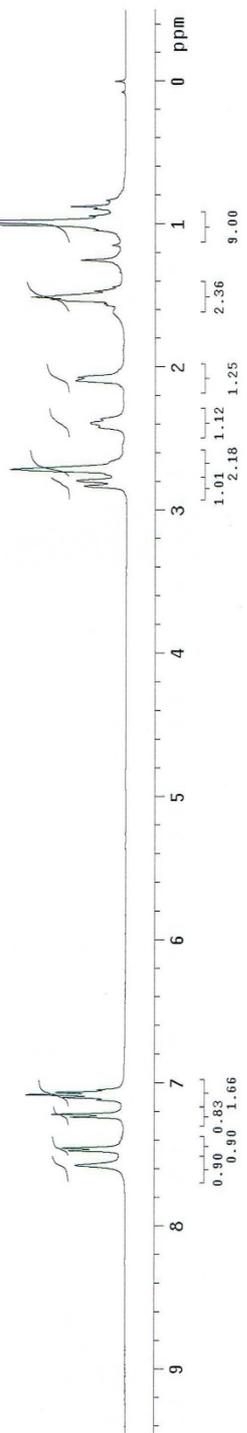
16 repetitions

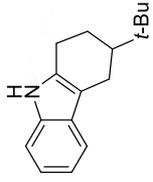
OBSERVE H1, 399.8112430 MHz

DATA PROCESSING

F1 size 32768

Total time 0 min, 55 sec





0Tf-4c-13c_CDCl3_130810_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vnmr2

Mercury-4000B "mercury"

Relax. delay 1.000 sec

Puls. delay 0.000 sec

Acq. time 1.390 sec

Width 24154.6 Hz

208 repetitions

OBSERVE C13, 100.5326038 MHz

DECOUPLE H1, 399.8132388 MHz

Power 46.00 dB

Gate 100.00 dB

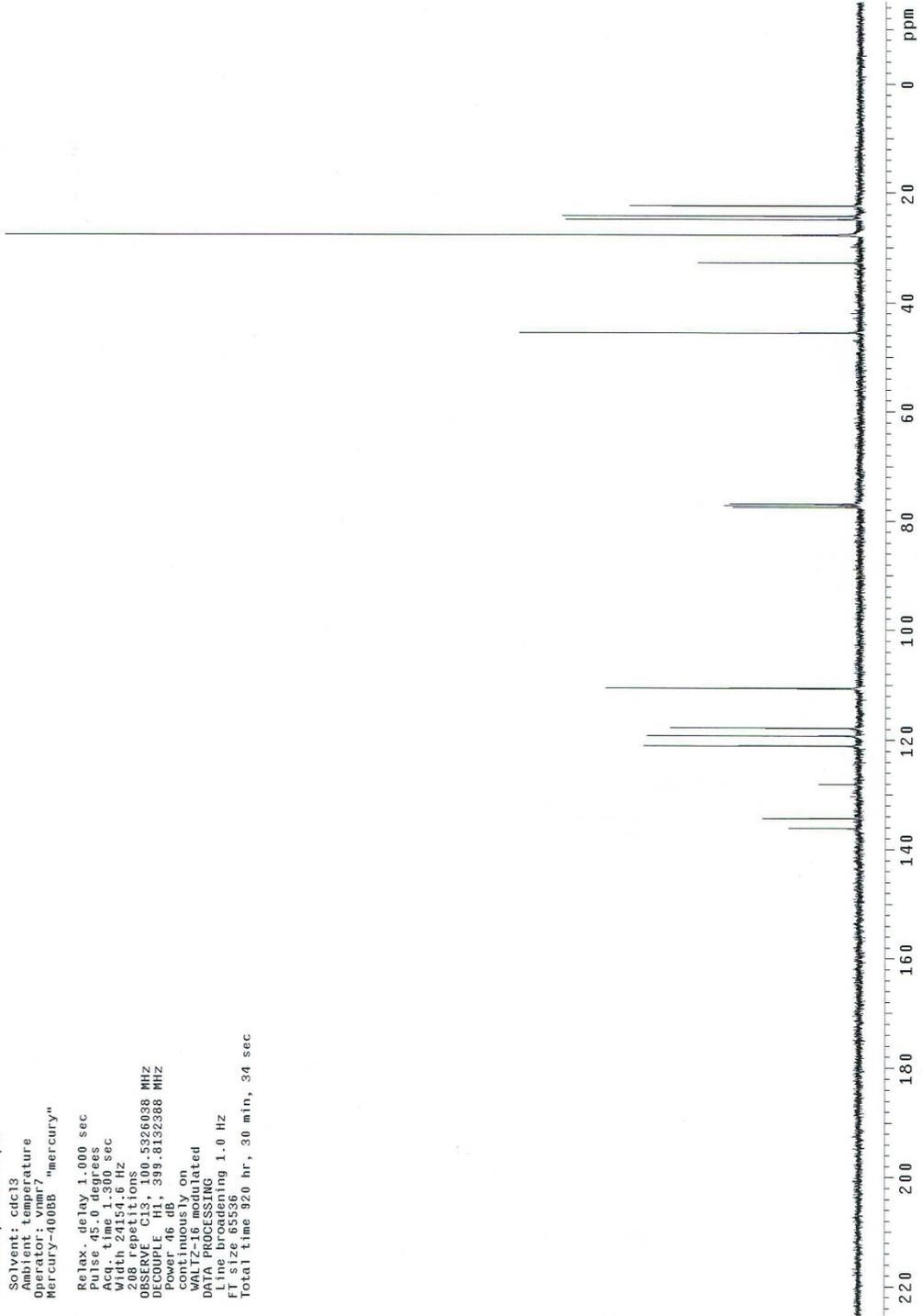
WALTZ-16 modulated

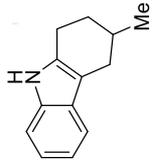
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

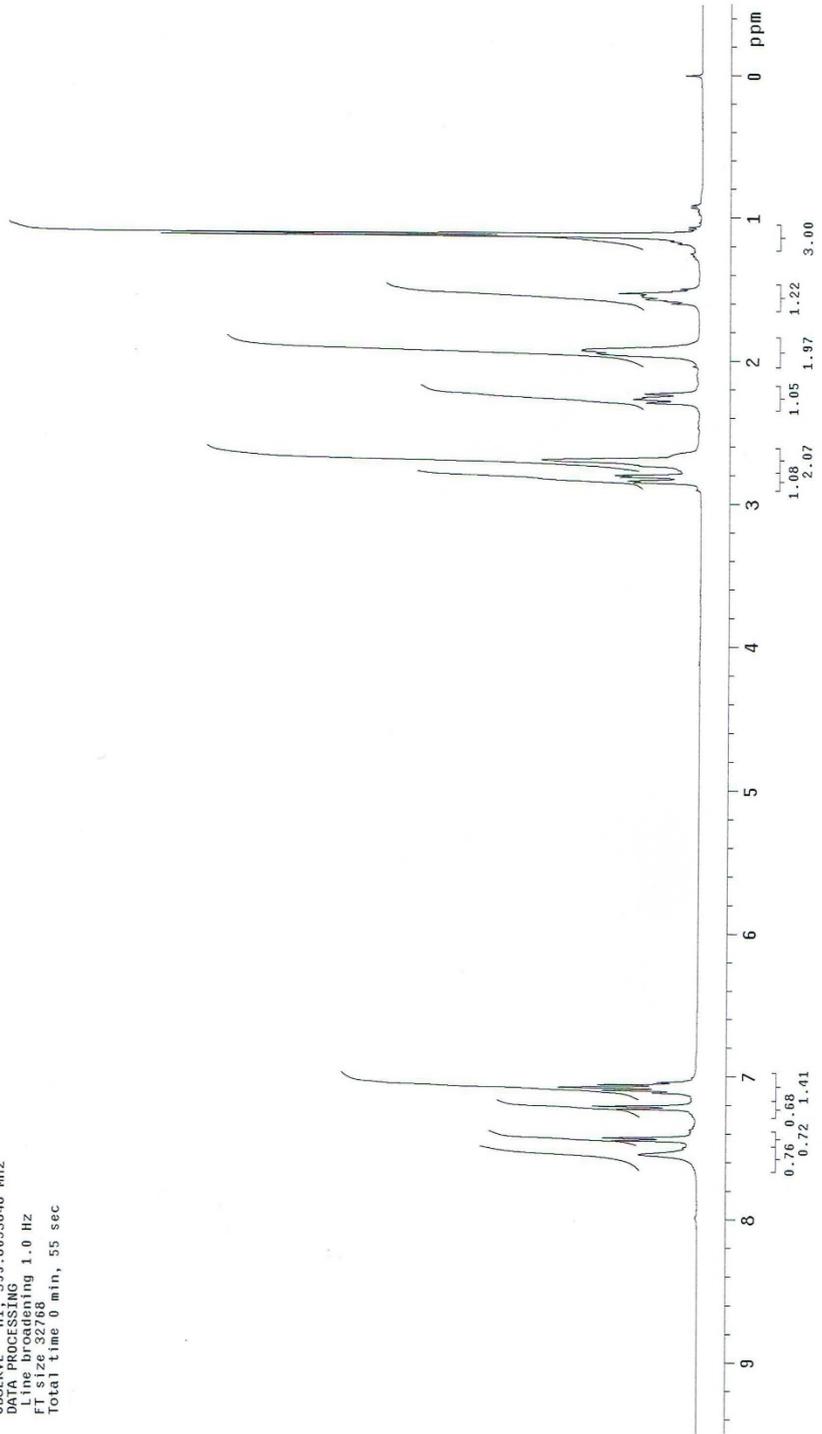
Total time 920 hr, 30 min, 34 sec

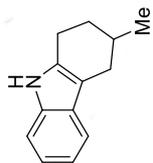




15c

OTf-4-Me-6R-1H-CDC13_131025_LBY
 File: home/vnmr7/vnmrSYS/data/13_05-14_03/Lm/OTf/4/OTf-4-Me-6R-1H-CDC13_131025_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Operator: vnmr7
 File: OTf-4-Me-6R-1H-CDC13_131025_LBY
 Mercury-400BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.988 sec
 Width 6396.0 Hz
 Observed H1
 OBSERVE H1 399.8053848 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





15c

OTf-4_4-Me-6R-13C_CDCl3_131025_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Orbitals: 1000

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

96 repetitions

DESCRIBE C13, 100.5511295 MHz

POWER 14, 399.8073389 MHz

Power 46 dB,

continuously on

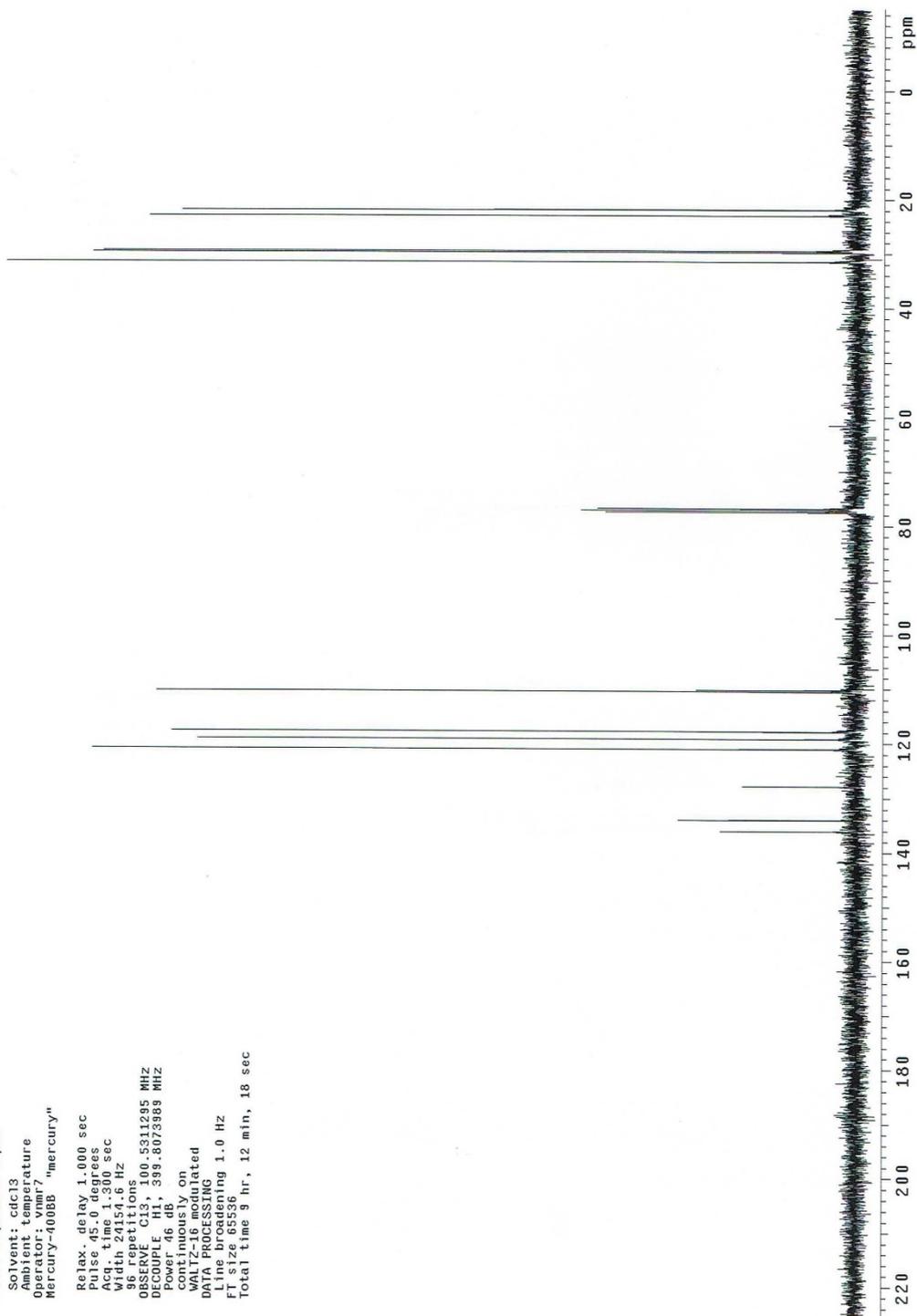
WALTZ-16 modulated

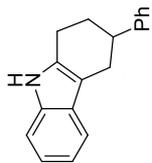
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





15d

OTf-4-Ph-6R-1H_CDCl3_140528_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Temp: 3.0 C / 276.1 K

Operator: jh

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

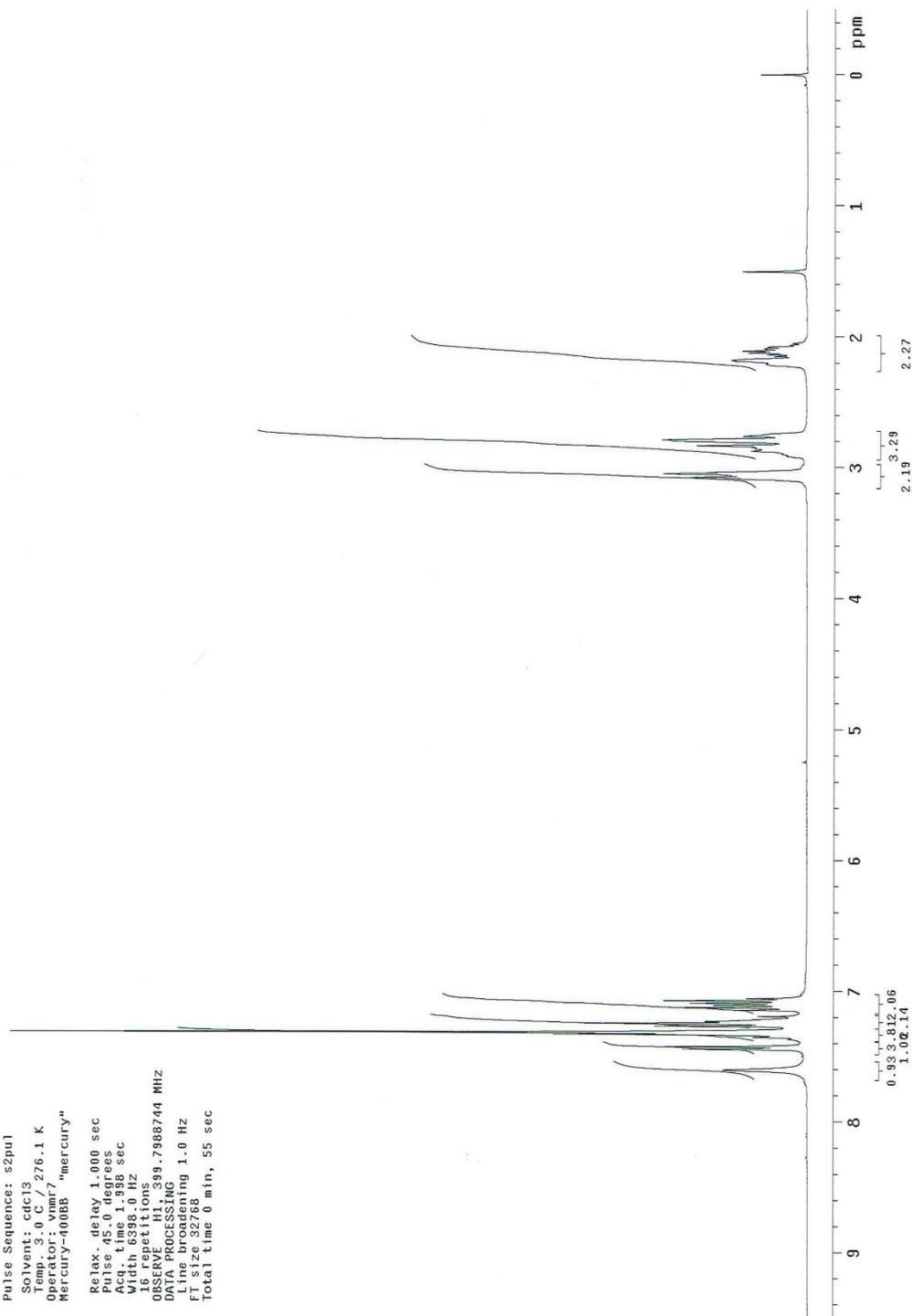
OBSERVE H1, 399.7888744 MHz

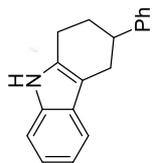
DATA PROCESSING

SI unit scaling 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





15d

0Tf-4-Ph-6R-13C_CDCl3_131023_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Operator: jant7 "mercury"

Mercury-400BB

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

100 repetitions

RESOLVE C13, 100.531391 MHz

DECODE C13, 399.8073383 MHz

Power 46 dB

continuously on

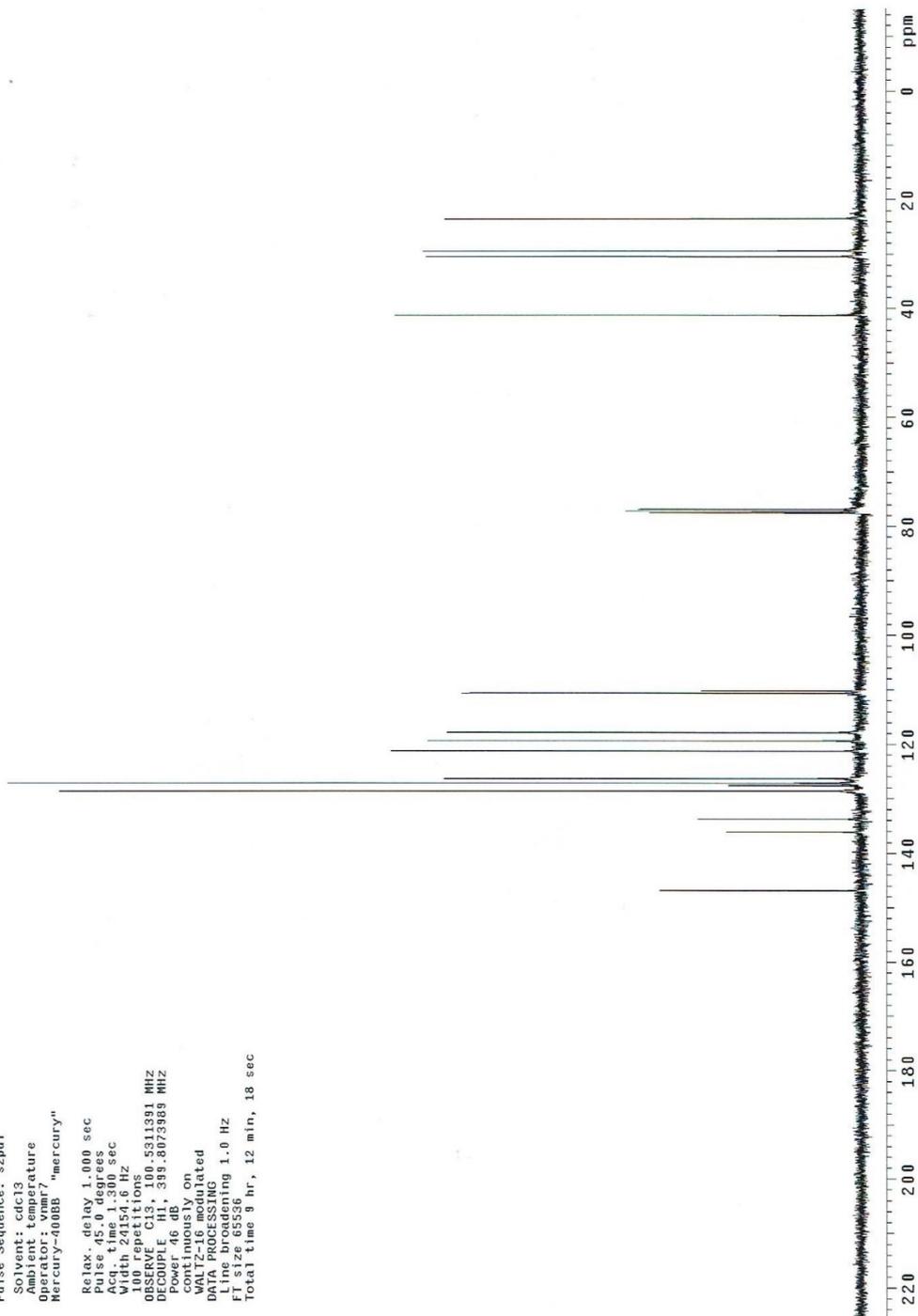
WALTZ-16 modulated

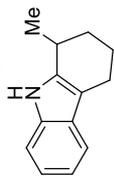
DATA PROCESSING

Line broadening 1.0 Hz

SI 2.8356

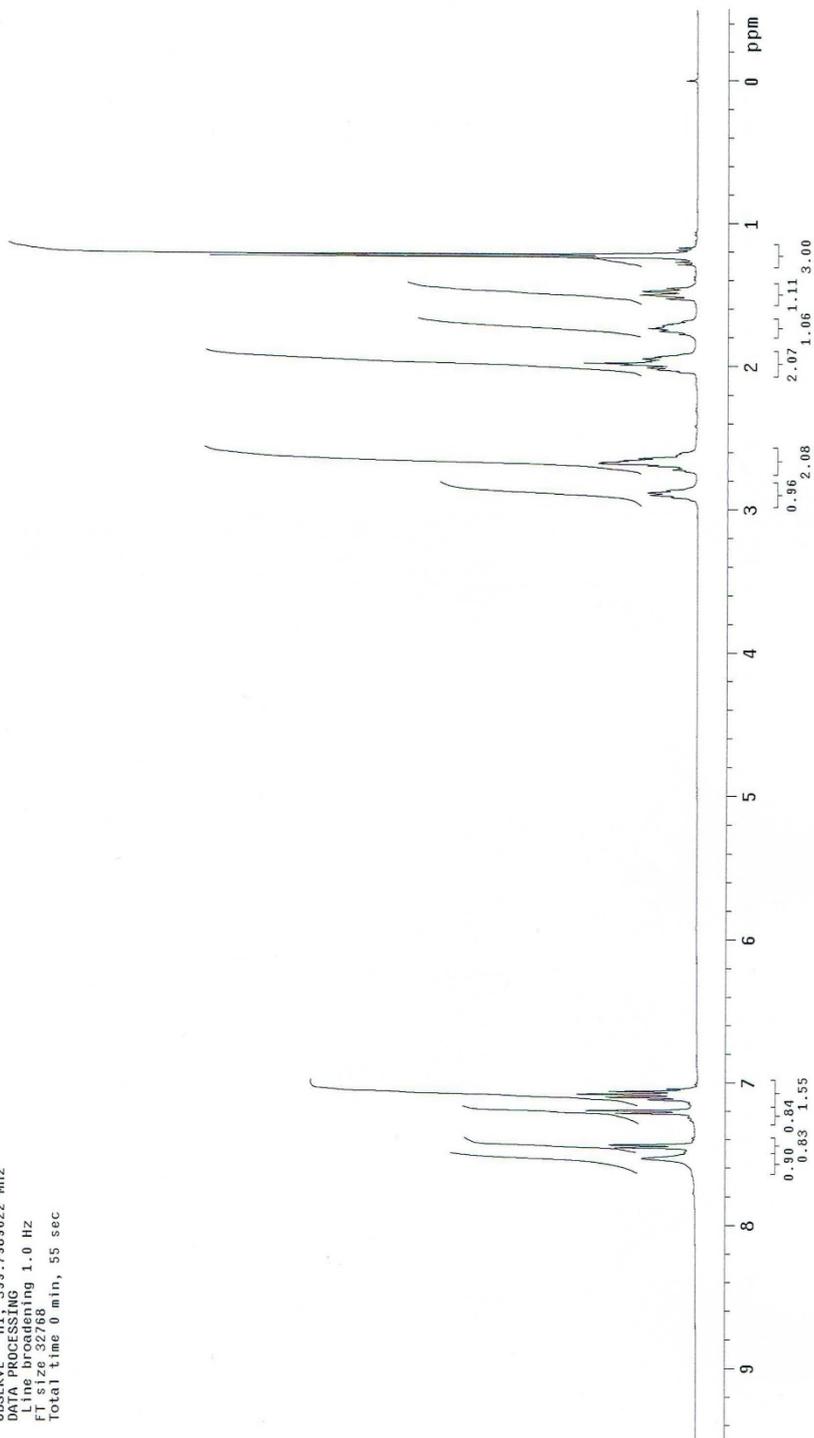
Total time 9 hr, 12 min, 18 sec

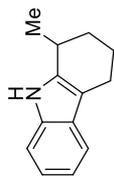




15e

OTf-4_6-Me-6R-1H_CDC13_140509_LBY
 File: home/vnmr7/vnmrSYS/data/14_05/L1m/OTf-4_6-Me-6R-1H_CDC13_140509_LBY.fid
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Acq. time 1.989 sec
 File: OTf-4_6-Me-6R-1H_CDC13_140509_LBY
 Mercury-4000BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.989 sec
 Width 6398.0 Hz
 16 repetitions
 OBSERVED F1 399.7989022 MHz
 OBSERVED F2 101.6250000 MHz
 Line broadening 1.0 Hz
 FT size 32768
 Total time 0 min, 55 sec





15e

0Tf-4_6-Me-6R-13C_CDCl3_140509_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Operator: jmr7

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

196 repetitions

OBSERVE C13, 100.5295010 MHz

DECOUPLE H1, 399.6008135 MHz

continuous on

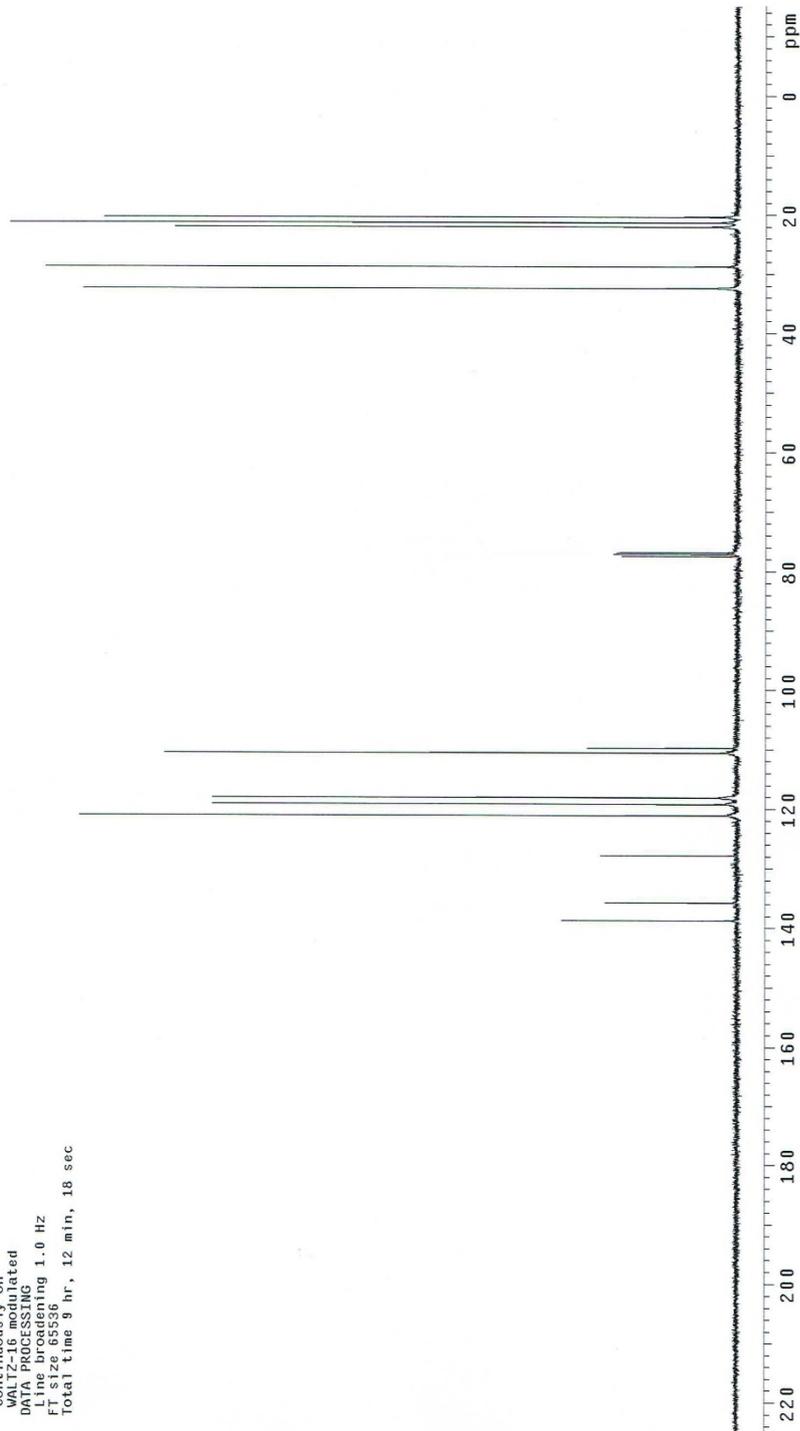
WALTZ-16 modulated

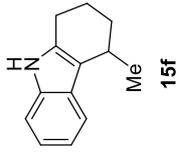
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





OTf-4_3-Me-6R-1H_CDCl3_140509_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdCl3

Ambient temperature

Acq. time 1.998 sec

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

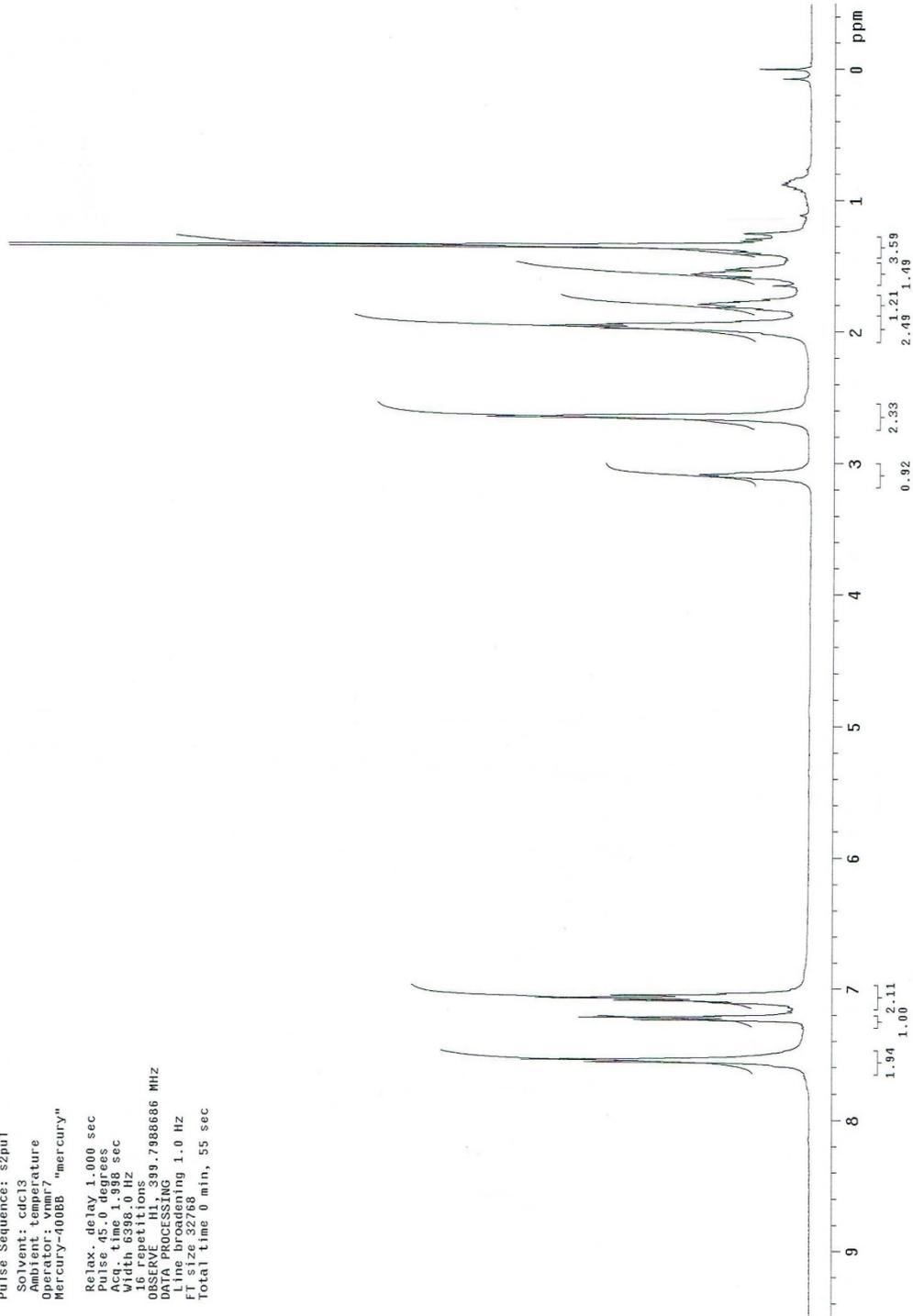
16 repetitions

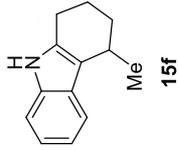
OBSERVE H1, 399.798686 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 55 sec





0Tf-4_3-Me-6R-13C_CDCl3_140509_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Temperature

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

168 repetitions

OBSERVE C13, 100.5294899 MHz

DECOUPLE H1, 399.6006135 MHz

continuously on

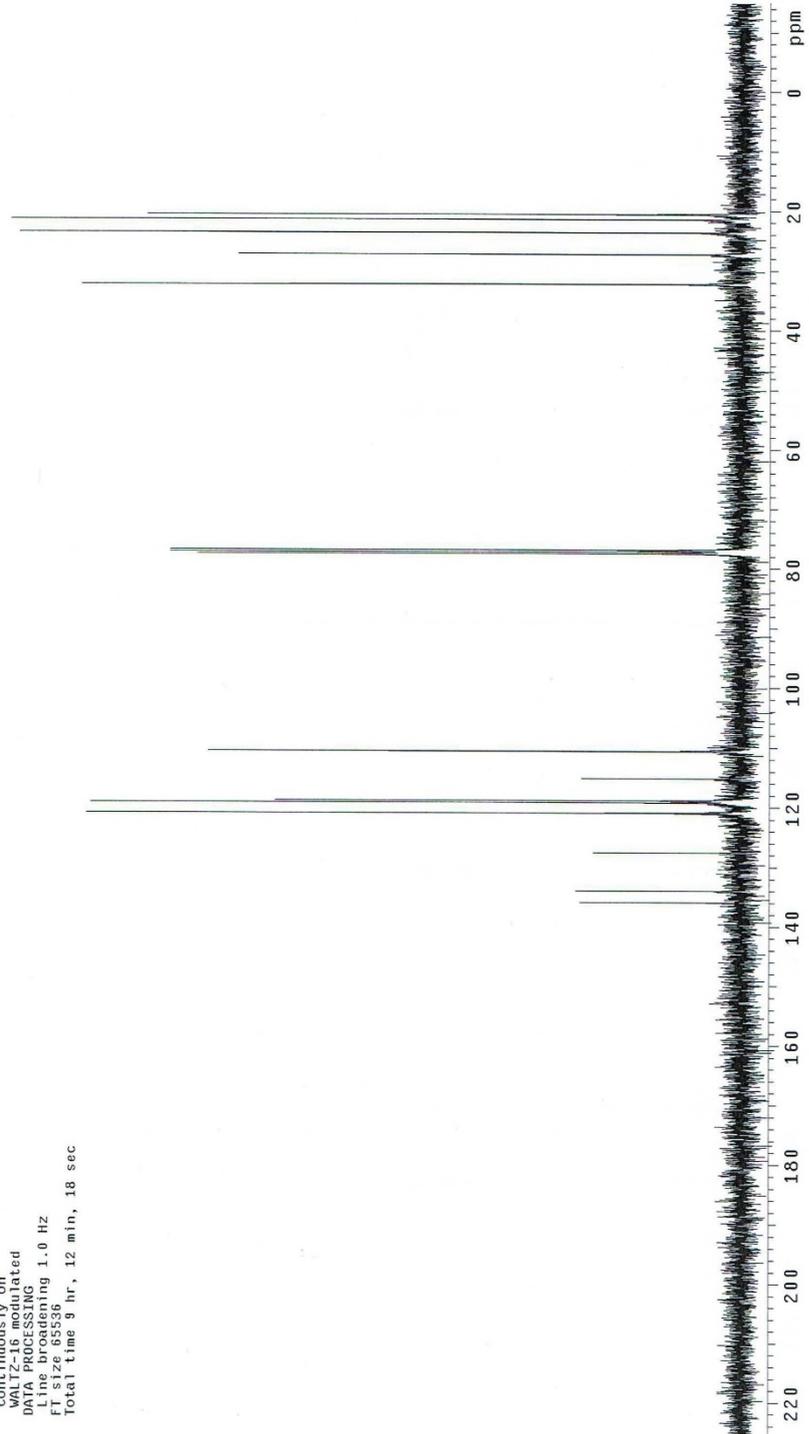
WALTZ-16 modulated

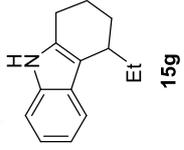
DATA PROCESSING

Line broadening 1.0 Hz

FI size 65536

Total time 9 hr, 12 min, 18 sec





DTF-4_3-Et-6R-1H_CDC13_140504_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Operator: ym

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.988 sec

Width 6398.0 Hz

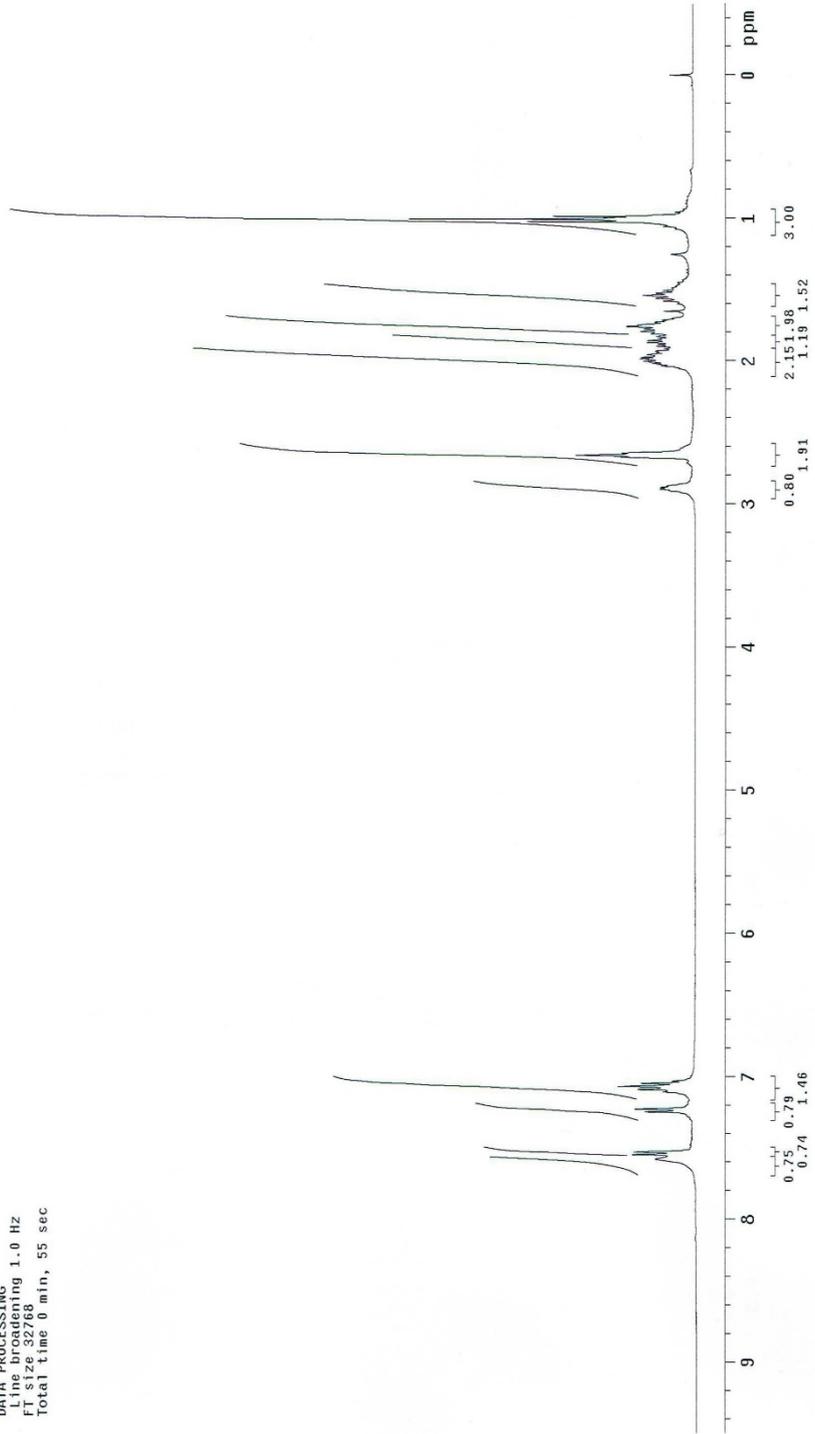
16 repetitions

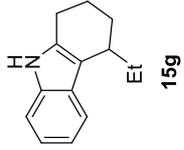
00SERVOLOCKING39.7988643 MHz

Line broadening 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





OTf-4_3-Et-6R-13C_CDCl3_140504_LBY

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Operator: dm

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

164 repetitions

OBSERVE C13, 100.5294892 MHz

DECOUPLE H1, 399.8006135 MHz

Phase 0.00000000

continuously on

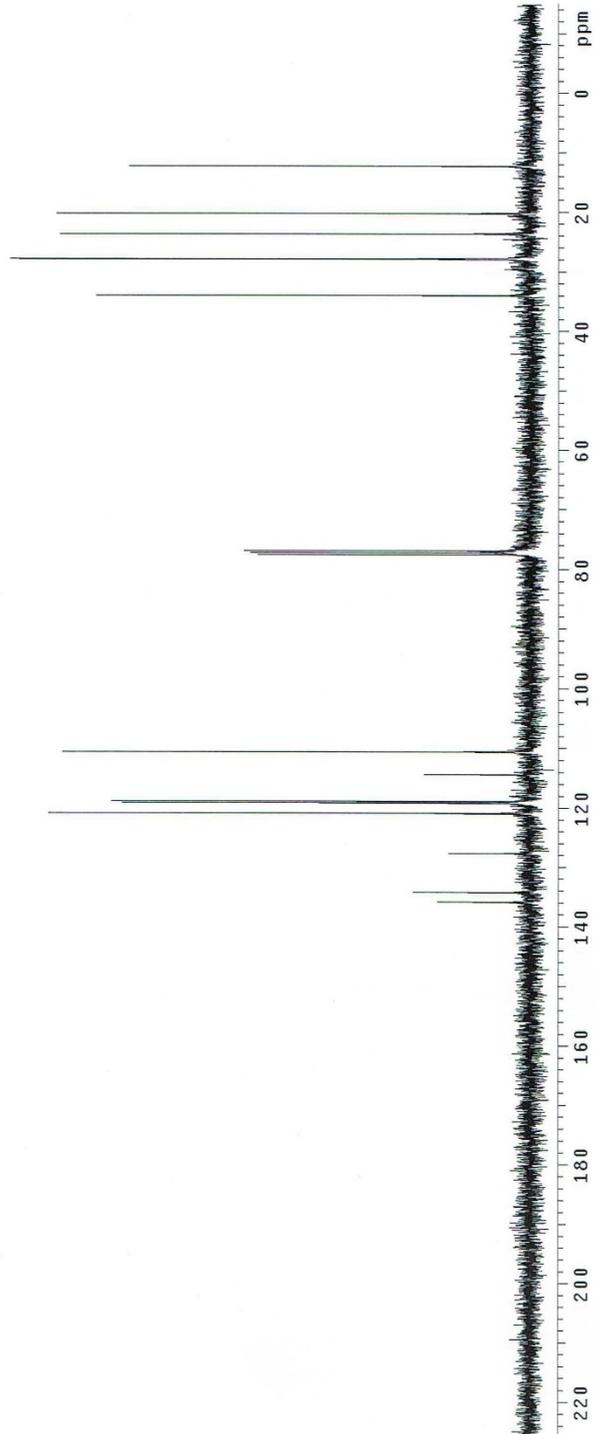
WALTZ-16 modulated

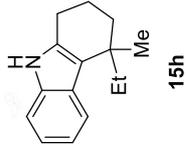
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec





OTf-4_3-Et-Me-6R-1H_CDC13_140506_LBY

File: XP

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

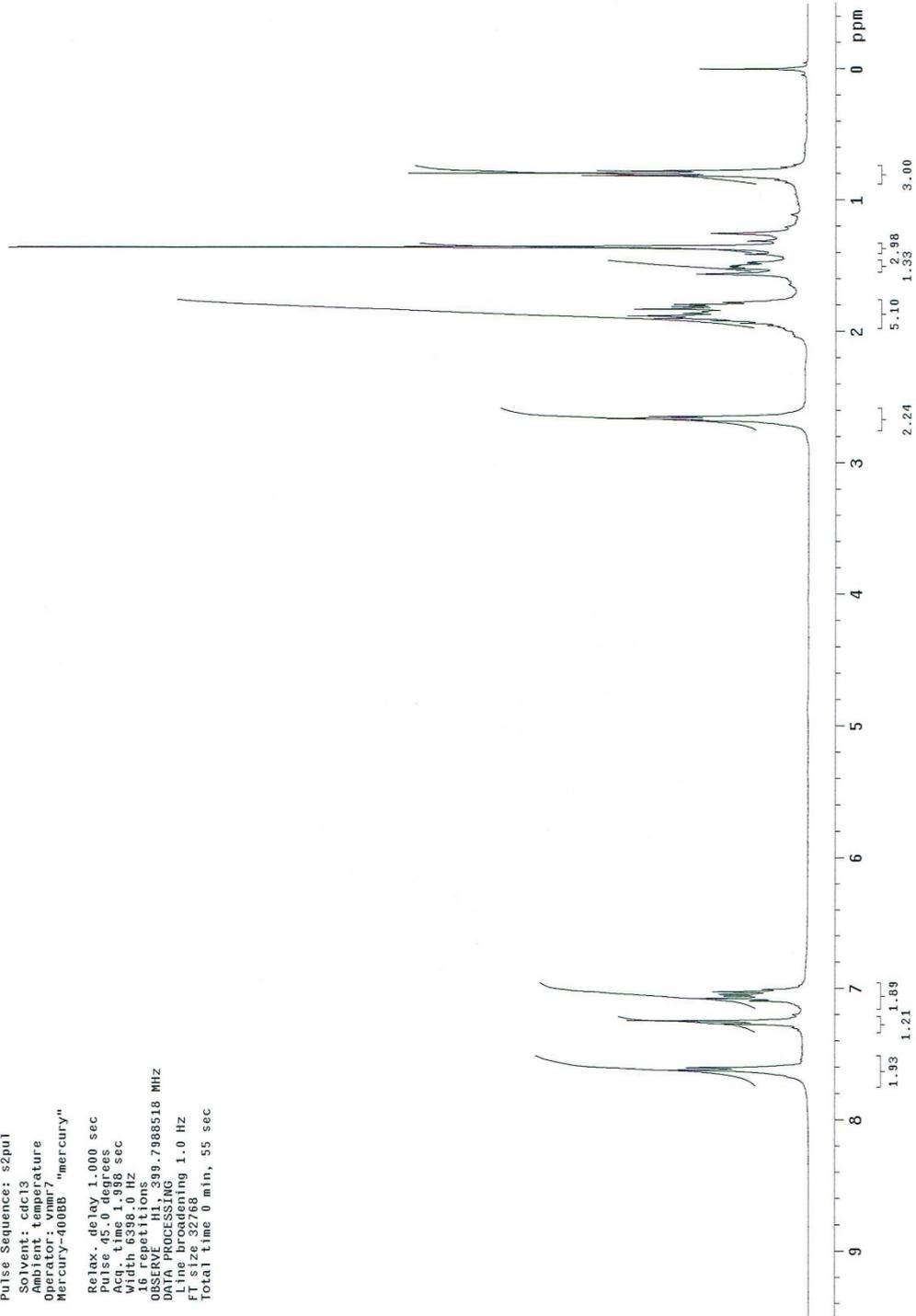
16 repetitions

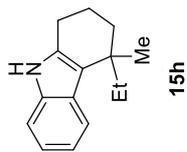
OBSERVE H1, 399.7988518 MHz

DATA PROCESSING 1.0 Hz

FT size 32768

Total time 0 min, 55 sec





OTf-4_3-Et-Me-6R-13C_CDCl3_140506_LBY

File: home/vnmr7/lmi150506.fid

Pulse Sequence: s2pu1

Solvent: cdcl3

Acq. temperature

File: Lmi150506

Mercury-40088 "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24154.6 Hz

18500 repetitions

OPROBE CH1, 5294855 MHz

DECOUPLE CH1, 399.8008135 MHz

Power 46 dB, continuously on

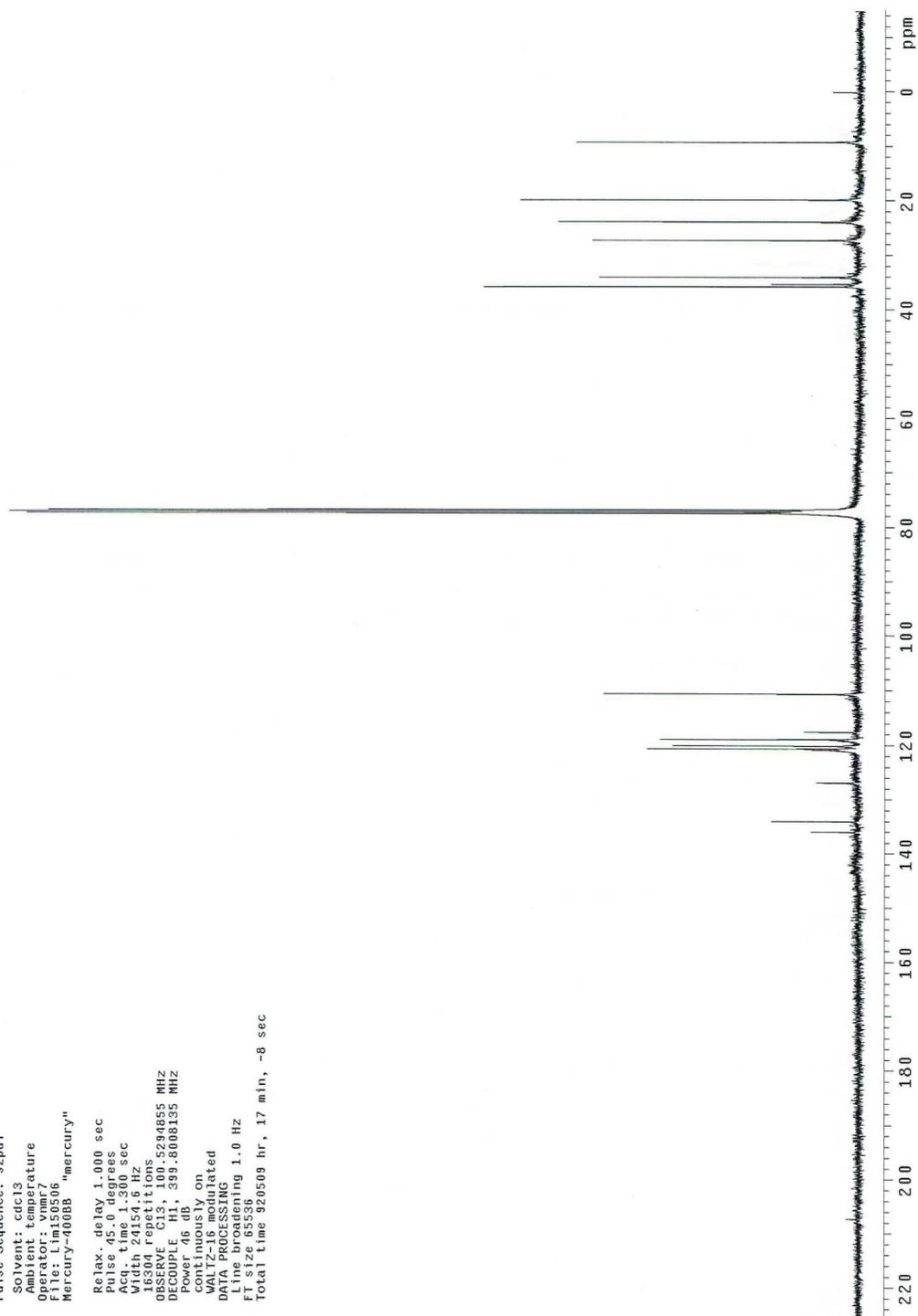
WALTZ-16 modulated

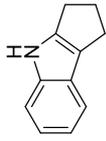
DATA PROCESSING

Line broadening 1.0 Hz

File size 633888

Total time 320509 hr, 17 min, -8 sec





15i

OTf-4a-1H_CDC13_140528_L8Y

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: kim

Mercury-400BB "mercury"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

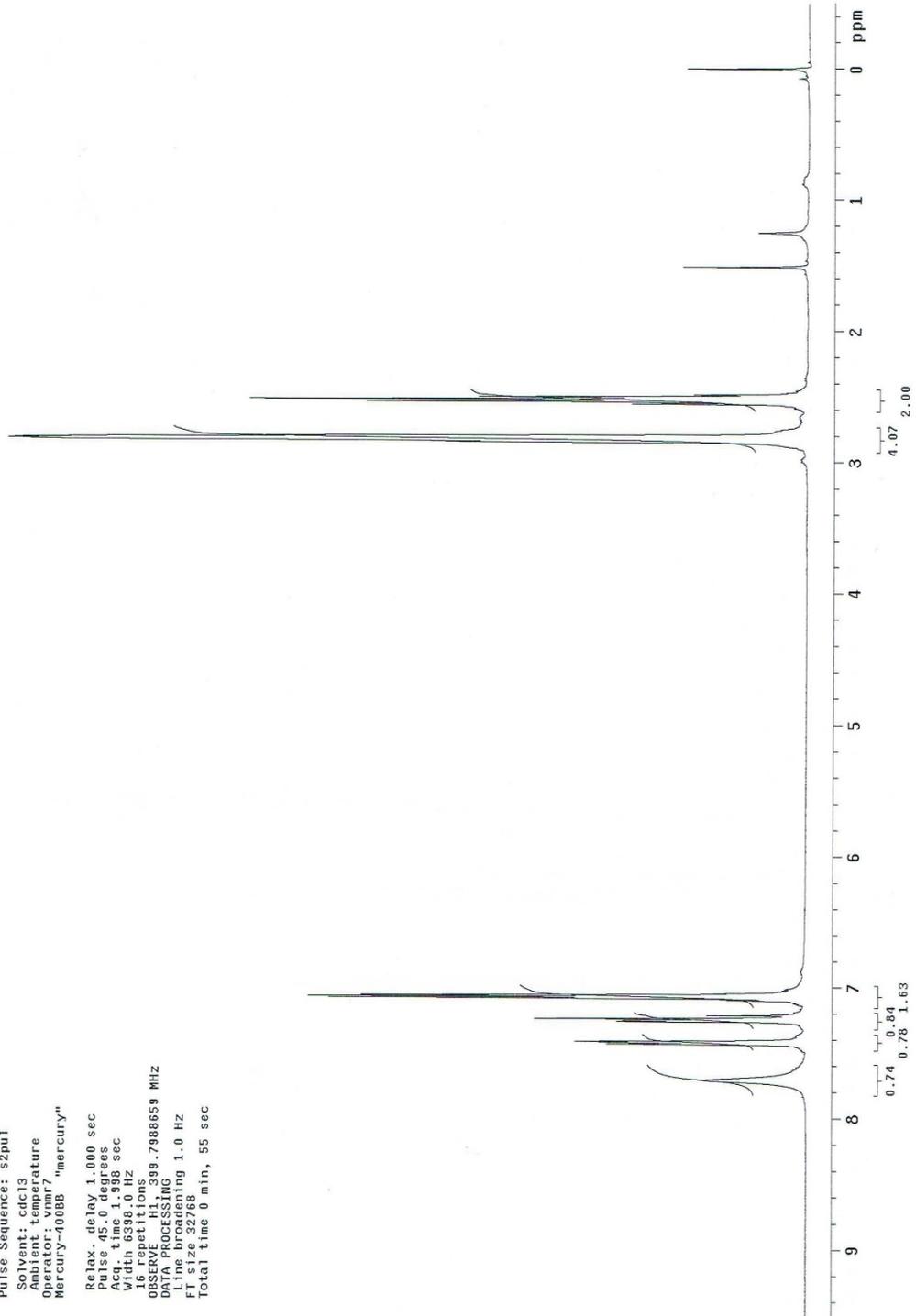
OBSERVE H1, 399.7988659 MHz

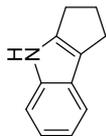
DATA PROCESSING

FT size 32768

Acquiring 1.0 Hz

Total time 0 min, 55 sec





15i

0Tf-4a-13C_CDCl3_1.40528_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vnmr7

Mercury-400BBB "mercury"

Relax. delay 1.000 sec

Acq. time 1.300 sec

Width 24154.6 Hz

280 repetitions

OBSERVE C13, 100.5294907 MHz

DECOUPLE H1, 399.8008135 MHz

Power 46 dB

Gate by on

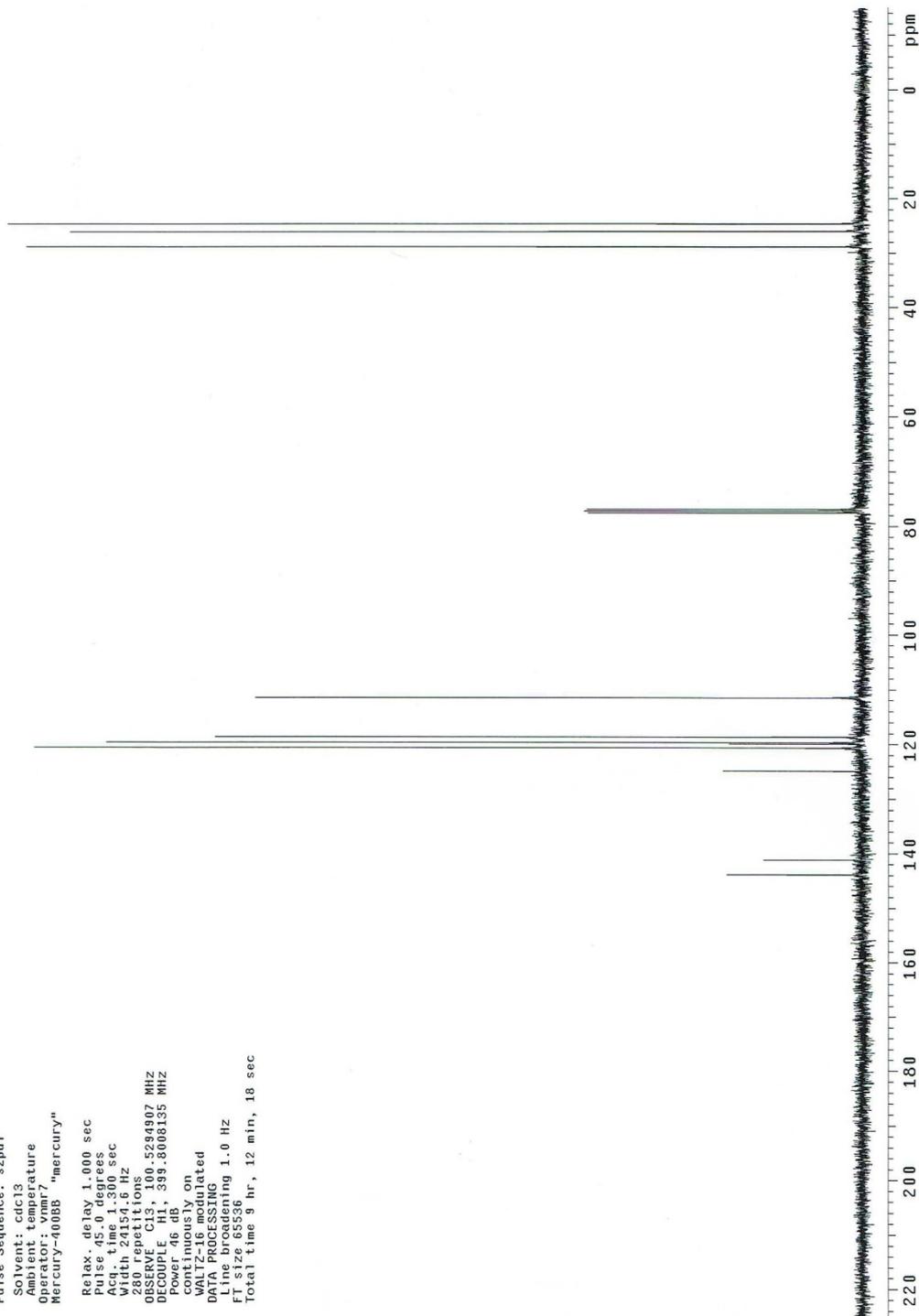
WALTZ-16 modulated

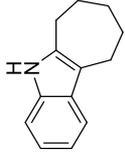
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec



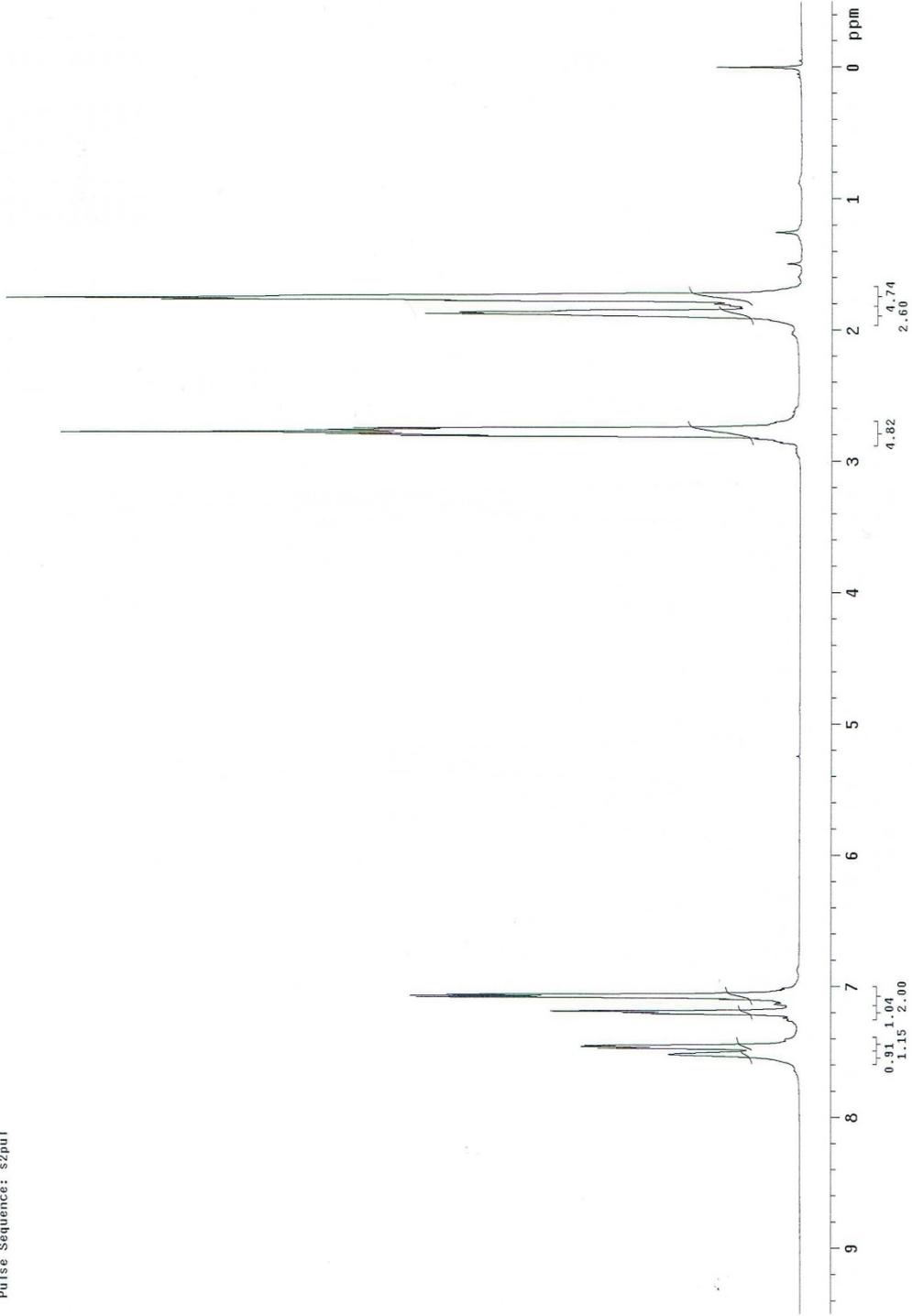


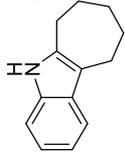
15j

140505-1H-CDC13-JBE

File: xp

Pulse Sequence: s2pul1



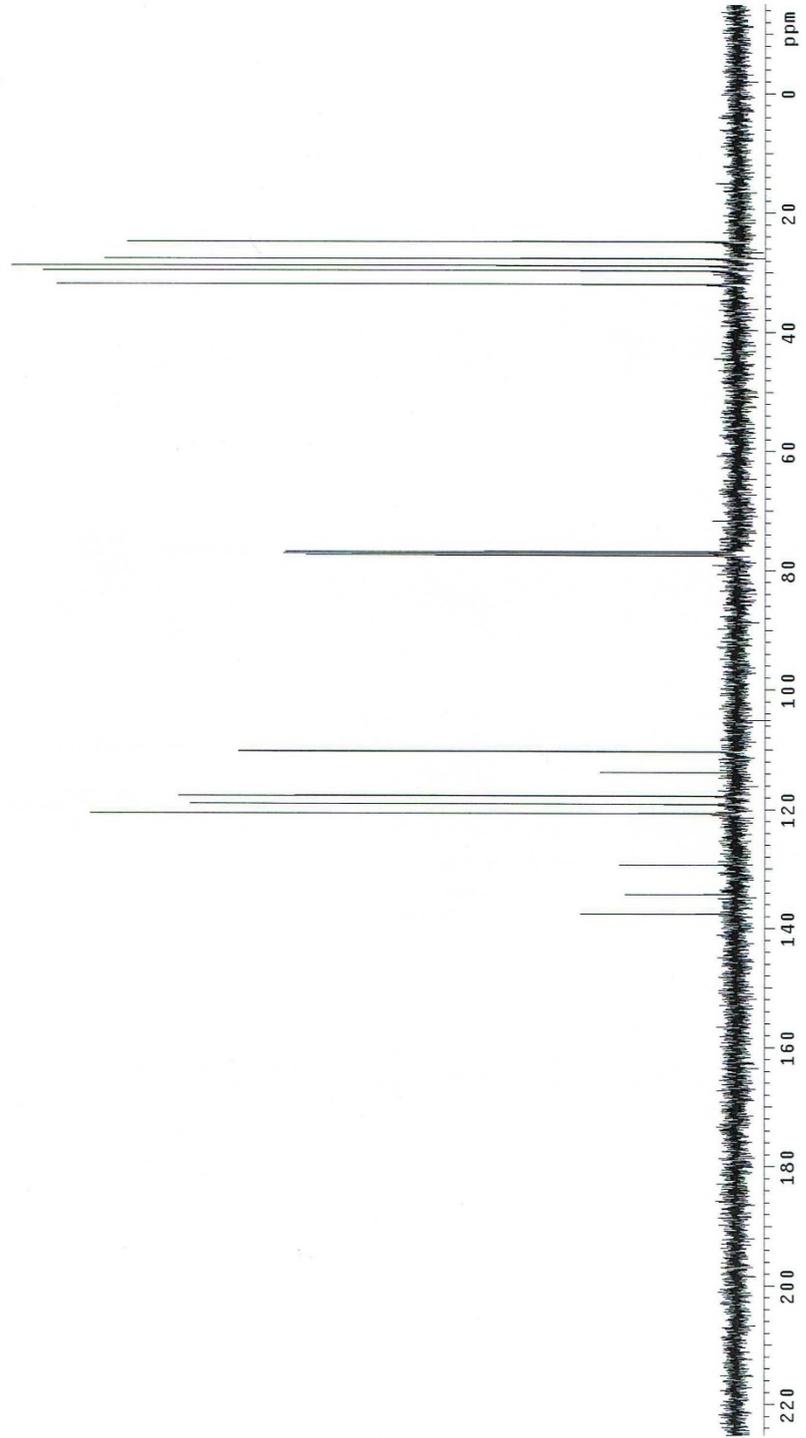


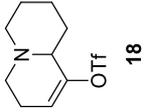
15j

140505-13C-CDCl3-JBE

File: home/vnmr7/vnmrSYS/data/14_05/JBE/140505-13C-CDCl3-JBE.f1d

Pulse Sequence: s2pu1





d_OTF-IH_CDCl3_140519_LBY

File: xp

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

Operator: vmm

Mercury-40058 "mercury"

Relax. delay 1.000 sec

Pulse 45 degrees

Acq. time 1.998 sec

Width 6398.0 Hz

16 repetitions

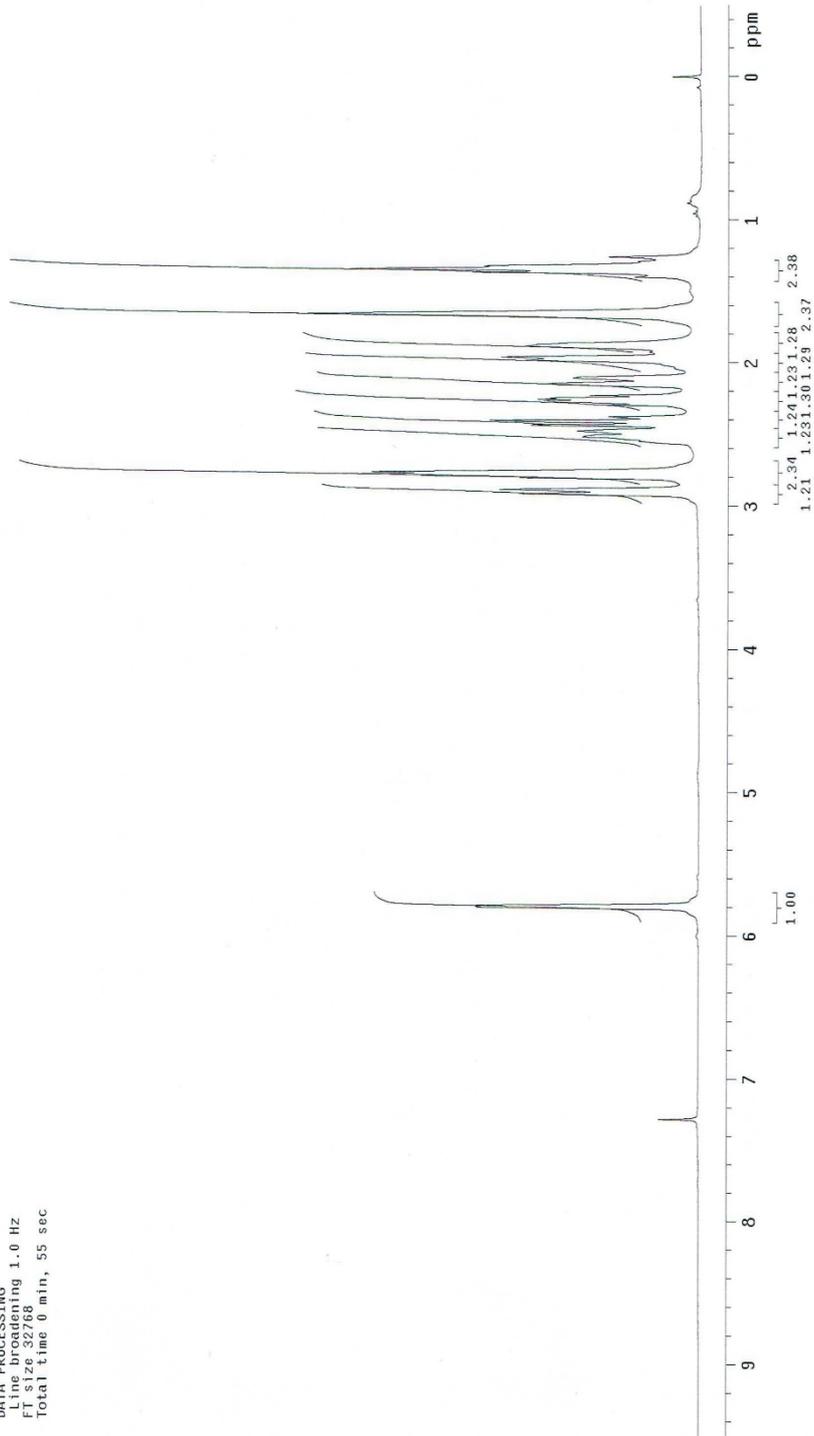
OBSERVE H1, 399.788397 MHz

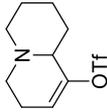
DATA PROCESSING

Using program 1.0 Hz

F1 size 32768

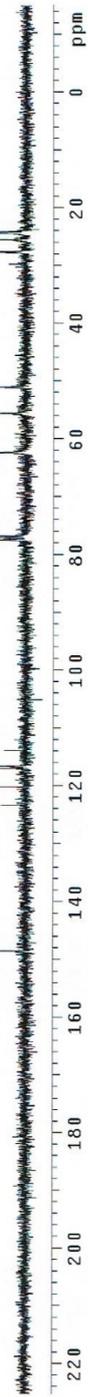
Total time 0 min, 55 sec

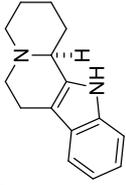




18

140220_OTf_13C_CDCl3
 File: home/vnmr7/vnmrSYS/data/13.05-14.03/Lim/Total Synthesis/10-DesbromoarborescidioloneA/140220_OTf_13C_CDCl3.fid
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient Temperature
 Observed F1: 100.628136 MHz
 File: 140220_OTf_13C_CDCl3
 Mercury-400BB "mercury"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24154.6 Hz
 File: 140220_OTf_13C_CDCl3
 OBSERVE C13, 100.5311236 MHZ
 DECOUPLE H1, 399.8073389 MHZ
 Power 46 dB
 continuously on
 WALTZ-18 modulated
 WALTZ-16 modulated
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 hr, 12 min, 18 sec





desbromoarborescicine A

140320_10-Desbromoarborescicine A_HH_cdc13_LBY

File: xp

Pulse Sequence: s2pul

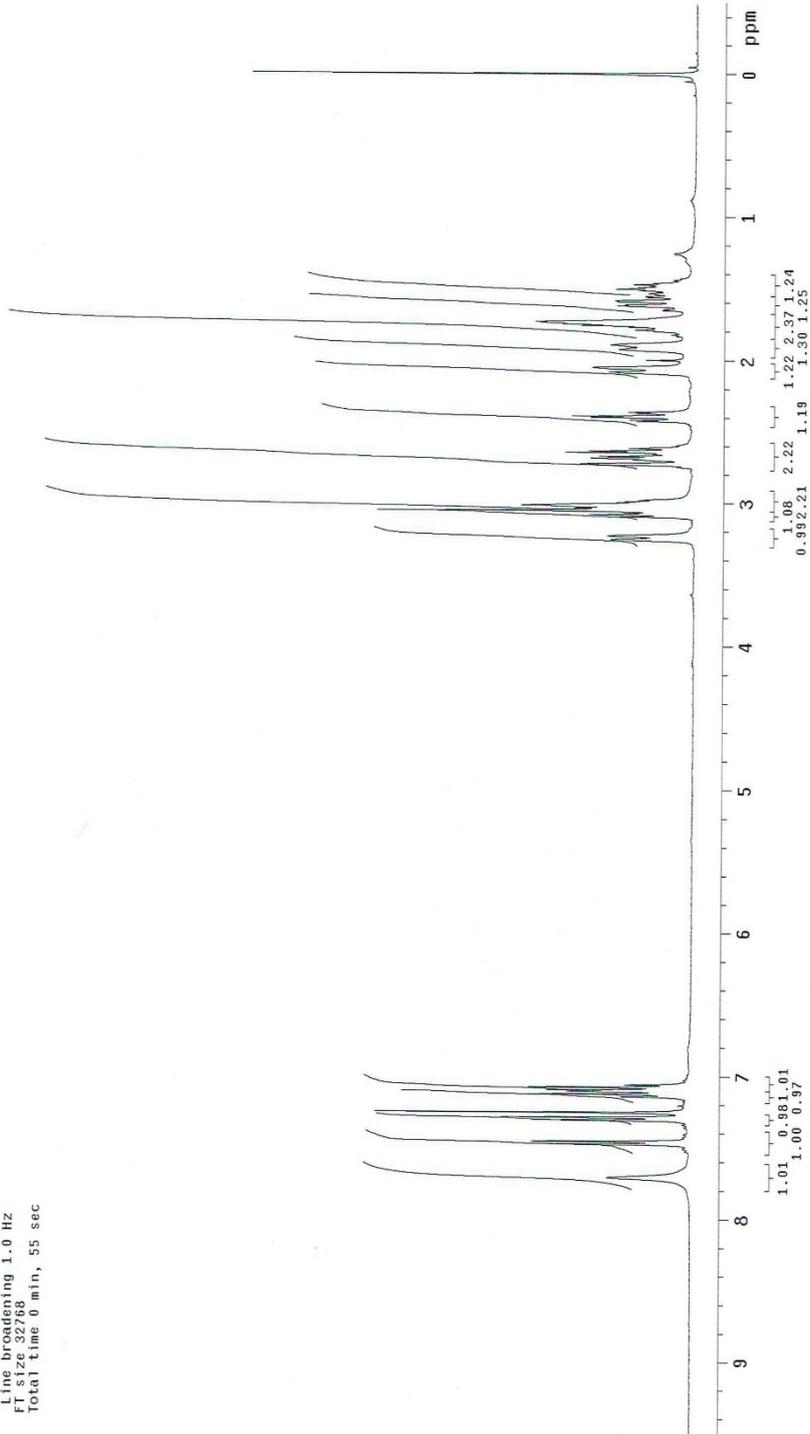
Solvent: cdc13

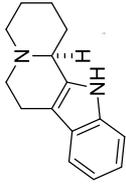
Ambient temperature

Operator: vnmr7

Mercury-400BB "mercury"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 0.100 sec
 Width 6398.0 Hz
 16 repetitions
 OBSERVE H1, 399.7988502 MHz
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 32768
 Total time 0 min, 55 sec





desbromoarborescine A

140317_Final_13C_CDC13_LBY

File: xp

Pulse Sequence: s2pul

Solvent: dmsd

Ambient temperature

Operator: vmmr

Mercury-40088B "mercury"

Relax. delay 1.000 sec

Pulse delay 0.000 sec

Acq. time 1.380 sec

Width 24154.6 Hz

292 repetitions

OBSERVE C13, 100.5294854 MHz

DECOUPLE H1, 399.8027126 MHz

Power 46 dB

Gate delay on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 12 min, 18 sec

