Supporting Information for:

Aerobic Palladium-Catalyzed sp^3 C–H Olefination: A Route to Both N-Heterocyclic Scaffolds and Alkenes

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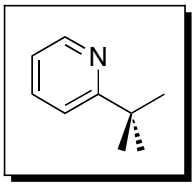
IA. General Procedures

NMR spectra were obtained on a Varian Inova 400 (399.96 MHz for ^{1}H; 100.57 MHz for ^{13}C; 376.34 MHz for ^{19}F) unless otherwise noted. ^{1}H NMR chemical shifts are reported in parts per million (ppm) relative to TMS, with the residual solvent peak used as an internal reference. The chloroform reference peak is set to 7.26 ppm for ^{1}H and 77.23 ppm for ^{13}C. In the case of benzene, the reference peak is set to 7.16 ppm for ^{1}H. Deuterated methanol is set to a reference peak of 3.31 ppm for ^{1}H and 49.00 ppm for ^{13}C. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), doublet of doublets of doublets (ddd), doublet of triplets (dt), triplet (t), quartet (q), quintet (quin), multiplet (m), and broad resonance (br). Apparent splitting is indicated with the abbreviation app. IR spectra were obtained on a Perkin-Elmer spectrum BX FT-IR spectrometer. Melting points were determined with a Mel-Temp 3.0 (Laboratory Devices Inc) and are uncorrected. HRMS data were obtained on a Micromass AutoSpec Ultima Magnetic Sector mass spectrometer. Optical rotations were measured on a Rudolph Research Autopol III automatic polarimeter.

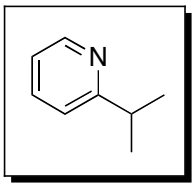
IB. Materials and Methods

$Pd(OAc)_2$ was obtained from Pressure Chemical and used as received, and the polyoxometalates ($H_6[PMo_9V_3O_{40}]$[1], and $H_4[PMo_{11}VO_{40}]$[2]) were synthesized according to literature procedures. Alkyl acrylates were obtained from Aldrich and used as received unless otherwise noted. Benzyl acrylate was synthesized according to literature procedure.[3] 2-Ethyl pyridine (**S2**) was purchased from Aldrich and used as received. Solvents were obtained from Fisher Chemical and used without further purification unless otherwise noted. THF was purified using an Innovative Technology (IT) solvent purification system composed of activated alumina, copper catalyst, and molecular sieves. Flash chromatography was performed on EM Science silica gel 60 (0.040–0.063 mm particle size, 230–400 mesh). Thin layer chromatography was performed on Merck TLC plates pre-coated with silica gel 60 F254 or on Baker-flex Aluminum Oxide-IB flexible sheets where noted.

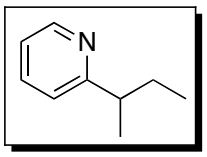
II. Synthesis and Characterization of Substrates S1, S3-S11



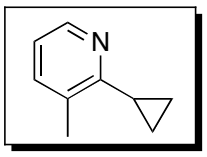
Substrate **S1** was prepared by Cu-catalyzed coupling between 2-bromopyridine (Aldrich) and ^{t}BuMgCl (Alfa Aesar) using a literature procedure.[4] **S1** was obtained as a yellow oil (R_f = 0.29 in 95:5 pentanes/Et_2O). Spectral data (^{1}H NMR ($CDCl_3$) and $^{13}C\{^1H\}$ NMR ($CDCl_3$)) for **S1** matched those reported in the literature.[5]



Substrate **S3** was prepared by alkylation of 2-ethylpyridine (Aldrich) with iodomethane (Aldrich) according to a literature procedure.[5] **S3** was obtained as a yellow oil (R_f = 0.16 in 95:5 pentanes/Et_2O). Spectral data (^{1}H NMR ($CDCl_3$) and $^{13}C\{^1H\}$ NMR ($CDCl_3$)) for **S3** matched those reported in the literature.[5]

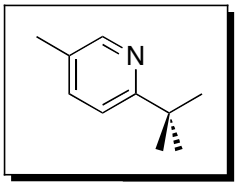


Substrate **S4** was prepared by alkylation of 2-*n*-propylpyridine (Lancaster Synthesis Inc.) with iodomethane (Aldrich) using a literature procedure.[5] **S4** was obtained as a yellow oil (R_f = 0.3 in 90:10 hexanes/ethyl acetate). ^{1}H NMR ($CDCl_3$): δ 8.54 (d, J = 4.0 Hz, 1H), 7.59 (dd, J = 7.9, 7.8 Hz, 1H), 7.13-7.07 (multiple peaks, 2H), 2.78 (tq, J = 7.2, 6.8 Hz, 1H), 1.76 (d app. quin, J = 13.6, 7.2 Hz, 1H), 1.63 (d app. quin, J = 13.6, 7.2 Hz, 1H), 1.28 (d, J = 6.8 Hz, 3H), 0.84 (t, J = 7.2 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 166.71, 149.37, 136.39, 121.78, 121.19, 43.90, 30.20, 20.60, 12.33. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_9H_{14}N^+$ 136.1121; found 136.1120.

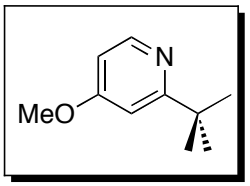


Substrate **S5** was prepared from 3-methyl-2-bromopyridine (Matrix Scientific) and cyclopropylboronic acid (Frontier Scientific) according to the literature procedure[6] with extended reaction time (overnight). After the reaction was complete, 3 M HCl solution was added to acidify the reaction mixture and the aqueous layer was extracted with EtOAc. The aqueous layer was basified with 3 M NaOH solution and the product was extracted with Et_2O. The products were then purified by flash chromatography using 90:10 petroleum ether/Et_2O. This product (**S5**) was obtained as a yellow oil (R_f = 0.28 in 90:10 hexanes/ethyl acetate). ^{1}H NMR ($CDCl_3$): δ 8.28 (d, J = 4.8

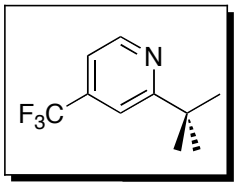
Hz, 1H), 7.36 (d, J = 7.2 Hz, 1H), 6.93 (dd, J = 7.2, 4.8 Hz, 1H), 2.41 (s, 3H), 2.08 (tt, J = 9.6, 5.2 Hz, 1H), 1.06 (m, 2H), 0.95 (m, 2H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 160.67, 146.77, 136.97, 131.20, 120.29, 19.06, 13.75, 9.00. HRMS electrospray (m/z): $[M-H]^+$ calcd for $C_9H_{10}N^+$ 132.0813; found 132.0816.



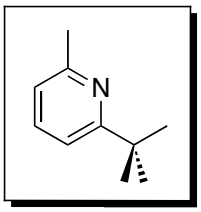
Substrate **S6** was prepared by Cu-catalyzed coupling between 2-bromo-5-methylpyridine (Matrix Scientific) and ^{t}BuMgCl using a literature procedure.[4] **S6** was obtained as a pale yellow oil (R_f = 0.32 in 95:5 pentanes/Et_2O). ^1H NMR ($CDCl_3$): δ 8.38 (d, J = 2.4 Hz, 1H), 7.41 (dd, J = 8.0, 2.4 Hz 1H), 7.22 (d, J = 8.0 Hz, 1H), 2.28 (s, 3H), 1.35 (s, 9H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 166.67, 149.20, 136.88, 129.87, 118.70, 37.16, 30.47, 18.14. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{10}H_{16}N^+$ 150.1277; found 150.1275.



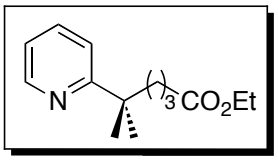
Substrate **S7** was prepared by double alkylation[5] using iodomethane (Aldrich) of 2-ethyl-4-methoxypyridine prepared according to a literature procedure.[7] **S7** was obtained as a yellow oil (R_f = 0.30 in 90:10 hexanes/ethyl acetate). ^1H NMR ($CDCl_3$): δ 8.40 (d, J = 5.6 Hz, 1H), 6.85 (s, 1H) 6.63 (d, J = 5.6 Hz, 1H), 3.84 (s, 3H), 1.35 (s, 9H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 171.33, 166.18, 150.19, 106.42, 105.94, 55.15, 37.55, 30.32. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{10}H_{16}NO^+$ 166.1226; found 166.1224.



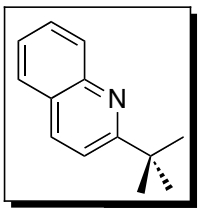
Substrate **S8** was prepared by Cu-catalyzed coupling between 2-bromo-4-trifluoromethylpyridine (Matrix Scientific) and ^{t}BuMgCl using a literature procedure.[4] **S8** was obtained as a yellow oil (R_f = 0.3 in 97:3 pentanes/Et_2O). ^1H NMR ($CDCl_3$): δ 8.73 (d, J = 4.8 Hz, 1H), 7.53 (s, 1H), 7.31 (d, J = 4.8 Hz, 1H), 1.40 (s, 9H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 171.34, 149.85, 138.70 (q, $^2J_{CF}$ = 34.0 Hz), 123.42 (q, $^1J_{CF}$ = 271.0 Hz), 116.52 (q, $^3J_{CF}$ = 4.0 Hz) 114.94 (q, $^3J_{CF}$ = 4.0 Hz), 38.13, 30.26. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –64.84. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{10}H_{13}F_3N^+$ 204.0995; found 204.0994.



Substrate **S9** was prepared by Cu-catalyzed coupling between 2-bromo-6-methylpyridine (Matrix Scientific) and ^{t}BuMgCl using a literature procedure.[4] **S9** was obtained as a yellow oil (R_f = 0.32 in 95:5 pentanes/Et_2O). ^{1}H NMR ($CDCl_3$): δ 7.47 (app. t, *J* = 7.6, Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 7.6, 1H), 2.28 (s, 3H), 1.35 (s, 9H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 168.84, 157.22, 136.32, 120.12, 115.83, 37.46, 30.45, 25.00. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{10}H_{16}N^+$ 150.1277; found 150.1276.



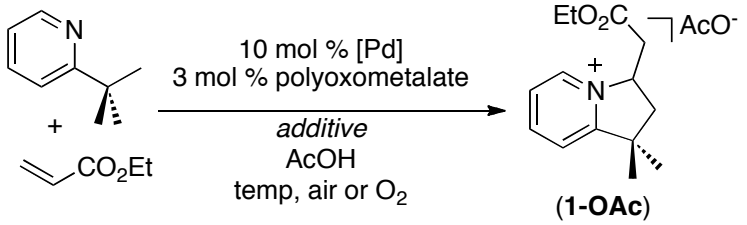
Substrate **S10** was prepared by hydrogenation (10 mol % Pd/C, 0.2 M EtOH, rt, 8 h, 90% yield) of **14**. **S10** was obtained as a colorless oil (R_f = 0.25 in 90:10 hexanes/ethyl acetate). ^{1}H NMR ($CDCl_3$): δ 8.57 (m, 1H), 7.60 (ddd, *J* = 7.4, 6.0, 1.2 Hz, 1H), 7.29 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.08 (ddd, *J* = 7.6, 4.8, 1.2, 1H), 4.09 (q, *J* = 7.2 Hz, 2H), 2.21 (t, *J* = 7.6 Hz, 2H), 1.74 (m, 2H), 1.40 (m, 2H), 1.36 (s, 6H), 1.23 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 173.92, 168.06, 148.95, 136.26, 120.85, 120.09, 60.36, 42.93, 40.53, 35.01, 28.02, 20.58, 14.44. IR (thin film): 1735 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{14}H_{22}NO_2^+$ 236.1645; found 236.1653.



Substrate **S11** was prepared by triple alkylation of quinaldine (Alfa Aesar) with iodomethane (Aldrich) using a literature procedure.[5] **S11** was obtained as a yellow oil (R_f = 0.3 in 90:10 hexanes/ethyl acetate). ^{1}H NMR ($CDCl_3$): δ 8.07 (d, *J* = 8.8 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.76 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.69 (ddd, *J* = 8.4, 6.8, 1.6 Hz, 1H), 7.53 (d, *J* = 8.4 Hz, 1H), 7.47 (ddd, *J* = 8.4, 6.8, 1.2 Hz, 1H), 1.48 (s, 9H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 169.45, 147.63, 136.04, 129.63, 129.17, 127.42, 126.64, 125.81, 118.42, 38.33, 30.35. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{13}H_{16}N^+$ 186.1277; found 186.1276.

III. Optimization of Reaction Conditions

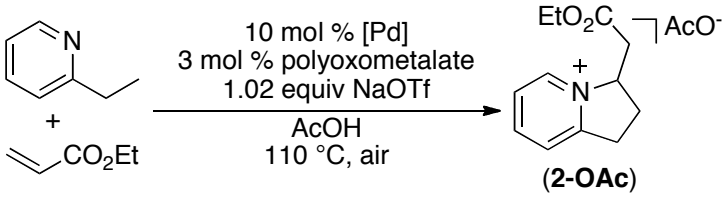
Table S1. Optimization of catalyst, polyoxometalate, additives and O_2 for S1



(**1-OAc**)

Entry	[Pd]	Polyoxometalate	Additives	O_2 or Air	Temperature	Yield
1	$Pd(OAc)_2$	$H_4[PMo_{11}VO_{40}]$	10 mol % acac 8 mol % NaOAc	O_2	90 ºC	49%
2	$Pd(OAc)_2$	$H_4[PMo_{11}VO_{40}]$	10 mol % acac 8 mol % NaOAc	air	90 ºC	40%
3	$Pd(OAc)_2$	$H_4[PMo_{11}VO_{40}]$	10 mol % acac 10 mol % NaOAc	O_2	110 ºC	81%
4	$Pd(OAc)_2$	$H_4[PMo_{11}VO_{40}]$	10 mol % acac 10 mol % NaOAc	air	110 ºC	83%
5	$Pd(OAc)_2$	$H_4[PMo_{11}VO_{40}]$	None	air	110 ºC	89%
6	$Pd(MeCN)_4(BF_4)_2$	$H_6[PMo_9V_3O_{40}]$	10 mol % NaOAc,	air	110 ºC	78%
7	$Pd(MeCN)_4(BF_4)_2$	$H_4[PMo_{11}VO_{40}]$	10 mol % NaOAc	air	110 ºC	92%

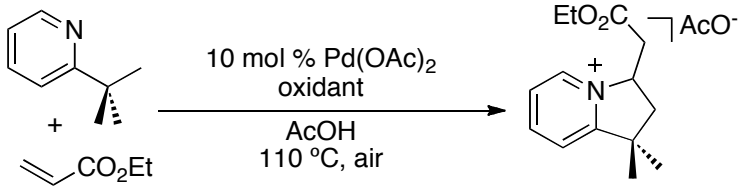
Table S2. Optimization of catalyst, polyoxometalate, additives and O_2 for 2-ethylpyridine



(**2-OAc**)

Entry	[Pd]	Polyoxometalate	Yield
1	$Pd(OAc)_2$	None	6%
2	$PdCl_2$	$H_4[PMo_{11}VO_{40}]$	0%
3	$Pd_2(dba)_3$	$H_4[PMo_{11}VO_{40}]$	39%
4	$Pd(OTf)_2$	$H_4[PMo_{11}VO_{40}]$	53%
5	$Pd(OAc)_2$	$H_4[PMo_{11}VO_{40}]$	68%
6	$Pd(MeCN)_4(BF_4)_2$	$H_6[PMo_9V_3O_{40}]$	88%
7	$Pd(MeCN)_4(BF_4)_2$	$H_4[PMo_{11}VO_{40}]$	89%

Table S3. Screening of other oxidants



(**1-OAc**)

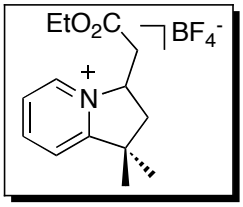
Entry	Oxidant	Yield
1	*t*-butyl hydroperoxide	No reaction
2	benzoquinone	25%
3	10 mol % $Cu(OAc)_2$	28%
4	3 mol % $H_4[PMo_{11}VO_{40}]$	75%

IV. Synthesis and Characterization of Cyclized Products 1-11, P1-P5

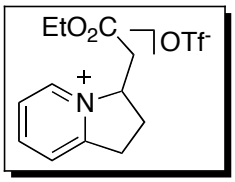
Standard Procedure A. In a 40 mL vial in air, NaOAc (2.1 mg, 0.025 mmol, 0.1 equiv), $Pd(MeCN)_4(BF_4)_2$ (11.11 mg, 0.025 mmol, 0.10 equiv) and $H_4[PMo_{11}VO_{40}]$ (13.36 mg, 0.0075 mmol, 0.03 equiv) were combined in AcOH (2 mL). The alkene (1.25 mmol, 5.0 equiv) was added, followed by addition of substrate (0.25 mmol, 1.0 equiv), after which the vial was sealed with a Teflon-lined cap, and the resulting solution was heated at 110 ºC for 18 h. The reaction was then cooled to room temperature and filtered through a short celite plug, which was washed with MeOH (1 mL). The AcOH and MeOH were removed under reduced pressure, and the remaining salts were dried under vacuum. Water (1 mL) was added to dissolve the cyclized product and the solution was filtered through a short celite plug to remove the polyoxometalate and catalyst. The celite was rinsed with 1 mL of water. The water layers were combined and the water was removed under reduced pressure followed by addition of 1 mL of saturated aqueous solution of $NaBF_4$ to exchange the AcO^- counterion. The organic BF_4^- salt was then extracted into CH_2Cl_2 (5 x 2 mL). The CH_2Cl_2 layers were combined and dried with $MgSO_4$. The solvent was removed under reduced pressure to provide the product.

Standard Procedure B. In a 40 mL vial in air, NaOTf (47.3 mg, 0.275 mmol, 1.1 equiv), $Pd(MeCN)_4(BF_4)_2$ (11.11 mg, 0.025 mmol, 0.10 equiv) and $H_4[PMo_{11}VO_{40}]$ (13.36 mg, 0.0075 mmol, 0.03 equiv) were combined in AcOH (2 mL). The alkene (1.25 mmol, 5.0 equiv) was added followed by addition of substrate (0.25 mmol, 1.0 equiv), after which the vial was sealed with a Teflon-lined cap, and the resulting solution was heated at 110 ºC for 18 h. The reaction was then cooled to room temperature and filtered through a short celite plug, which was washed with MeOH (1 mL). The AcOH and MeOH were removed under reduced pressure, and the remaining salts were dried under vacuum. Water (1 mL) was added to dissolve the cyclized product and the solution was filtered through a short celite plug to remove the polyoxometalate and catalyst. The celite was rinsed with 1 mL of water. The water layers were combined and the water was removed under reduced pressure. The remaining salt was dissolved in a saturated $NaHCO_3$ solution and the TfO^- salt of the product was extracted into CH_2Cl_2 (5 x 2 mL). The CH_2Cl_2 layers were combined and dried with $MgSO_4$. The solvent was removed under reduced pressure to provide the product.

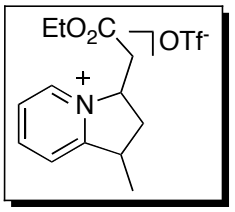
Products **3-5** were isolated as inseparable mixtures of diastereomers. Analysis by COSY and HSQC allowed for assignment of the ^1H and ^{13}C NMR resonances associated with each diastereomer.



Product **1** was prepared from **S1** and ethyl acrylate using standard procedure **A** and was obtained in 90% yield as an orange oil. ^{1}H NMR ($CDCl_3$): δ 8.80 (d, *J* = 6.4 Hz, 1H), 8.38 (app. t, *J* = 7.6 Hz, 1H), 7.87 (dd, *J* = 7.6, 6.4 Hz, 1H), 7.76 (d, *J* = 7.6 Hz, 1H), 5.46 (m, 1H), 4.09 (q, *J* = 7.2 Hz, 2H), 3.31 (dd, *J* = 17.2, 3.8 Hz, 1H), 3.27 (dd, *J* = 17.2, 4.6 Hz, 1H), 2.60 (dd, *J* = 13.1, 7.7 Hz, 1H), 2.24 (dd, *J* = 13.1, 9.4 Hz, 1H), 1.56 (s, 3H), 1.42 (s, 3H), 1.19 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 169.64, 165.45, 146.42, 140.26, 126.82, 122.58, 65.14, 61.73, 44.46, 42.39, 37.07, 28.44, 27.36, 14.22. ^{19}F{^{1}H} NMR ($CDCl_3$): δ –152.15, –152.20. IR (thin film): 1731 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{14}H_{20}NO_2^+$ 234.1489; found 234.1488.

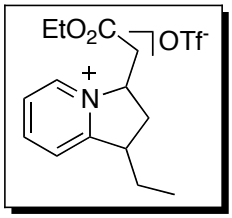


Product **2** was prepared from **S2** and ethyl acrylate using standard procedure **B** and was obtained in 89% yield as a pale yellow oil. ^{1}H NMR ($CDCl_3$): δ 8.89 (d, *J* = 6.2 Hz, 1H), 8.29 (app. t, *J* = 7.8 Hz, 1H), 7.85-7.80 (multiple peaks, 2H), 5.50 (m, 1H), 4.10 (dq, *J* = 10.8, 7.2 Hz, 1H), 4.07 (dq, *J* = 10.8, 7.2 Hz, 1H), 3.61 (dd, *J* = 19.2, 7.6 Hz, 1H), 3.54 (dd, *J* = 19.2, 7.2 Hz, 1H), 3.26 (dd, *J* = 17.8, 5.8 Hz, 1H), 3.20 (dd, *J* = 17.8, 4.6 Hz, 1H), 2.82 (m, 1H), 2.30 (m, 1H), 1.18 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 169.55, 159.24, 145.53, 140.64, 126.35, 125.03, 120.77 (q, $^{1}J_{CF}$ = 318 Hz), 67.56 61.62, 38.10, 31.53, 26.93, 14.09. ^{19}F{^{1}H} NMR ($CDCl_3$): δ –78.43. IR (thin film): 1730 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{12}H_{16}NO_2^+$ 206.1176; found 206.1172.

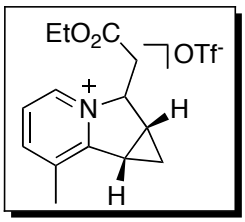


Product **3** was prepared from **S3** and ethyl acrylate using standard procedure **B** and was obtained in 81% yield as an orange oil. This compound was isolated as a 1.2:1 mixture of inseparable diastereomers. ^{1}H NMR ($CDCl_3$) *major diastereomer:* δ 8.93 (d, *J* = 5.6 Hz, 1H), 8.35 (dd, *J* = 8.0, 7.6 Hz, 1H), 7.88-7.80 (multiple peaks, 2H), 5.51 (m, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 3.89 (m, 1H), 3.18-3.06 (multiple peaks, 2H), 2.54 (m, 1H), 2.44 (m, 1H), 1.48 (d, *J* = 7.2 Hz, 3H), 1.19 (t, *J* = 7.2 Hz, 3H). *minor diastereomer*: δ 8.83 (d, *J* = 5.6 Hz, 1H), 8.35 (app. t, *J* = 7.6 Hz, 1H), 7.85-7.80 (multiple peaks, 2H), 5.39 (m, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 3.79 (m, 1H), 3.35-3.22 (multiple peaks, 2H), 2.97 (m, 1H), 1.94 (m, 1H), 1.52 (d, *J* = 6.8 Hz, 3H), 1.19 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$) *major diastereomer*: δ 169.72, 162.43,

146.26, 140.83, 126.66, 124.06, 120.81 (q, $^1J_{CF}$ = 318 Hz), 66.54, 61.66, 38.40, 38.24, 35.65, 18.11, 14.14. *minor diastereomer*: δ 169.49, 162.46, 145.78, 140.02, 126.44, 123.74, 120.81 (q, $^1J_{CF}$ = 318 Hz), 66.26, 61.66, 38.18, 37.06, 36.15, 17.67, 14.14. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –78.35. IR (thin film): 1732 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{13}H_{18}NO_2^+$ 220.1332; found 220.1331.



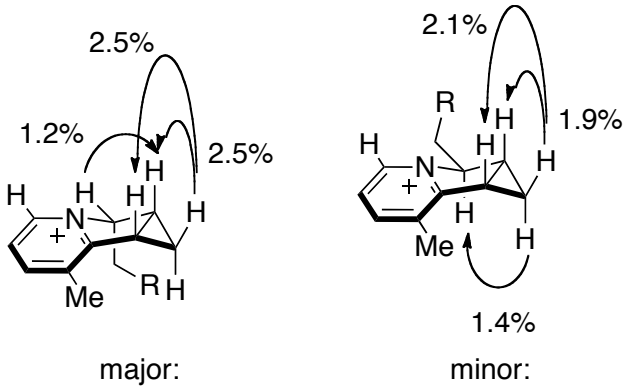
Product **4** was prepared from **S4** and ethyl acrylate using standard procedure **B** and was obtained in 55% yield as an orange oil. This compound was isolated as a 1.2:1 mixture of inseparable diastereomers. ^1H NMR ($CDCl_3$) *major diastereomer:* δ 8.93 (d, *J* = 6.0 Hz, 1H), 8.41 (app. t, *J* = 8.0 Hz, 1H), 7.91-7.84 (multiple peaks, 2H), 5.52 (m, 1H), 4.11 (d app. q, *J* = 7.2, 5.6 Hz, 2H), 3.77 (m, 1H), 3.17-3.15 (multiple peaks, 2H), 2.53-2.49 (multiple peaks, 2H), 2.07 (m, 1H), 1.70 (m, 1H), 1.22 (t, *J* = 7.2 Hz, 3H), 1.08 (t, *J* = 4.8 Hz, 3H). *minor diastereomer*: δ 8.85 (d, *J* = 6.4 Hz, 1H), 8.39 (app. t, *J* = 7.6 Hz, 1H), 7.91-7.84 (multiple peaks, 2H), 5.40 (m, 1H), 4.11 (d app. q, *J* = 7.2, 5.6 Hz, 2H), 3.69 (m, 1H), 3.32-3.30 (multiple peaks, 2H), 2.97 (dt, *J* = 12.8, 8.4 Hz, 1H), 2.19 (m, 1H), 1.97 (m, 1H), 1.70 (m, 1H), 1.22 (t, *J* = 7.2 Hz, 3H), 1.05 (t, *J* = 4.9 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$) *major diastereomer*: δ 169.73, 161.58, 145.91, 140.50, 126.77, 124.17, 120.80 (q, $^1J_{CF}$ = 318 Hz), 66.57, 61.68, 44.67, 38.46, 33.45, 25.64, 14.14, 11.40. *minor diastereomer*: δ 169.39, 161.48, 145.50, 141.23, 126.50, 123.82, 120.80 (q, $^1J_{CF}$ = 318 Hz), 66.31, 61.68, 44.85, 37.03, 33.06, 26.35, 14.14, 11.50. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –78.40. IR (thin film): 1733 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{14}H_{20}NO_2^+$ 234.1486; found 234.1489.

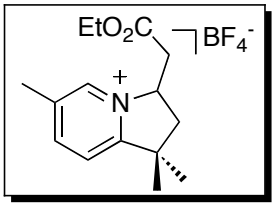


Product **5** was prepared from **S5** and ethyl acrylate using standard procedure **B** and was obtained in 43% yield as a green oil. This compound was isolated as a 2.4:1 mixture of inseparable diastereomers. The stereochemical assignment was determined by nOe as shown in Figure 1. ^1H NMR ($CDCl_3$) *major diastereomer*: δ 8.77 (d, *J* = 6.0 Hz, 1H), 8.12 (d, *J* = 7.6 Hz, 1H), 7.70 (app. t, *J* = 7.6 Hz, 1H), 5.51 (m, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 3.24 (dd, *J* = 17.0, 6.0 Hz, 1H), 3.14 (dd, *J* = 17.0, 4.4 Hz, 1H), 3.06 (m, 1H), 2.61 (s, 3H), 2.41 (app. q, *J* = 5.2 Hz, 1H), 1.68 (app. q, *J* = 8.0 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H), 0.93 (m, 1H). *minor diastereomer*: δ 8.64 (d, *J* = 6.0 Hz, 1H), 8.12 (d, *J* = 7.4 Hz, 1H), 7.68 (app. t, *J* = 7.4 Hz, 1H), 5.66 (m, 1H), 4.20 (dd, *J* = 7.0, 2.0 Hz, 1H), 4.17 (dd, *J* = 7.0, 2.0 Hz, 1H), 3.46 (m, 1H), 3.06 (m, 1H), 2.94 (dd, *J* = 16.8, 8.8 Hz, 1H), 2.78 (m, 1H), 2.60 (s, 3H), 1.52 (app. q, *J* = 6.0 Hz, 1H), 1.29 (t, *J* =

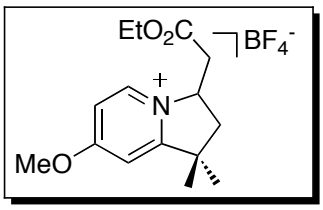
7.2 Hz, 3H), 0.93 (m, 1H). $^{13}C\{^1H\}$ NMR ($CDCl_3$) *major diastereomer*: δ 169.02, 159.30, 145.91, 139.02, 134.80, 125.44, 120.78 (q, $^1J_{CF}$ = 318 Hz), 69.71, 61.58, 39.98, 23.06, 20.74, 18.21, 17.07, 14.13, *minor diastereomer*: δ 169.62, 159.30, 145.91, 138.74, 135.45, 125.63, 120.78 (q, $^1J_{CF}$ = 318 Hz), 68.07, 61.85, 37.44, 22.01, 20.59, 18.11, 14.20 13.56. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –78.50. IR (thin film): 1734 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{14}H_{18}NO_2^+$ 232.1332; found 232.1332.

Figure S1. Observed nOes for **5**:



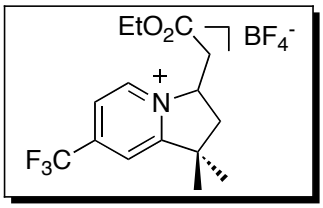


Product **6** was prepared from **S6** and ethyl acrylate using standard procedure **A** and was obtained in 70% yield as an orange oil. ^1H NMR ($CDCl_3$): δ 8.69 (s, 1H), 8.19 (d, *J* = 8.2 Hz, 1H), 7.68 (d, *J* = 8.2 Hz, 1H), 5.47 (m, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.36 (dd, *J* = 18.1, 6.0 Hz, 1H), 3.32 (dd, *J* = 18.1, 4.6 Hz, 1H), 2.62 (dd, *J* = 13.2, 7.6 Hz, 1H), 2.57 (s, 3H), 2.25 (dd, *J* = 13.2, 9.6 Hz, 1H), 1.59 (s, 3H), 1.43 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 169.65, 162.61, 147.18, 139.31, 138.17, 121.87, 64.94, 61.60, 44.01, 42.45, 37.04, 28.47, 27.40, 18.40, 14.19. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –151.97, –152.03. IR (thin film): 1733 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{15}H_{22}NO_2^+$ 248.1645; found 248.1646.

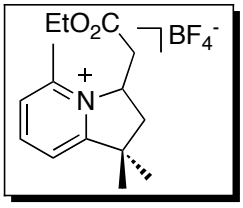


Product **7** was prepared from **S7** and ethyl acrylate using standard procedure **A** and was obtained in 71% yield as an orange oil. ^1H NMR ($CDCl_3$): δ 8.55 (d, *J* = 7.2 Hz, 1H), 7.30 (dd, *J* = 7.2, 2.8 Hz, 1H), 7.11 (d, *J* = 2.8 Hz, 1H), 5.24

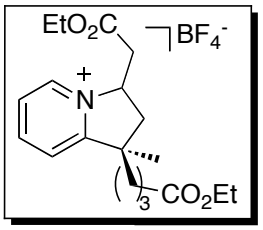
(m, 1H), 4.13 (q, J = 7.2 Hz, 2H), 4.10 (s, 3H), 3.21 (dd, J = 17.6, 4.8 Hz, 1H), 3.15 (dd, J = 17.6, 6.0 Hz, 1H), 2.57 (dd, J = 13.2, 7.2 Hz, 1H), 2.18 (dd, J = 13.2, 9.2, 1H), 1.55 (s, 3H), 1.41 (s, 3H), 1.23 (t, J = 7.2 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 172.51, 169.82, 167.32, 140.90, 113.06, 107.10, 63.06, 61.72, 58.16, 44.17, 43.07, 37.34, 28.32, 27.24, 14.19. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –152.48, –152.54. IR (thin film): 1733 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{15}H_{22}NO_3^+$ 264.1594; found 264.1596.



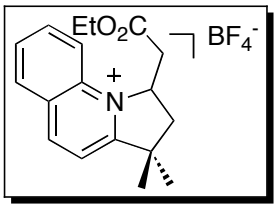
Product **8** was prepared from **S8** and ethyl acrylate using standard procedure **A** and was obtained in 75% yield as an orange oil. ^1H NMR ($CDCl_3$): δ 9.02 (d, J = 6.4 Hz, 1H), 8.04 (d, J = 6.0 Hz, 1H), 7.92 (s, 1H), 5.49 (m, 1H), 4.05 (q, J = 7.2 Hz, 2H), 3.30 (dd, J = 17.8, 4.4 Hz, 1H), 3.24 (dd, J = 17.8, 6.8 Hz, 1H), 2.62 (dd, J = 13.2, 8.0 Hz, 1H), 2.23 (dd, J = 13.2, 9.6 Hz, 1H), 1.72 (s, 3H), 1.60 (s, 3H), 1.21 (t, J = 7.2 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$ with minimal CD_3OD added for solubility): δ 169.60, 167.69, 146.42 (q, $^2J_{CF}$ = 36.1 Hz), 142.55 (q, $^3J_{CF}$ = 3.1 Hz), 123.36, 121.13 (q, $^1J_{CF}$ = 273.2 Hz), 119.04 (q, $^3J_{CF}$ = 3.4 Hz), 66.26, 61.80, 44.99, 42.25, 36.53, 28.02, 27.00, 13.94. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –65.24, –152.68, –152.74. IR (thin film): 1733 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{15}H_{19}F_3NO_2^+$ 302.1362; found 302.1362.



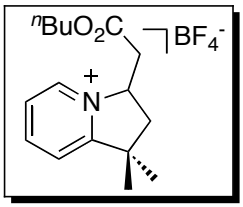
Product **9** was prepared from **S9** and ethyl acrylate using standard procedure **A** and was obtained in 36% yield as an orange oil. ^1H NMR ($CDCl_3$): δ 8.28 (dd, J = 8.0, 7.6 Hz, 1H), 7.66-7.61 (multiple peaks, 2H), 5.59 (m, 1H), 4.18 (q, J = 7.2 Hz, 2H), 3.08 (dd, J = 16.6, 2.4 Hz, 1H), 2.94 (dd, J = 16.4, 9.6 Hz, 1H), 2.85 (s, 3H), 2.77 (dd, J = 14.0, 9.6 Hz, 1H), 2.19 (dd, J = 14.0, 2.4 Hz, 1H), 1.56 (s, 3H), 1.52 (s, 3H), 1.27 (t, J = 7.2 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 168.98, 165.89, 152.74, 146.25, 128.52, 120.70, 64.47, 61.87, 44.80, 41.32, 39.28, 30.28, 29.49, 19.97, 14.23. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –153.22, –153.27. IR (thin film): 1733 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{15}H_{22}NO_2^+$ 248.1645; found 248.1645.



Product **10** was prepared from **S10** and ethyl acrylate using standard procedure **A** and was obtained in 49% yield as a 1:1 mixture of diastereomers as an orange oil. However, the assignment of specific NMR peaks to each diastereomer was not possible due to the number of overlapping resonances. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.83 (d, *J* = 4.8 Hz, 1H), 8.80 (d, *J* = 4.8 Hz, 1H), 8.40 (app t. *J* = 6.4 Hz, 2H), 7.89 (m, 2H), 7.80 (d, *J* = 6.4 Hz, 1H), 7.77 (d, *J* = 6.8 Hz, 1H), 5.46-5.41 (multiple peaks, 2H), 4.11-4.04 (multiple peaks, 8H), 3.29-3.27 (multiple peaks, 4H), 2.75 (dd, *J* = 10.8, 6.4 Hz, 1H), 2.49 (dd, *J* = 10.8, 6.4 Hz, 1H), 2.33 (m, 2H), 2.26 (t, *J* = 5.6 Hz, 2H), 2.15 (dd, *J* = 11.0, 7.4 Hz, 1H), 1.95-1.82 (multiple peaks, 2H), 1.76-1.38 (multiple peaks, 13H), 1.23-1.18 (multiple peaks, 12H). ^{13}C{^{1}H} NMR ($CDCl_3$, 125 MHz): δ 173.16, 173.08, 169.72 (2C), 169.53, 164.62, 146.13, 145.96, 140.66 (2C), 126.98, 126.86, 123.01, 122.71, 65.42, 65.08, 61.77, 60.77, 47.96, 47.91, 40.03, 40.01, 39.33, 38.34, 37.20, 36.50, 33.77, 33.59, 26.28, 25.21, 23.19, 19.89, 19.72, 14.43, 14.40, 14.21. ^{19}F{^{1}H} NMR ($CDCl_3$): δ –151.92, –151.97. IR (thin film): 1727 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{19}H_{28}NO_4^+$ 334.2013; found 334.2008.

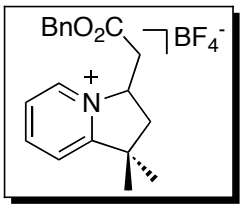


Product **11** was prepared from **S11** and ethyl acrylate using standard procedure **A** and was obtained in 39% yield as a yellow oil. ^{1}H NMR ($CDCl_3$): δ 8.99 (d, *J* = 8.4 Hz, 1H), 8.26 (d, *J* = 8.0 Hz, 1H), 8.22-8.14 (multiple peaks, 2H), 7.92-7.88 (multiple peaks, 2H), 6.13 (m, 1H), 4.17-4.07 (multiple peaks, 2H), 3.28 (dd, *J* = 16.8, 2.4 Hz, 1H), 3.07 (dd, *J* = 16.4, 9.6 Hz, 1H), 2.96 (dd, *J* = 13.6, 9.6 Hz, 1H), 2.37 (dd, *J* = 13.6, 2.0 Hz, 1H), 1.69 (s, 3H), 1.65 (s, 3H), 1.27 (t, *J* = 6.8 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 169.24 (2C), 148.92, 136.05, 135.88, 131.12, 129.94, 129.46, 118.76, 117.96, 63.93, 61.99, 46.62, 41.34, 39.47, 29.57 (2C), 14.26. ^{19}F{^{1}H} NMR ($CDCl_3$): δ –153.22, –153.27. IR (thin film): 1733 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{18}H_{22}NO_2^+$ 284.1645; found 284.1645.

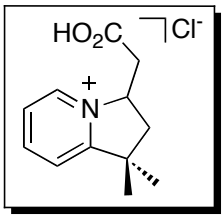


Product **P1** was prepared from **S1** and *n*-butyl acrylate using standard procedure **A** and was obtained in 80% yield as an orange oil. ^{1}H NMR ($CDCl_3$): δ 8.84 (d, *J* = 6.2 Hz, 1H), 8.43 (dd, *J* = 8.0, 7.6 Hz, 1H), 7.91 (dd, *J* = 7.2, 6.2 Hz, 1H), 7.81 (d,

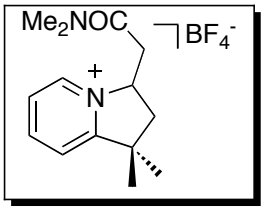
8.0 Hz, 1H), 5.50 (m, 1H), 4.07 (app. t, J = 10.8 Hz, 2H), 3.33 (app. d, J = 4.8 Hz, 2H), 2.64 (dd, J = 13.2, 7.6 Hz, 1H), 2.27 (dd, J = 13.2, 9.4 Hz, 1H), 1.61-1.55 (multiple peaks, 5H), 1.46 (s, 3H), 1.38-1.31 (multiple peaks, 2H), 0.92 (t, J = 6.8 Hz, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 169.61, 165.41, 146.63, 139.94, 126.74, 122.84, 65.47, 65.09, 44.39, 42.34, 37.12, 30.52, 28.27, 27.30, 19.13, 13.78. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –152.51, –152.57. IR (thin film): 1731 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{16}H_{24}NO_2^+$ 262.1802; found 262.1802.



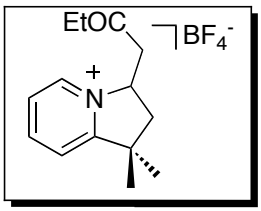
Product **P2** was prepared from **S1** and benzyl acrylate using standard procedure **A** and was obtained in 75% yield as a yellow oil. ^1H NMR ($CDCl_3$): δ 8.77 (d, J = 6.4 Hz, 1H), 8.28 (dd, J = 8.0, 7.6 Hz, 1H), 7.79 (app. t, J = 6.4 Hz, 1H), 7.65 (d, 8.0 Hz, 1H), 7.31-7.22 (multiple peaks, 5H), 5.46 (m, 1H), 5.05 (s, 2H), 3.35 (dd, J = 15.8, 3.4 Hz, 1H), 3.32 (dd, J = 15.8, 2.6 Hz, 1H), 2.54 (dd, J = 13.2, 7.6 Hz, 1H), 2.19 (dd, J = 13.2, 9.6 Hz, 1H), 1.49 (s, 3H), 1.38 (s, 3H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 169.42, 165.34, 146.26, 140.35, 135.19, 128.89, 128.83, 128.81, 126.74, 122.37, 67.48, 65.12, 44.39, 42.29, 36.96, 28.42, 27.24. $^{19}F\{^1H\}$ NMR ($CDCl_3$): δ –152.00, –152.05. IR (thin film): 1735 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{19}H_{22}NO_2^+$ 296.1645; found 296.1646.



Product **P3** was prepared from **S1** and acrylic acid (Aldrich) using standard procedure **A**. After removal of the acetic acid and methanol, NMR analysis of crude mixture shows 69% yield; however, the carboxylic acid product was inseparable from acrylic acid oligomers. A pure sample of **P3** for characterization was obtained as the chloride salt through hydrolysis of **1**.[8] ^1H NMR (CD_3OD) for **P3**: δ 8.84 (d, J = 6.0 Hz, 1H), 8.44 (app. t, J = 7.6 Hz, 1H), 7.99 (d, J = 8.0 Hz, 1H), 7.87 (app. t, J = 6.4 Hz, 1H), 5.37 (m, 1H), 3.27 (dd, J = 18.6, 4.8 Hz, 1H), 3.14 (dd, J = 17.6, 6.8 Hz, 1H), 2.60 (dd, J = 14.2, 5.6 Hz, 1H), 2.19 (dd, J = 13.2, 8.8 Hz, 1H), 1.53 (s, 3H), 1.39 (s, 3H). $^{13}C\{^1H\}$ NMR (CD_3OD): δ 171.09, 165.76, 146.29, 139.69, 126.16, 122.82, 65.28, 43.99, 42.14, 36.98, 27.01, 26.22. IR (thin film): 2971, 1732 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{12}H_{16}NO_2^+$ 206.1176; found 206.1172.

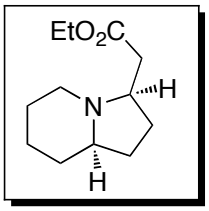


Product **P4** was prepared from **S1** and N,N-dimethyl acrylamide (Aldrich) using standard procedure **A**. After removal of the acetic acid and methanol, NMR analysis of crude mixture shows 55% yield. Water was added and the solution was washed with (5 x 2 mL) of CH_2Cl_2 to remove the acrylamide oligomers, polyoxometalate and catalyst. The water was removed under reduced pressure and 1 mL of saturated $NaBF_4$ solution was added. The product was extracted with (5 x 1 mL) CH_2Cl_2 as a BF_4^- salt. The CH_2Cl_2 layers were combined, dried with $MgSO_4$, filtered and evaporated to afford the product in 40% yield as a yellow oil. ^{1}H NMR ($CDCl_3$): δ 8.80 (d, *J* = 6.0 Hz, 1H), 8.38 (app. t, *J* = 7.6 Hz, 1H), 7.82-7.78 (multiple peaks, 2H), 5.47 (m, 1H), 3.42 (dd, *J* = 17.6, 5.2 Hz, 1H), 3.23 (dd, *J* = 17.6, 5.2 Hz, 1H), 3.04 (s, 3H), 2.91 (s, 3H), 2.57 (dd, *J* = 13.0, 7.6 Hz, 1H), 2.36 (dd, *J* = 13.0, 10.0 Hz, 1H), 1.59 (s, 3H), 1.41 (s, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 168.66, 165.51, 146.12, 140.22, 126.55, 122.47, 65.78, 44.23, 42.80, 37.28, 36.68, 35.65, 28.33, 27.03. ^{19}F{^{1}H} NMR ($CDCl_3$): δ –152.07, –152.13. IR (thin film): 1652 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{14}H_{21}N_2O^+$ 233.1648; found 233.1648.



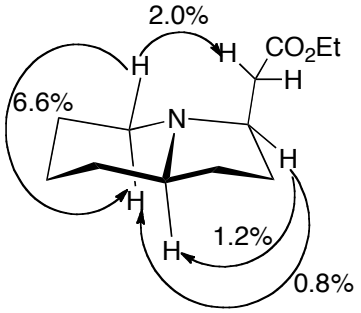
Product **P5** was prepared from **S1** and ethyl vinyl ketone (Aldrich, freshly distilled prior to use) using standard procedure **A** and was obtained in 40% yield as a yellow solid. Mp = 102-105 °C. ^{1}H NMR ($CDCl_3$): δ 8.74 (d, *J* = 6.4 Hz, 1H), 8.41 (app. t, *J* = 7.2 Hz, 1H), 7.85 (app t., *J* = 7.6 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 5.39 (m, 1H), 3.60 (dd, *J* = 19.2, 4.8 Hz, 1H), 3.38 (dd, *J* = 18.8, 6.4 Hz, 1H), 2.64 (dd, *J* = 13.2, 7.6 Hz, 1H), 2.56 (m, 2H), 2.19 (dd, *J* = 13.2, 9.6 Hz, 1H), 1.59 (s, 3H), 1.43 (s, 3H), 1.02 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 208.34, 165.39, 146.30, 140.09, 126.82, 122.59, 64.90, 44.89, 44.46, 42.80, 36.21, 28.30, 27.34, 7.55. ^{19}F{^{1}H} NMR ($CDCl_3$): δ –152.12, –152.17. IR (thin film): 1714 cm^{-1}. HRMS electrospray (m/z): $[M]^+$ calcd for $C_{14}H_{20}NO^+$ 218.1539; found 218.1537.

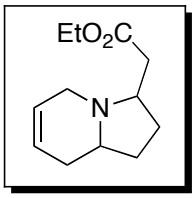
V. Reductions of Pyridinium Salts



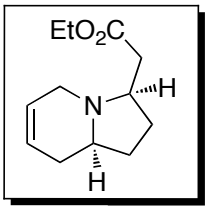
To a 25 mL flask were added **2** (147 mg, 0.50 mmol), PtO_2 (11 mg, 0.048 mmol, 10 mol %, Aldrich), and ethanol (5 mL). Hydrogen was bubbled through the resulting suspension for 5 min, and then the mixture was stirred at room temperature for 14 h under a balloon of hydrogen. The resulting suspension was filtered through a plug of glass wool, and the solvent was removed under vacuum. The crude product was dissolved in CH_2Cl_2 (30 mL) and washed with 1 M aqueous NaOH (1 x 20 mL). The aqueous layer was extracted with CH_2Cl_2 (2 x 30 mL), and the CH_2Cl_2 extracts were combined, dried over Na_2SO_4, and concentrated under vacuum. ^{1}H NMR spectroscopic analysis of the crude product showed a 28:1 ratio of two diastereomeric products. The crude product was purified by chromatography on basic alumina by gradient elution starting with 95:5 and ending with 90:10 hexanes/ethyl acetate to provide diastereomerically pure **12** as a colorless oil (79 mg, 75% yield, R_f = 0.31 in 90:10 hexanes/ethyl acetate on an alumina TLC plate). The stereochemistry of **12** was determined by nOe analysis (Figure 2). ^{1}H NMR ($CDCl_3$): δ 4.13 (q, *J* = 7.2 Hz, 2H), 3.05 (m, 1H), 2.68 (dd, *J* = 14.8, 4.0 Hz, 1H), 2.53 (dtd, *J* = 9.2, 7.8, 4.0 Hz, 1H), 2.26 (dd, *J* = 14.8, 9.2 Hz, 1H), 1.99-1.86 (multiple peaks, 2H), 1.86 (ddd, *J* = 12.4, 10.8, 3.2 Hz, 1H), 1.80-1.71 (multiple peaks, 3H), 1.65 (m, 1H), 1.53-1.41 (multiple peaks, 2H), 1.35 (qd, *J* = 11.2, 6.8 Hz, 1H), 1.25 (t, *J* = 7.2 Hz, 3H), 1.23-1.15 (multiple peaks, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 172.66, 65.31, 61.51, 60.40, 51.46, 39.18, 31.39, 29.24, 28.81, 25.62, 24.46, 14.42. IR (thin film): 1736 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{12}H_{22}NO_2^+$ 212.1645; found 212.1647.

Figure S2. Observed nOes for **12**:



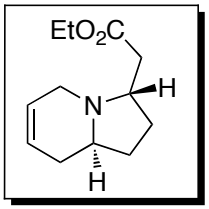


To a 25 mL flask were added **2** (147 mg, 0.50 mmol) and ethanol (5 mL). The solution was cooled to 0˚C in an ice bath and $NaBH_4$ (49 mg, 1.3 mmol, 2.6 equiv, Aldrich) was added slowly over 5 min. The reaction mixture was stirred at 0 ºC for 6 h, warmed to room temperature, and stirred for an additional 4 h. Water (1 mL) was added, and the reaction was concentrated to ~2 mL. The resulting white slurry was partitioned between CH_2Cl_2 (15 mL) and water (15 mL), and the pH of the aqueous layer was adjusted to ~10 by addition of K_2CO_3. The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The CH_2Cl_2 extracts were combined, dried with Na_2SO_4, and concentrated under vacuum to give a colorless oil. ^{1}H NMR analysis of the crude product showed a 2.3:1 ratio of two diastereomeric products. The crude products were purified by chromatography on basic alumina by gradient elution starting with 98:2 and ending with 90:10 hexanes/ethyl acetate to provide the products **13A** and **13B**.



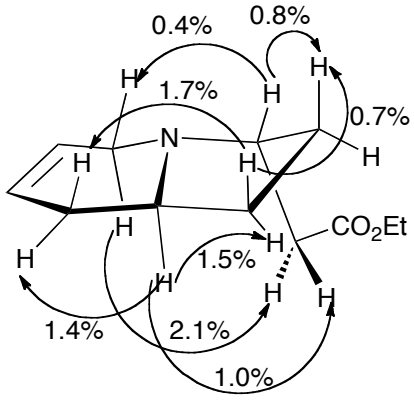
Product **13A** was isolated as a colorless oil (60 mg, 57% yield, R_f = 0.33 in 95:5 CH_2Cl_2/MeOH on a silica gel TLC plate). ^{1}H NMR ($CDCl_3$): δ 5.75 (m, 1H), 5.71 (m, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.44 (ddd, *J* = 16.0, 4.0, 2.4 Hz, 1H), 2.73 (m, 1H), 2.72 (dd, *J* = 14.4, 4.8 Hz, 1H), 2.67 (dddd, *J* = 12.4, 8.8, 8.0, 4.4 Hz, 1H), 2.32 (dd, *J* = 14.4, 8.8 Hz, 1H), 2.36-2.21 (multiple peaks, 2H), 2.08-1.90 (multiple peaks, 3H), 1.53 (dddd, *J* = 12.4, 10.8, 6.4, 4.8 Hz, 1H), 1.42 (dddd, *J* = 12.4, 10.8, 8.4, 6.4 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 172.54, 125.57, 125.29, 62.32, 60.53, 60.50, 51.50, 39.55, 33.07, 29.64, 29.05, 14.45. IR (thin film): 3033, 1734, 1656 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{12}H_{20}NO_2^+$ 210.1489; found 210.1487.

The stereochemistry of **13A** was confirmed by hydrogenation to form **12** in the following manner: To a 25 mL flask were added **13A** (21 mg, 0.10 mmol), PtO_2 (2 mg, 0.009 mmol, 9 mol %), and ethanol (1 mL). Hydrogen was bubbled through the resulting suspension, and then the mixture was stirred at room temperature for 14 h under a balloon of hydrogen. The suspension was filtered through a plug of glass wool, and the solvent was removed under vacuum. The ^{1}H NMR spectrum of the crude product matched that of **12**.



Product **13B** was isolated as a colorless oil (25 mg, 24% yield, R_f = 0.32 in 90:10 CH_2Cl_2/MeOH on a silica gel TLC plate). The stereochemistry of **13B** was determined by nOe analysis (Figure 3). ^{1}H NMR (C_6D_6): δ 5.65 (ddddd, *J* = 10.0, 4.0, 2.8, 2.4, 2.0 Hz, 1H), 5.55 (ddq, *J* = 10.0, 4.0, 2.0 Hz, 1H), 3.97 (q, *J* = 7.2 Hz, 2H), 3.70 (dddd, *J* = 9.2, 8.0, 4.8, 3.2 Hz, 1H), 3.16 (dddt, *J* = 16.8, 4.0, 3.6, 2.0 Hz, 1H), 3.08 (ddq, *J* = 16.8, 4.0, 2.0 Hz, 1H), 2.68 (ddt, *J* = 8.8, 6.8, 5.6 Hz, 1H), 2.46 (dd, *J* = 14.4, 4.8 Hz, 1H), 2.04 (dd, *J* = 14.4, 9.2 Hz, 1H), 2.03 (dddd, *J* = 12.8, 10.4, 8.0, 6.8 Hz, 1H), 1.87-1.82 (multiple peaks, 2H), 1.78 (ddt, *J* = 12.4, 10.0, 6.8 Hz, 1H), 1.56 (dddd, *J* = 12.8, 10.0, 4.4, 3.2 Hz, 1H), 1.24 (dddd, *J* = 12.4, 10.4, 5.6, 4.4 Hz, 1H), 0.97 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 173.05, 125.44, 125.21, 60.59, 57.75, 54.84, 46.19, 35.53, 31.75, 29.84, 28.56, 14.45. IR (thin film): 3030, 1733, 1659 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{12}H_{20}NO_2^+$ 210.1489; found 210.1488.

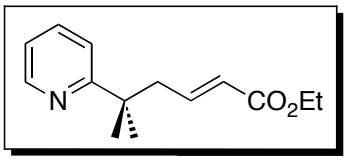
Figure S3. Observed nOes for **13B**:



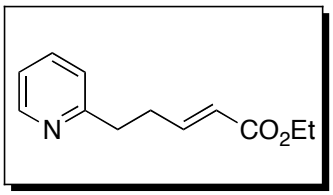
VI. Formation of the Linear Heck Products

Standard procedure for Formation of the Linear Heck Product

To a 25 mL flask charged with the appropriate pyridinium salt (0.50 mmol, 1.0 equiv) under nitrogen was added 10 mL of dry CH_2Cl_2 followed by 1,8-diazabicyclo[5.4.0]undec-7-ene (150 μL, 1.0 mmol, 2.0 equiv, Aldrich). The reaction was stirred for 1h at room temperature, after which the solution was diluted with CH_2Cl_2 (30 mL). The CH_2Cl_2 solution was washed with saturated aqueous $NaHCO_3$ (2 x 20 mL), and the combined aqueous layers were extracted once with CH_2Cl_2 (20 mL). The CH_2Cl_2 layers were combined, washed with 20 mL of brine, dried over Na_2SO_4, concentrated under vacuum, and chromatographed as indicated below.



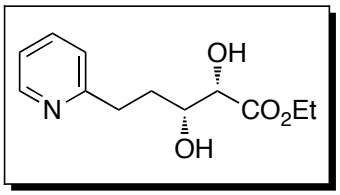
Product **14** was made from pyridinium salt **1** and was purified by chromatography on basic alumina, which had been flushed with 99:1 hexanes/triethylamine, eluting with 69:30:1 hexanes/ethyl acetate/triethylamine to provide the product **14** as a colorless oil (96 mg, 82% yield, R_f = 0.39 in 78:20:2 hexanes/ethyl acetate/triethylamine on an alumina TLC plate to which 98:2 hexanes/triethylamine had been applied and allowed to evaporate prior to spotting with the sample). ^{1}H NMR ($CDCl_3$): δ 8.57 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.61 (ddd, *J* = 8.0, 7.6, 2.0 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 1H), 7.10 (ddd, *J* = 7.6, 4.8, 0.8 Hz, 1H), 6.74 (dt, *J* = 15.6, 7.6 Hz, 1H), 5.77 (dt, *J* = 15.6, 1.2 Hz, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.65 (dd, *J* = 7.6 Hz, 1.2 Hz, 2H), 1.36 (s, 6H), 1.24 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 167.06, 166.66, 149.07, 146.52, 136.50, 123.69, 121.23, 119.88, 60.31, 45.72, 40.93, 27.96, 14.45. IR (thin film): 3052, 1718, 1652 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{14}H_{20}NO_2^+$ 234.1489; found 234.1486.



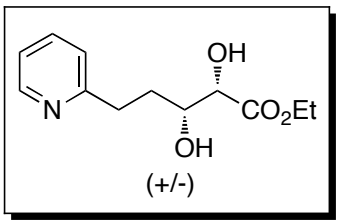
Product **15** was made from pyridinium salt **2** and was purified by chromatography on basic alumina eluting with 70:30 hexanes/ethyl acetate to provide the product **15** as a colorless oil (98 mg, 95% yield, R_f = 0.28 in 80:20 hexanes/ethyl acetate on an alumina TLC plate). ^{1}H NMR ($CDCl_3$): δ 8.53 (ddd, *J* = 4.8, 1.6, 1.2 Hz, 1H), 7.59 (ddd, *J* = 8.0, 7.6, 2.0 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 1H), 7.12 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 7.00 (dt, *J* = 15.6, 6.8 Hz, 1H), 5.84 (dt, *J* = 15.6, 1.6 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.94 (dd, *J* = 8.0, 7.2 Hz, 2H), 2.66 (dddd, 8.0, 7.2, 6.8, 1.6 Hz, 2H), 1.27 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 166.78, 160.53, 149.64, 148.09, 136.62, 123.06, 122.12, 121.56, 60.40, 36.74, 32.12, 14.45. IR (thin film): 3064, 1718,

1654 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{12}H_{16}NO_2^+$ 206.1176; found 206.1174.

VII. Derivatizations of the Linear Heck Product



To a 20 mL vial was added AD-mix β (700 mg, Aldrich), methanesulfonamide (95 mg, 1.00 mmol, 2.0 equiv, Aldrich), *t*-BuOH (1 mL), and water (1 mL). The mixture was stirred and cooled to 0 ºC in an ice bath. **15** (103 mg, 0.50 mmol) was added, and the mixture was stirred at 0 ºC for 24 h. A 10% aqueous solution of Na_2SO_3 (5 mL) was added with stirring followed by the addition of another 5 mL of water. The mixture was extracted with CH_2Cl_2 (8 x 10 mL), and the CH_2Cl_2 extracts were combined and concentrated under vacuum. The crude product was purified by chromatography on silica gel, eluting with 95:5 CH_2Cl_2/MeOH to provide the product **16** as a colorless oil (110 mg, 92% yield, 97% ee as determined by Mosher ester analysis, R_f = 0.19 in 95:5 CH_2Cl_2/MeOH). The absolute stereochemistry of the diol was assumed based on the mnemonic rule for the facial selectivity of the Sharpless dihydroxylation reaction.[9] ^{1}H NMR ($CDCl_3$): δ 8.46 (d, *J* = 5.4 Hz, 1H), 7.63 (td, *J* = 7.6, 2.0 Hz, 1H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.14 (dd, *J* = 7.6, 5.4 Hz, 1H), 5.98 (br s, 1H), 4.30 (dq, *J* = 10.4, 7.2 Hz, 1H), 4.28 (dq, *J* = 10.4, 7.2 Hz, 1H), 4.11-4.04 (multiple peaks, 2H), 3.29 (br d, *J* = 5.6 Hz, 1H), 3.10 (ddd, *J* = 15.2, 7.6, 4.8 Hz, 1H), 3.06 (ddd, *J* = 15.2, 7.2, 5.2 Hz, 1H), 2.15 (m, 1H), 2.06 (m, 1H), 1.32 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 173.61, 161.24, 148.54, 137.24, 123.53, 121.52, 74.19, 72.80, 61.84, 34.76, 32.46, 14.36. IR (thin film): 3368, 1734 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{12}H_{18}NO_4^+$ 240.1230; found 240.1228. $[\alpha]_D^{25}$ = –11.8 (*c* = 2.00, $CHCl_3$).

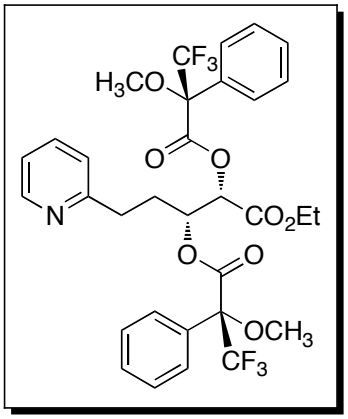


To a solution of **15** (103 mg, 0.50 mmol) in 9:1 acetone/water (4 mL) in a 20 mL vial was added 4-methylmorpholine *N*-oxide (117 mg, 1.00 mmol, 2.0 equiv, Aldrich) and aqueous 4% OsO_4 (32 μL, 0.0050 mmol, 1.0 mol %, Aldrich). The mixture was stirred at room temperature for 12 h. A 10% aqueous solution of Na_2SO_3 (5 mL) was added with stirring followed by the addition of another 5 mL of water. The mixture was extracted with CH_2Cl_2 (8 x 10 mL), and the CH_2Cl_2 extracts were combined and concentrated under vacuum. The crude product was purified by chromatography on silica gel, eluting with 95:5 CH_2Cl_2/MeOH to provide the racemic product ***rac*-16** as a colorless oil (105 mg, 88% yield). The spectroscopic data matched that of the diol **16** made by Sharpless asymmetric dihydroxylation.

Mosher analysis[10] of 16:

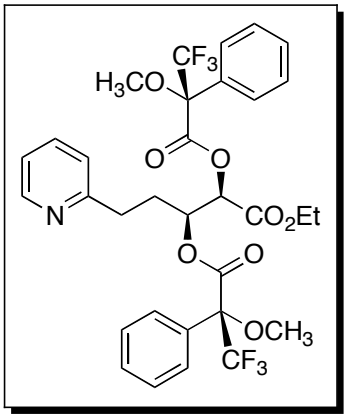
The (*R*)-Mosher diester of **16** was prepared from **16** and (*R*)-(+)-α-methoxy-α-trifluoromethylphenylacetic acid ((*R*)-(+)-MTPA) as follows: To a 25 mL flask was added ***rac*-16** (24 mg, 0.10 mmol). Toluene (5 mL) was added and evaporated under high vacuum to remove water as an azeotrope. (*R*)-MTPA, (70 mg, 0.30 mmol, 3.0 equiv, Matrix Scientific) and dry CH_2Cl_2 (1 mL) were added under nitrogen. 1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride (58 mg, 0.30 mmol, 3.0 equiv, TCI America), 4-(dimethylamino)pyridine (3.0 mg, 0.025 mmol, 0.25 equiv, Aldrich), and *N*-ethyldiisopropylamine (85 μL, 0.50 mmol, 5.0 equiv, Alfa Aesar) were added sequentially. The reaction mixture was stirred at room temperature for 24 h, diluted with 10 mL of CH_2Cl_2, and then this CH_2Cl_2 solution was washed with saturated aqueous NH_4Cl (10 mL), water (10 mL), saturated aqueous $NaHCO_3$ (10 mL), and brine (10 mL). The CH_2Cl_2 solution was dried over Na_2SO_4, and the solvent was evaporated under vacuum. The crude product was purified by chromatography on silica gel, eluting with 98:2 CH_2Cl_2/MeOH, and collecting all of the fractions that contained the Mosher ester products according to TLC. The fractions were concentrated to provide the product mixture (**bis-(*R*)-MTPA esters of *rac*-16**) as a colorless oil (63 mg, 94% yield, R_f = 0.29 in 98:2 CH_2Cl_2/MeOH).

The Mosher esterification reaction above was performed on diol **16** made by Sharpless asymmetric dihydroxylation to provide predominantly the **bis-(*R*)-MTPA ester of *(2S, 3R)*-16**. Integration of the doublets at 5.41 ppm and 5.49 ppm in the ^{1}H NMR spectrum of the product mixture indicated that the ee of **16** was 97% (Figure 4).



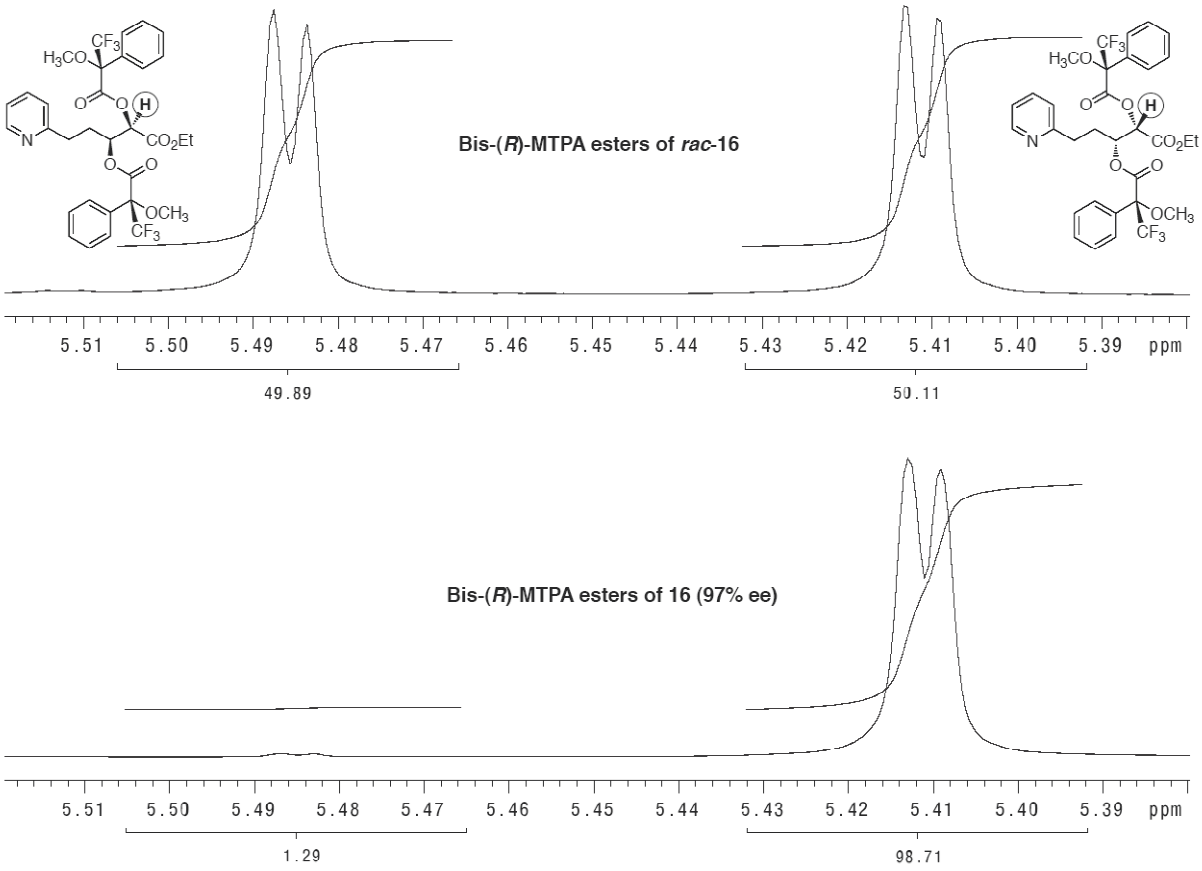
Bis-(*R*)-MTPA ester of *(2S, 3R)*-16: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.51 (br d, *J* = 5.0 Hz, 1H), 7.58 (br d, *J* = 7.5 Hz, 2H), 7.57 (td, *J* = 7.5, 1.5 Hz, 1H), 7.51 (br d, *J* = 7.5 Hz, 2H), 7.41-7.31 (multiple peaks, 6H), 7.12 (dd, *J* = 7.5, 5.0 Hz, 1H), 6.94 (d, *J* = 7.5 Hz, 1H), 5.68 (td, *J* = 7.0, 2.0 Hz, 1H), 5.41 (d, *J* = 2.0 Hz, 1H), 4.18 (dq, *J* = 11.0, 7.0 Hz, 1H), 4.16 (dq, *J* = 11.0, 7.0 Hz, 1H), 3.48 (s, 3H), 3.38 (s, 3H), 2.65 (dt, *J* = 14.0, 7.5 Hz, 1H), 2.62 (dt, *J* = 14.0, 7.5 Hz, 1H), 2.16 (q, *J* = 7.0 Hz, 2H), 1.23 (t, *J* = 7.0 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 166.42, 166.34, 166.13, 159.55, 149.68, 136.73, 132.02, 131.38, 129.98, 129.87, 128.60, 128.58, 128.06,

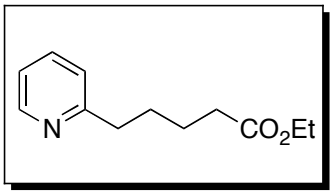
127.55, 123.33 (q, $^{1}J_{CF}$ = 288.5 Hz), 123.27 (q, $^{1}J_{CF}$ = 288.5 Hz), 123.07, 121.76, 85.14 (q, $^{2}J_{CF}$ = 28.0 Hz), 84.70 (q, $^{2}J_{CF}$ = 27.7 Hz), 74.24, 73.98, 62.76, 55.73 (2C), 33.33, 30.33, 14.04. $^{19}F\{^{1}H\}$ NMR ($CDCl_3$): δ –71.64, –72.21. IR (thin film): 1754 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{32}H_{32}F_6NO_8^+$ 672.2027; found 672.2020.



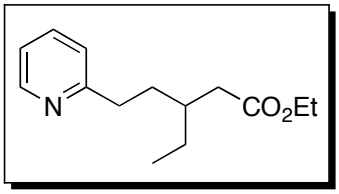
Bis-(*R*)-MTPA ester of *(2R, 3S)*-16: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.52 (br d, *J* = 5.0 Hz, 1H), 7.66 (br d, *J* = 7.5 Hz, 2H), 7.58 (td, *J* = 7.5, 1.5 Hz, 1H), 7.51 (br d, *J* = 7.5 Hz, 2H), 7.41-7.31 (multiple peaks, 6H), 7.13 (dd, *J* = 7.5, 5.0 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 5.64 (td, *J* = 7.0, 2.0 Hz, 1H), 5.49 (d, *J* = 2.0 Hz, 1H), 4.08 (q, *J* = 7.0 Hz, 2H), 3.60 (s, 3H), 3.42 (s, 3H), 2.72 (ddd, *J* = 14.5, 8.0, 7.0 Hz, 1H), 2.69 (ddd, *J* = 14.5, 8.0, 7.0 Hz, 1H), 2.11 (ddt, *J* = 15.0, 7.5, 7.0 Hz, 1H), 2.01 (ddt, *J* = 15.0, 7.5, 7.0 Hz, 1H), 1.19 (t, *J* = 7.0 Hz, 3H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$): δ 166.21, 166.18, 165.78, 159.47, 149.62, 136.74, 131.90, 131.58, 129.88, 129.87, 128.62, 128.54, 127.73, 127.67, 123.36 (q, $^{1}J_{CF}$ = 288.9 Hz, 1C), 123.26 (q, $^{1}J_{CF}$ = 288.5 Hz, 1C), 122.98, 121.77, 84.76 (q, $^{2}J_{CF}$ = 28.0 Hz, 1C), 84.74 (q, $^{2}J_{CF}$ = 27.7 Hz, 1C), 74.56, 73.68, 62.63, 55.88, 55.50, 33.47, 29.72, 14.01. $^{19}F\{^{1}H\}$ NMR ($CDCl_3$): δ –71.79, –72.06.

Figure S4. Comparison of (*R*)-MTPA esters of *rac*-**16** and (*R*)-MTPA esters of **16** made by Sharpless dihydroxylation.





To a 20 mL scintillation vial was added palladium on carbon (10.6 mg, 10 wt% Pd/C, 0.01 mmol, 10 mol % Pd, Aldrich), **15** (20.7 mg, 0.1 mmol) and MeOH (0.5 mL). The vial was fitted with a 24/40 septum and was stirred at room temperature for 10 h under a balloon of hydrogen. The solvent was evaporated, and the product was purified by chromatography on silica gel, eluting with 60:40 hexanes/ethyl acetate. Compound **17** was obtained as a colorless oil (9.6 mg, 90% yield, R_f = 0.3 in 60:40 hexanes/ethyl acetate). ^{1}H NMR ($CDCl_3$): δ 8.52 (dd, *J* = 4.8, 0.8 Hz, 1H), 7.59 (app. td, *J* = 7.6, 2.0 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.10 (dd, *J* = 7.6, 5.2 Hz, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.81 (t, *J* = 7.2 Hz, 2H), 2.34 (t, *J* = 7.6 Hz, 2H), 1.80-1.68 (multiple peaks, 4H), 1.25 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 173.84, 161.99, 149.47, 136.50, 122.94, 121.21, 60.44, 38.19, 34.40, 29.44, 24.86, 14.45. IR (thin film): 1732 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{12}H_{18}NO_2^+$ 208.1332; found 208.1331.



Copper(I) bromide (7 mg, 0.05 mmol, 10 mol %, Fisher) and lithium chloride (4 mg, 0.1 mmol, 20 mol %, Fisher) were added to an oven-dried 5 mL Schlenk tube under nitrogen. The tube was evacuated, heated with a heat gun, allowed to cool to room temperature under vacuum, and filled with nitrogen. Dry THF (1 mL) was added, and the mixture stirred for 10 min. The tube was placed in an ice bath, and after 10 min, a solution of **15** (103 mg, 0.50 mmol, 1 equiv) in dry THF (1 mL) was added followed by TMSCl (70 μL, 0.55 mmol, 1.1 equiv, Aldrich). The solution was stirred at 0 ºC for 15 min, and then 3 M EtMgBr in Et_2O (250 μL, 0.75 mmol, 1.5 equiv, Aldrich) was added dropwise. The solution was stirred for 1 h at 0 ºC and then poured into 10 mL of saturated aqueous NH_4Cl. The mixture was extracted with Et_2O (3 x 20 mL). The Et_2O extracts were combined, washed with brine, dried over Na_2SO_4, and concentrated under vacuum. The crude product was purified by chromatography on silica gel, eluting with 80:20 hexanes/ethyl acetate, to provide product **18** as a colorless oil (222 mg, 94% yield, R_f = 0.22 in 70:30 hexanes/ethyl acetate). ^{1}H NMR ($CDCl_3$): δ 8.51 (ddd, *J* = 4.8, 2.0, 0.8 Hz, 1H), 7.58 (app. td, *J* = 7.6, 2.0 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.09 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 2.79 (t, *J* = 4.0 Hz, 2H), 2.31 (d, *J* = 6.8 Hz, 2H), 1.90 (tq, *J* = 6.8, 6.4 Hz, 1H), 1.82-1.66 (multiple peaks, 2H), 1.51-1.34 (multiple peaks, 2H), 1.24 (t, *J* = 7.2 Hz, 3H), 0.90 (t, *J* = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR ($CDCl_3$): δ 173.57, 162.36, 149.41, 136.48, 122.83, 121.14, 60.33, 38.83, 36.43, 35.75, 33.75, 26.37, 14.44, 10.89. IR (thin film): 1734 cm^{-1}. HRMS electrospray (m/z): $[M+H]^+$ calcd for $C_{14}H_{22}NO_2^+$ 236.1645; found 236.1645.

VIII. References

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9kjsetme-sm

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Sample directory:

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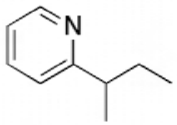
Solvent: cdcl3
Data collected on: Jan 11 2011

Temp. 23.0 C / 296.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206274 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S4

9kjsetme-smC

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Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

Sample directory:

FidFile: 9kjsetme-smC

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 11 2011

Temp. 23.0 C / 296.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
160 repetitions
OBSERVE C13, 100.7109708 MHz
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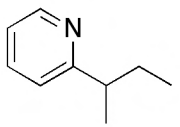
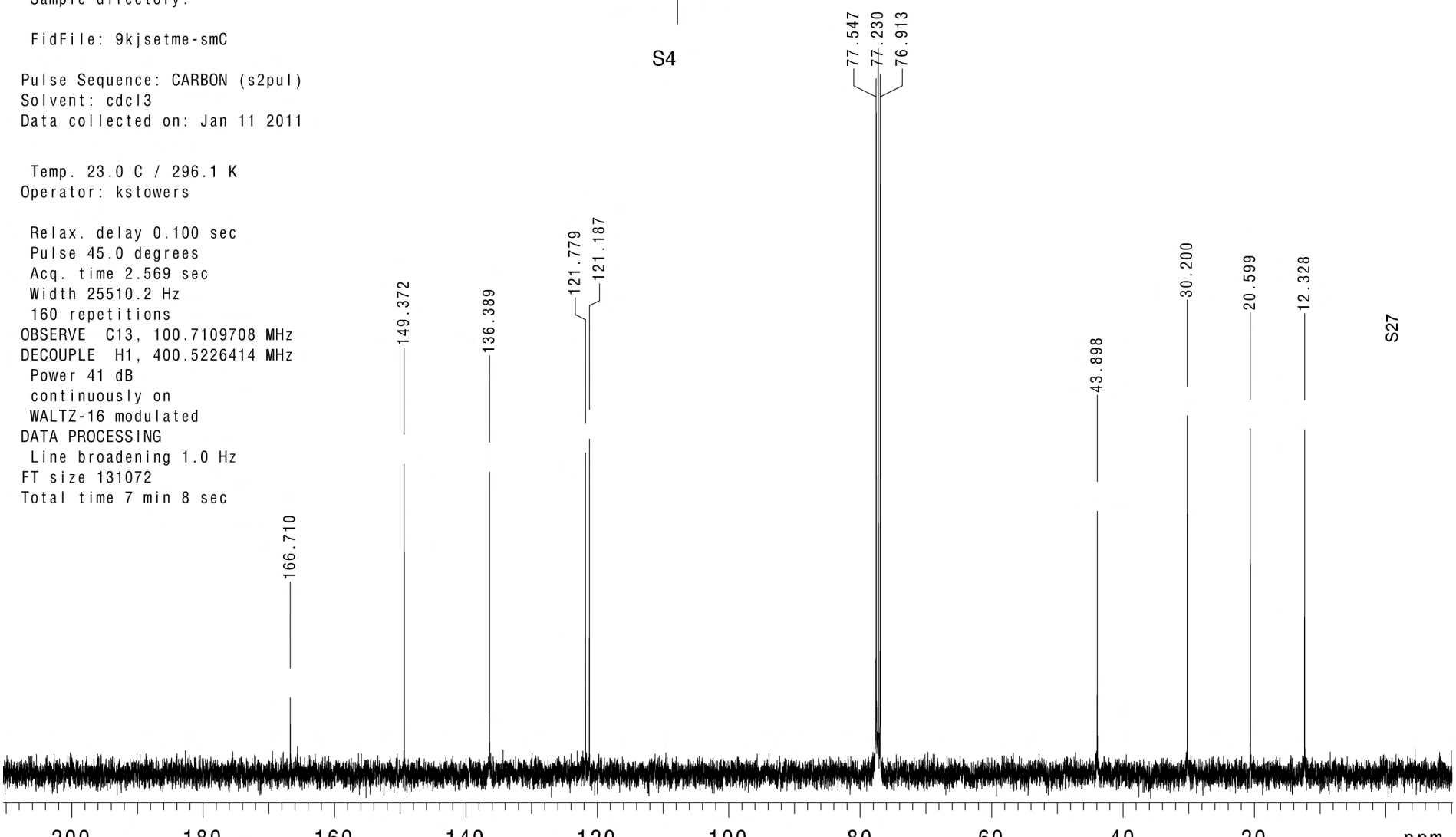
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 min 8 sec



S4



9kjscyclopr-sm

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

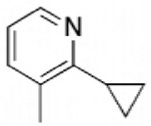
Sample directory:

FidFile: 9kjscyclopr-sm

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 11 2011

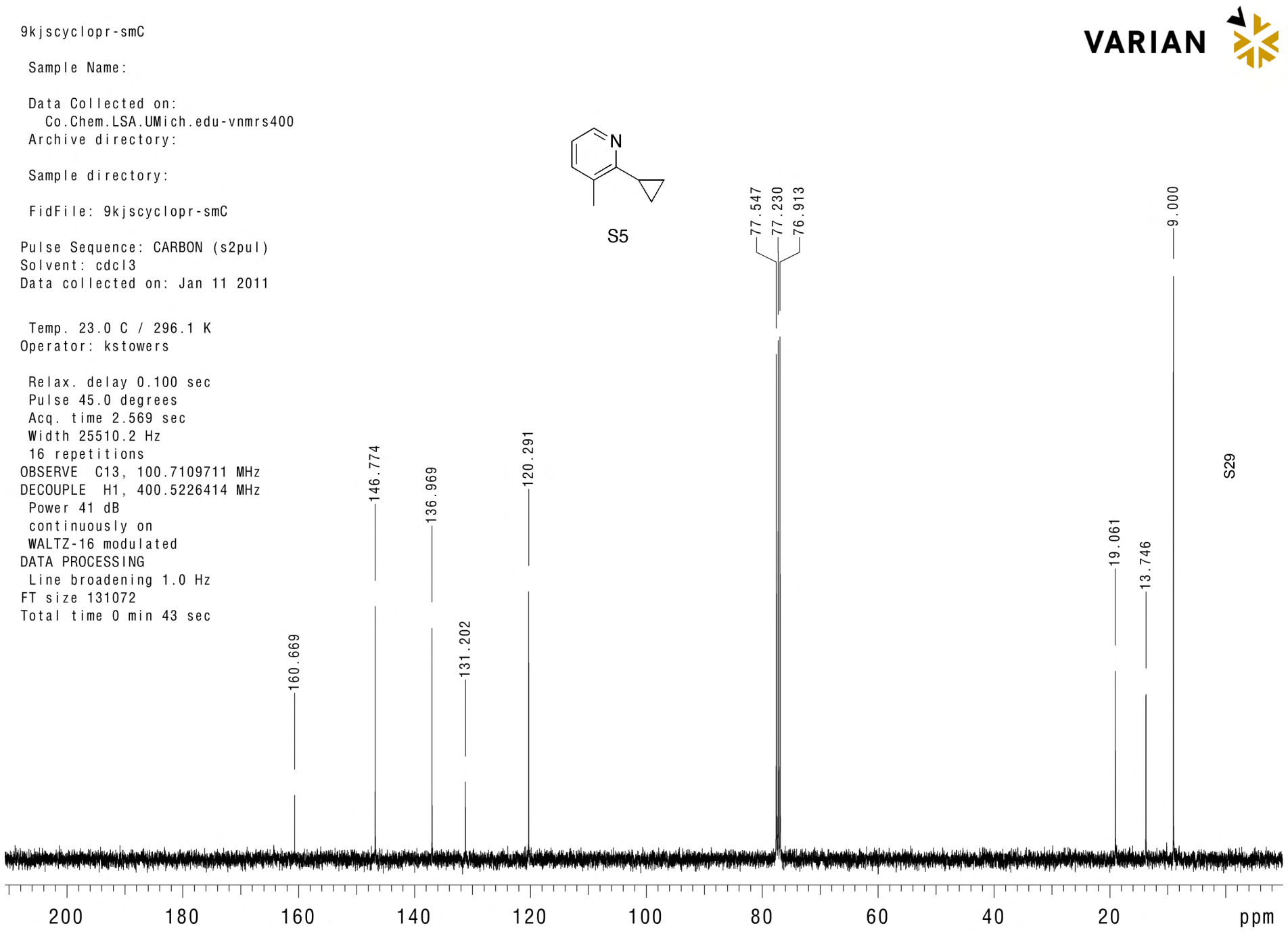
Temp. 23.0 C / 296.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206311 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S5

S28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Archive directory:
Sample directory:
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Solvent: cdcl3
Data collected on: Jan 11 2011
Temp. 23.0 C / 296.1 K
Operator: kstowers
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
16 repetitions
OBSERVE C13, 100.7109711 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min 43 sec
VARIAN
N
S5
S29
160.669
146.774
136.969
131.202
120.291
77.547
77.230
76.913
19.061
13.746
9.000
200
180
160
140
120
100
80
60
40
20
ppm

9kjs6x-mMe

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

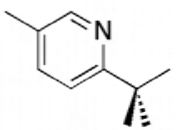
Sample directory:

FidFile: 9kjs6x-mMe

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2011

Temp. 31.0 C / 304.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389149 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 24 sec



S6

S30

9kjs6x-mMe-C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

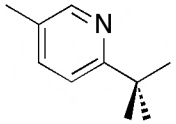
Sample directory:

FidFile: 9kjs6x-mMe-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2011

Temp. 31.0 C / 304.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
624 repetitions
OBSERVE C13, 100.4641163 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 27 min



S6

S31

8kjsA-OMe-1HNMR

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

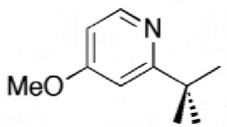
Sample directory:

FidFile: 8kjsA-OMe-1HNMR

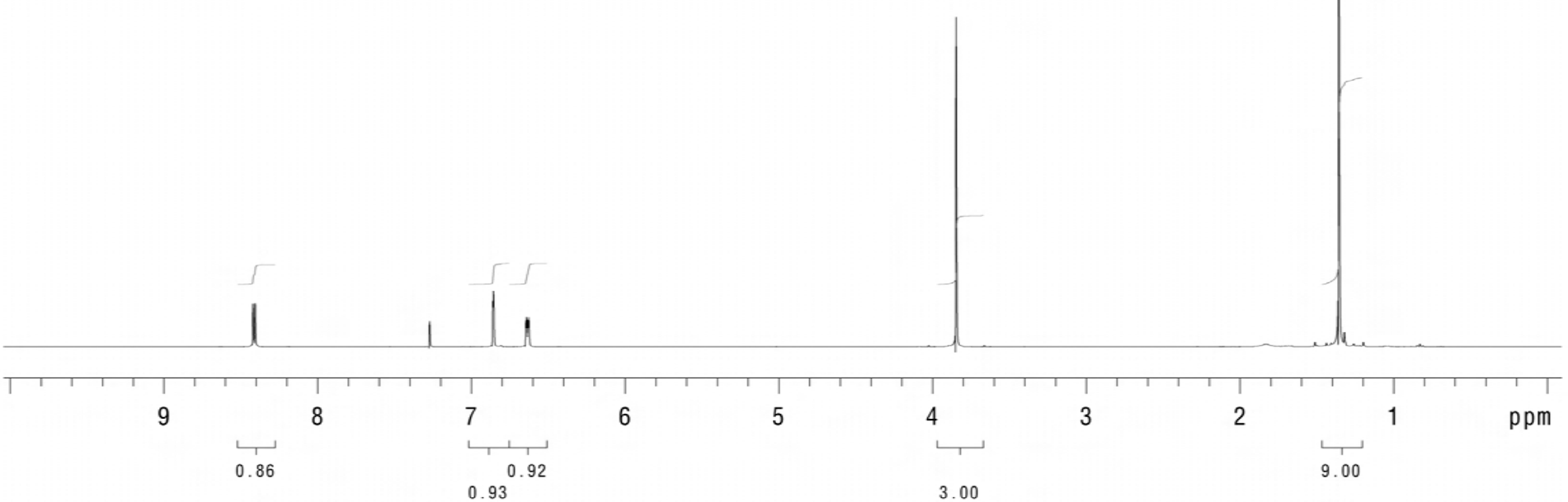
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Nov 30 2010

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206274 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S7



8kjsA-OMe-CNMR

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

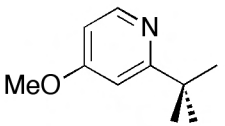
Sample directory:

FidFile: 8kjsA-OMe-CNMR

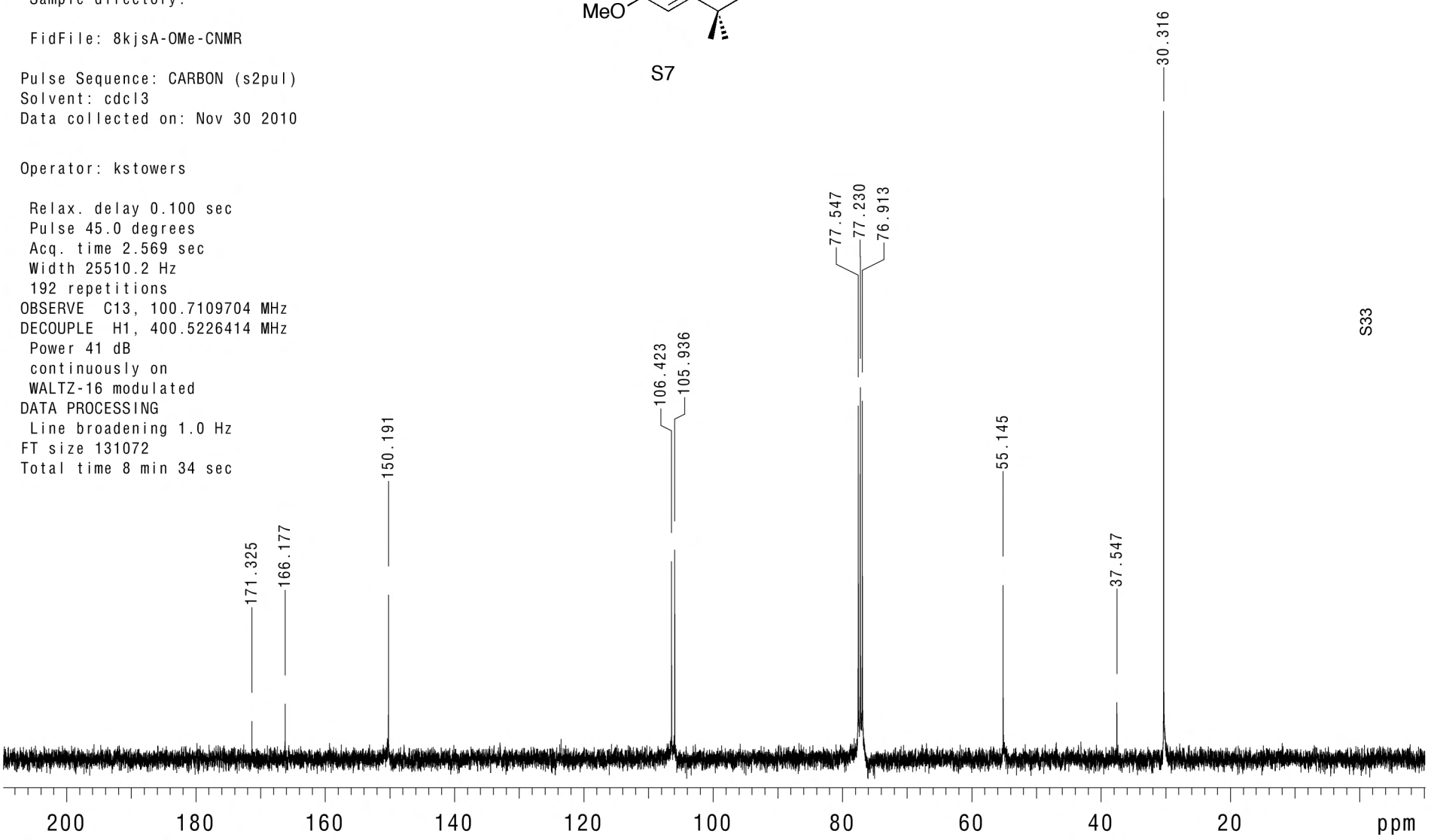
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 30 2010

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
192 repetitions
OBSERVE C13, 100.7109704 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 8 min 34 sec



S7



9kjsCF3-smtst

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

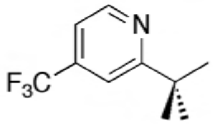
Sample directory:

FidFile: 9kjsCF3-smtst

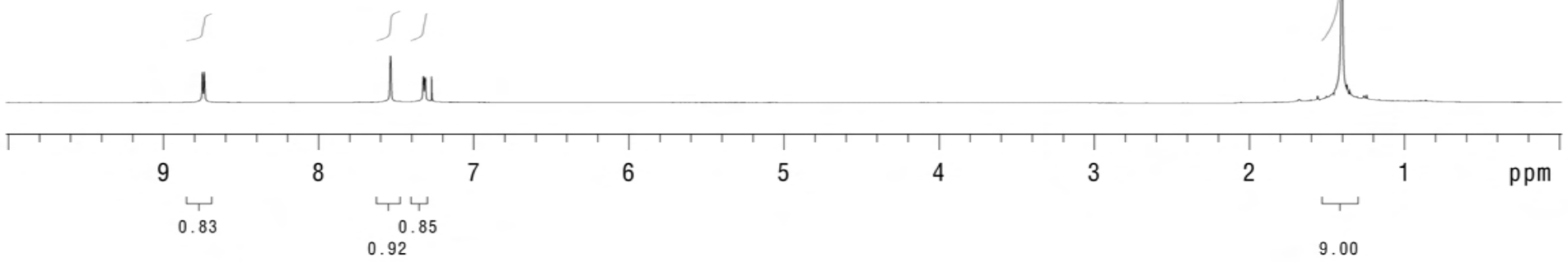
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 26 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
Single scan
OBSERVE H1, 399.5389148 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 12 sec



S8



STANDARD CARBON PARAMETERS

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

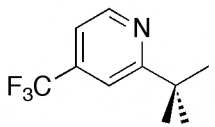
Sample directory:

FidFile: carbon

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 27 2011

Temp. 31.0 C / 304.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
10000 repetitions
OBSERVE C13, 100.4641107 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 26 min



S8

S35

9kjsCF3sm-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

Sample directory:

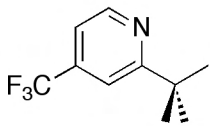
FidFile: 9kjsCF3sm-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Temp. 45.0 C / 318.1 K
Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec

-64.839



S8

S36

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

9kjsomethylsm

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

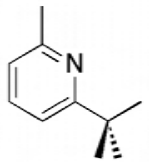
Sample directory:

FidFile: 9kjso-methylsm

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Temp. 44.0 C / 317.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.5389140 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 40 sec



S9

VARIAN

S37

9kjs-o-MeC

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

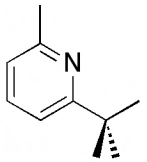
Sample directory:

FidFile: 9kjs-o-MeC

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 18 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
816 repetitions
OBSERVE C13, 100.4641194 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 36 min



S9

S38

168.836
157.224
136.317
120.118
115.828
77.548
77.230
76.912
37.461
30.445
24.997

200 180 160 140 120 100 80 60 40 20 ppm

9kjs63-1H

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

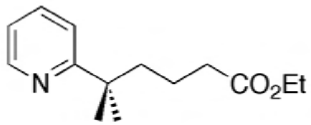
Sample directory:

FidFile: 9kjs63-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 4 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S10

9kjs63-13C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

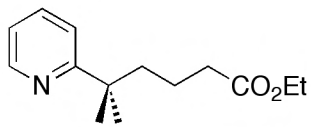
Sample directory:

FidFile: 9kjs63-13C

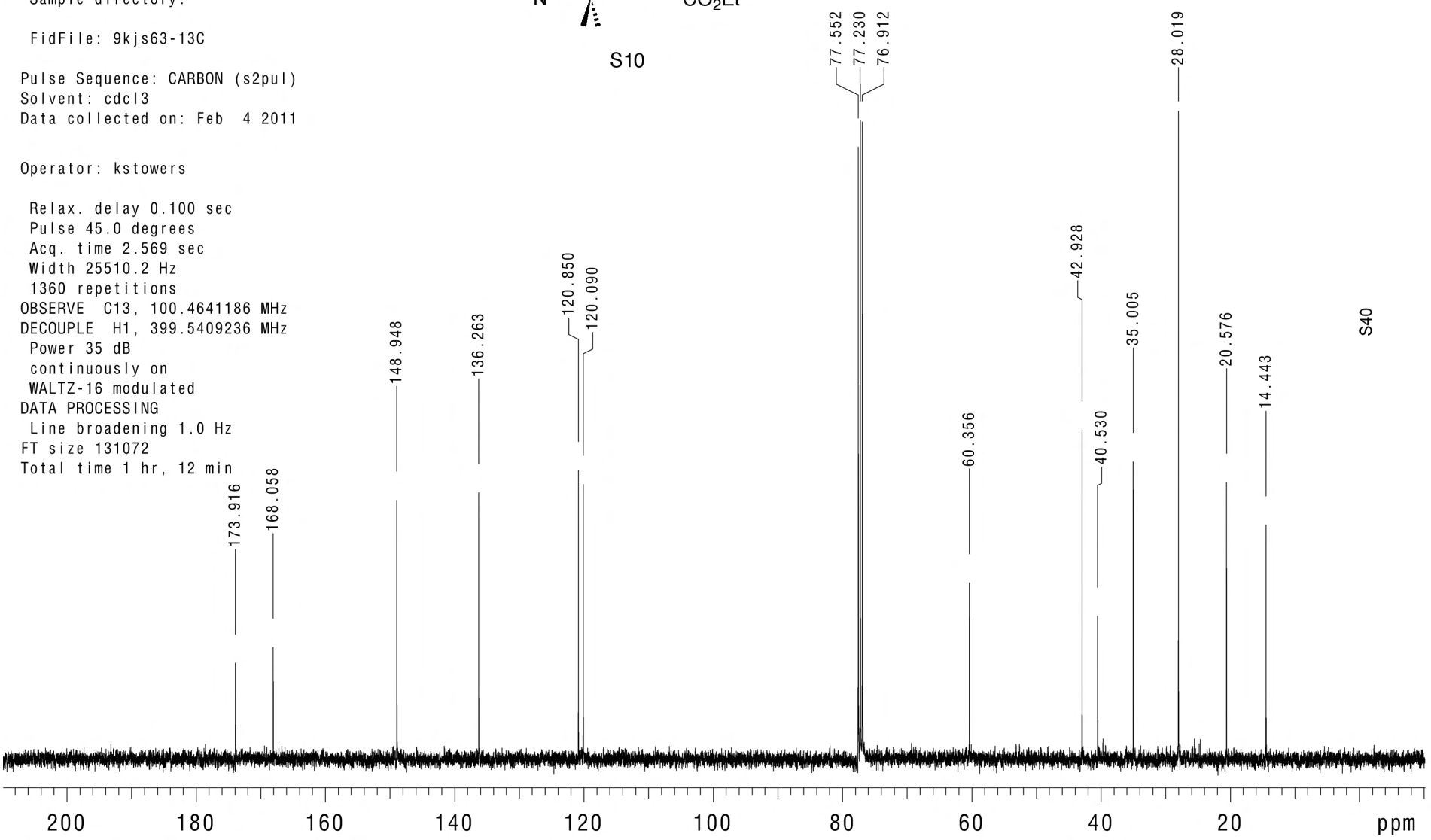
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 4 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1360 repetitions
OBSERVE C13, 100.4641186 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 12 min



S10



S40

8kjs190-1

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

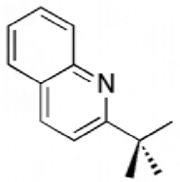
Sample directory:

FidFile: 8kjs190-1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 9 2010

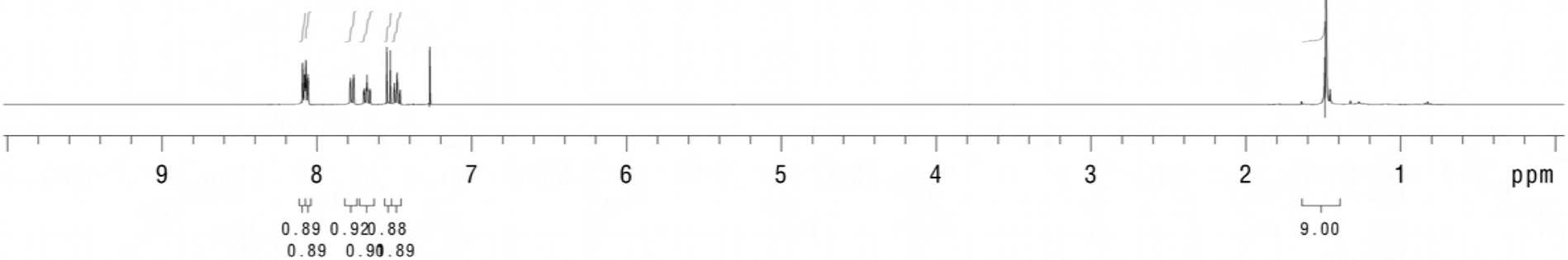
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206278 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



S11

S41



9kjs90-tBuQuinC13

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

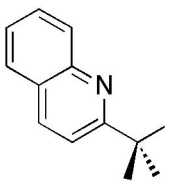
Sample directory:

FidFile: 9kjs90-tBuQuinC13

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 8 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
496 repetitions
OBSERVE C13, 100.4641209 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 22 min



S11

S42

9kjs-tBuHeckH

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

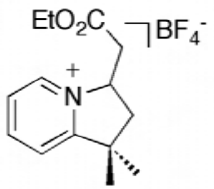
Sample directory:

FidFile: 9kjs-tBuHeckH

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 18 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389157 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



1

9kjs72-C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

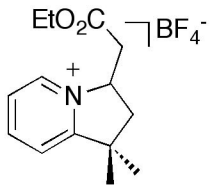
Sample directory:

FidFile: 9kjs72-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1072 repetitions
OBSERVE C13, 100.4641199 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 47 min



1

9kjs-tBuHeckF

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

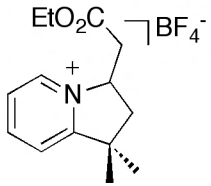
Sample directory:

FidFile: 9kjs-tBuHeckF

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 18 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



1

9kjs78-3

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

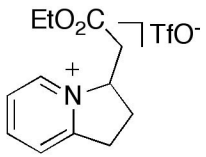
Sample directory:

FidFile: 9kjs78-3

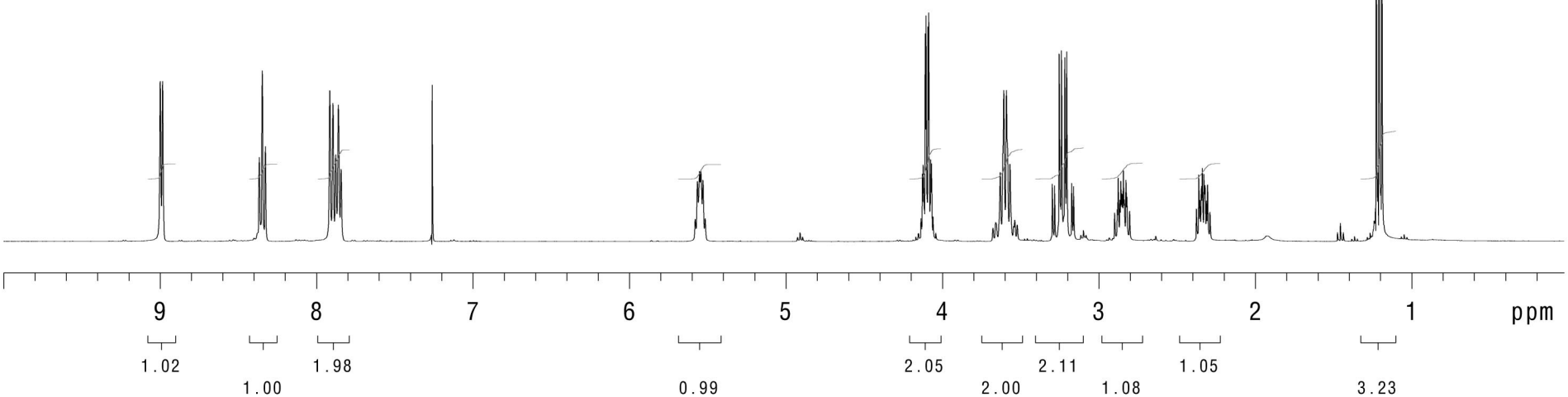
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 3 2011

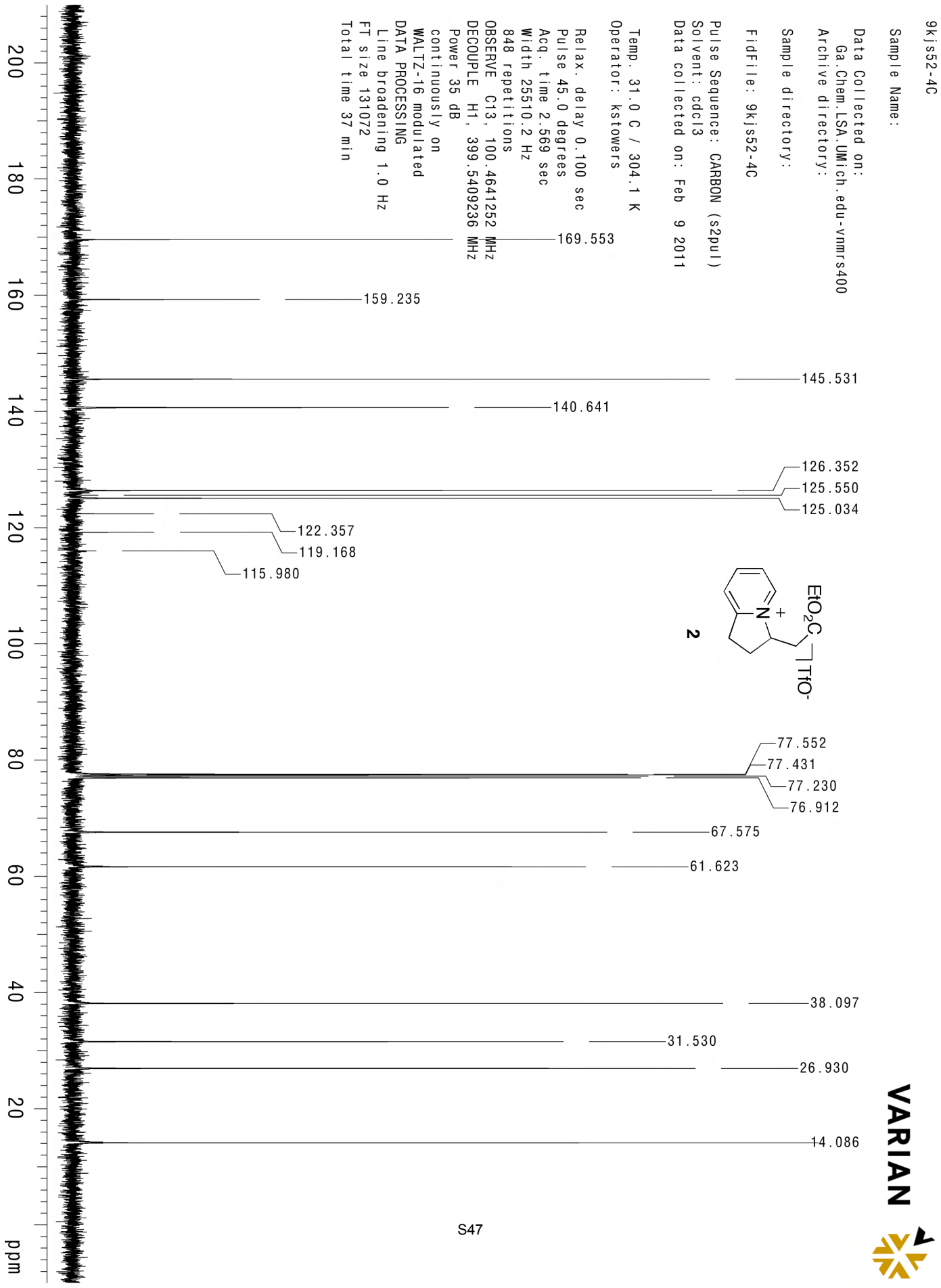
Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206315 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



2




9kjs52-4C
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: 9kjs52-4C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 9 2011
Temp. 31.0 C / 304.1 K
Operator: kstowers
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
848 repetitions
OBSERVE C13, 100.4641252 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 37 min
169.553
159.235
145.531
140.641
126.352
125.550
125.034
122.357
119.168
115.980
2
77.552
77.431
77.230
76.912
67.575
61.623
38.097
31.530
26.930
14.086
200
180
160
140
120
100
80
60
40
20
ppm
VARIAN

9kjs47-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

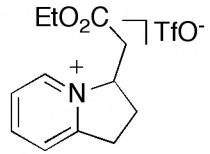
Sample directory:

FidFile: 9kjs47-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Feb 1 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



2

-78.432

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

9kjs-ipr1

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

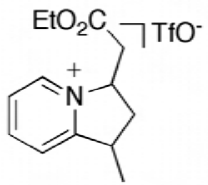
Sample directory:

FidFile: 9kjs-ipr-1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
Single scan
OBSERVE H1, 400.5206436 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 12 sec



3

9kjs-47-C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

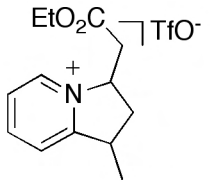
Sample directory:

FidFile: 9kjs-47-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 26 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1520 repetitions
OBSERVE C13, 100.7109804 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 7 min



3

9kjs47-2F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

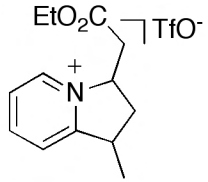
Sample directory:

FidFile: 9kjs47-2F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 29 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



3

-78.352

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

9kjs85-1

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

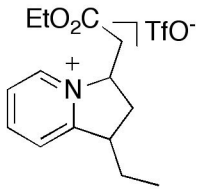
Sample directory:

FidFile: 9kjs85-1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2011

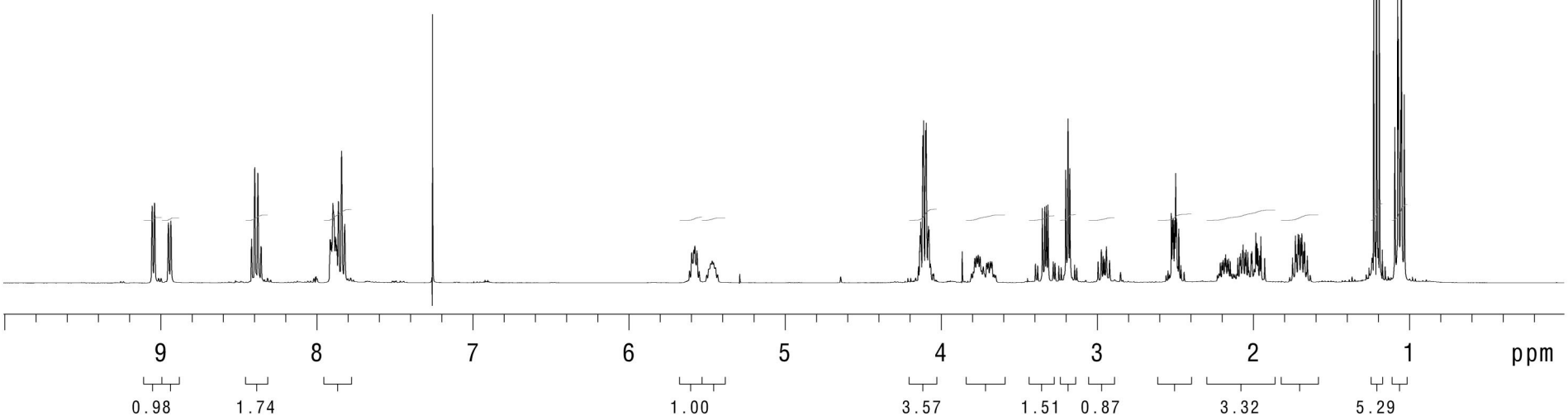
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389198 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



4

S52



9kjs25-1C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

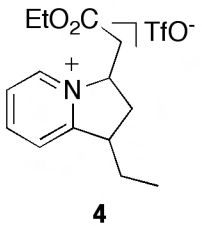
Sample directory:

FidFile: 9kjs25-1C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 10 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
736 repetitions
OBSERVE C13, 100.7109739 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 32 min



4

9kjs54-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

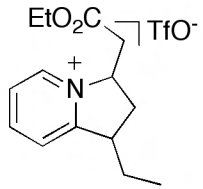
Sample directory:

FidFile: 9kjs54-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Feb 1 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



4

-78.399

S54

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

9kjs48-1

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

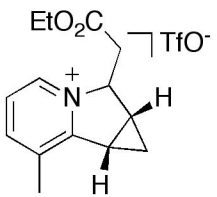
Sample directory:

FidFile: 9kjs48-1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 1 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389198 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



5

S55

9kjs34-C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

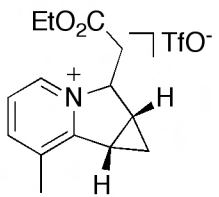
Sample directory:

FidFile: 9kjs34-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1664 repetitions
OBSERVE C13, 100.7109754 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 14 min



5

VARIAN

S56

9kjs48-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

Sample directory:

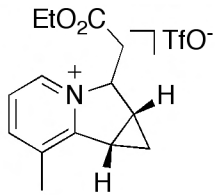
FidFile: 9kjs48-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Feb 1 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec

-78.504



5

S57

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

9kjs39-1H

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

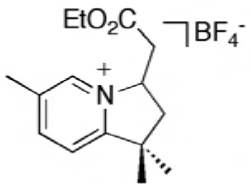
Sample directory:

FidFile: 9kjs39-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 26 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389151 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



6

8kjs105-C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

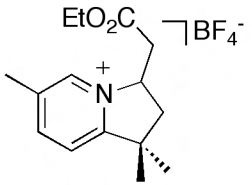
Sample directory:

FidFile: 8kjs105-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
720 repetitions
OBSERVE C13, 100.4641229 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 32 min



6

VARIAN

169.346
162.614
147.181
139.312
138.169
121.873
77.548
77.230
76.912
64.944
61.596
44.006
42.448
37.039
28.469
27.395
18.399
14.187

S59

200 180 160 140 120 100 80 60 40 20 ppm

9kjs39-1F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

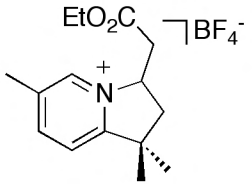
Sample directory:

FidFile: 9kjs39-1F

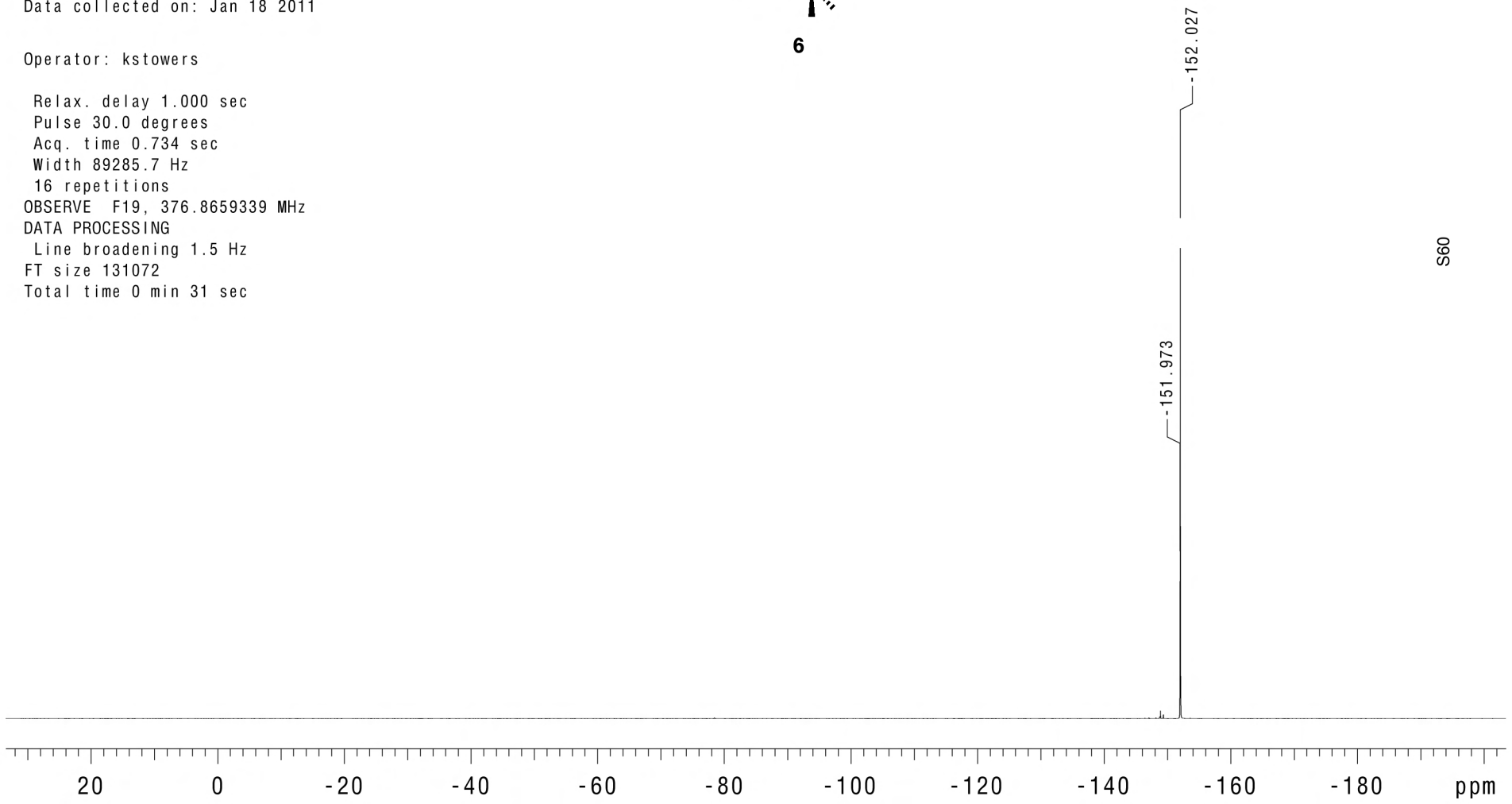
Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 18 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



6



8kjs171-HNMR1

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

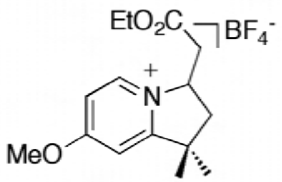
Sample directory:

FidFile: 8kjs171-HNMR1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 15 2010

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206278 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



7

8kjs171-1-CNMR

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

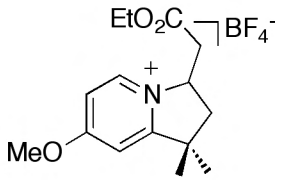
Sample directory:

FidFile: 8kjs171-1-CNMR

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 30 2010

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
384 repetitions
OBSERVE C13, 100.7109723 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 17 min



7

VARIAN

S62

9kjs171-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

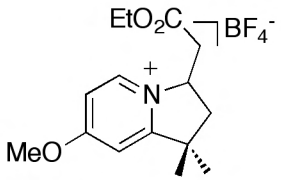
Sample directory:

FidFile: 9kjs171-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



7

9kjs180-Ctst

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

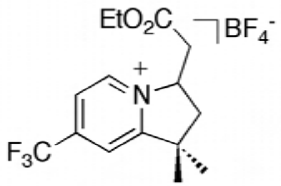
Sample directory:

FidFile: 9kjs180-Ctst

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 14 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
Single scan
OBSERVE H1, 400.5206276 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 12 sec



8

9kjs180-C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

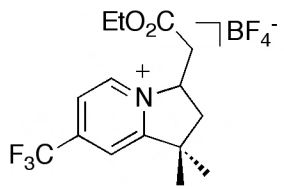
Sample directory:

FidFile: 9kjs180-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 14 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
9728 repetitions
OBSERVE C13, 100.7109778 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 14 min



8

169.601
167.692
146.600
146.241
142.550
123.371
123.340
122.494
119.761
119.089
119.054
119.019
77.551
77.431
77.230
76.913
66.261
61.801
49.877
49.664
49.452
49.239
49.023
48.810
48.598
44.991
42.251
36.527
28.020
26.996
13.943

S65

200 180 160 140 120 100 80 60 40 20 ppm

9kjs180-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

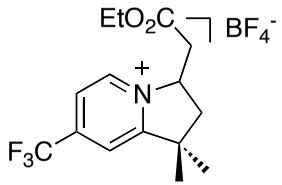
Sample directory:

FidFile: 9kjs180-F

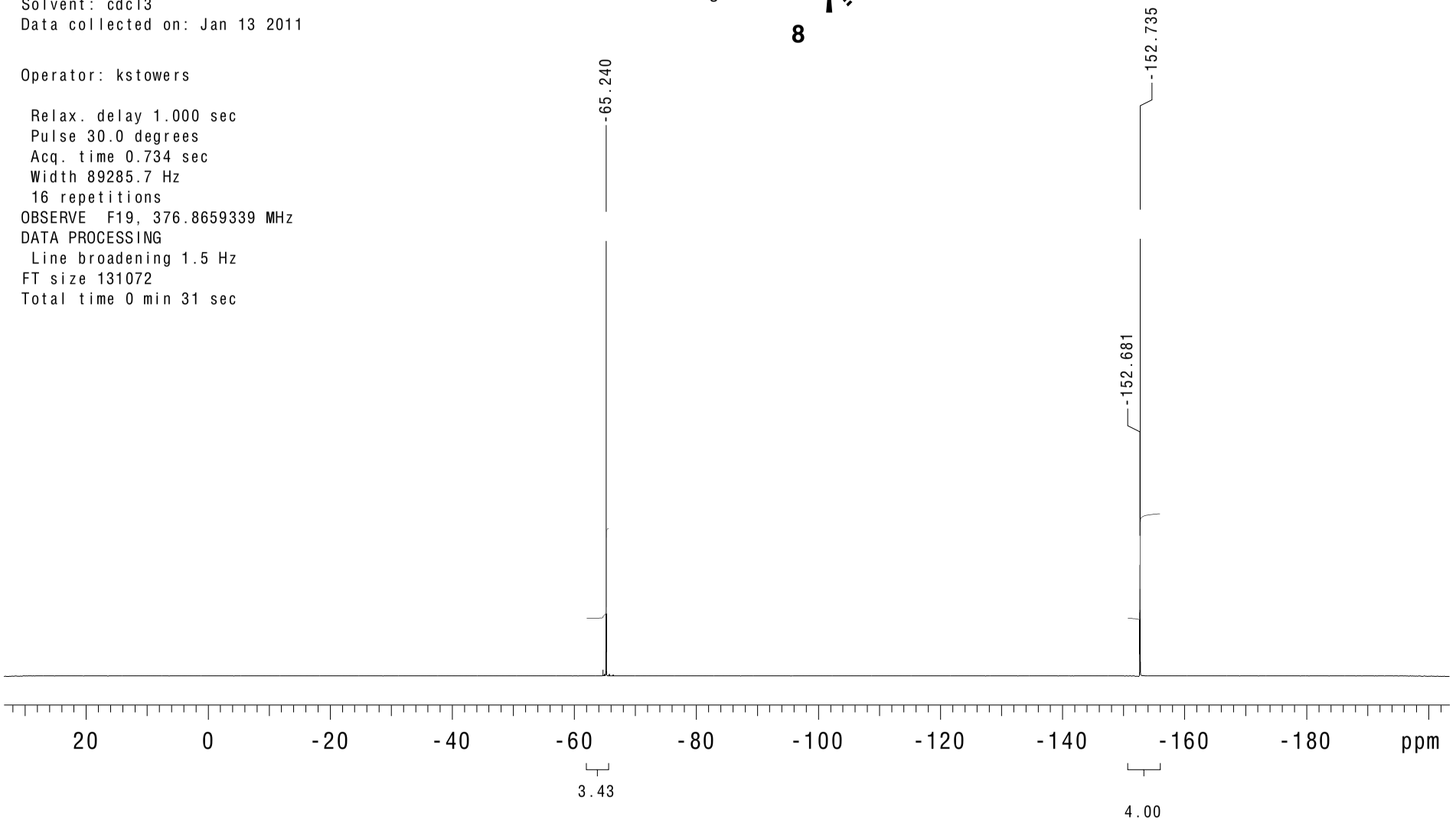
Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



8



9kjs79-1

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

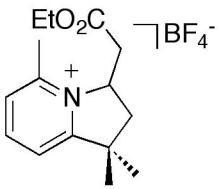
Sample directory:

FidFile: 9kjs79-1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 4 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389198 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



9

S67

8kjs79-2C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

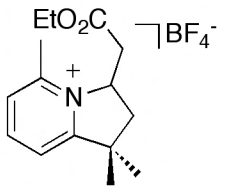
Sample directory:

FidFile: 8kjs79-2C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 18 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
848 repetitions
OBSERVE C13, 100.7109746 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 37 min



9

VARIAN

8kjs198-1F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

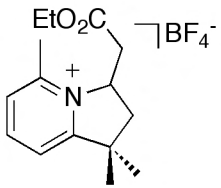
Sample directory:

FidFile: 8kjs198-1F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Dec 18 2010

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



9

9kjs74-311

Sample Name:

Data Collected on:
Te-vnmrs500
Archive directory:

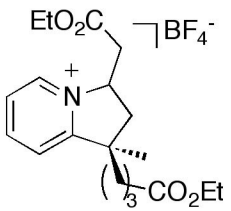
Sample directory:

FidFile: 9kjs74-311

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Mar 11 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 8012.8 Hz
16 repetitions
OBSERVE H1, 500.0931710 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



10

S70

9kjs74-C

Sample Name:

Data Collected on:
Te-vnmrs500
Archive directory:

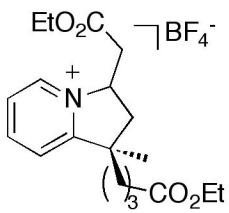
Sample directory:

FidFile: 9kjs74-C

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Mar 12 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 32051.3 Hz
3872 repetitions
OBSERVE C13, 125.7485012 MHz
DECOUPLE H1, 500.0956704 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 18 min



10

VARIAN

S71

9kjs53-1F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

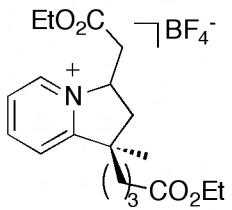
Sample directory:

FidFile: 9kjs53-1F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 31 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



10

9kjs4xquin-1

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

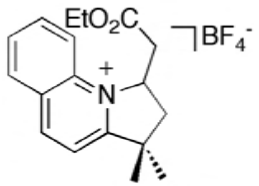
Sample directory:

FidFile: 9kjs4xquin-1

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 26 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389153 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



11

8kjs157-C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

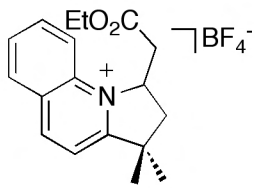
Sample directory:

FidFile: 8kjs157-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 19 2010

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1248 repetitions
OBSERVE C13, 100.7109719 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 55 min



11

VARIAN

S74

8kjs157-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

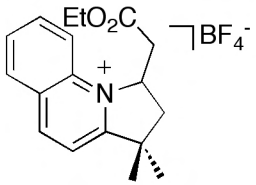
Sample directory:

FidFile: 8kjs157-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Feb 2 2011

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



11

VARIAN

-153.270

-153.216

S75

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

8kjs116-H

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

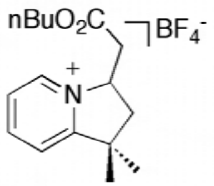
Sample directory:

FidFile: 8kjs116-H

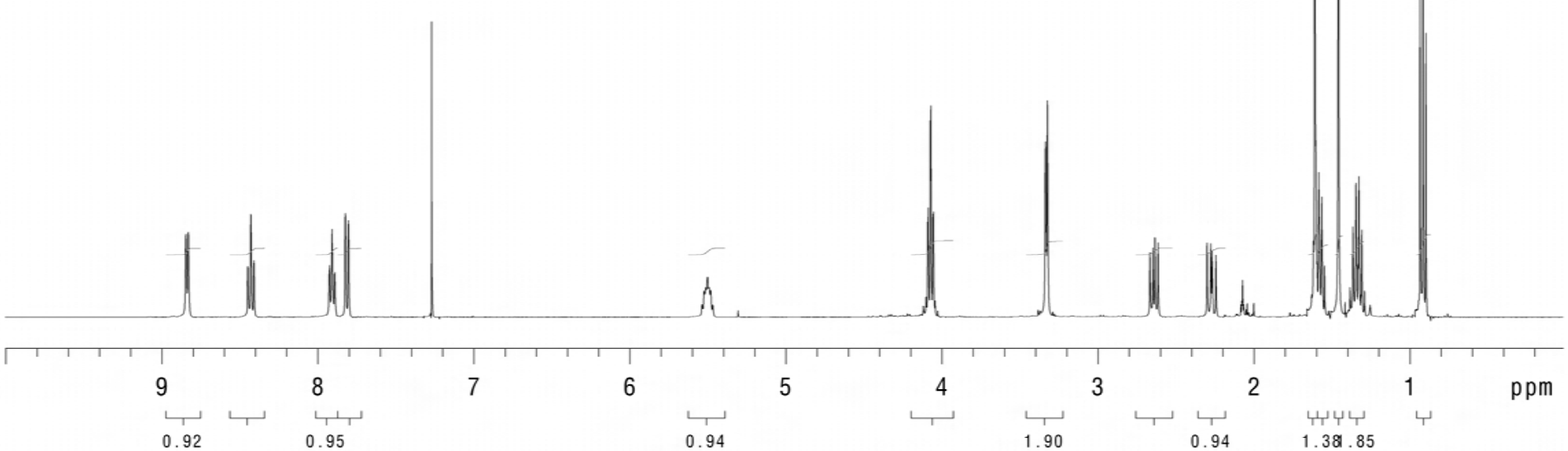
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 23 2010

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.5389153 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 40 sec



P1



9kjs77-C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

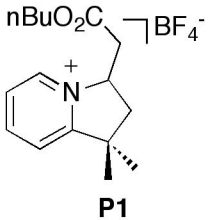
Sample directory:

FidFile: 9kjs77-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
592 repetitions
OBSERVE C13, 100.4641235 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 26 min



P1

9kjsnBu-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

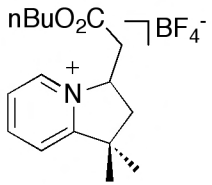
Sample directory:

FidFile: 9kjsnBu-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



P1

VARIAN

9kjsOBn-tst

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

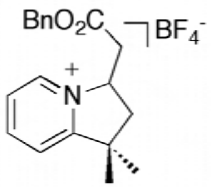
Sample directory:

FidFile: 9kjsOBn-tst

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
Single scan
OBSERVE H1, 400.5206388 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 12 sec



P2

8kjs107-C

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

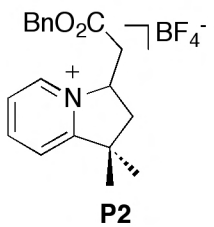
Sample directory:

FidFile: 8kjs107-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2010

Temp. 25.0 C / 298.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
912 repetitions
OBSERVE C13, 100.7109708 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 40 min



P2

VARIAN

S80

169.420
165.342
146.256
140.354
135.194
128.891
128.829
128.809
126.742
122.374
77.547
77.230
76.913
67.478
65.117
44.389
42.286
36.956
28.418
27.235

200 180 160 140 120 100 80 60 40 20 ppm

9kjsOBn-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

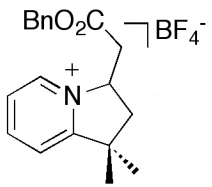
Sample directory:

FidFile: 9kjsOBn-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



P2

9kjs75-3

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

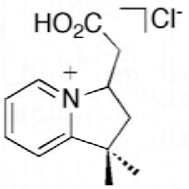
Sample directory:

FidFile: 9kjs75-3

Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Feb 16 2011

Temp. 30.0 C / 303.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5222121 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



P3

9kjs70-2C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

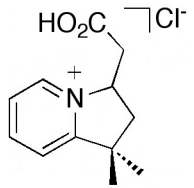
Sample directory:

FidFile: 9kjs70-2C

Pulse Sequence: CARBON (s2pul)
Solvent: cd3od
Data collected on: Feb 12 2011

Temp. 31.0 C / 304.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1000 repetitions
OBSERVE C13, 100.4645386 MHz
DECOUPLE H1, 399.5424978 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 44 min



P3

VARIAN

S83

171.093
165.758
146.285
139.694
126.160
122.817
65.283
48.309
48.096
47.883
47.670
47.457
47.240
47.026
43.993
42.137
36.984
27.014
26.220

180 160 140 120 100 80 60 40 20 ppm

9kjs59-4

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

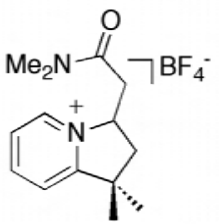
Sample directory:

FidFile: 9kjs59-4

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 12 2011

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389249 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



P4

9kjs59-4C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

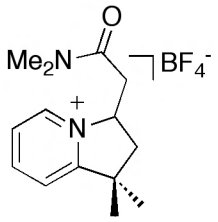
Sample directory:

FidFile: 9kjs59-4C

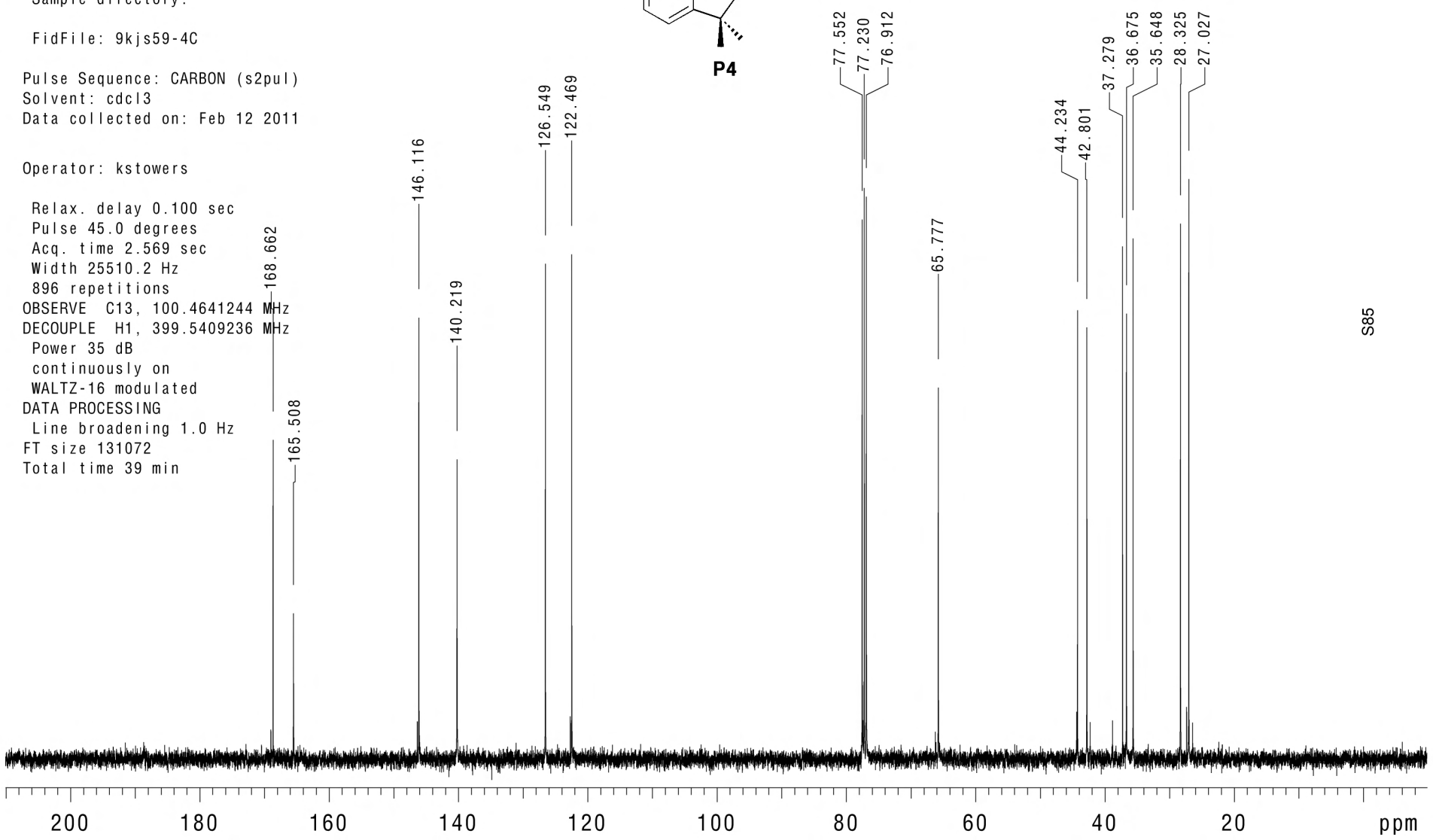
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 12 2011

Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
896 repetitions
OBSERVE C13, 100.4641244 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 39 min



P4



9kjs27-1F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

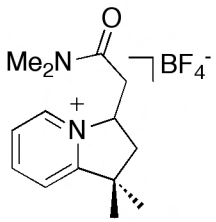
Sample directory:

FidFile: 9kjs27-1F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 31 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



P4

VARIAN

-152.128

-152.074

S86

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

9kjs60-2

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

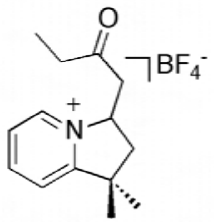
Sample directory:

FidFile: 9kjs60-2

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 9 2011

Temp. 31.0 C / 304.1 K
Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389151 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



P5

VARIAN

S87

9kjs60-2C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

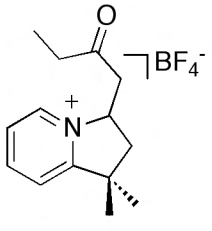
Sample directory:

FidFile: 9kjs60-2C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 9 2011

Temp. 31.0 C / 304.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.568 sec
Width 25510.2 Hz
832 repetitions
OBSERVE C13, 100.4641217 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 37 min



P5

VARIAN

9kjsCOEt-F

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

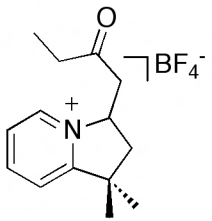
Sample directory:

FidFile: 9kjsCOEt-F

Pulse Sequence: FLUORINE (s2pul)
Solvent: cdcl3
Data collected on: Jan 28 2011

Operator: kstowers

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
16 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 0 min 31 sec



P5

VARIAN

-152.168
-152.117

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

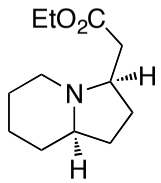
Sample directory:

FidFile: KCF-3-123-1H

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Dec 18 2010

Operator: kfortner

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
32 repetitions
OBSERVE H1, 400.5206316 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 2 min 16 sec



12

VARIAN

S90

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

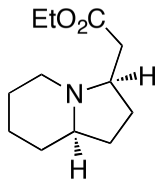
Sample directory:

FidFile: KCF-3-123-13C

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Dec 18 2010

Operator: kfortner

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
128 repetitions
OBSERVE C13, 100.7109736 MHz
DECOUPLE H1, 400.5226417 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 min 43 sec



12

VARIAN

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

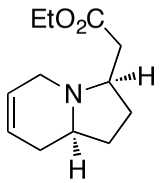
Sample directory:

FidFile: KCF-3-125-1H

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Dec 19 2010

Operator: kfortner

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.5206318 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



13A

S92

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

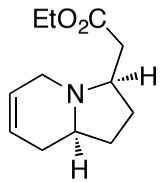
Sample directory:

FidFile: KCF-3-125-13C

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Dec 21 2010

Operator: kfortner

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
2176 repetitions
OBSERVE C13, 100.7109708 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 37 min



13A

STANDARD 1H OBSERVE

Selective band center: 6.08 (ppm); width: 50.7 (Hz)

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

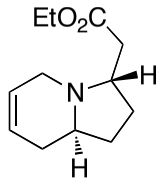
Sample directory:

FidFile: KCF-3-137-1H

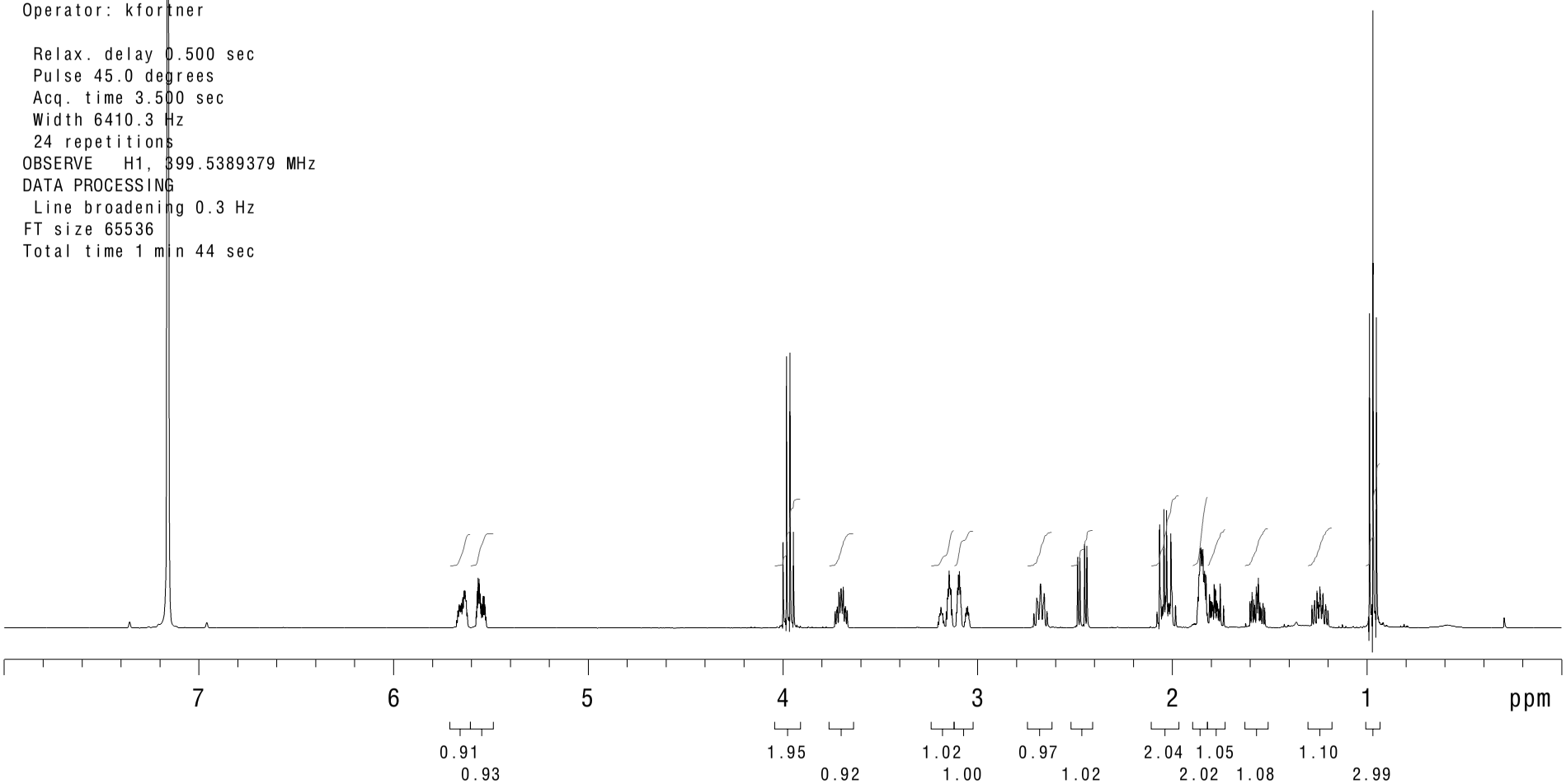
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jan 20 2011

Temp. 23.0 C / 296.1 K
Operator: kfortner

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
24 repetitions
OBSERVE H1, 399.5389379 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 44 sec



13B



Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

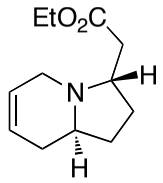
Sample directory:

FidFile: KCF-3-137-13C

Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Jan 20 2011

Operator: kfortner

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
2336 repetitions
OBSERVE C13, 100.7109704 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 44 min



13B

S95

STANDARD 1H OBSERVE

Selective band center: 6.08 (ppm); width: 50.7 (Hz)

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

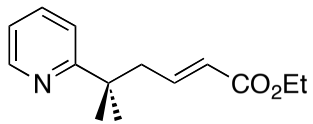
Sample directory:

FidFile: KCF-3-147-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 6 2011

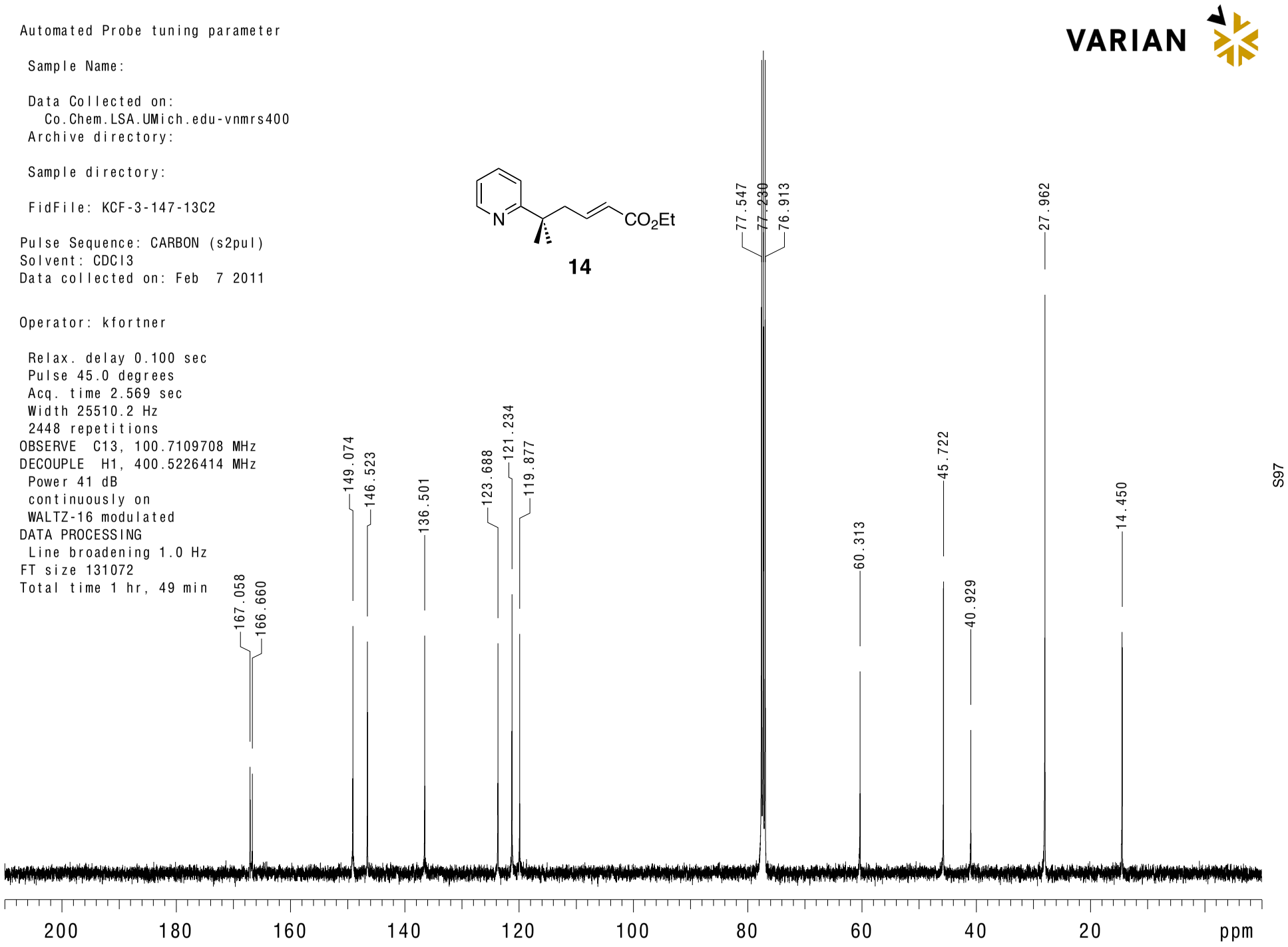
Operator: kfortner

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
48 repetitions
OBSERVE H1, 399.5389189 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 3 min 20 sec



14

S96


Automated Probe tuning parameter
Sample Name:
Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: KCF-3-147-13C2
Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Feb 7 2011
Operator: kfortner
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
2448 repetitions
OBSERVE C13, 100.7109708 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min
N
CO2Et
14
167.058
166.660
149.074
146.523
136.501
123.688
121.234
119.877
77.547
77.230
76.913
60.313
45.722
40.929
27.962
14.450
S97
200
180
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Selective band center: 6.08 (ppm); width: 50.7 (Hz)

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

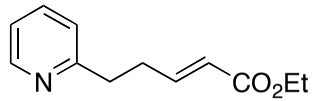
Sample directory:

FidFile: KCF-3-145-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 6 2011

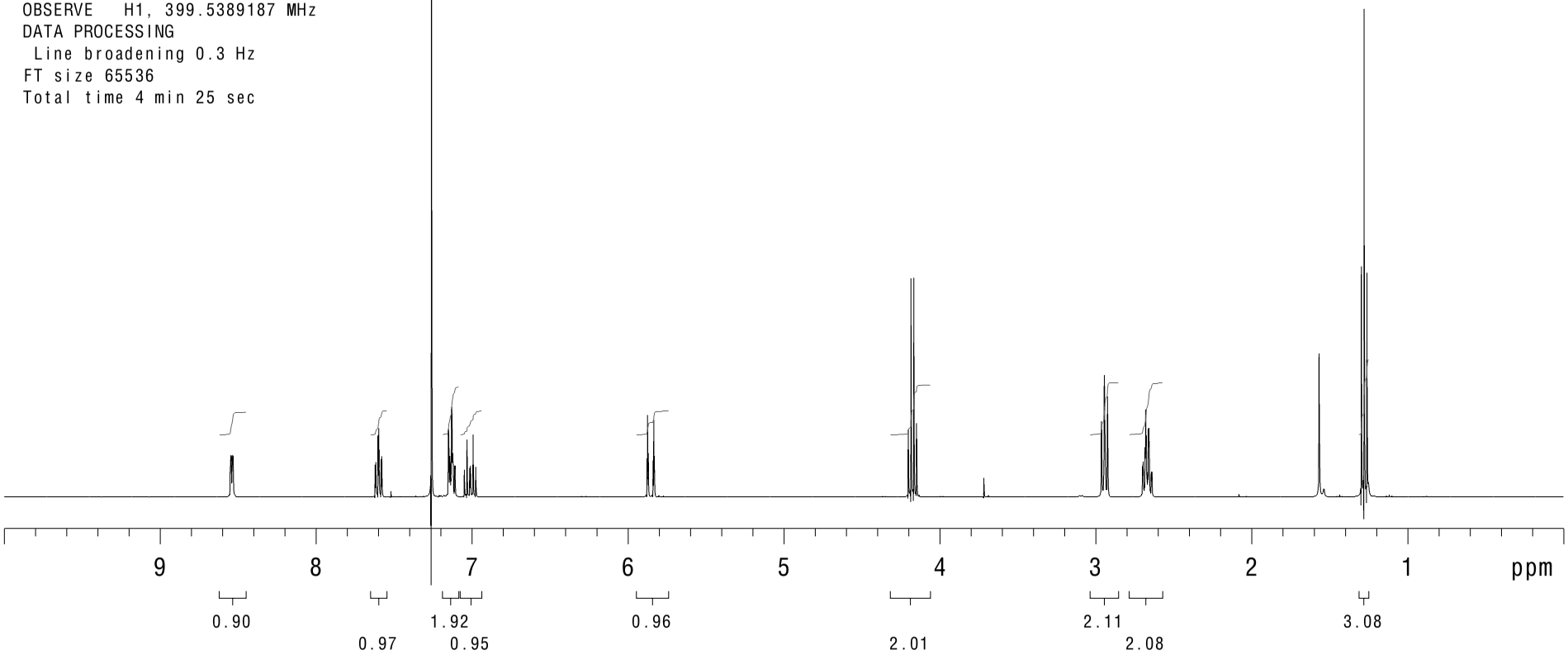
Operator: kfortner

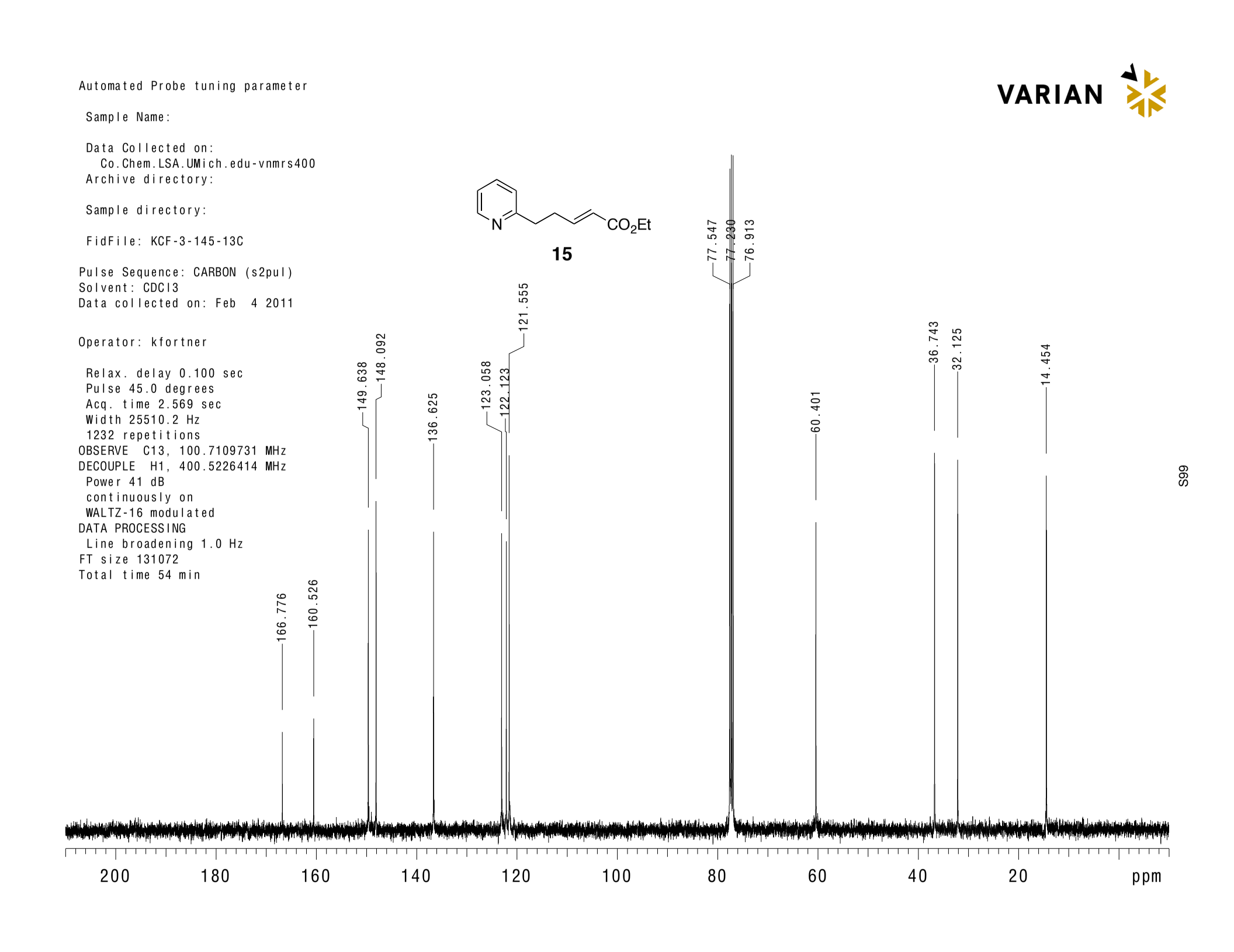
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
64 repetitions
OBSERVE H1, 399.5389187 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 4 min 25 sec



15

S98




Automated Probe tuning parameter
Sample Name:
Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: KCF-3-145-13C
Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Feb 4 2011
Operator: kfortner
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1232 repetitions
OBSERVE C13, 100.7109731 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 54 min
N
CO2Et
15
166.776
160.526
149.638
148.092
136.625
123.058
122.123
121.555
77.547
77.230
76.913
60.401
36.743
32.125
14.454
S99
200
180
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Selective band center: 6.08 (ppm); width: 50.7 (Hz)

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

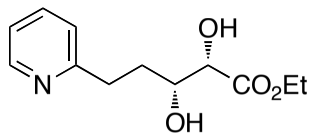
Sample directory:

FidFile: KCF-3-117-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 11 2010

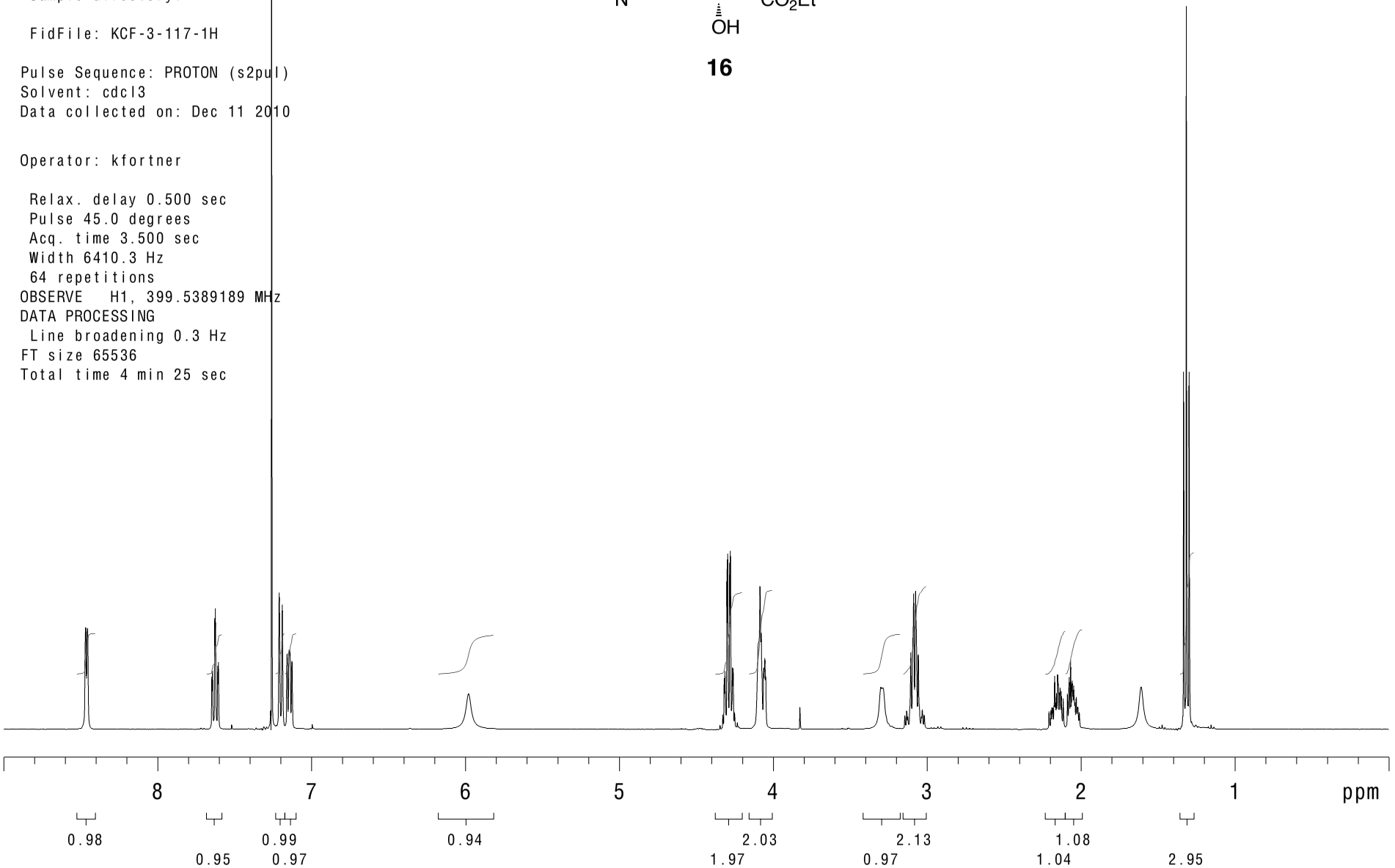
Operator: kfortner

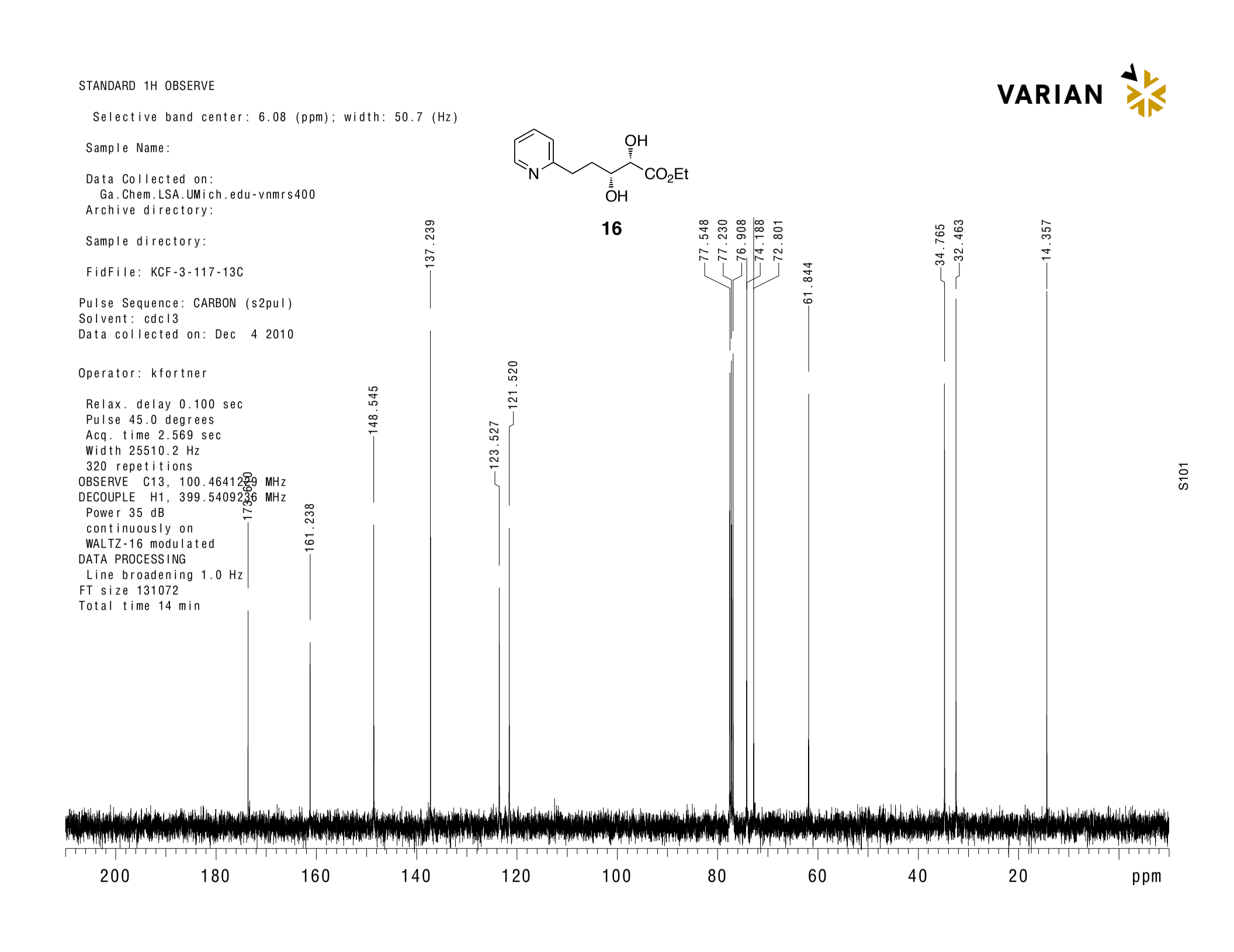
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
64 repetitions
OBSERVE H1, 399.5389189 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 4 min 25 sec



16

S100




STANDARD 1H OBSERVE
Selective band center: 6.08 (ppm); width: 50.7 (Hz)
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: KCF-3-117-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 4 2010
Operator: kfortner
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
320 repetitions
OBSERVE C13, 100.4641239 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 14 min
OH
N
CO2Et
OH
16
173.660
161.238
148.545
137.239
123.527
121.520
77.548
77.230
76.908
74.188
72.801
61.844
34.765
32.463
14.357
S101
200
180
160
140
120
100
80
60
40
20
ppm

STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:

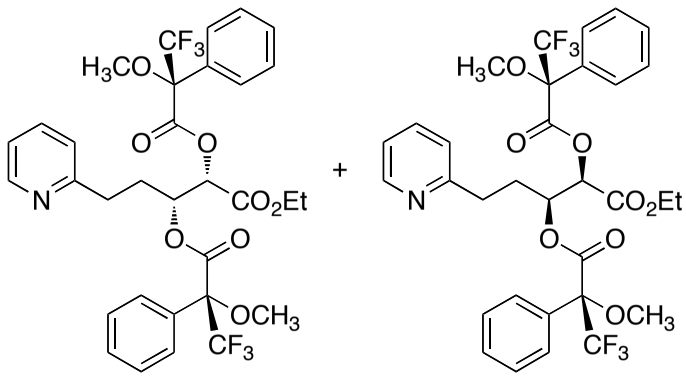
Sample directory:

FidFile: KCF-3-129-1H

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Jan 7 2011

Operator: kfortner

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 7998.4 Hz
64 repetitions
OBSERVE H1, 499.9042609 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 4 min 25 sec



+

Bis-(*R*)-MTPA esters of *rac*-16

S102

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

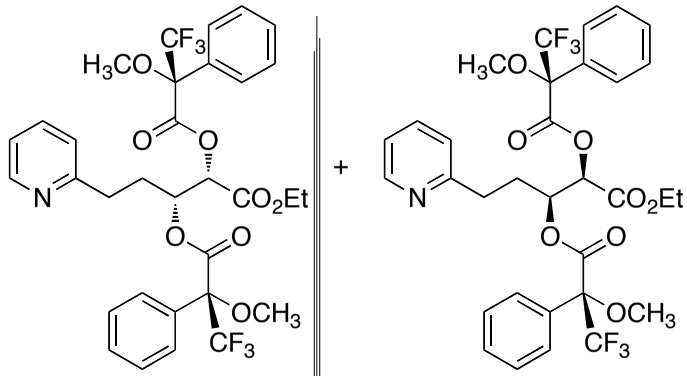
Sample directory:

FidFile: KCF-3-129-13C

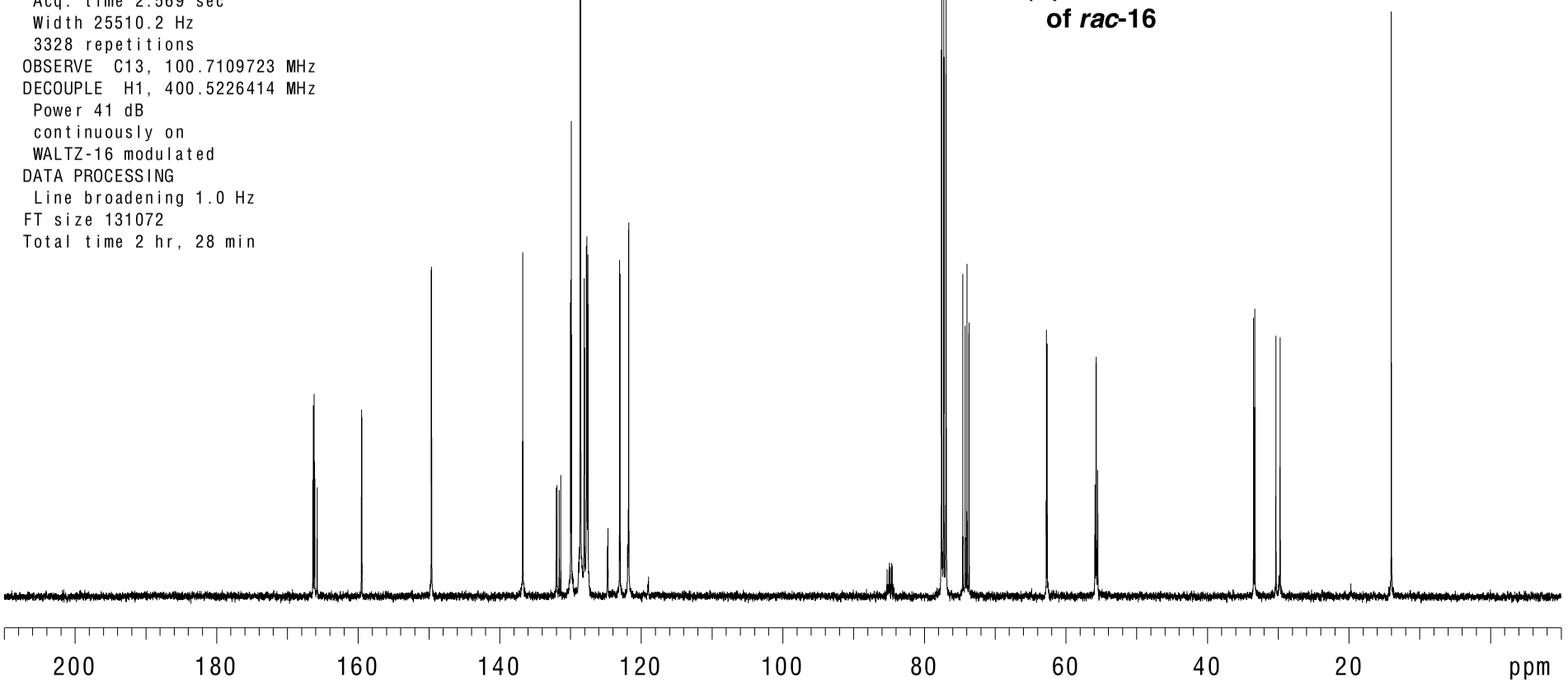
Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Jan 2 2011

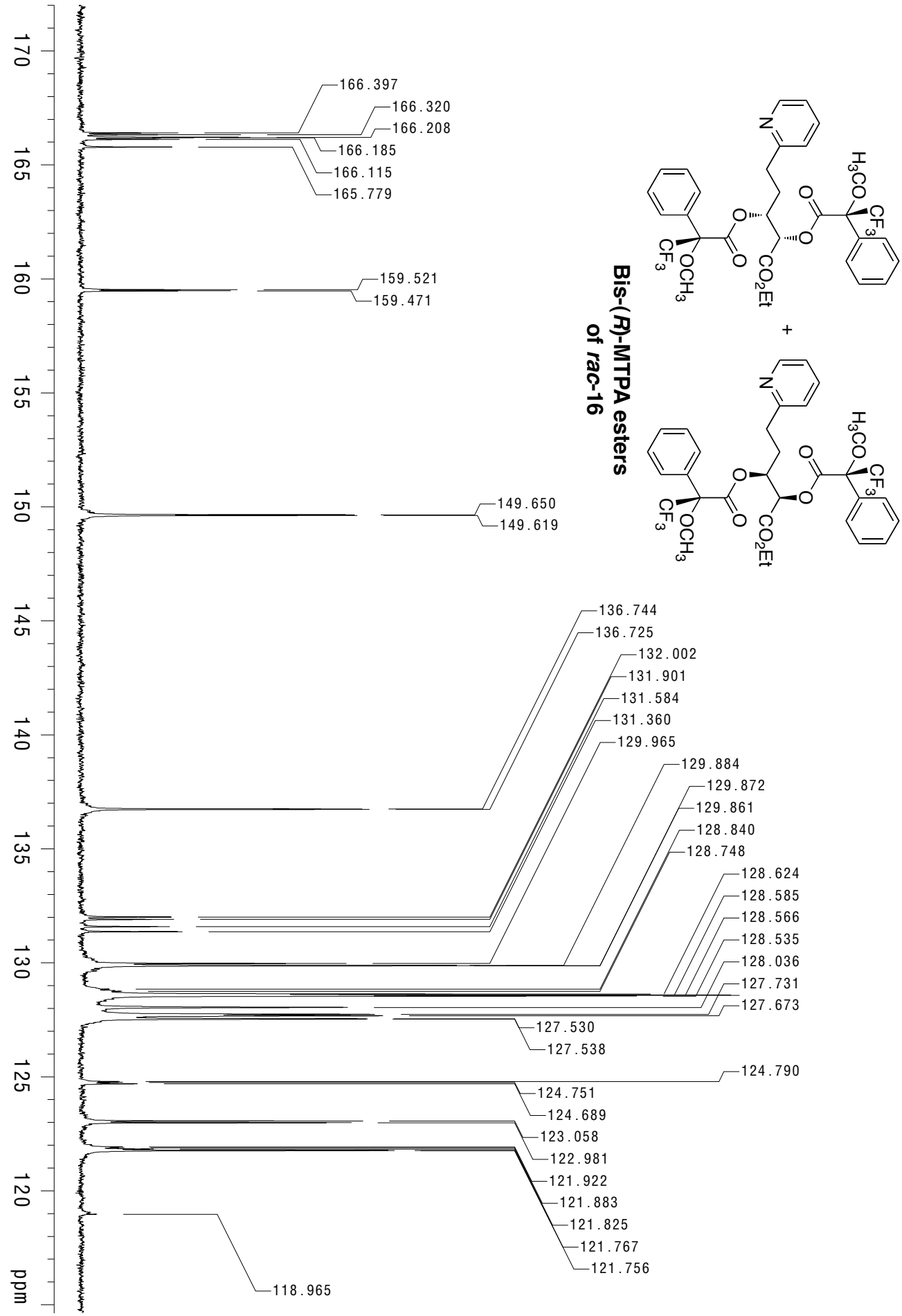
Operator: kfortner

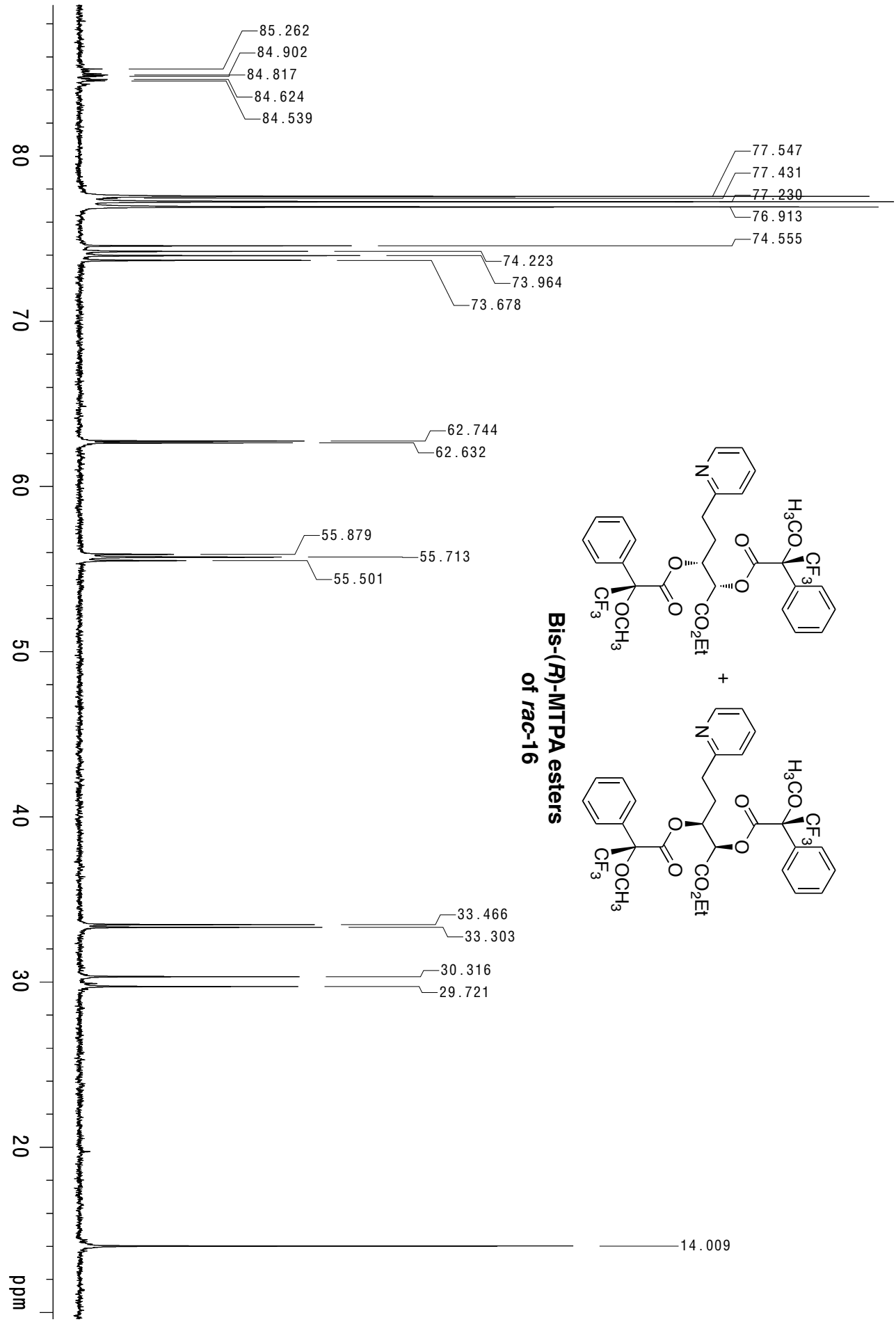
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
3328 repetitions
OBSERVE C13, 100.7109723 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 28 min



Bis-(*R*)-MTPA esters of *rac*-16




Bis-(R)-MTPA esters
of rac-16
166.397
166.320
166.208
166.185
166.115
165.779
159.521
159.471
149.650
149.619
136.744
136.725
132.002
131.901
131.584
131.360
129.965
129.884
129.872
129.861
128.840
128.748
128.624
128.585
128.566
128.535
128.036
127.731
127.673
127.530
127.538
124.790
124.751
124.689
123.058
122.981
121.922
121.883
121.825
121.767
121.756
118.965
ppm


85.262
84.902
84.817
84.624
84.539
77.547
77.431
77.230
76.913
74.555
74.223
73.964
73.678
62.744
62.632
55.879
55.713
55.501
33.466
33.303
30.316
29.721
14.009
80
70
60
50
40
30
20
ppm
Bis-(R)-MTPA esters
of rac-16
+

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

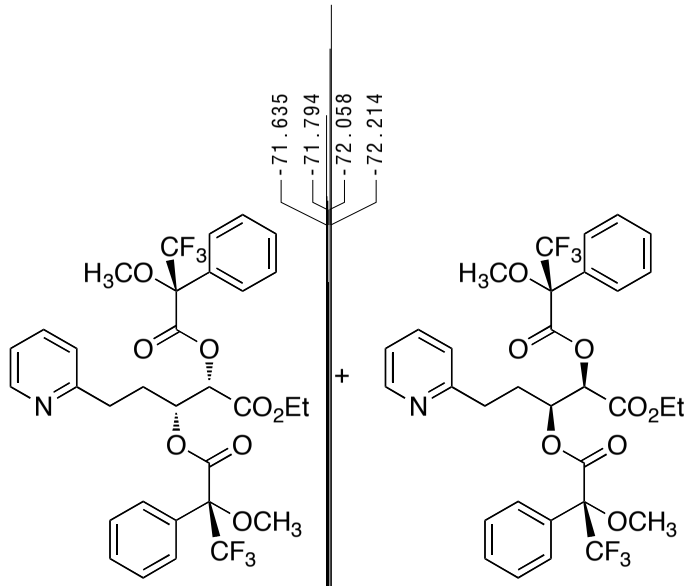
Sample directory:

FidFile: KCF-3-129-19F

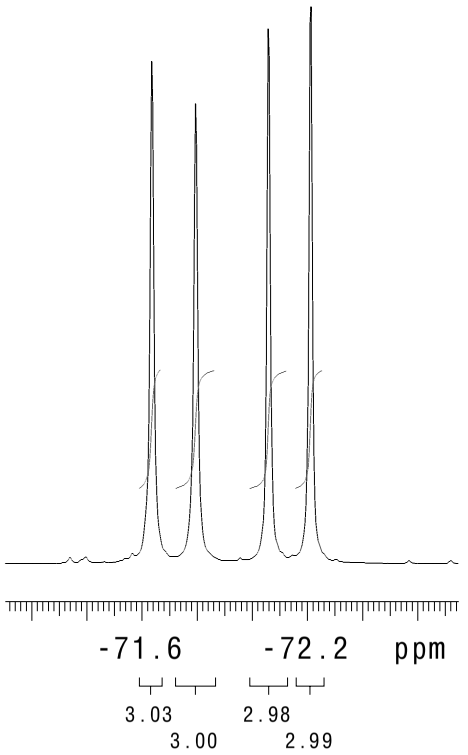
Pulse Sequence: FLUORINE (s2pul)
Solvent: CDCl3
Data collected on: Dec 21 2010

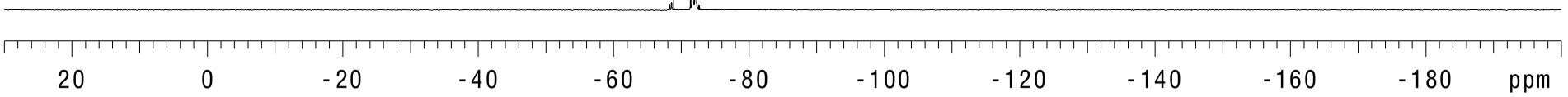
Operator: kfortner

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
64 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 min 55 sec



Bis-(*R*)-MTPA esters of *rac*-16





STANDARD PROTON PARAMETERS

Sample Name:

Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:

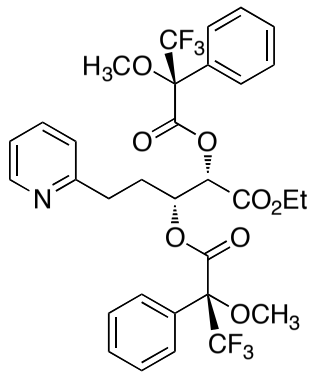
Sample directory:

FidFile: KCF-3-131-1H

Pulse Sequence: PROTON (s2pul)
Solvent: CDCl3
Data collected on: Jan 6 2011

Operator: kfortner

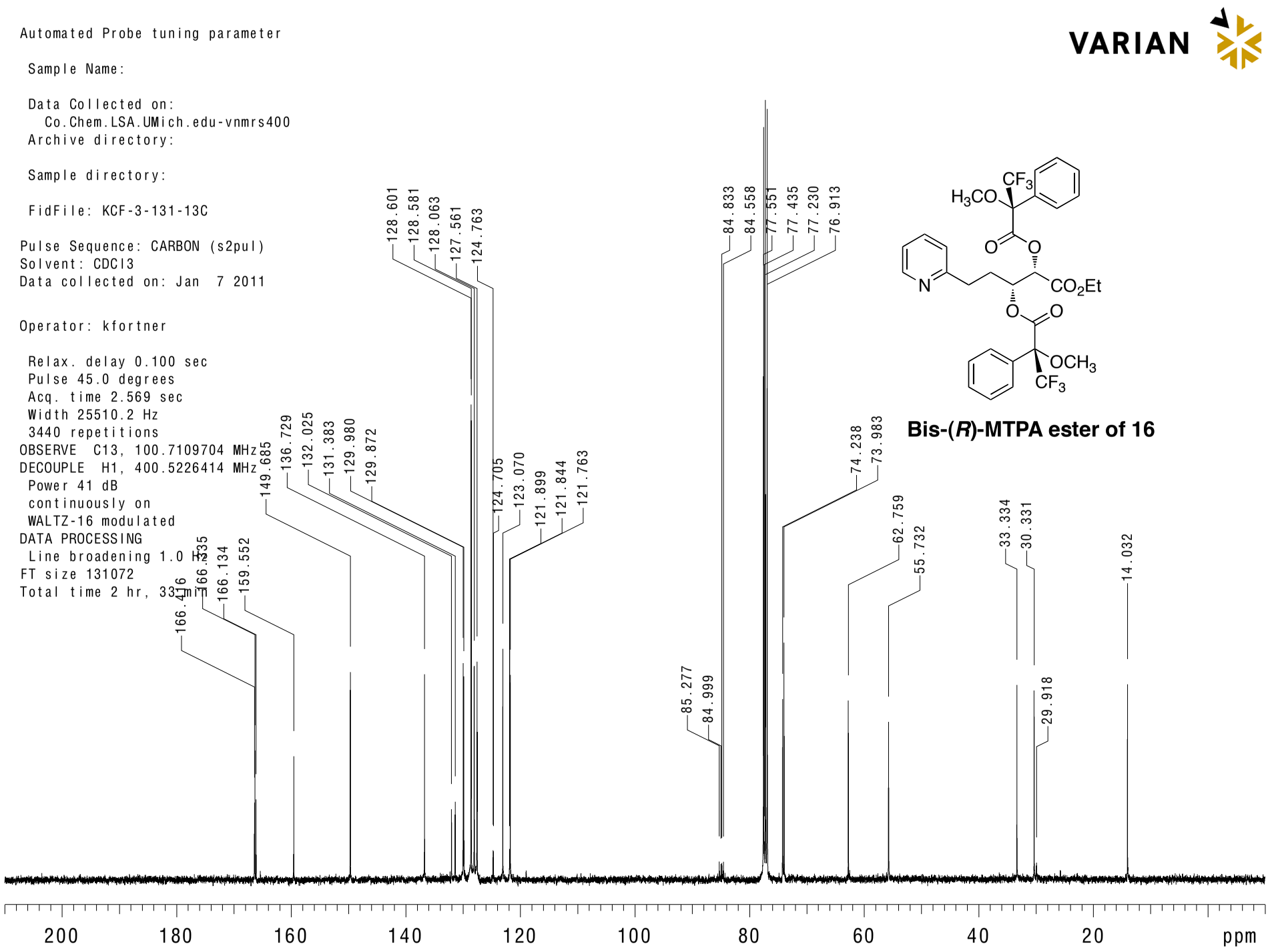
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 7998.4 Hz
112 repetitions
OBSERVE H1, 499.9042611 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 7 min 37 sec



Bis-(*R*)-MTPA ester of 16

8 7 6 5 4 3 2 1 ppm

0.90 1.99 0.95 1.00 1.98 2.96 2.03
2.93 6.06 0.96 0.99 3.00 2.10 3.15


Automated Probe tuning parameter
Sample Name:
Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: KCF-3-131-13C
Pulse Sequence: CARBON (s2pul)
Solvent: CDCl3
Data collected on: Jan 7 2011
Operator: kfortner
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
3440 repetitions
OBSERVE C13, 100.7109704 MHz
DECOUPLE H1, 400.5226414 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min
VARIAN
Bis-(R)-MTPA ester of 16
S108
166.416
166.735
166.134
159.552
149.685
136.729
132.025
131.383
129.980
129.872
128.601
128.581
128.063
127.561
124.763
124.705
123.070
121.899
121.844
121.763
85.277
84.999
84.833
84.558
77.551
77.435
77.230
76.913
74.238
73.983
62.759
55.732
33.334
30.331
29.918
14.032
200
180
160
140
120
100
80
60
40
20
ppm

Automated Probe tuning parameter

Sample Name:

Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

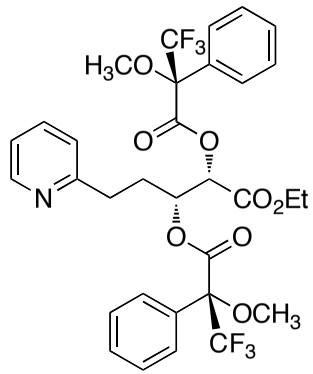
Sample directory:

FidFile: KCF-3-131-19F

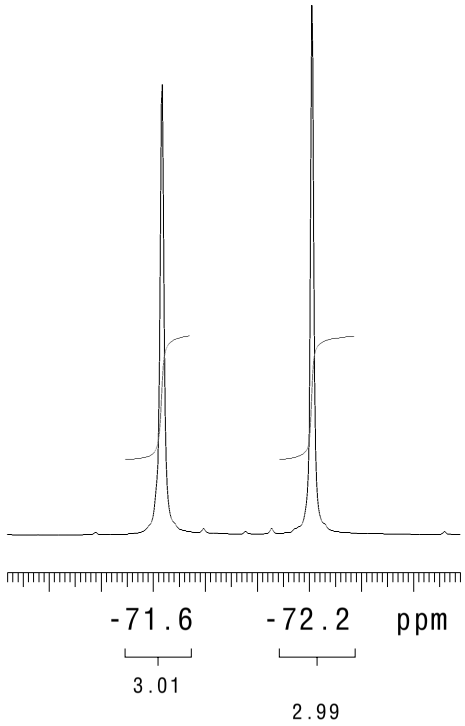
Pulse Sequence: FLUORINE (s2pul)
Solvent: CDCl3
Data collected on: Jan 7 2011

Operator: kfortner

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.734 sec
Width 89285.7 Hz
272 repetitions
OBSERVE F19, 376.8659339 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 7 min 58 sec



Bis-(*R*)-MTPA ester of 16



7kjs22-2

Sample Name:

Data Collected on:
Zr.Chem.LSA.UMich.edu-inova400
Archive directory:

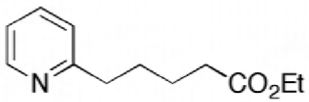
Sample directory:

FidFile: 7kjs22-2

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 11 2010

Operator: kstowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6399.5 Hz
8 repetitions
OBSERVE H1, 399.9649442 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min 40 sec



17

7kjs22-C

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

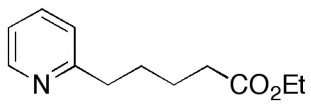
Sample directory:

FidFile: 7kjs22-C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 11 2010

Temp. 23.0 C / 296.1 K
Operator: kstowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
656 repetitions
OBSERVE C13, 100.4641198 MHz
DECOUPLE H1, 399.5409236 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 29 min



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STANDARD 1H OBSERVE

Selective band center: 6.08 (ppm); width: 50.7 (Hz)

Sample Name:

Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:

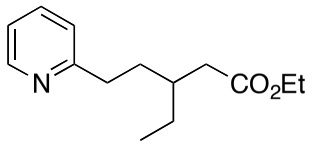
Sample directory:

FidFile: KCF-3-113-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 4 2010

Operator: kfortner

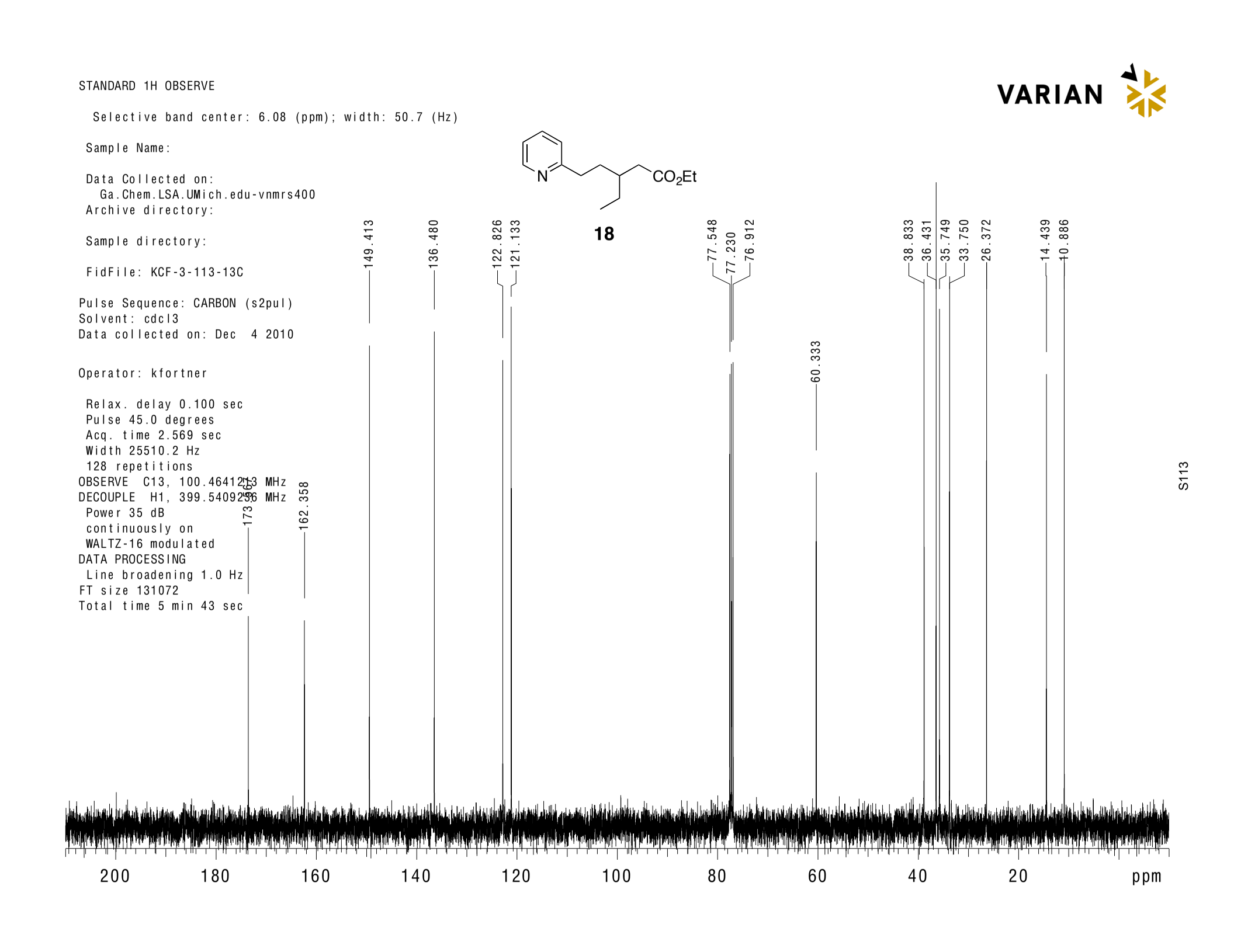
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.5389189 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min 12 sec



18

VARIAN

S112


STANDARD 1H OBSERVE
Selective band center: 6.08 (ppm); width: 50.7 (Hz)
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: KCF-3-113-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 4 2010
Operator: kfortner
Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
128 repetitions
OBSERVE C13, 100.4641273 MHz
DECOUPLE H1, 399.5409256 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 min 43 sec
N
CO2Et
18
173.167
162.358
149.413
136.480
122.826
121.133
77.548
77.230
76.912
60.333
38.833
36.431
35.749
33.750
26.372
14.439
10.886
S113
200
180
160
140
120
100
80
60
40
20
ppm