Catalytic Asymmetric Reductive Acyl Cross-Coupling: Synthesis of Enantioenriched Acyclic α,α-Disubstituted Ketones

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Supporting Information 1 (Experimental Procedures):

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1. Materials and Methods

Unless otherwise stated, reactions were performed under a nitrogen atmosphere using freshly dried solvents. Tetrahydrofuran (THF), methylene chloride (CH_2Cl_2), and acetonitrile (MeCN), were dried by passing through activated alumina columns. Anhydrous dimethylacetamide (DMA) was purchased from Aldrich and stored under inert atmosphere. Manganese powder (-325 mesh, 99.3%) was purchased from Alfa Aesar. Unless otherwise stated, chemicals and reagents were used as received. All reactions were monitored by thin-layer chromatography using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm) and were visualized by UV, *p*-anisaldehyde, or $KMnO_4$ staining. Flash column chromatography was performed as described by Still et al.[1] using silica gel (partical size 0.032-0.063) purchased from Silicycle. Optical rotations were measured on a Jasco P-2000 polarimeter using a 100 mm path-length cell at 589 nm. ^1H and ^{13}C NMR spectra were recorded on a Varian 400 MR (at 400 MHz and 101 MHz, respectively) or a Varian Inova 500 (at 500 MHz and 126 MHz, respectively), and are reported relative to internal $CHCl_3$ (^1H, δ = 7.26) or acetone (^1H, δ = 2.05), and $CDCl_3$ (^{13}C, δ = 77.0) or acetone (^{13}C, δ = 29.8). Data for ^1H NMR spectra are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Multiplicity and qualifier abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, app = apparent. IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in frequency of absorption (cm^{-1}). HRMS were acquired using an Agilent 6200 Series TOF with an Agilent G1978A Multimode source in electrospray ionization (ESI) or mixed (MM) ionization mode, or obtained from the Caltech Mass Spectral Facility in fast-atom bombardment mode (FAB). Analytical SFC was performed with a Mettler SFC supercritical CO_2 analytical chromatography system with Chiralcel AD-H, OD-H, AS-H, OB-H, and OJ-H columns (4.6 mm x 25 cm) with visualization at 210 nm. Analytical achiral GC was performed with an Agilent 6850 GC utilizing an Agilent DB-WAX (30.0 m x 0.25 mm) column (1.0 mL/min He carrier gas flow).

Abbreviations used: DMA – dimethylacetamide; dme – dimethoxyethane; IPA – isopropanol; MeCN – acetonitrile; THF – tetrahydrofuran; 2,6-DMBA – 2,6-dimethylbenzoic acid; COD – cyclooctadiene; ee – enantiomeric excess; dr – diastereomeric ratio; TDAE – tetrakis(dimethylamino)ethylene

2. Optimization of Reaction Parameters

A. General Procedure 1 (Table 1)

On a bench-top, to a 1/2 dram vial was added the appropriate ligand (0.044 mmol, 22 mol %), carboxylic acid (0.15 mmol, 0.75 equiv), 3 Å mol sieves (30 mg/0.2 mmol benzyl chloride), reductant (0.6 mmol, 3 equiv), and nickel source (0.02 mmol, 10 mol %). Under an inert atmosphere in a glovebox, the vial was charged with the appropriate solvent (0.53 mL, 0.375 M) followed by benzyl chloride (**2**, 0.2 mmol, 1 equiv), acid chloride (**1**, 0.24 mmol, 1.2 equiv), and dodecane (internal standard). The mixture was stirred at 240 rpm, ensuring that the

reductant was uniformly suspended. Stirring continued at 20 °C under inert atmosphere for 24 h. The black slurry was transferred to a separatory funnel using 1 M HCl (5 mL) and diethyl ether (10 mL). The mixture was diluted with H_2O (10 mL) and the aqueous and organic layers were separated. The aqueous layer was extracted with diethyl ether (2 X 10 mL) and the combined organic layers were washed with brine (1 X 15 mL) and dried ($MgSO_4$), filtered, and concentrated. The crude residue was analyzed by GC.

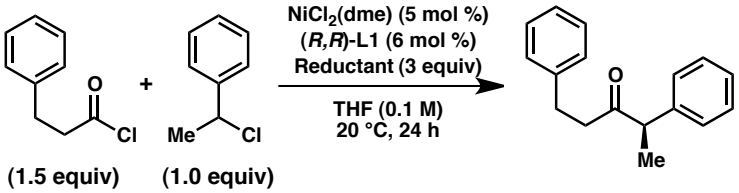
The following response factors relative to (1-chloroethyl)benzene were measured and calculated based on three runs of varied concentration:

Ketone **3a** (Product): Response Factor = 0.37

Dibenzyl **4** (Homocoupling): Response Factor = 0.73

Dodecane was used as an internal standard. GC samples were analyzed by flame ionization detection and yields calculated based on the above factors.

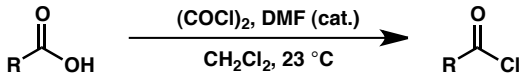
B. Alternative Reductants



Entry	Reductant	Conversion	Yield (%)	ee (%)
1	Mg^0	Full	Trace	20
2	Co^0	0	0	--
3	Fe^0	0	0	--
4	$CrCl_2$	Full	0	--
5	$CoCp_2$	0	0	--
6	TDAE	0	0	--

3. Substrate Preparation.

A. General Procedure 2: Acid Chloride Synthesis

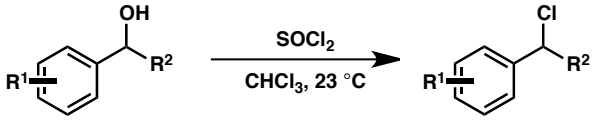


A flask was charged with the appropriate carboxylic acid (1.0 equiv) and CH_2Cl_2 (0.5 M). Two drops of DMF and oxalyl chloride (1.2 equiv) were added dropwise. The solution was stirred at 23 °C for 3 h and then concentrated. The crude acid chloride was used without any further purification.

B. 3-(4-methoxyphenyl)propanoic 2,6-dimethylbenzoic anhydride (1b)

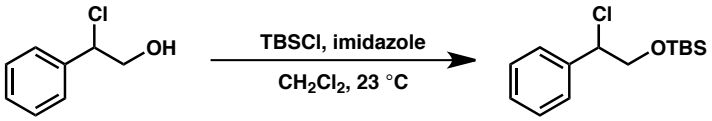
A flame-dried flask was charged with 2,6-dimethylbenzoic acid (1.0 mmol, 1 equiv) and CH_2Cl_2 (0.33 M). To the solution was added NaH (60% dispersion in oil, 1.05 mmol, 1.05 equiv) and the reaction was allowed to stir for 3 h. 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 1.0 mmol, 1 equiv) was added dropwise to the reaction mixture and the reaction was stirred overnight. The crude mixture was filtered through a small plug of celite and concentrated to afford a light yellow oil (291.1 mg, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 (t, *J* = 7.7 Hz, 1H), 7.13 (d, *J* = 8.7 Hz, 2H), 7.06 (d, *J* = 7.5 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 4H), 3.79 (s, 3H), 2.97 (t, *J* = 7.6 Hz, 2H), 2.82 (dd, *J* = 4879.7, 7.5 Hz, 4H), 2.37 (d, *J* = 0.7 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.5, 165.1, 158.2, 135.8, 131.7, 131.6, 130.4, 129.3, 127.9, 114.0, 55.3, 37.5, 29.4, 20.0; FTIR (NaCl, thin film): 2955, 2931, 2836, 1811, 1740, 1612, 1595, 1584, 1513, 1466, 1301, 1248, 1179, 1124, 1079, 1036, 990, 827, 775 cm^{-1}; LRMS (ESI) calc'd for $[M+Na]^+$ 335.1, found 335.1.

C. General Procedure 3: Benzyl Chloride Synthesis



A flask was charged with the appropriate benzyl alcohol (1.0 equiv) and $CHCl_3$ (1.5 M). Thionyl chloride (1.05 equiv) was added dropwise. Evolved gas was quenched via cannula by aqueous $NaHCO_3$. The solution was stirred at 23 °C for 12 h and then concentrated to afford a yellow oil. The crude residue was purified by Kugelrohr distillation to isolate a clear oil. Spectral data for all compounds matched those reported in the literature.

[1-chloro-2-(*t*-butyldimethylsiloxy)ethyl]benzene (2m).



To a flask was added 2-chloro-2-phenylethanol (8.5 mmol, 1.0 equiv) and CH_2Cl_2 (18 mL, 0.5 M) followed by imidazole (10.2 mmol, 1.2 equiv) and *tert*-butyldimethylsilyl chloride (10.2 mmol, 1.2 equiv). The reaction was stirred at 23 °C for 24 h and then quenched by pouring into water (40 mL). The aqueous and organic layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 X 20 mL). The combined organic layers were washed with brine (1 X 20 mL) and dried (Na_2SO_4), filtered, and concentrated. The crude residue was filtered through a thick pad of silica with hexanes and concentrated to afford a clear oil (2.21 g, 96% yield). ^{1}H

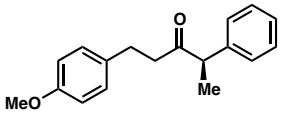
NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.27 (m, 5H), 4.87 (t, J = 6.6 Hz, 1H), 4.00 (dd, J = 10.7, 6.8 Hz, 1H), 3.92 (dd, J = 10.7, 6.5 Hz, 1H), 0.85 (s, 9H), 0.01 (s, 3H), –0.04 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.0, 128.39, 128.37, 127.6, 68.5, 63.3, 25.7, -5.4, -5.5; FTIR (NaCl, thin film): 2955, 2928, 2884, 2856, 1494, 1472, 1361, 1257, 1123, 1080, 837, 778 cm^{-1}; HRMS (FAB) calc'd for $[M+H]^+$ 271.1279, found 271.1290.

4. Enantioselective Reductive Cross-Coupling

General Procedure 4: Enantioselective Reductive Coupling of Benzyl Chlorides and Acid Chlorides

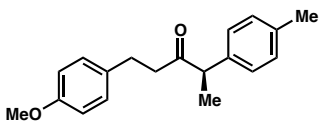
On a bench-top, to a 1/2 dram vial was added (*R*,*R*)-**L1** (0.044 mmol, 22 mol %), 2,6-DMBA (**5**, 0.15 mmol, 0.75 equiv), 3 Å mol sieves (30 mg/0.2 mmol benzyl chloride), manganese powder (0.6 mmol, 3 equiv), and $NiCl_2$(dme) (0.02 mmol, 10 mol %). Under an inert atmosphere in a glovebox, the vial was charged with 30% v/v DMA/THF (0.53 mL, 0.375 M) followed by benzyl chloride (**2**, 0.2 mmol, 1 equiv) and acid chloride (**1a** or **6**, Table 2: 0.3 mmol, 1.5 equiv, Table 3: 0.24 mmol, 1.2 equiv). The mixture was stirred at 240 rpm, ensuring that the manganese powder was uniformly suspended. Stirring continued at 20 °C under inert atmosphere for 24 h. The black slurry was transferred to a separatory funnel using 1 M HCl (5 mL) and diethyl ether (10 mL). The mixture was diluted with H_2O (10 mL) and the aqueous and organic layers were separated. The aqueous layer was extracted with diethyl ether (2 X 10 mL) and the combined organic layers were washed with brine (1 X 15 mL) and dried ($MgSO_4$), filtered, and concentrated. The crude residue was purified by flash chromatography.

(*R*)-1-(4-methoxyphenyl)-4-Phenylpentan-3-one (3a)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3a** (42.3 mg, 79% yield) in 93% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (minor) = 9.2 min, t_R (major) = 9.8 min. $[\alpha]_D^{25}$ = – 102.3° (c = 1.10, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.21 (m, 3H), 7.22 – 7.14 (m, 2H), 7.05 – 6.96 (m, 2H), 6.84 – 6.75 (m, 2H), 3.79 (s, 3H), 3.72 (q, *J* = 7.0 Hz, 1H), 2.88 – 2.57 (m, 4H), 1.39 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.0, 157.8, 140.4, 133.0, 129.2, 128.9, 127.8, 127.1, 113.7, 55.2, 53.2, 42.8, 29.1, 17.3; FTIR (NaCl, thin film): 3060, 3027, 2973, 2931, 2834, 1713, 1611, 1513, 1493, 1452, 1300, 1247 cm^{-1}; HRMS (MM) calc'd for $[M–H]^-$ 267.1391, found 267.1391.

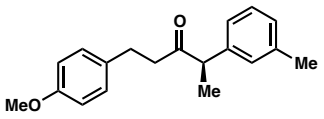
(*R*)-1-(4-methoxyphenyl)-4-(*p*-tolyl)Pentan-3-one (3b)



Prepared from 1-(1-chloroethyl)-4-methylbenzene (**2b**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 33 mol % (*R*,*R*)-**L1** (0.066 mmol). The crude residue was purified by

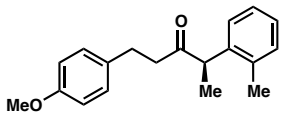
silica gel chromatography (5% ethyl acetate/hexanes) to yield **3b** (41.8 mg, 74%% yield) in 93% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (minor) = 9.0 min, t_R (major) = 9.8 min. $[\alpha]_D^{25} = -84.9°$ (c = 1.37, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (d, J = 7.9 Hz, 2H), 7.05 (d, J = 7.9 Hz, 2H), 6.99 (d, J = 9.0 Hz, 2H), 6.77 (d, J = 8.6 Hz, 2H), 3.77 (s, 3H), 3.66 (q, J = 6.9 Hz, 1H), 2.84 – 2.55 (m, 4H), 2.33 (s, 3H), 1.35 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.2, 157.8, 137.4, 136.7, 133.1, 129.6, 129.2, 127.7, 113.8, 55.2, 52.8, 42.8, 29.1, 21.0, 17.3; FTIR (NaCl, thin film): 2930, 2834, 1713, 1612, 1584, 1513, 1454, 1300, 1246, 1178, 1036, 824 cm^{-1}; HRMS (MM) calc'd for $[M+H]^+$ 283.1647, found 283.1693.

(*R*)-1-(4-methoxyphenyl)-4-(*m*-tolyl)Pentan-3-one (3c)



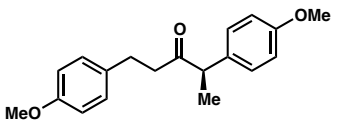
Prepared from 1-(1-chloroethyl)-3-methylbenzene (**2c**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 33 mol % (*R*,*R*)-**L1** (0.066 mmol). The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3c** (42.5 mg, 75% yield) in 93% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (minor) = 9.1 min, t_R (major) = 9.9 min. $[\alpha]_D^{25} = -90.4°$ (c = 1.46, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (t, J = 7.5 Hz, 1H), 7.09 – 7.01 (m, 1H), 7.02 – 6.92 (m, 4H), 6.77 (d, J = 8.5 Hz, 2H), 3.77 (s, 3H), 3.66 (q, J = 6.9 Hz, 1H), 2.84 – 2.56 (m, 4H), 2.31 (s, 3H), 1.36 (d, J = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.1, 157.8, 140.4, 138.6, 133.1, 129.2, 128.8, 128.6, 127.9, 125.0, 113.8, 55.2, 53.1, 42.8, 29.1, 21.4, 17.3; FTIR (NaCl, thin film): 2931, 2834, 1714, 1611, 1584, 1513, 1453, 1300, 1246, 1178, 1036, 825 cm^{-1}; HRMS (MM) calc'd for $[M+H]^+$ 283.1693, found 283.1557.

(*R*)-1-(4-methoxyphenyl)-4-(*o*-tolyl)Pentan-3-one (3d)



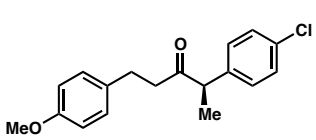
Prepared from 1-(1-chloroethyl)-2-methylbenzene (**2d**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 33 mol % (*R*,*R*)-**L1** (0.066 mmol). The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3d** (19.8 mg, 35% yield) in 72% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 10% IPA in CO_2, λ = 210 nm): t_R (minor) = 5.3 min, t_R (major) = 5.7 min. $[\alpha]_D^{25} = -72.3°$ (c = 0.56, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 – 7.09 (m, 3H), 7.02 – 6.92 (m, 3H), 6.77 (d, J = 8.6 Hz, 2H), 3.87 (q, J = 6.9 Hz, 1H), 3.76 (s, 3H), 2.85 – 2.68 (m, 2H), 2.64 – 2.47 (m, 2H), 2.33 (s, 3H), 1.32 (d, J = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.4, 157.9, 140.0, 135.7, 133.1, 130.8, 129.2, 127.0, 126.6, 113.8, 55.2, 49.2, 42.8, 29.2, 19.7, 16.7; FTIR (NaCl, thin film): 2931, 2834, 1712, 1611, 1513, 1491, 1463, 1300, 1246, 1171, 1036, 828 cm^{-1}; HRMS (MM) calc'd for M^{*+} 282.1614, found 282.1543.

(*R*)-1,4-bis(4-methoxyphenyl)Pentan-3-one (3e)



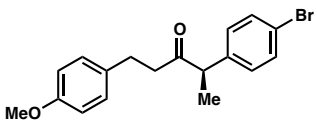
Prepared from 1-(1-chloroethyl)-4-methoxybenzene (**2e**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 33 mol % (*R*,*R*)-**L1** (0.066 mmol). The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **2e** (33.4 mg, 56% yield) in 86% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OB, 2.5 mL/min, 10% IPA in CO_2, λ = 210 nm): t_R (minor) = 6.8 min, t_R (major) = 7.4 min. $[\alpha]_D^{25} = -77.2°$ (c = 1.22, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.10 (d, J = 8.3 Hz, 2H), 6.98 (d, J = 8.0 Hz, 2H), 6.83 (d, J = 9.0 Hz, 2H), 6.76 (d, J = 9.0 Hz, 2H), 3.79 (s, 3H), 3.77 (s, 3H), 3.64 (q, J = 6.9 Hz, 1H), 2.83 – 2.54 (m, 4H), 1.34 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.3, 158.7, 157.9, 133.1, 132.4, 129.2, 128.9, 114.3, 113.8, 55.24, 55.23, 52.3, 42.7, 29.1, 17.3 ; FTIR (NaCl, thin film): 2930, 2834, 1710, 1611, 1582, 1512, 1463, 1301, 1246, 1177, 1034, 827 cm^{-1}; HRMS (MM) calc'd for M^{*+} 298.1563, found 298.1622.

(*R*)-4-(4-chlorophenyl)-1-(4-methoxyphenyl)Pentan-3-one (3f)



Prepared from 1-chloro-4-(1-chloroethyl)benzene (**2f**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3f** (45.9 mg, 76% yield) in 91% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 3% IPA in CO_2, λ = 210 nm): t_R (minor) = 19.6 min, t_R (major) = 20.6 min. $[\alpha]_D^{25} = -64.1°$ (c = 0.79, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (d, J = 8.8 Hz, 2H), 7.06 (d, J = 8.8 Hz, 2H), 6.97 (d, J = 8.8 Hz, 2H), 6.76 (d, J = 8.4 Hz, 2H), 3.77 (s, 3H), 3.67 (q, J = 7.0 Hz, 1H), 2.83 – 2.55 (m, 4H), 1.34 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.4, 157.9, 138.8, 133.0, 132.8, 129.2, 129.0, 113.8, 55.2, 52.5, 42.9, 29.0, 17.3; FTIR (NaCl, thin film): 2932, 1713, 1611, 1513, 1491, 1300, 1247, 1178, 1093, 1036, 1014, 825 cm^{-1}; HRMS (MM) calc'd for M^+ 302.1068, found 302.1001.

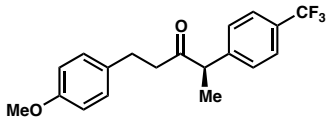
(*R*)-4-(4-bromophenyl)-1-(4-methoxyphenyl)Pentan-3-one (3g)



Prepared from 1-bromo-4-(1-chloroethyl)benzene (**2g**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 1.25 equiv 2,6-DMBA (0.25 mmol). The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3g** (51.0 mg, 73% yield) in 86% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (minor) = 25.4 min, t_R (major) = 27.0 min. $[\alpha]_D^{25} = -53.5°$ (c = 1.44, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, J = 8.6 Hz, 2H), 7.01 (d, J = 8.4 Hz, 2H), 6.97 (d, J = 8.6 Hz, 2H), 6.76 (d, J = 9.2 Hz, 2H), 3.77 (s, 3H),

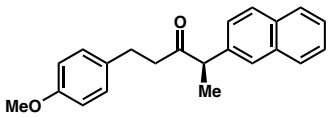
3.65 (q, J = 7.0 Hz, 1H), 2.83 – 2.55 (m, 4H), 1.34 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.3, 157.9, 139.3, 132.8, 132.0, 129.6, 129.2, 121.1, 113.8, 55.2, 52.6, 42.9, 29.0, 17.3; FTIR (NaCl, thin film): 2932, 2834, 1714, 1611, 1513, 1487, 1453, 1300, 1247, 1178, 1036, 1010, 825 cm^{-1}; HRMS (MM) calc'd for M^{*+} 346.0563, found 346.0463.

(*R*)-1-(4-methoxyphenyl)-4-(4-(trifluoromethyl)phenyl)Pentan-3-one (3h)



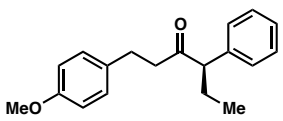
Prepared from 1-(1-chloroethyl)-4-(trifluoromethyl)benzene (**2h**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 20% v/v DMA/THF. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3h** (42.8 mg, 64% yield) in 82% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OJ, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (major) = 6.0 min, t_R (minor) = 7.3 min. $[\alpha]_D^{25} = -50.8°$ (c = 1.01, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, J = 7.8 Hz, 2H), 7.25 (d, J = 7.7 Hz, 2H), 6.97 (d, J = 8.8 Hz, 2H), 6.80 (d, J = 9.0 Hz, 2H), 3.80 – 3.74 (m, 4H), 2.85 – 2.60 (m, 4H), 1.38 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.0, 158.0, 144.2, 132.7, 129.3, 129.2, 128.2, 125.8, 113.9, 113.8, 55.2, 53.0, 43.1, 28.9, 17.3; FTIR (NaCl, thin film): 2934, 2837, 1717, 1616, 1584, 1513, 1419, 1326, 1247, 1165, 1124, 1070, 1036, 825 cm^{-1}; HRMS (MM) calc'd for M^{*+} 336.1332, found 336.1342.

(*R*)-1-(4-methoxyphenyl)-4-(naphthalen-2-yl)Pentan-3-one (3i)



Prepared from 2-(1-chloroethyl)naphthalene (**2i**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 33 mol % (*R*,*R*)-**L1** (0.066 mmol). The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3i** (41.7 mg, 65% yield) in 91% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AS, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (minor) = 10.7 min, t_R (major) = 11.3 min. $[\alpha]_D^{25} = -100.4°$ (c = 1.00, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 – 7.73 (m, 3H), 7.59 (s, 1H), 7.52 – 7.42 (m, 2H), 7.29 – 7.23 (m, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.71 (d, *J* = 8.8 Hz, 2H), 3.86 (q, *J* = 6.9 Hz, 1H), 3.73 (s, 3H), 2.85 – 2.60 (m, 4H), 1.46 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.0, 157.8, 137.9, 133.6, 132.9, 132.5, 129.2, 128.7, 127.7, 127.6, 126.6, 126.2, 125.9, 113.7, 55.2, 53.3, 42.9, 29.0, 17.3; FTIR (NaCl, thin film): 3055, 2972, 2931, 2834, 1713, 1611, 1583, 1511, 1455, 1374, 1300, 1245, 1178, 1035, 822, 750 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 319.2, found 319.2.

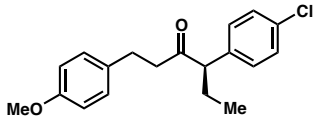
(*R*)-1-(4-methoxyphenyl)-4-Phenylhexan-3-one (3j)



Prepared from (1-chloropropyl)benzene (**2j**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4.

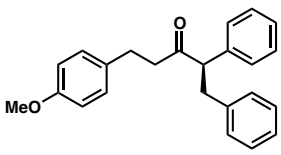
The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3j** (28.1 mg, 50% yield) in 94% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OB, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (minor) = 6.2 min, t_R (major) = 6.9 min. $[\alpha]_D^{25} = -97.9°$ (c = 0.96, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.20 (m, 3H), 7.19 – 7.12 (m, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 6.76 (d, *J* = 8.5 Hz, 2H), 3.76 (s, 3H), 3.48 (t, *J* = 7.4 Hz, 1H), 2.84 – 2.56 (m, 4H), 2.11 – 1.99 (m, 1H), 1.77 – 1.64 (m, 1H), 0.80 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.7, 157.8, 138.8, 133.1, 129.2, 128.8, 128.3, 127.1, 113.8, 61.0, 55.2, 43.6, 29.0, 25.1, 12.1; FTIR (NaCl, thin film): 2961, 2932, 1711, 1611, 1513, 1492, 1453, 1300, 1247, 1178, 1036, 821 cm^{-1}; HRMS (MM) calc'd for M^{*+} 282.1614, found 282.1631.

(*R*)-4-(4-chlorophenyl)-1-(4-methoxyphenyl)Hexan-3-one (3k)



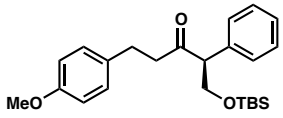
Prepared from 1-chloro-4-(1-chloropropyl)benzene (**2k**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3k** (41.2 mg, 65% yield) in 91% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 3% IPA in CO_2, λ = 210 nm): t_R (minor) = 18.1 min, t_R (major) = 19.4 min. $[\alpha]_D^{25} = -79.7°$ (c = 1.85, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.6 Hz, 2H), 7.06 (d, *J* = 8.9 Hz, 2H), 6.97 (d, *J* = 9.1 Hz, 2H), 6.76 (d, *J* = 8.6 Hz, 2H), 3.77 (s, 3H), 3.48 – 3.41 (m, 1H), 2.83 – 2.55 (m, 4H), 2.01 (dp, *J* = 14.4, 7.3 Hz, 1H), 1.72 – 1.62 (m, 1H), 0.78 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.2, 157.9, 137.1, 133.0, 132.8, 129.6, 129.2, 128.9, 113.7, 60.3, 55.2, 43.7, 28.9, 25.1, 12.0; FTIR (NaCl, thin film): 2962, 2932, 2834, 1711, 1611, 1583, 1512, 1490, 1463, 1300, 1246, 1178, 1092, 1036, 1014, 819 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 317.1, found 317.1.

(*R*)-5-(4-methoxyphenyl)-1,2-Diphenylpentan-3-one (3l)



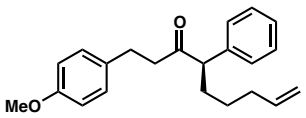
Prepared from (1-chloroethane-1,2-diyl)dibenzene (**2l**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3l** (54.6 mg, 79% yield) in 92% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AS, 2.5 mL/min, 10% IPA in CO_2, λ = 210 nm): t_R (major) = 4.5 min, t_R (minor) = 5.3 min. $[\alpha]_D^{25} = -166.8°$ (c = 0.85, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.08 (m, 8H), 7.06 – 6.96 (m, 2H), 6.92 (d, J = 8.3 Hz, 2H), 6.74 (d, J = 8.3 Hz, 2H), 3.87 (t, J = 7.4 Hz, 1H), 3.77 (s, 3H), 3.42 (dd, J = 13.7, 7.7 Hz, 1H), 2.90 (dd, J = 13.7, 7.0 Hz, 1H), 2.80 – 2.59 (m, 3H), 2.58 – 2.45 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.0, 157.8, 139.7, 138.3, 132.9, 129.1, 129.0, 128.9, 128.4, 128.2, 127.3, 126.1, 113.8, 61.1, 55.2, 44.1, 38.6, 28.9; FTIR (NaCl, thin film): 3027, 2930, 2834, 1712, 1611, 1583, 1513, 1495, 1453, 1300, 1247, 1178, 1035, 824 cm^{-1}; HRMS (MM) calc'd for $[M+H]^+$ 345.1849, found 345.1831.

(*S*)-1-((*tert*-butyldimethylsilyl)oxy)-5-(4-methoxyphenyl)-2-Phenylpentan-3-one (3m)



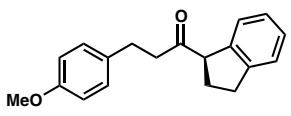
Prepared from [1-chloro-2-(*t*-butyldimethylsiloxy)ethyl]benzene (**2m**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4 except using 50% v/v DMA/THF. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3m** (40.4 mg, 51% yield) in 89% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AS, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (major) = 3.3 min, t_R (minor) = 3.8 min. $[\alpha]_D^{25} = -50.0°$ (c = 0.90, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.23 (m, 3H), 7.20 (dd, J = 8.1, 1.6 Hz, 2H), 7.01 (d, J = 8.8 Hz, 2H), 6.77 (d, J = 8.8 Hz, 2H), 4.23 (dd, J = 9.7, 8.5 Hz, 1H), 3.92 (dd, J = 8.5, 5.7 Hz, 1H), 3.77 (s, 3H), 3.73 (dd, J = 9.7, 5.7 Hz, 1H), 2.88 – 2.68 (m, 4H), 0.84 (s, 9H), –0.01 (s, 3H), –0.03 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.8, 157.8, 135.9, 133.1, 129.2, 128.7, 128.5, 127.5, 113.8, 65.0, 61.0, 55.2, 45.1, 28.6, 25.8, 18.2, –5.57, –5.60; FTIR (NaCl, thin film): 2953, 2928, 2855, 1718, 1612, 1583, 1513, 1463, 1361, 1248, 1099, 835 cm^{-1}; HRMS (MM) calc'd for $[M+H]^+$ 399.2350, found 399.2198.

(*R*)-1-(4-methoxyphenyl)-4-Phenylnon-8-en-3-one (3n)



Prepared from (1-chlorohex-5-en-1-yl)benzene (**2n**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3n** (24.6 mg, 38% yield) in 92% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AD, 2.5 mL/min, 5% IPA in CO_2, λ = 210 nm): t_R (major) = 10.9 min, t_R (minor) = 11.9 min. $[\alpha]_D^{25} = -90.9°$ (c = 0.47, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.20 (m, 3H), 7.18 – 7.11 (m, 2H), 6.98 (d, J = 8.4 Hz, 2H), 6.76 (d, J = 8.9 Hz, 2H), 5.73 (ddt, J = 16.9, 10.2, 6.7 Hz, 1H), 5.02 – 4.88 (m, 2H), 3.76 (s, 3H), 3.55 (t, J = 7.4 Hz, 1H), 2.84 – 2.54 (m, 4H), 2.09 – 1.93 (m, 3H), 1.74 – 1.63 (m, 1H), 1.37 – 1.15 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.6, 157.9, 138.8, 138.4, 133.0, 129.2, 128.9, 128.3, 127.2, 114.7, 113.8, 59.1, 55.2, 43.6, 33.6, 31.4, 29.0, 26.7; FTIR (NaCl, thin film): 2930, 1712, 1640, 1611, 1583, 1513, 1453, 1300, 1247, 1177, 1036, 824 cm^{-1}; HRMS (MM) calc'd for $[M+H]^+$ 323.2006, found 323.1945.

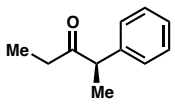
(*R*)-1-(2,3-dihydro-1*H*-inden-1-yl)-3-(4-methoxyphenyl)Propan-1-one (3o)



Prepared from 1-chloro-2,3-dihydro-1*H*-indene (**2o**, 0.20 mmol) and 3-(4-methoxyphenyl)propanoyl chloride (**1a**, 0.30 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **3o** (38.3 mg, 68% yield) in 78% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AD, 2.5 mL/min, 10% IPA in CO_2, λ = 210 nm): t_R (minor) = 7.9 min, t_R

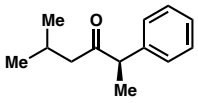
(major) = 8.9 min. $[\alpha]_D^{25} = 11.3°$ (c = 0.1.79, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.10 (m, 4H), 7.07 (d, *J* = 8.9 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 3H), 4.08 (t, *J* = 7.1 Hz, 1H), 3.78 (s, 3H), 3.05 (d, *J* = 7.9 Hz, 1H), 2.98 – 2.67 (m, 5H), 2.37 – 2.18 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.0, 157.9, 144.6, 140.8, 133.2, 129.3, 127.5, 124.9, 124.8, 113.9, 113.8, 58.4, 55.3, 42.4, 31.9, 28.9, 28.5; FTIR (NaCl, thin film): 2932, 2849, 1709, 1611, 1583, 1513, 1458, 1300, 1247, 1178, 1036, 826, 755 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 281.2, found 281.1.

(*R*)-2-Phenylpentan-3-one (7a)



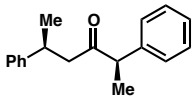
Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and propionyl chloride (**6a**, 0.24 mmol) according to General Procedure 4 except using 20% v/v DMA/THF. The crude residue was purified by silica gel chromatography (2% ethyl acetate/hexanes) to yield **7a** (19.5 mg, 60% yield) in 91% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AS, 2.5 mL/min, 1% IPA in CO_2, λ = 210 nm): t_R (minor) = 1.8 min, t_R (major) = 2.0 min. $[\alpha]_D^{25} = –225.9°$ (c = 0.57, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 2H), 7.28 – 7.23 (m, 1H), 7.23 – 7.19 (m, 2H), 3.76 (q, J = 7.0 Hz, 1H), 2.42 – 2.33 (m, 2H), 1.39 (d, J = 7.0 Hz, 3H), 0.97 (t, J = 7.3 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 211.5, 140.9, 128.8, 127.8, 127.0, 52.7, 34.2, 17.5, 8.0; FTIR (NaCl, thin film): 3027, 2976, 2935, 1716, 1600, 1494, 1453, 1374, 1130, 1070, 1029, 957, 758 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 163.1, found 163.1.

(*R*)-5-Methyl-2-phenylhexan-3-one (7b)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and isovaleroyl chloride (**6b**, 0.24 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (2% ethyl acetate/hexanes) to yield **7b** (27.5 mg, 73% yield) in 88% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 1% IPA in CO_2, λ = 210 nm): t_R (minor) = 2.2 min, t_R (major) = 2.7 min. $[\alpha]_D^{25} = –205.8°$ (c = 0.92, $CHCl_3$); ^1H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 2H), 7.28 – 7.23 (m, 1H), 7.23 – 7.18 (m, 2H), 3.72 (q, J = 6.9 Hz, 1H), 2.29 – 2.16 (m, 2H), 2.10 (hept, J = 6.7 Hz, 1H), 1.38 (d, J = 7.0 Hz, 3H), 0.84 (d, J = 6.6 Hz, 3H), 0.75 (d, J = 6.6 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.5, 140.5, 128.8, 127.9, 127.0, 53.3, 50.0, 24.3, 22.6, 22.2, 17.4; FTIR (NaCl, thin film): 3027, 2957, 2871, 1712, 1600, 1493, 1453, 1366, 1143, 1071, 1024, 761 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 191.1, found 191.2.

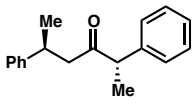
(2*R*,5*S*)-2,5-Diphenylhexan-3-one ((*R*,*S*)-7c)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and (*S*)-3-phenylbutyryl chloride (**(*S*)-6c**, 0.24 mmol) according to General Procedure 4. The crude residue was purified by silica gel chromatography (2% ethyl acetate/hexanes) to yield **(*R*,*S*)-7c** (34.8 mg, 69% yield) as a clear oil and as a 20:1 mixture of diastereomers (determined by NMR analysis of the purified product). $[\alpha]_D^{25} = –122.2°$

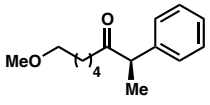
(c = 1.71, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.17 (m, 5H), 7.17 – 7.12 (m, 1H), 7.10 – 7.02 (m, 4H), 3.69 (q, J = 7.0 Hz, 1H), 3.30 (h, J = 7.0 Hz, 1H), 2.70 (dd, J = 16.8, 6.8 Hz, 1H), 2.58 (dd, J = 16.8, 7.5 Hz, 1H), 1.34 (d, J = 6.9 Hz, 3H), 1.20 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.3, 146.1, 140.2, 128.8, 128.3, 127.0, 126.74, 126.73, 126.1, 53.5, 49.2, 35.2, 21.9, 17.2; FTIR (NaCl, thin film): 3061, 3027, 2967, 2930, 1714, 1601, 1493, 1452, 1373, 1125, 1069, 1029, 759 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 253.2, found 253.2.

(2*S*,5*S*)-2,5-Diphenylhexan-3-one ((*S*,*S*)-7c)



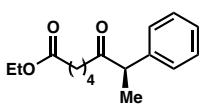
Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and (*S*)-3-phenylbutyryl chloride (**(*S*)-6c**, 0.24 mmol) according to General Procedure 4 except using (*S*,*S*)-**L1**. The crude residue was purified by silica gel chromatography (2% ethyl acetate/hexanes) to yield **(*S*,*S*)-7c** (33.7 mg, 67% yield) as a clear oil and as a 12:1 mixture of diastereomers (determined by NMR analysis of the purified product). $[\alpha]_D^{25}$ = 121.3° (c = 1.59, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 2H), 7.31 – 7.24 (m, 3H), 7.22 – 7.13 (m, 5H), 3.54 (q, J = 6.9 Hz, 1H), 3.29 (h, J = 7.3 Hz, 1H), 2.67 (dd, J = 16.3, 6.4 Hz, 1H), 2.56 (dd, J = 16.3, 7.9 Hz, 1H), 1.32 (d, J = 6.9 Hz, 3H), 1.11 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.5, 146.3, 140.3, 128.9, 128.5, 128.0, 127.1, 126.8, 126.2, 53.4, 49.6, 35.4, 21.5, 17.2; FTIR (NaCl, thin film): 3061, 3027, 2968, 2930, 1714, 1601, 1494, 1452, 1374, 1125, 1068, 1029, 1004, 763 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 253.2, found 253.1.

(*R*)-8-Methoxy-2-phenyloctan-3-one (7d)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and 6-methoxyhexanoyl chloride (**6d**, 0.24 mmol) according to General Procedure 4 except using 20% v/v DMA/THF. The crude residue was purified by silica gel chromatography (5-10% ethyl acetate/hexanes) to yield **7d** (35.0 mg, 75% yield) in 85% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 3% IPA in CO_2, λ = 210 nm): t_R (minor) = 5.4 min, t_R (major) = 5.8 min. $[\alpha]_D^{25}$ = –146.0° (c = 1.14, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 2H), 7.28 – 7.23 (m, 1H), 7.22 – 7.18 (m, 2H), 3.74 (q, J = 7.0 Hz, 1H), 3.31 – 3.25 (m, 5H), 2.38 – 2.32 (m, 2H), 1.57 – 1.42 (m, 2H), 1.38 (d, J = 7.0 Hz, 3H), 1.26 – 1.17 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.9, 140.7, 128.9, 127.9, 127.1, 72.5, 58.5, 53.0, 40.9, 29.3, 25.6, 23.6, 17.4; FTIR (NaCl, thin film): 2931, 2866, 2360, 1714, 1600, 1494, 1453, 1373, 1119, 1072, 1029, 761 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 235.2, found 235.2.

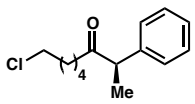
(*R*)-Ethyl 6-oxo-7-phenyloctanoate (7e)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and ethyl 6-chloro-6-oxohexanoate (**6e**, 0.24 mmol) according to General Procedure 4 except using 10% v/v DMA/THF. The crude residue was purified by silica gel chromatography (5% ethyl acetate/hexanes) to yield **7e**

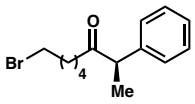
(33.8 mg, 64% yield) in 92% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (AD, 2.5 mL/min, 4% IPA in CO_2, λ = 210 nm): t_R (minor) = 4.9 min, t_R (major) = 5.3 min. $[\alpha]_D^{25} = -146.8°$ (c = 0.85, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 2H), 7.28 – 7.23 (m, 1H), 7.22 – 7.18 (m, 2H), 4.09 (q, J = 7.1 Hz, 2H), 3.73 (q, J = 7.0 Hz, 1H), 2.44 – 2.28 (m, 2H), 2.25 – 2.15 (m, 2H), 1.58 – 1.44 (m, 4H), 1.38 (d, J = 7.0 Hz, 3H), 1.22 (t, J = 7.1 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.4, 173.4, 140.6, 128.9, 127.8, 127.1, 60.2, 53.0, 40.5, 34.0, 24.3, 23.2, 17.4, 14.2; FTIR (NaCl, thin film): 2977, 2932, 1733, 1714, 1600, 1494, 1453, 1375, 1248, 1181, 1029, 761 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 263.2, found 263.2.

(*R*)-8-Chloro-2-phenyloctan-3-one (7f)



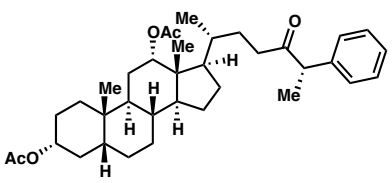
Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and 6-chlorohexanoyl chloride (**6f**, 0.24 mmol) according to General Procedure 4 except using 20% v/v DMA/THF. The crude residue was purified by silica gel chromatography (2% ethyl acetate/hexanes) to yield **7f** (36.3 mg, 76% yield) in 92% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 3% IPA in CO_2, λ = 210 nm): t_R (minor) = 5.8 min, t_R (major) = 6.5 min. $[\alpha]_D^{25} = -163.3°$ (c = 0.78, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.30 (m, 2H), 7.29 – 7.23 (m, 1H), 7.23 – 7.18 (m, 2H), 3.74 (q, J = 7.0 Hz, 1H), 3.45 (t, J = 6.7 Hz, 2H), 2.46 – 2.28 (m, 2H), 1.73 – 1.61 (m, 2H), 1.57 – 1.44 (m, 2H), 1.39 (d, J = 7.0 Hz, 3H), 1.34 – 1.24 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.6, 140.6, 128.9, 127.8, 127.2, 53.1, 44.8, 40.6, 32.3, 26.2, 23.0, 17.4; FTIR (NaCl, thin film): 2932, 2867, 2360, 1711, 1599, 1493, 1452, 1374, 1122, 1069, 1029, 760 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 239.1, found 239.1.

(*R*)-8-Bromo-2-phenyloctan-3-one (7g)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and 6-bromohexanoyl chloride (**6g**, 0.24 mmol) according to General Procedure 4 except using 10% v/v DMA/THF. The crude residue was purified by silica gel chromatography (2% ethyl acetate/hexanes) to yield **7g** (40.8 mg, 72% yield) in 86% ee as a clear oil. The enantiomeric excess was determined by chiral SFC analysis (OD, 2.5 mL/min, 3% IPA in CO_2, λ = 210 nm): t_R (minor) = 7.3 min, t_R (major) = 8.1 min. $[\alpha]_D^{25} = -146.8°$ (c = 1.57, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 2H), 7.29 – 7.24 (m, 1H), 7.23 – 7.18 (m, 2H), 3.74 (q, J = 7.0 Hz, 1H), 3.32 (t, J = 6.8 Hz, 2H), 2.46 – 2.28 (m, 2H), 1.80 – 1.70 (m, 2H), 1.56 – 1.44 (m, 2H), 1.39 (d, J = 7.0 Hz, 3H), 1.37 – 1.24 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 210.5, 140.6, 128.9, 127.9, 127.2, 53.1, 40.6, 33.6, 32.4, 27.5, 22.9, 17.4; FTIR (NaCl, thin film): 2932, 2867, 1713, 1600, 1494, 1453, 1373, 1252, 1069, 1029, 761 cm^{-1}; LRMS (ESI) calc'd for $[M+H]^+$ 283.1, found 283.1.

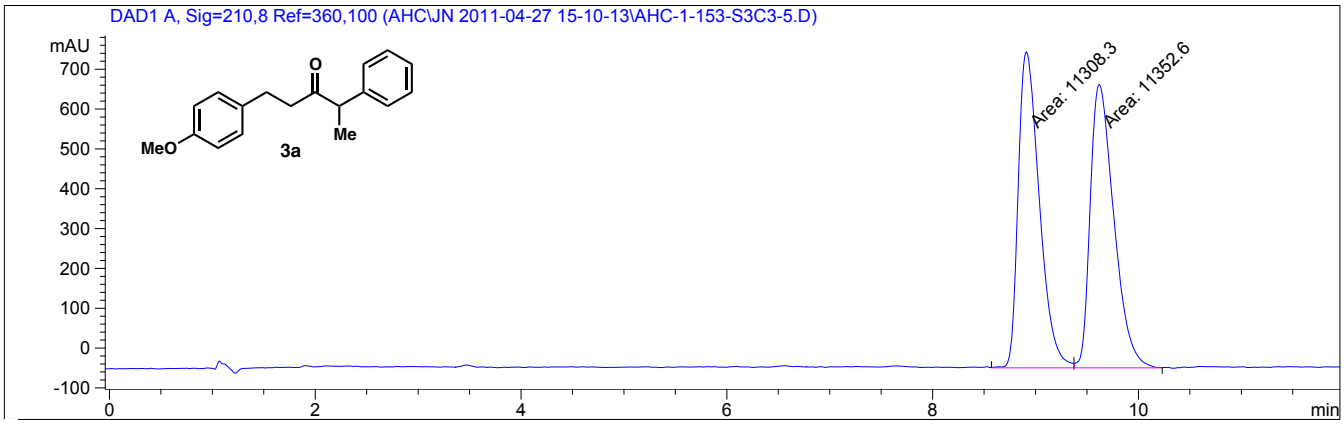
(3*R*,5*R*,8*R*,9*S*,10*S*,12*S*,13*R*,14*S*,17*R*)-10,13-Dimethyl-17-((2*R*,6*S*)-5-oxo-6-phenylheptan-2-yl)hexadecahydro-1*H*-cyclopenta[*a*]phenanthrene-3,12-diyl diacetate (7h)



Prepared from (1-chloroethyl)benzene (**2a**, 0.20 mmol) and acid chloride **6h** (0.24 mmol) according to General Procedure 4 except using 10% v/v DMA/THF and (*S*,*S*)-**L1.** Following extraction, the combined organic layers were washed with sat. aq. $NaHCO_3$ (1 X 10 mL) and brine (1 X 15 mL). The crude residue was purified by silica gel chromatography (15% ethyl acetate/hexanes) to yield **7h** (72.5 mg, 64% yield) as a fluffy white solid and as a 14:1 mixture of diastereomers (determined by NMR analysis of the purified product). $[\alpha]_D^{25} = 146.0°$ (c = 2.05, $CHCl_3$); ^{1}H NMR (500 MHz, Acetone-d_6) δ 7.39 – 7.31 (m, 2H), 7.30 – 7.22 (m, 3H), 4.99 (t, J = 3.0 Hz, 1H), 4.63 (tt, J = 11.4, 4.6 Hz, 1H), 3.90 (q, J = 6.9 Hz, 1H), 2.45 – 2.29 (m, 2H), 2.01 (s, 3H), 1.98 – 1.40 (m, 17H), 1.37 – 0.99 (m, 13H), 0.95 (s, 3H), 0.72 (s, 3H), 0.69 (d, J = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 209.8, 169.5, 169.3, 141.3, 128.7, 127.8, 126.9, 75.2, 73.5, 52.4, 49.4, 47.4, 44.9, 41.7, 37.3, 35.6, 34.6, 34.5, 34.3, 33.9, 32.1, 29.6, 27.0, 26.7, 26.4, 25.8, 25.3, 23.2, 22.5, 20.4, 20.3, 17.1, 16.9, 11.8; FTIR (NaCl, thin film): 2937, 2869, 1735, 1493, 1452, 1377, 1363, 1245, 1194, 1029, 971 cm^{-1}; LRMS (ESI) calc'd for $[M+H_2O]^+$ 582.4, found 582.4.

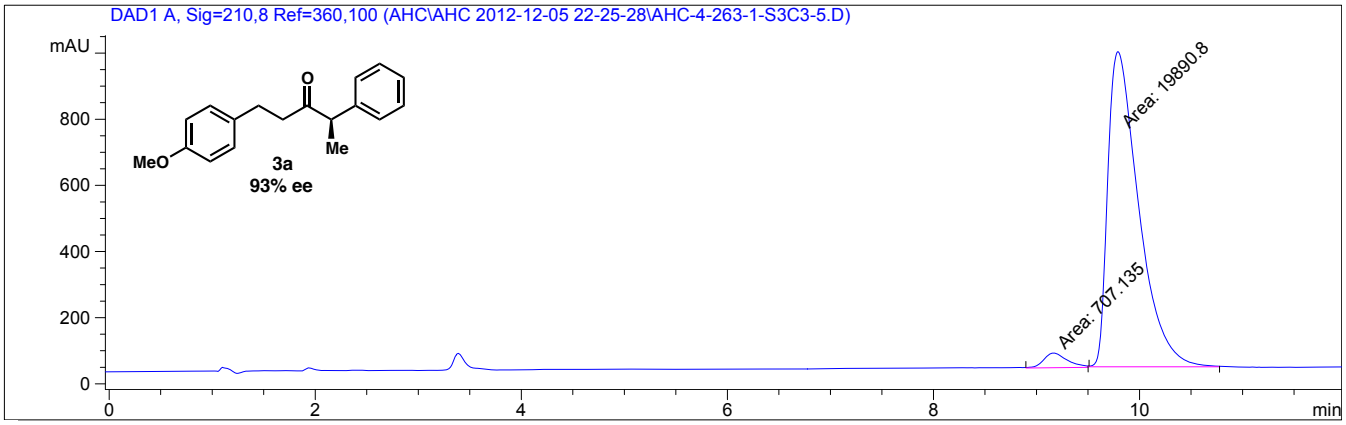
5. SFC Traces of Racemic and Enantioenriched Ketone Products

3a (Table 2, entry 1): racemic



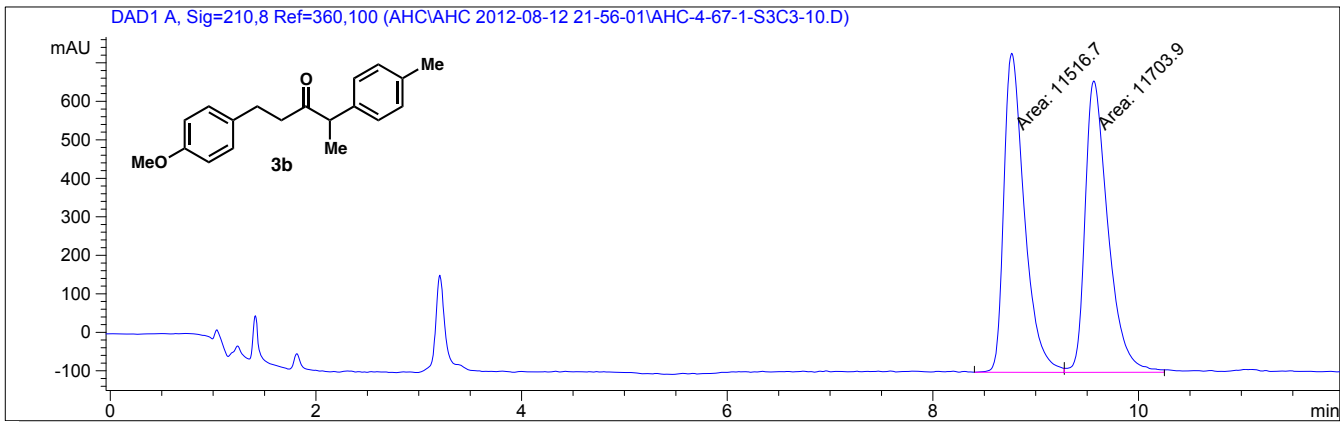
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.911	MM	0.2376	1.13083e4	793.28162	49.9021
2	9.619	MM	0.2660	1.13526e4	711.29791	50.0979

3a (Table 2, entry 1): enantioenriched, 93% ee



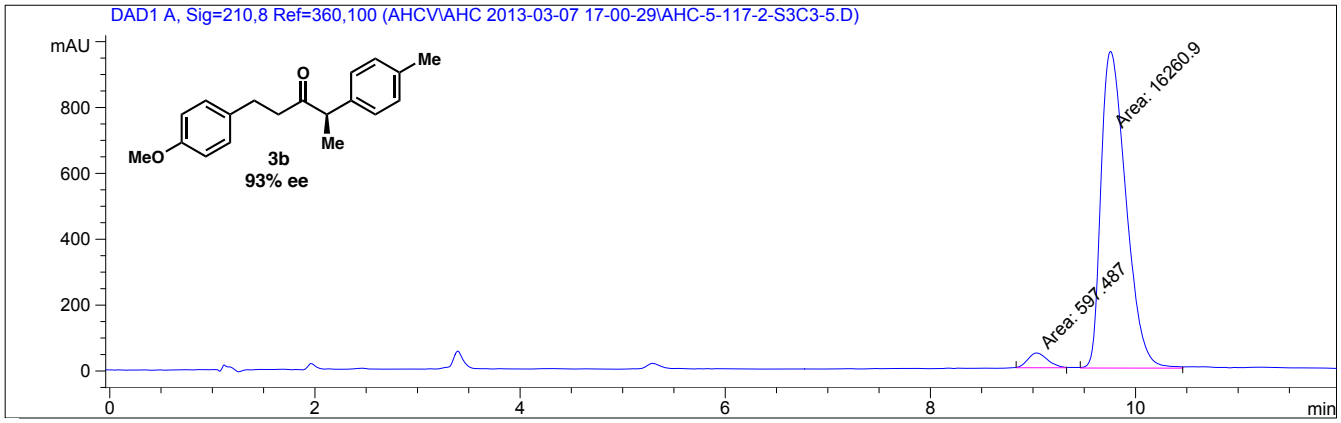
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.164	MM	0.2646	707.13519	44.53406	3.4330
2	9.790	MM	0.3479	1.98908e4	952.85913	96.5670

3b (Table 2, entry 2): racemic



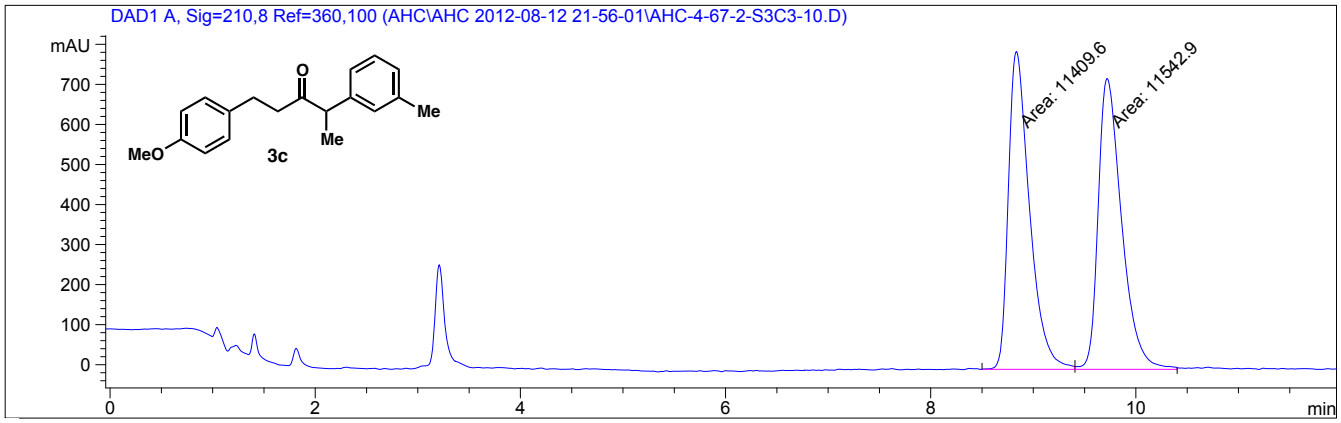
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.766	MM	0.2315	1.15167e4	829.08398	49.5970
2	9.564	MM	0.2576	1.17039e4	757.13312	50.4030

3b (Table 2, entry 2): enantioenriched, 93% ee



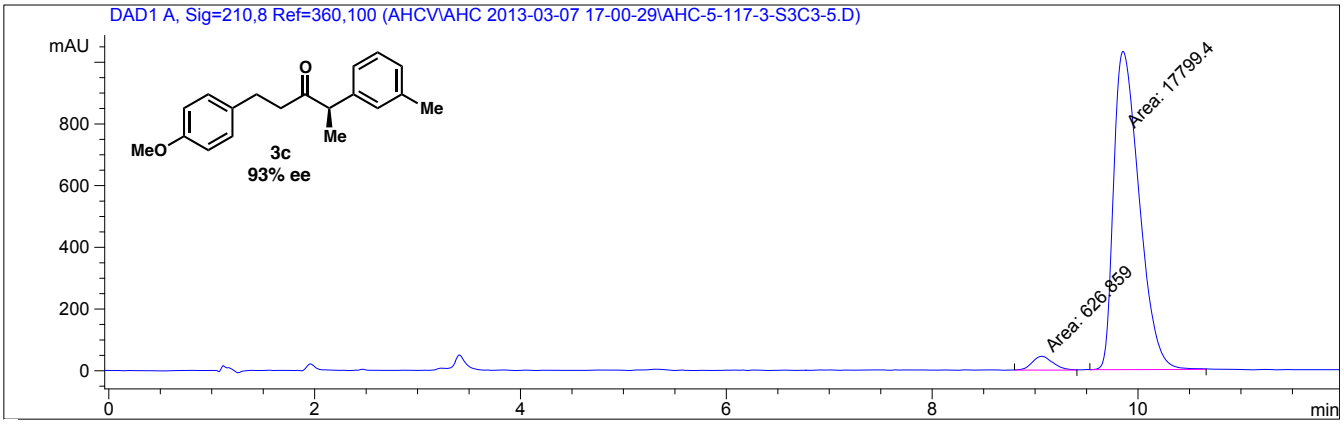
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.036	MM	0.2206	597.48749	45.14814	3.5442
2	9.756	MM	0.2819	1.62609e4	961.42609	96.4558

3c (Table 2, entry 3): racemic



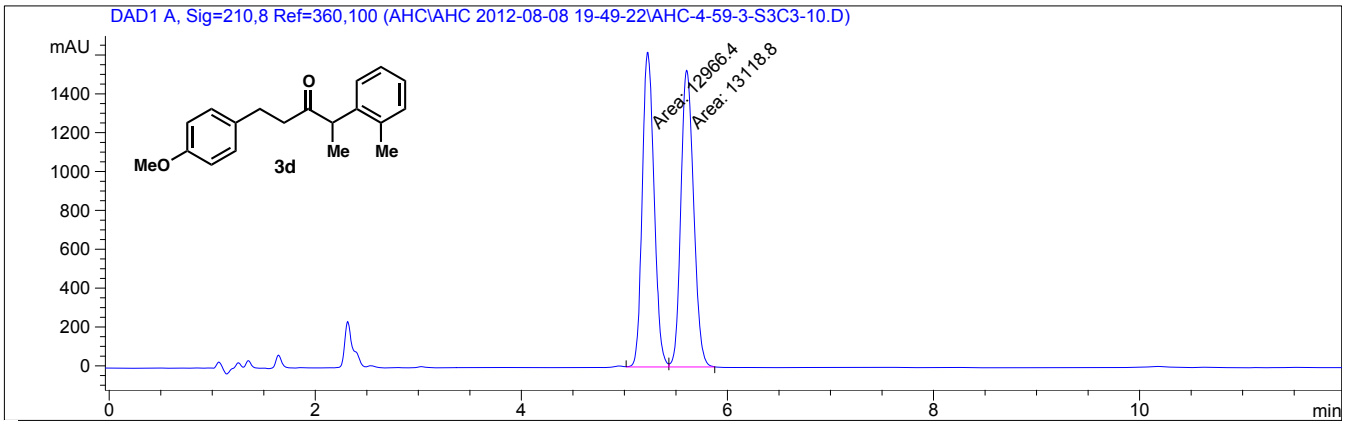
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.835	MM	0.2395	1.14096e4	794.09918	49.7095
2	9.720	MM	0.2647	1.15429e4	726.79602	50.2905

3c (Table 2, entry 3): enantioenriched, 93% ee



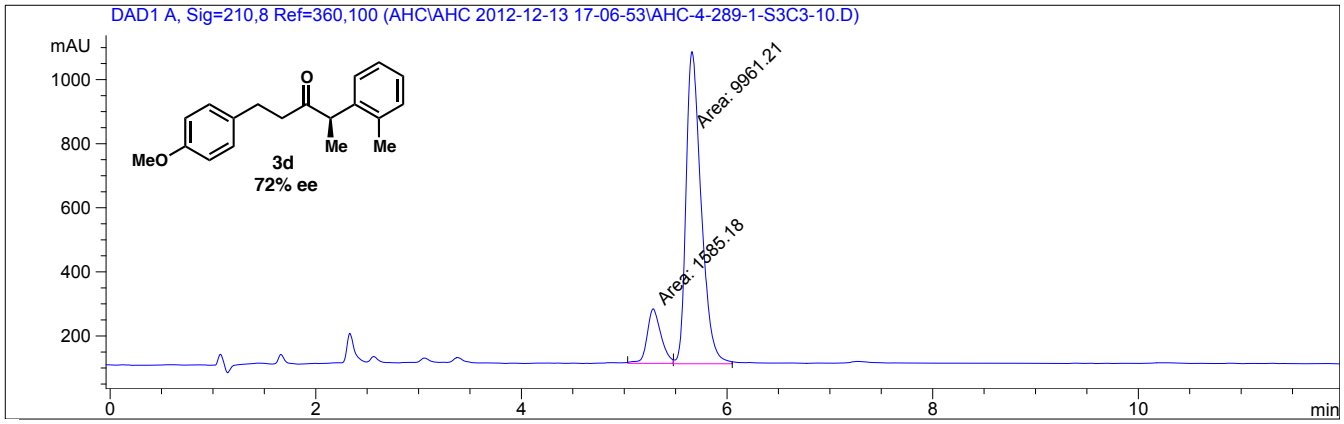
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.064	MM	0.2335	626.85852	44.73974	3.4020
2	9.854	MM	0.2876	1.77994e4	1031.51978	96.5980

3d (Table 2, entry 4): racemic



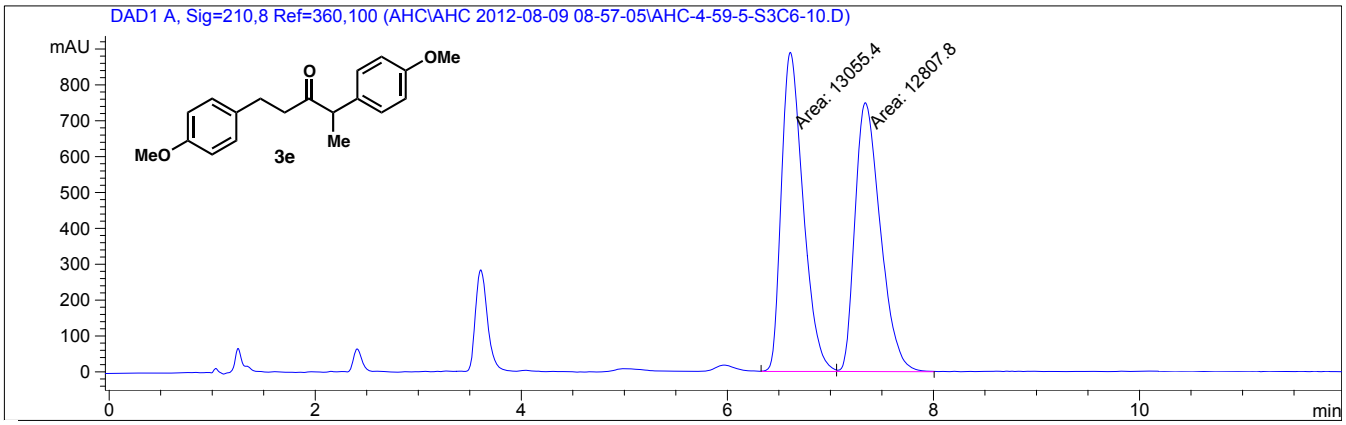
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.225	MM	0.1332	1.29664e4	1622.72693	49.7079
2	5.604	MM	0.1430	1.31188e4	1529.25037	50.2921

3d (Table 2, entry 4): enantioenriched, 72% ee



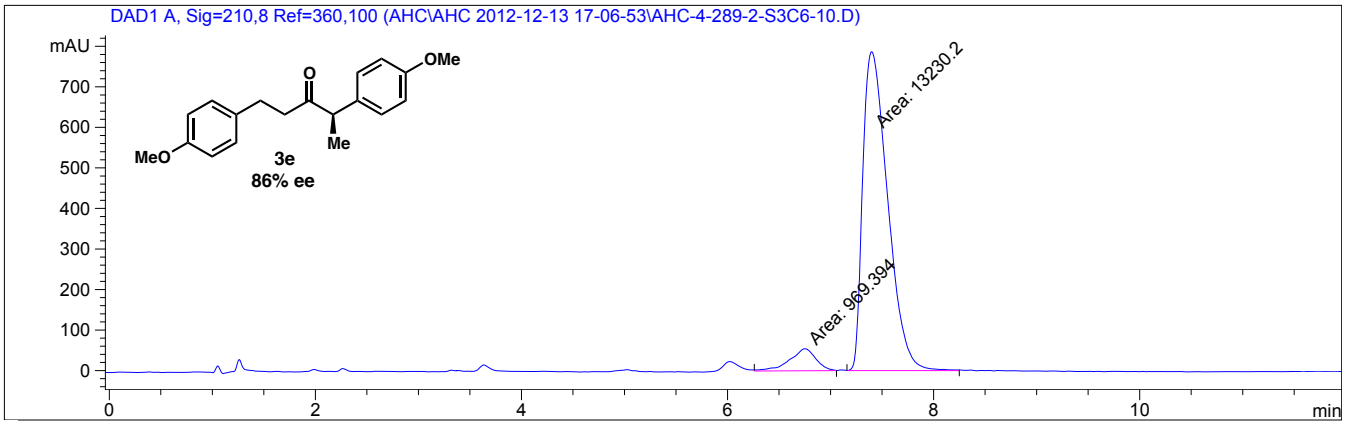
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.282	MM	0.1551	1585.18311	170.37230	13.7288
2	5.659	MM	0.1703	9961.21484	974.66937	86.2712

3e (Table 2, entry 5): racemic



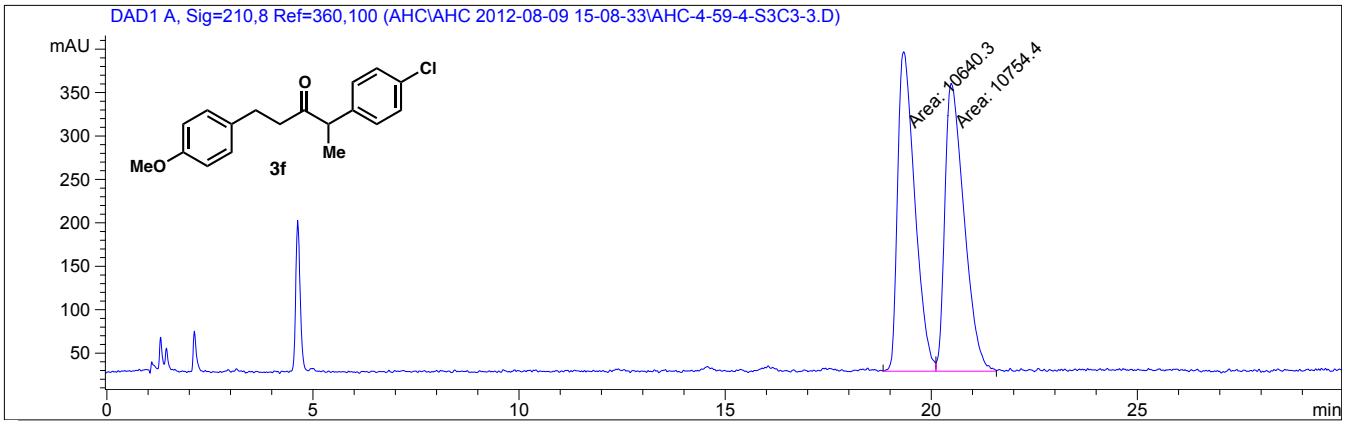
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.610	MM	0.2445	1.30554e4	889.75983	50.4785
2	7.339	MM	0.2848	1.28078e4	749.64392	49.5215

3e (Table 2, entry 5): enantioenriched, 86% ee



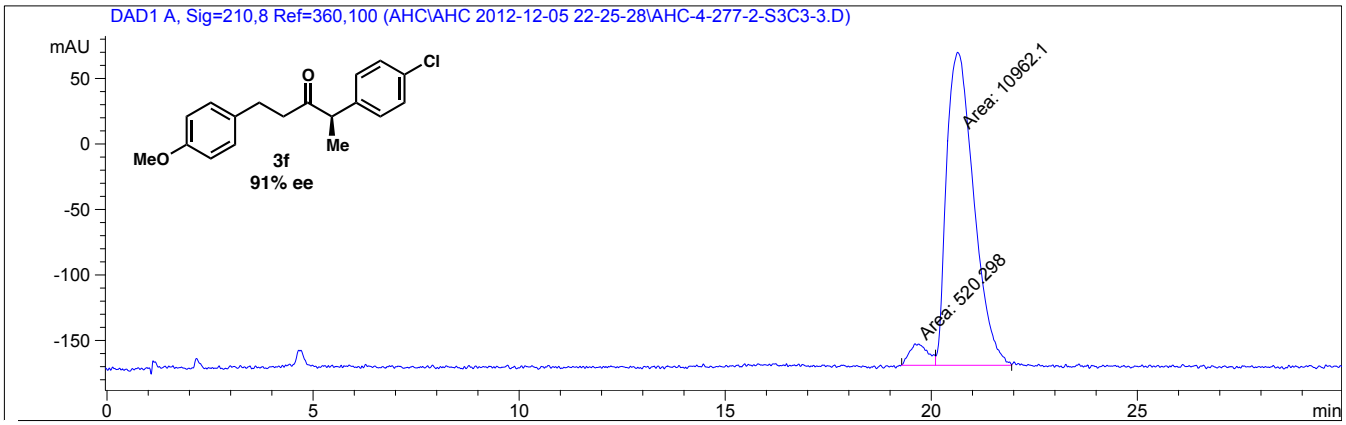
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.750	MM	0.2970	969.39368	54.40509	6.8269
2	7.399	MM	0.2801	1.32302e4	787.11755	93.1731

3f (Table 2, entry 6): racemic



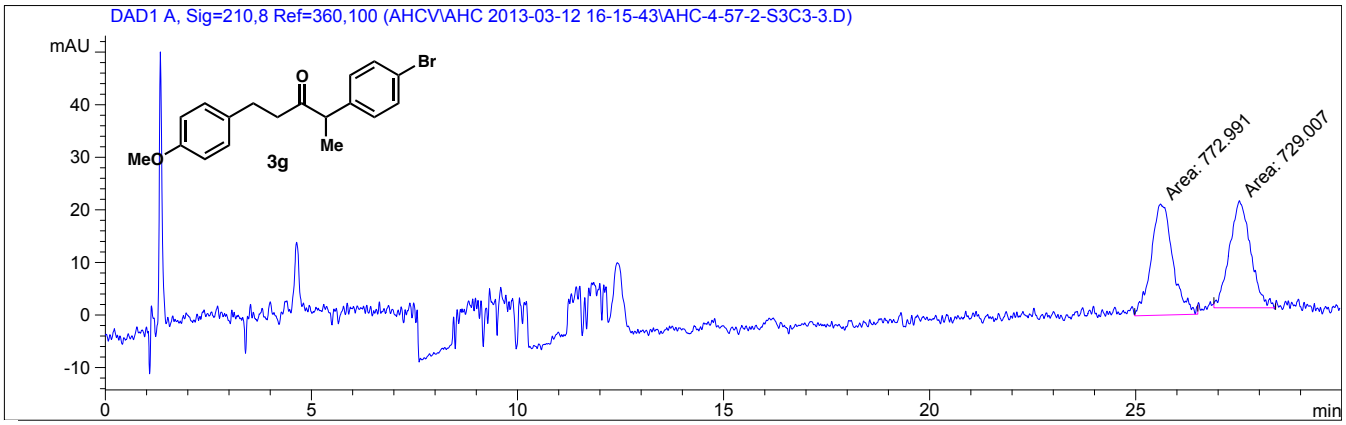
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.330	MM	0.4822	1.06403e4	367.73328	49.7335
2	20.480	MM	0.5409	1.07544e4	331.38272	50.2665

3f (Table 2, entry 6): enantioenriched, 91% ee



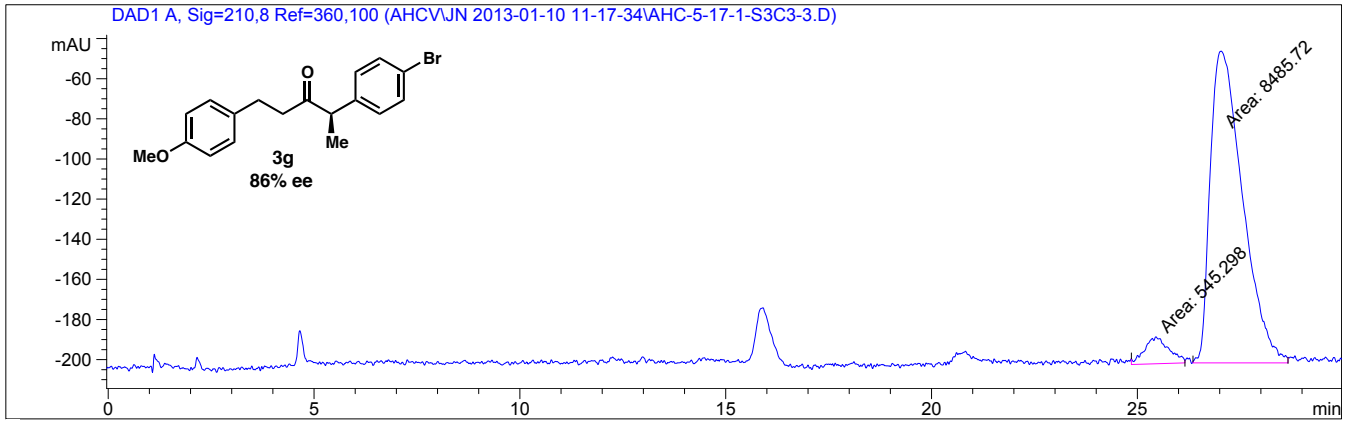
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.605	MM	0.5260	520.29840	16.48751	4.5313
2	20.639	MM	0.7645	1.09621e4	238.97188	95.4687

3g (Table 2, entry 7): racemic



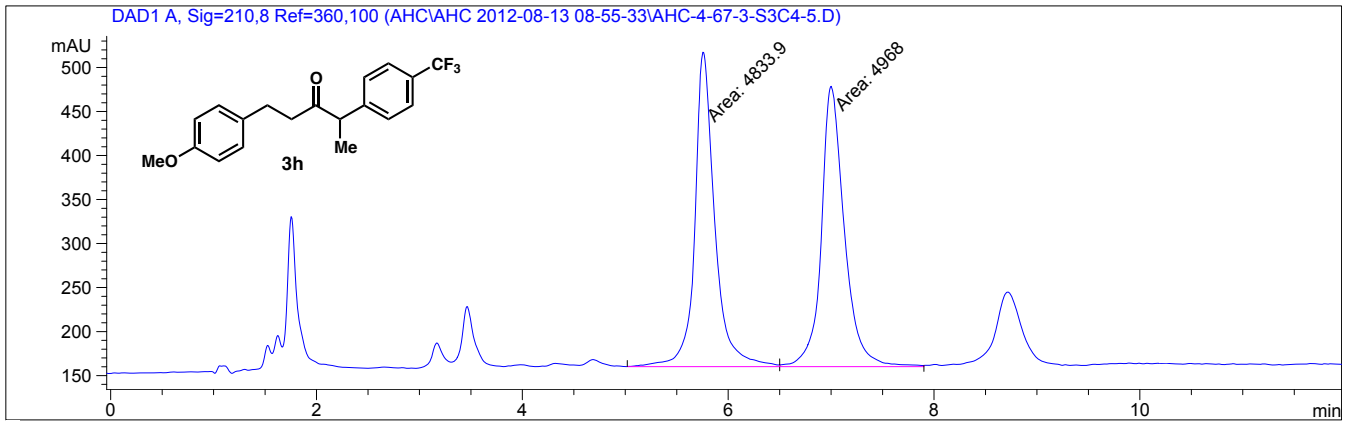
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.604	MM	0.6090	772.99146	21.15472	51.4642
2	27.518	MM	0.5962	729.00677	20.37799	48.5358

3g (Table 2, entry 7): enantioenriched, 86% ee



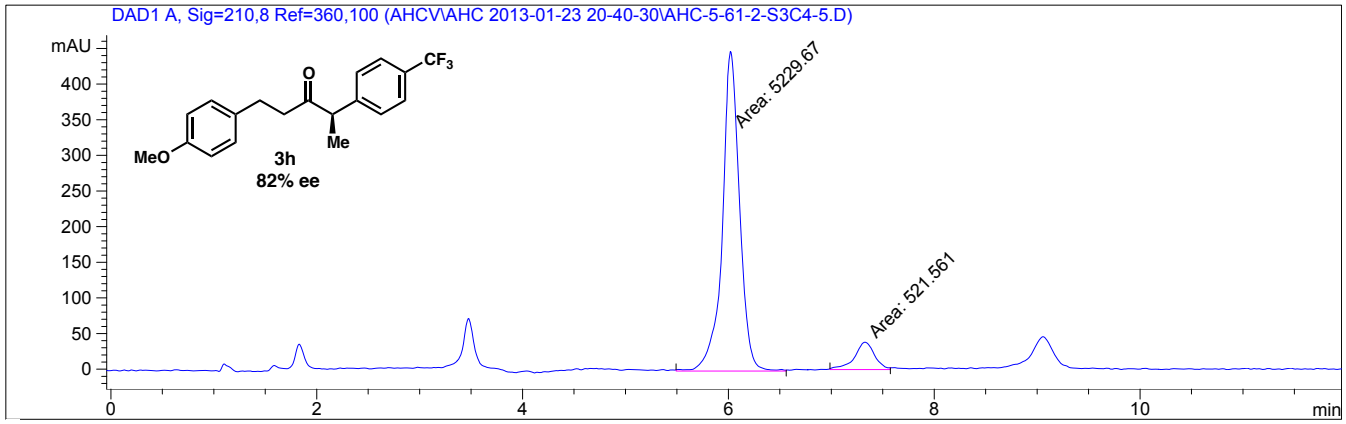
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.449	MM	0.6766	545.29791	13.43136	6.0381
2	27.030	MM	0.9105	8485.72363	155.33823	93.9619

3h (Table 2, entry 8): racemic



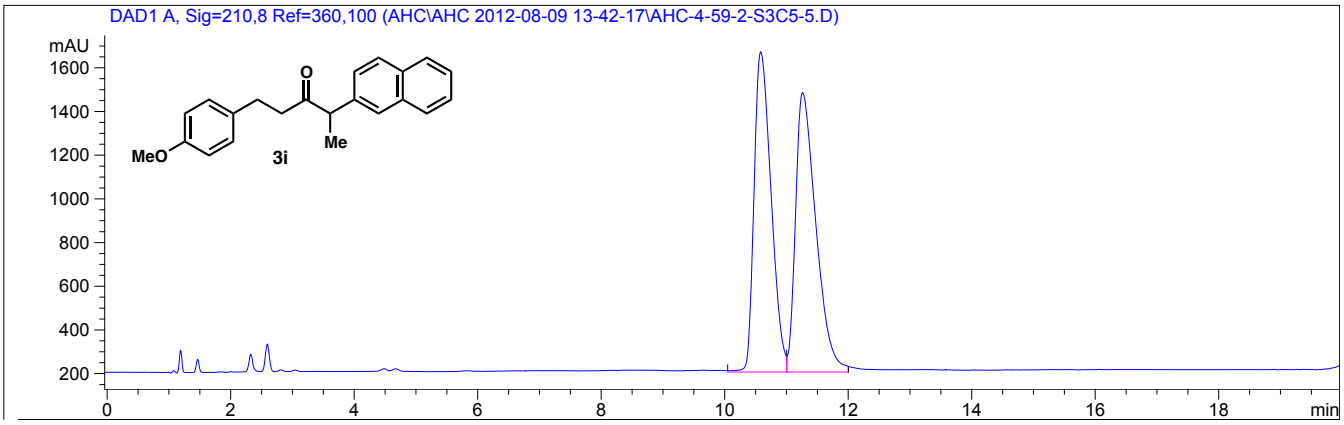
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.757	MM	0.2253	4833.89893	357.65167	49.3159
2	6.999	MM	0.2598	4967.99951	318.66217	50.6841

3h (Table 2, entry 8): enantioenriched, 82% ee



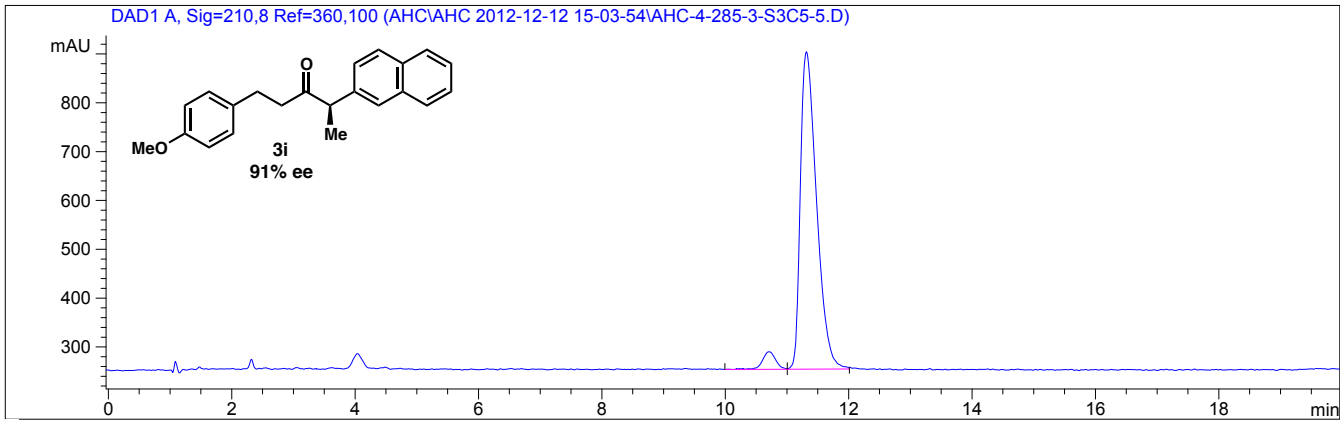
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.020	MM	0.1943	5229.66602	448.69507	90.9313
2	7.327	MM	0.2264	521.56061	38.39449	9.0687

3i (Table 2, entry 9): racemic



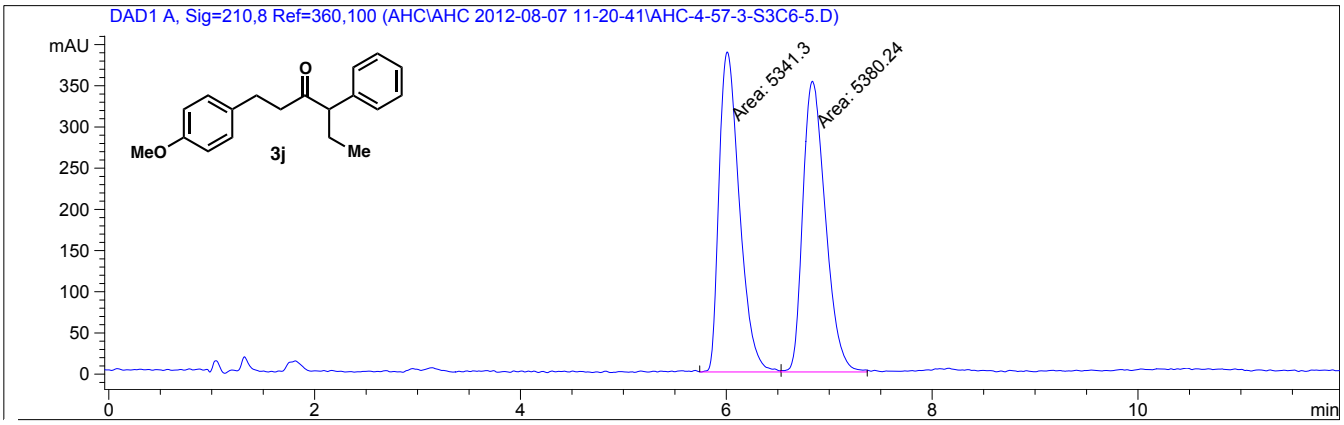
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.584	VV	0.3032	2.80499e4	1465.87903	48.9009
2	11.263	VBA	0.3473	2.93108e4	1279.38000	51.0991

3i (Table 2, entry 9): enantioenriched, 91% ee



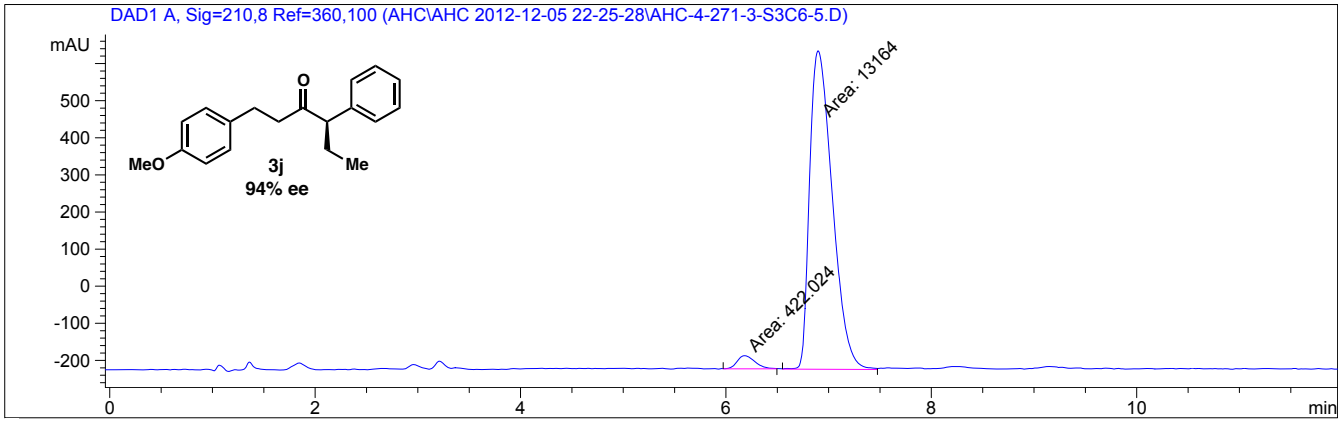
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.708	VV	0.2364	545.21796	36.05198	4.4227
2	11.316	VBA	0.2797	1.17825e4	649.47711	95.5773

3j (Table 2, entry 10): racemic



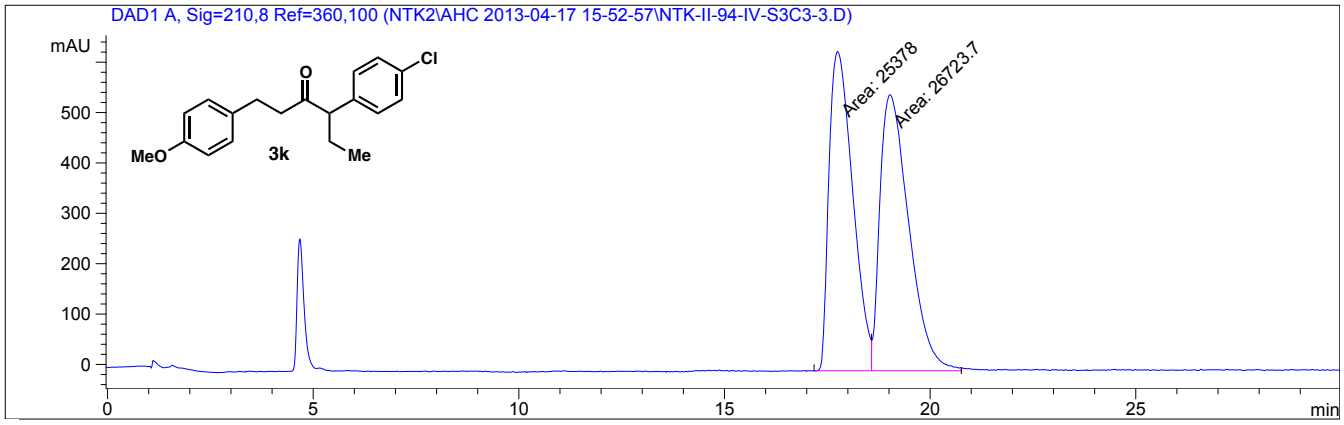
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.009	MM	0.2291	5341.29736	388.53415	49.8184
2	6.837	MM	0.2540	5380.24316	353.03430	50.1816

3j (Table 2, entry 10): enantioenriched, 94% ee



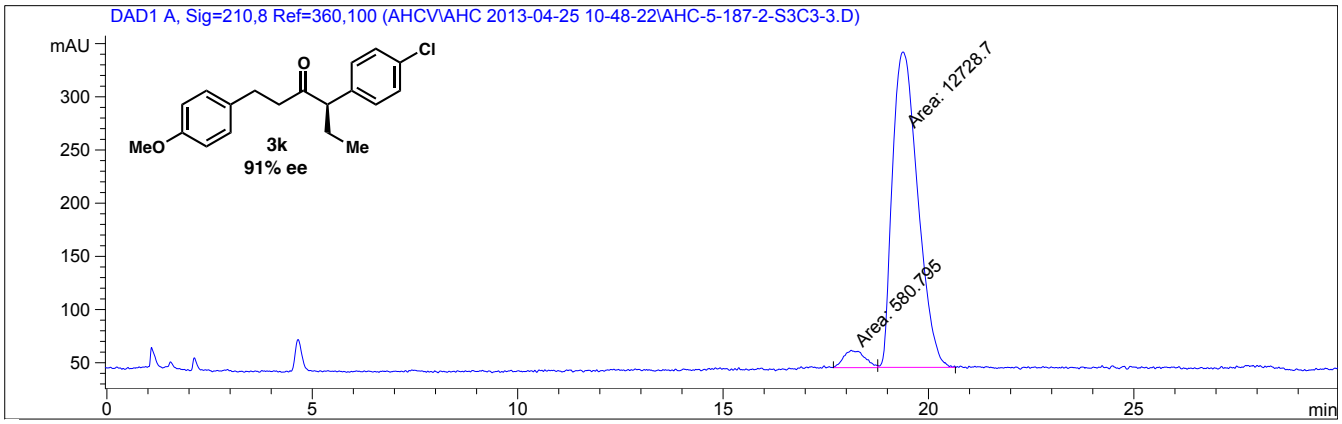
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.179	MM	0.1983	422.02435	35.47295	3.1063
2	6.898	MM	0.2556	1.31640e4	858.35632	96.8937

3k (Table 2, entry 11): racemic



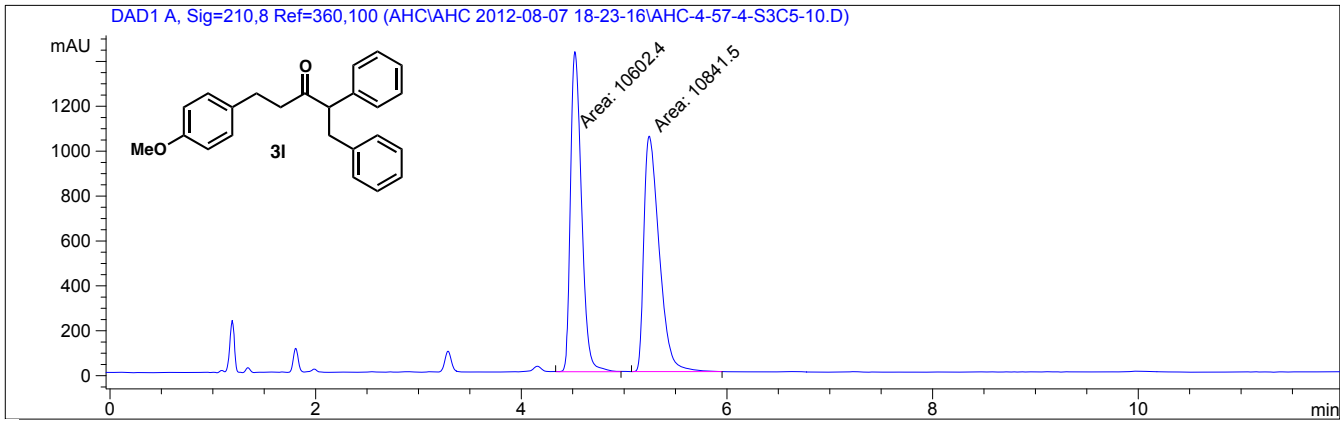
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.749	MM	0.6673	2.53780e4	633.81763	48.7086
2	19.022	MM	0.8126	2.67237e4	548.09839	51.2914

3k (Table 2, entry 11): enantioenriched, 91% ee



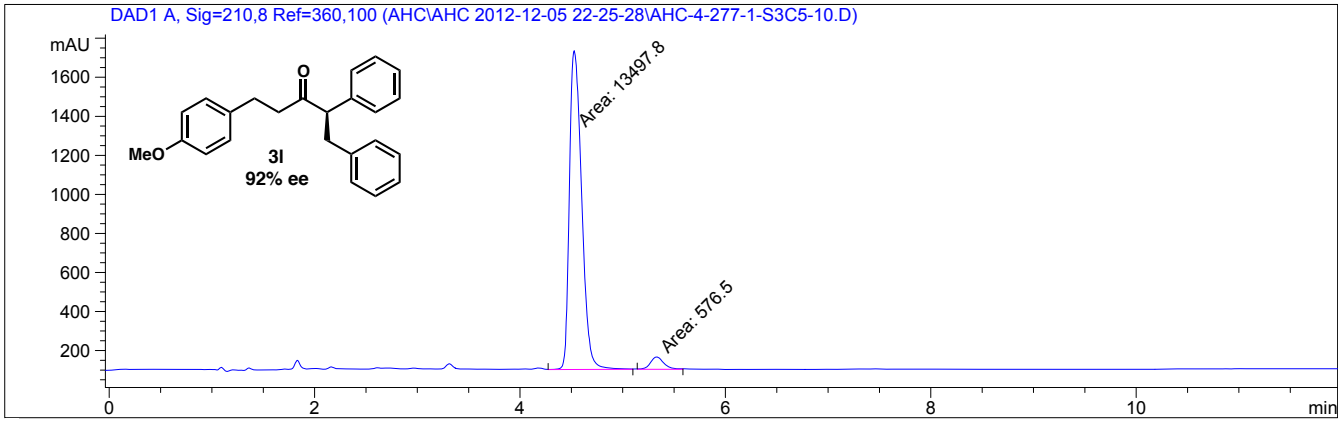
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.118	MM	0.5944	580.79498	16.28630	4.3637
2	19.381	MM	0.7154	1.27287e4	296.55020	95.6363

3l (Table 2, entry 12): racemic



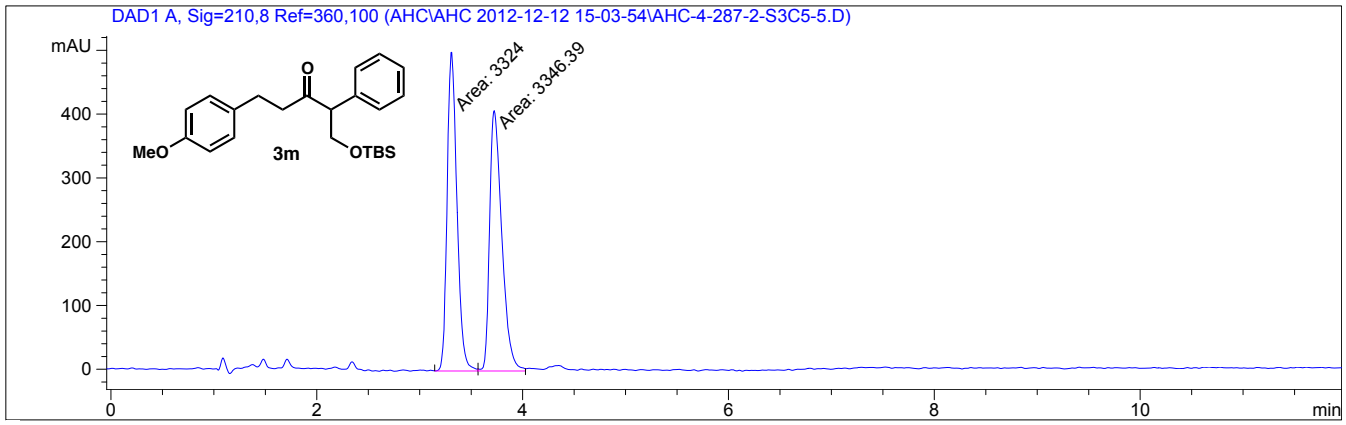
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.522	MM	0.1239	1.06024e4	1426.70251	49.4425
2	5.244	MM	0.1721	1.08415e4	1050.01685	50.5575

3l (Table 2, entry 12): enantioenriched, 92% ee



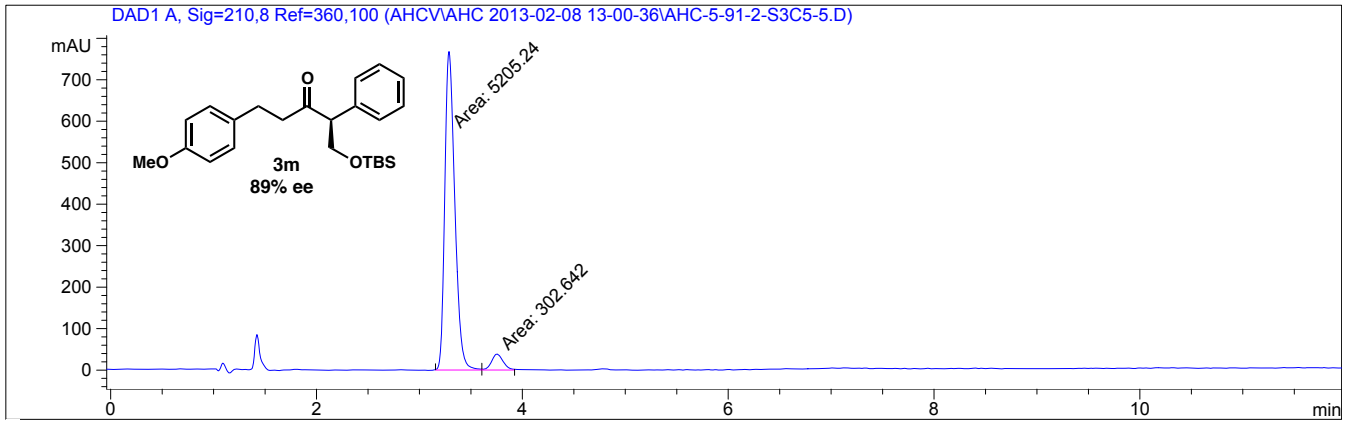
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.528	MM	0.1377	1.34978e4	1634.09204	95.9039
2	5.330	MM	0.1513	576.49988	63.49461	4.0961

3m (Table 2, entry 13): racemic



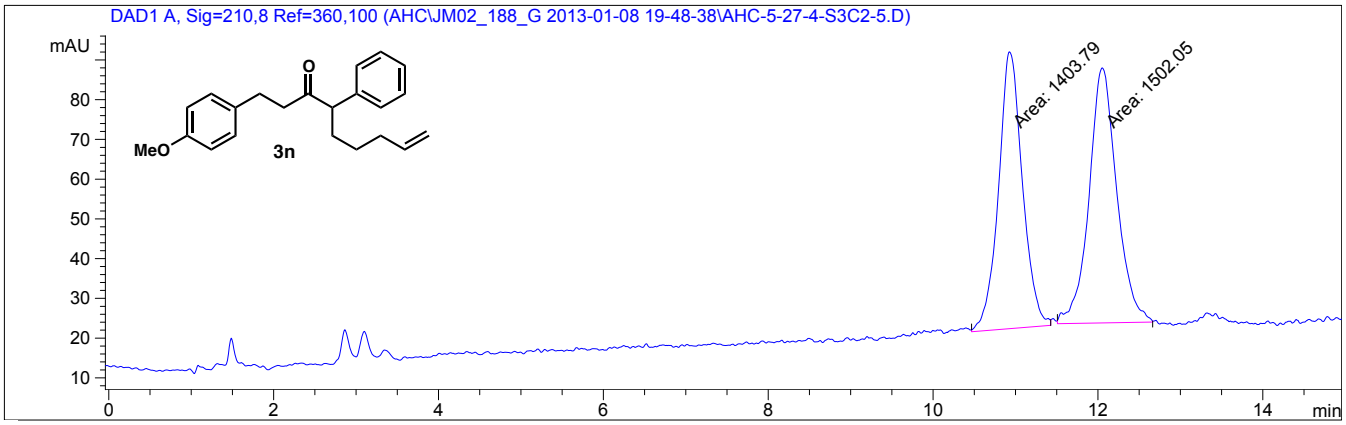
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.308	MM	0.1105	3324.00073	501.56870	49.8322
2	3.723	MM	0.1365	3346.39185	408.55920	50.1678

3m (Table 2, entry 13): enantioenriched, 89% ee



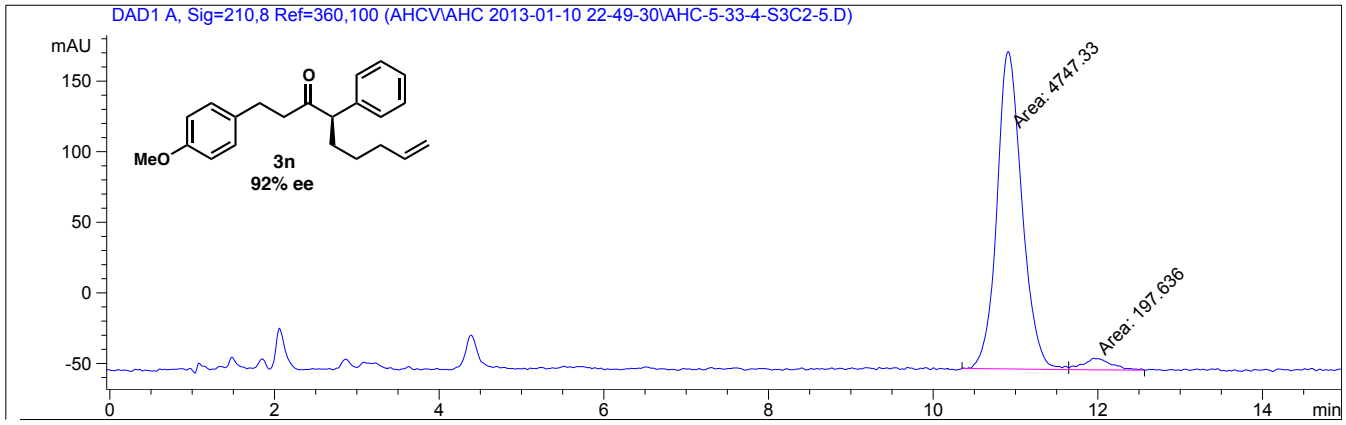
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.287	MM	0.1129	5205.23877	768.54687	94.5053
2	3.754	MM	0.1313	302.64224	38.40394	5.4947

3n (Table 2, entry 14): racemic



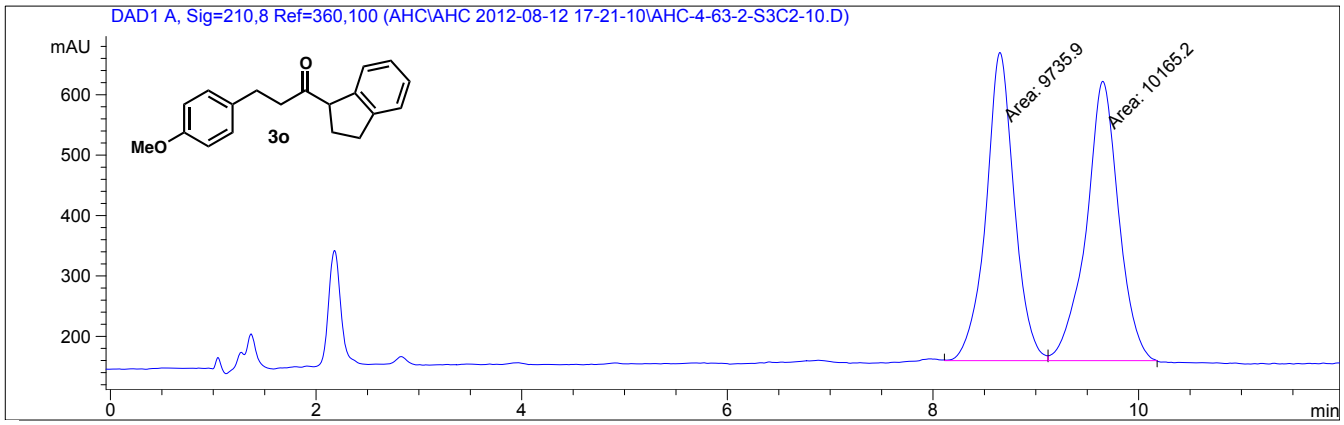
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.925	MM	0.3357	1403.79077	69.69751	48.3093
2	12.051	MM	0.3898	1502.04858	64.21613	51.6907

3n (Table 2, entry 14): enantioenriched, 92% ee



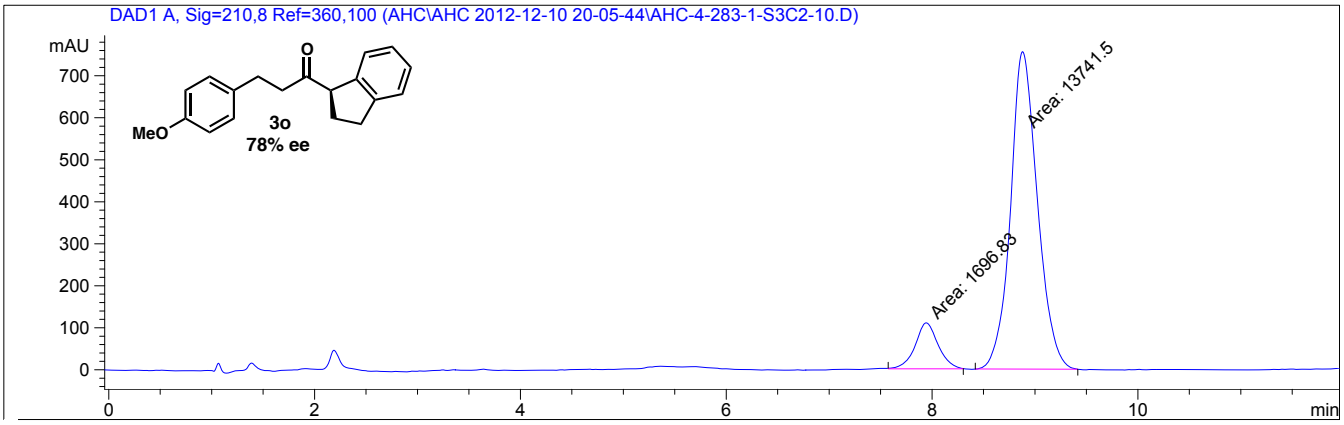
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.911	MM	0.3519	4747.33496	224.86562	96.0033
2	11.946	MM	0.4028	197.63620	8.17804	3.9967

3o (Table 2, entry 15): racemic



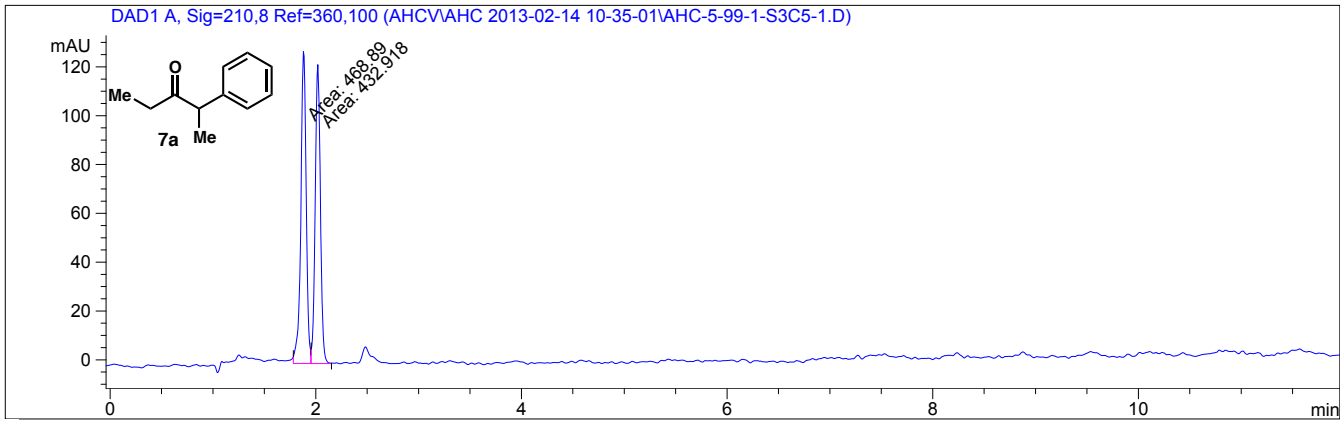
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.651	MM	0.3179	9735.89941	510.35568	48.9213
2	9.651	MM	0.3663	1.01652e4	462.52780	51.0787

3o (Table 2, entry 15): enantioenriched, 78% ee



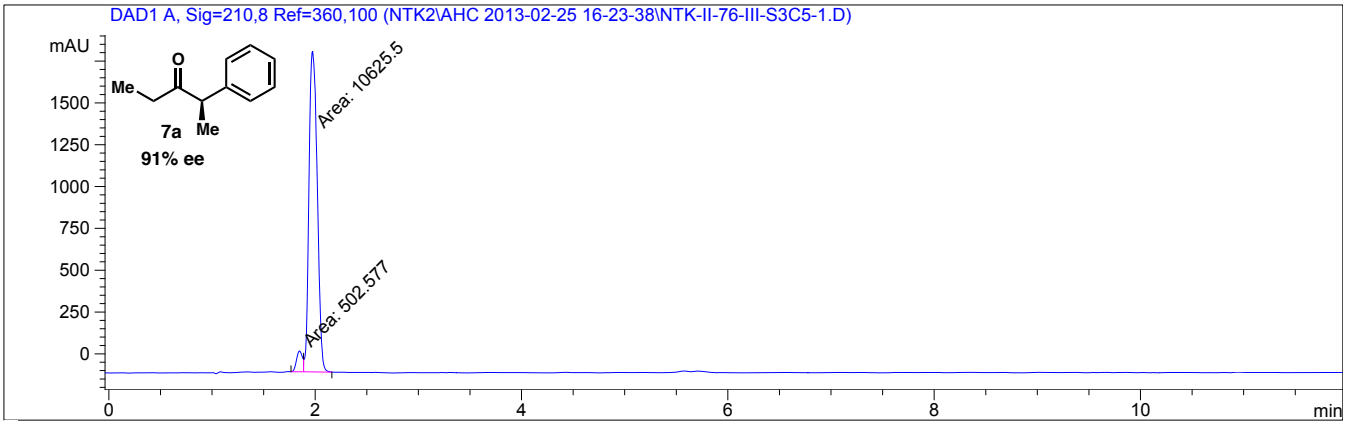
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.943	MM	0.2586	1696.82703	109.37256	10.9910
2	8.880	MM	0.3030	1.37415e4	755.81702	89.0090

7a (Table 3): racemic



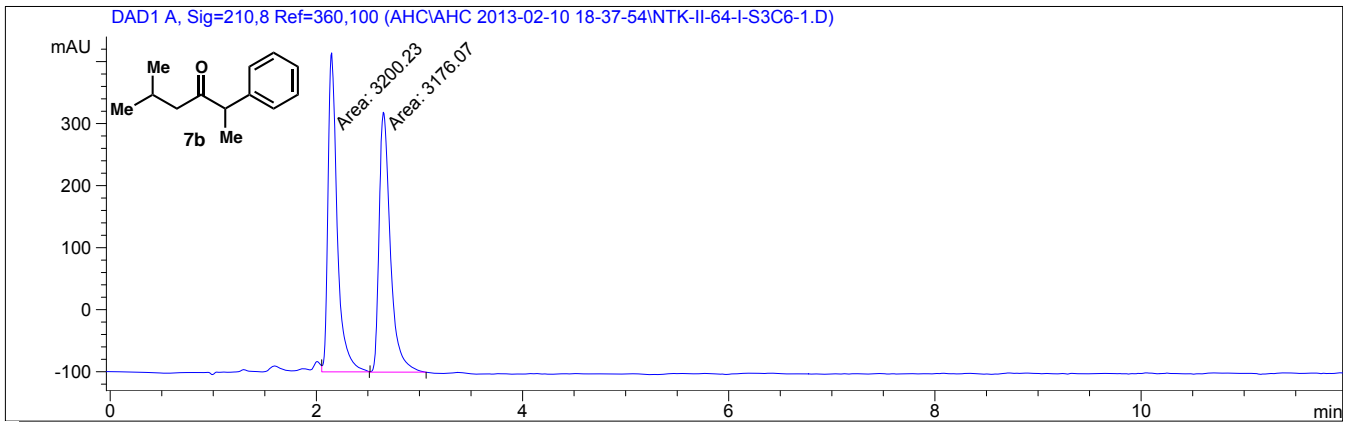
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.882	MM	0.0607	468.89008	128.69202	51.9944
2	2.020	MM	0.0587	432.91794	122.82136	48.0056

7a (Table 3): enantioenriched, 89% ee



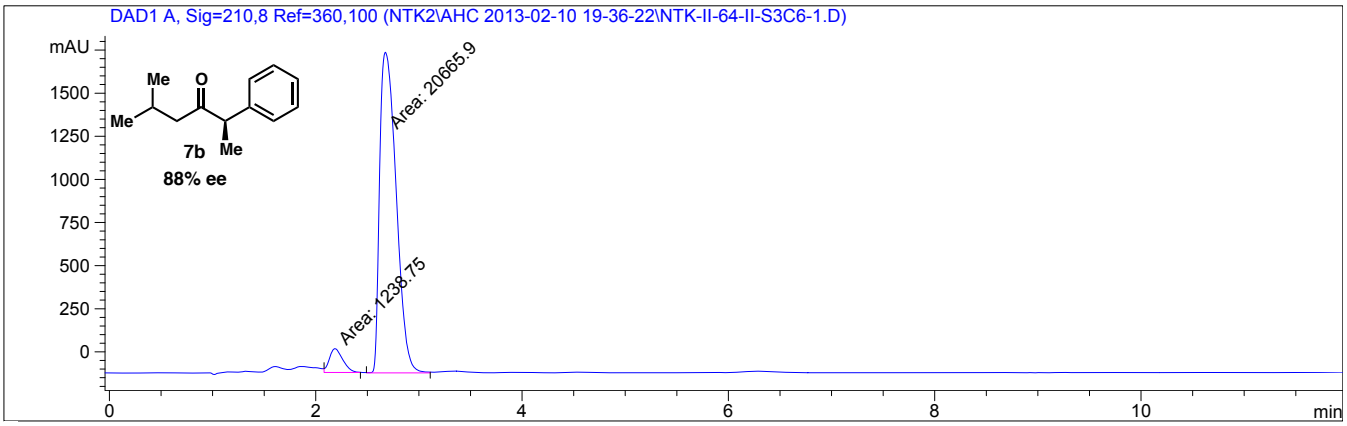
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.848	MM	0.0672	502.57666	124.59120	4.5163
2	1.973	MM	0.0923	1.06255e4	1918.92407	95.4837

7b (Table 3): racemic



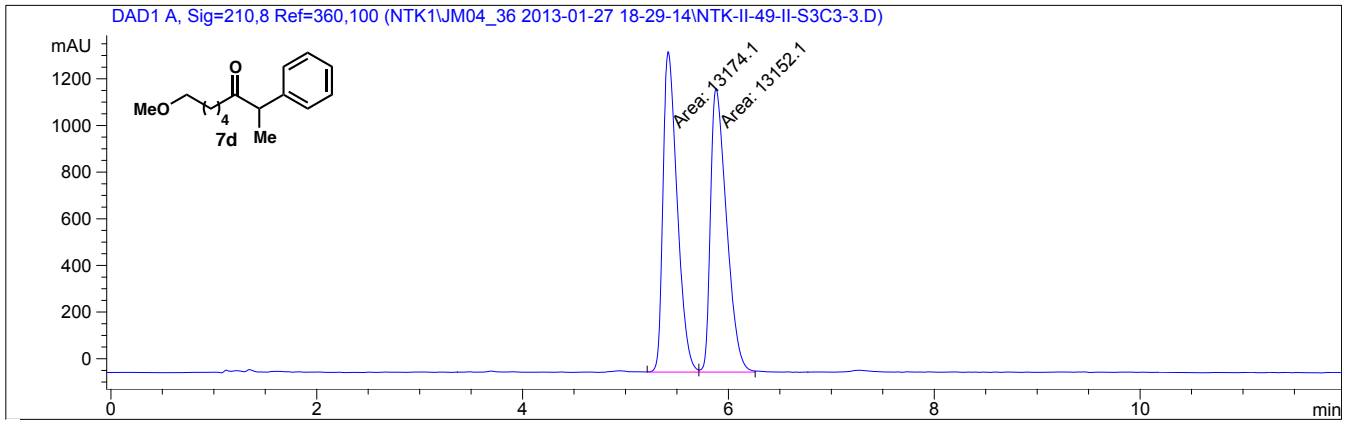
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.147	MM	0.1035	3200.22705	515.47467	50.1894
2	2.651	MM	0.1261	3176.07007	419.70117	49.8106

7b (Table 3): enantioenriched, 88% ee



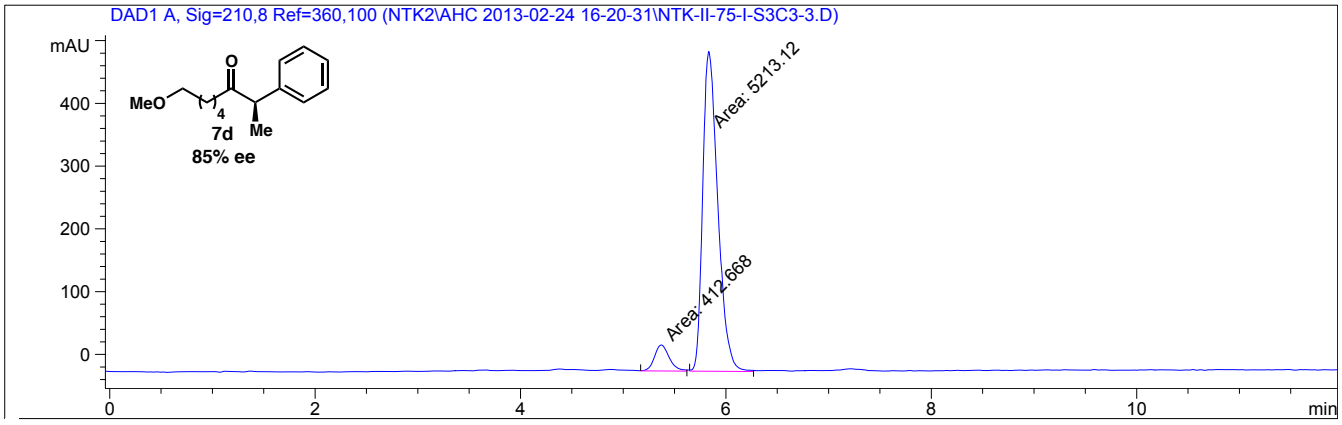
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.186	MM	0.1493	1238.74841	138.30533	5.6552
2	2.675	MM	0.1852	2.06659e4	1859.45630	94.3448

7d (Table 3): racemic



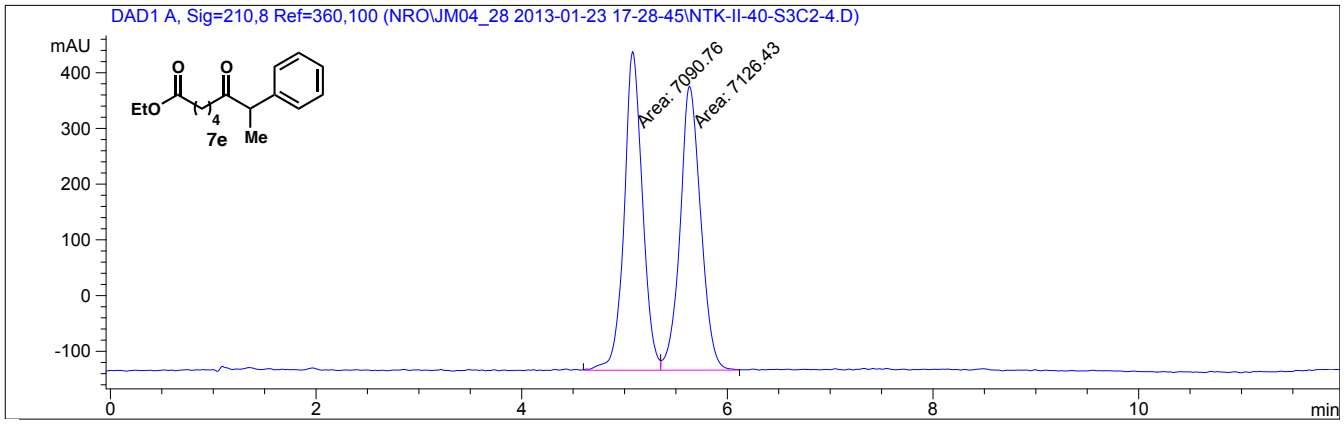
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.415	MM	0.1596	1.31741e4	1375.35034	50.0417
2	5.880	MM	0.1802	1.31521e4	1216.19214	49.9583

7d (Table 3): enantioenriched, 85% ee



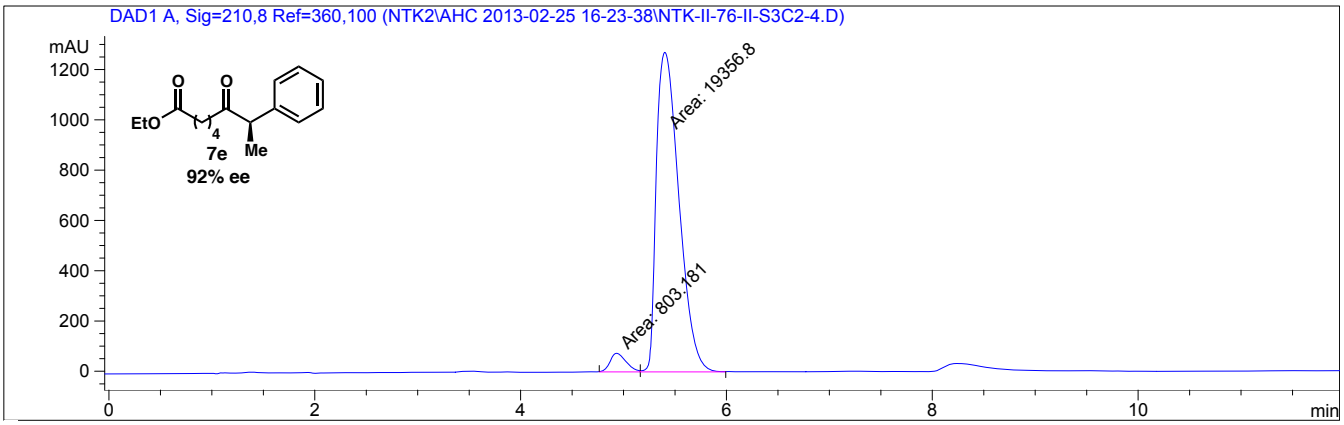
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.371	MM	0.1661	412.66797	41.40784	7.3353
2	5.833	MM	0.1703	5213.12402	510.12839	92.6647

7e (Table 3): racemic



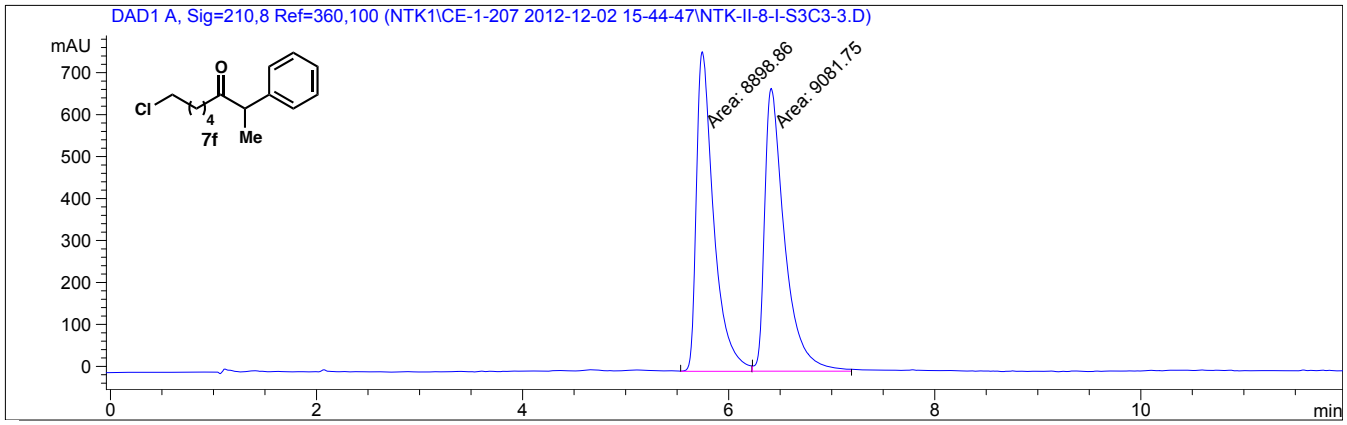
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.079	MM	0.2066	7090.76221	572.01874	49.8746
2	5.631	MM	0.2330	7126.43066	509.79788	50.1254

7e (Table 3): enantioenriched, 92% ee



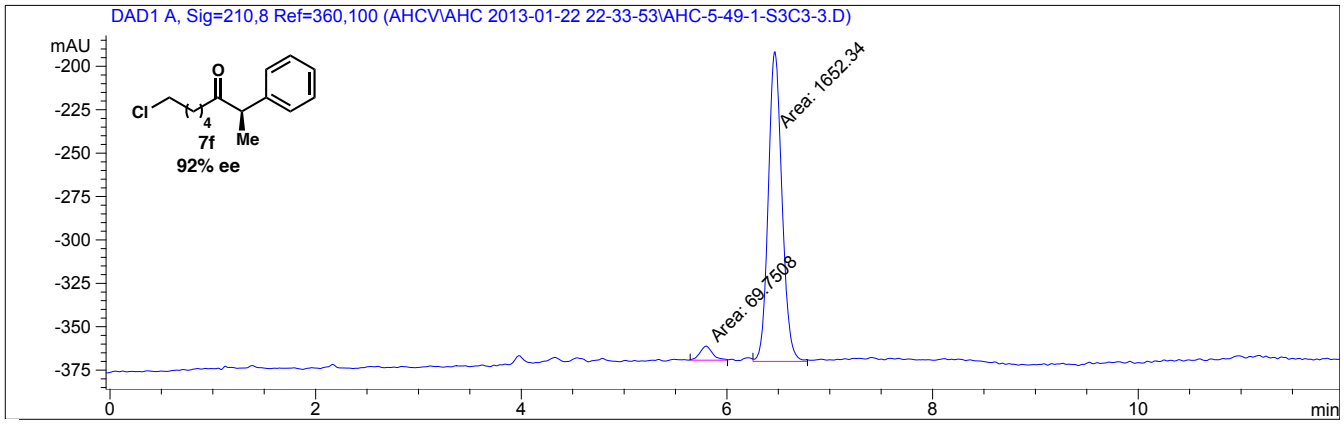
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.932	MM	0.1805	803.18146	74.16300	3.9840
2	5.401	MM	0.2538	1.93568e4	1271.19958	96.0160

7f (Table 3): racemic



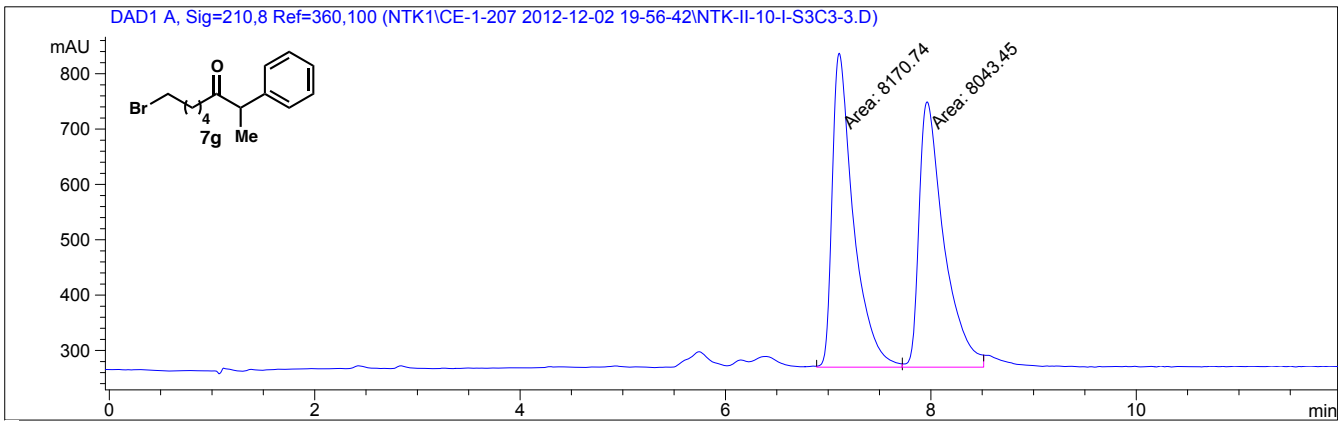
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.744	MM	0.1945	8898.86133	762.63263	49.4914
2	6.413	MM	0.2244	9081.74902	674.62744	50.5086

7f (Table 3): enantioenriched, 92% ee



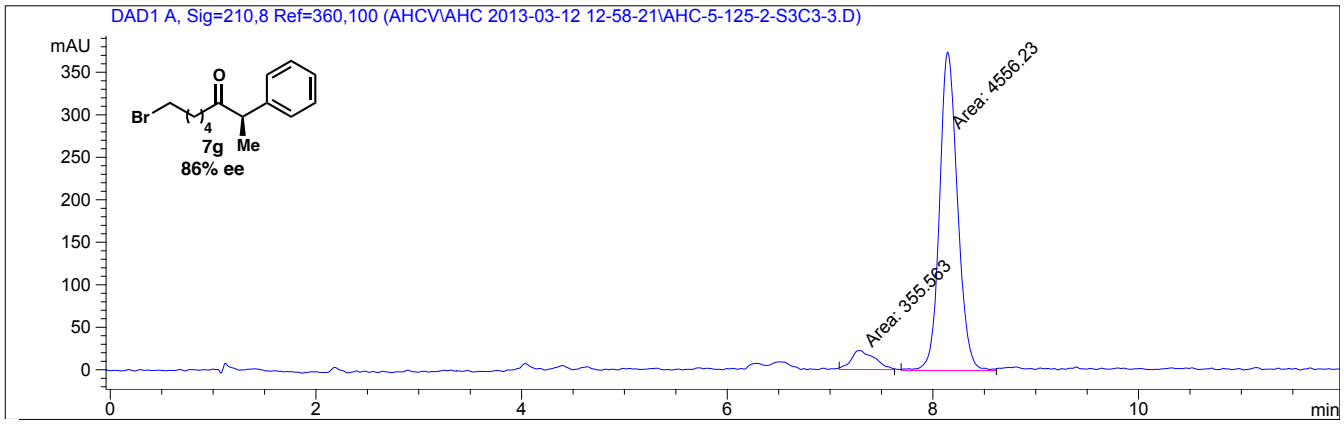
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.798	MM	0.1418	69.75076	8.19656	4.0504
2	6.465	MM	0.1542	1652.33777	178.58195	95.9496

7g (Table 3): racemic



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.107	MM	0.2399	8170.74316	567.72528	50.3925
2	7.963	MM	0.2795	8043.45215	479.56842	49.6075

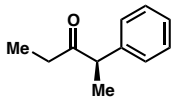
7g (Table 3): enantioenriched, 86% ee



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.295	MM	0.2652	355.56323	22.34372	7.2390
2	8.144	MM	0.2027	4556.22852	374.71005	92.7610

6. Assignment of Absolute Configuration

(*R*)-2-Phenylpentan-3-one (7a) The optical rotation of the product generated in the presence of (*R*,*R*)-**L1** was measured: $[\alpha]_D^{25} = -225.9°$ (c = 0.57, $CHCl_3$). (*R*) isomer: Lit. $[\alpha]_D^{25} = -76$ (c = 1.2, $CHCl_3$; 95% ee),[2] $[\alpha]_D^{21} = -47.2$ (c = 1.00, $CHCl_3$; 73% ee).[3]

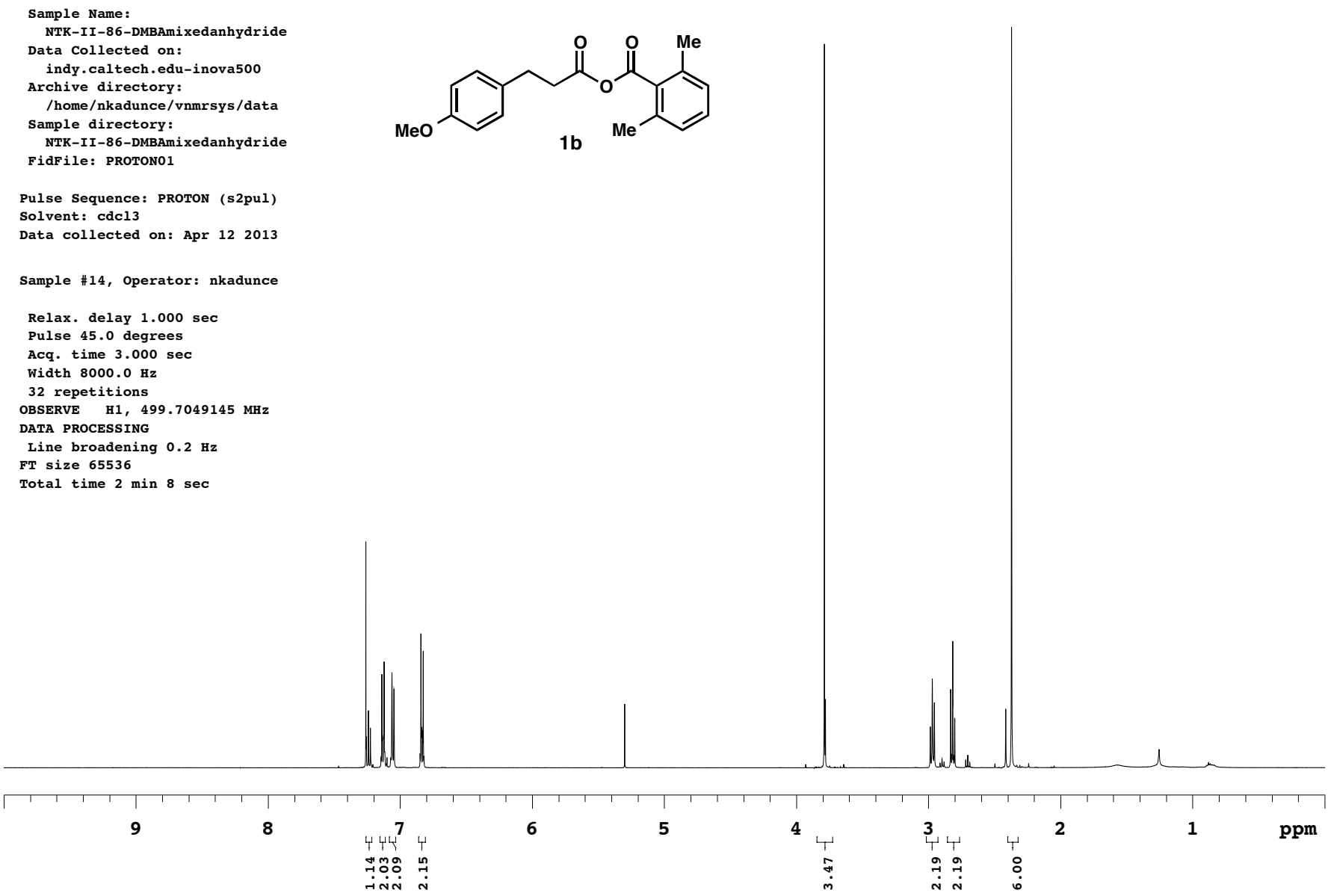


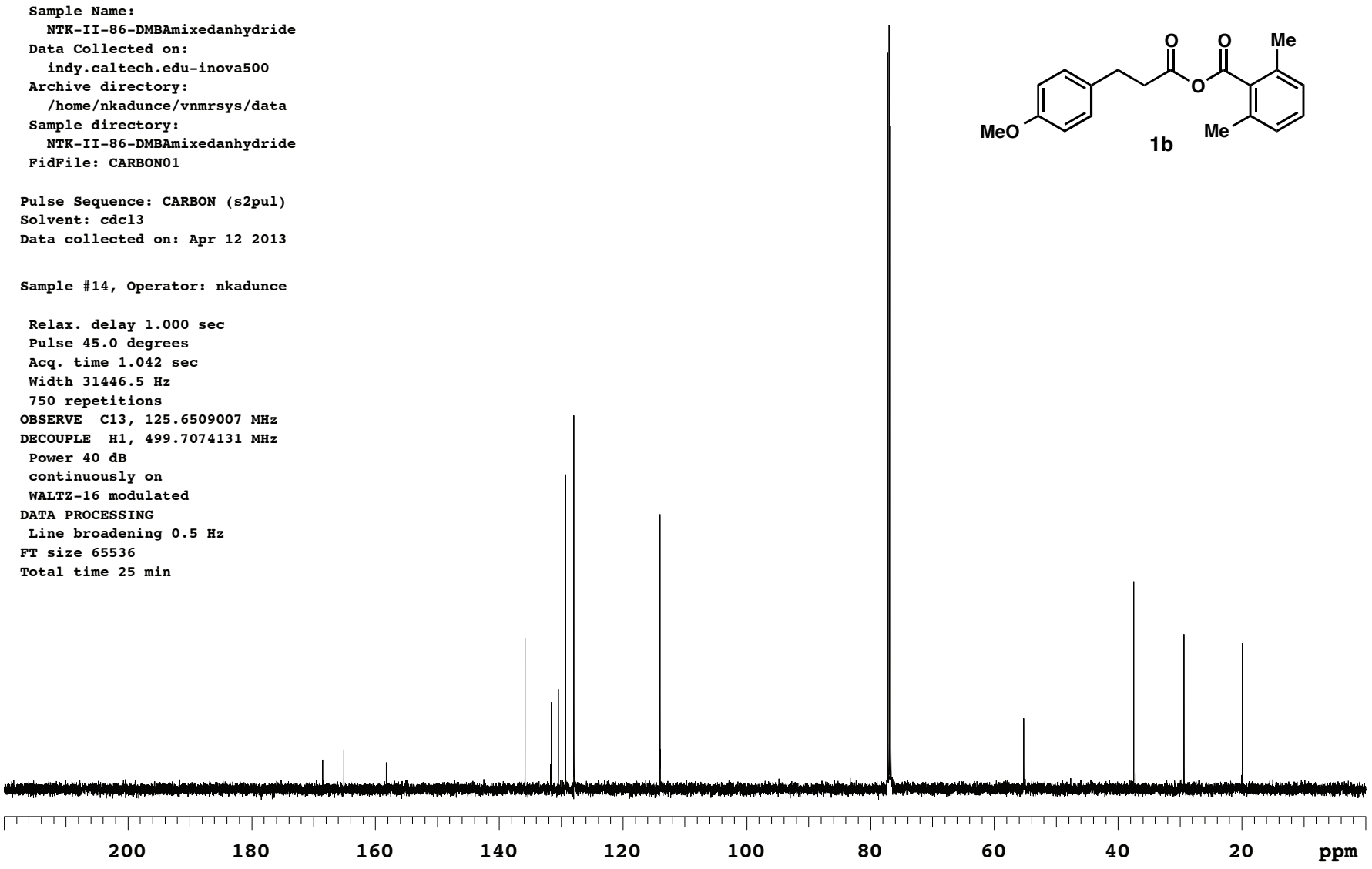
References

[1] Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem*. **1978**, *43*, 2923.

[2] Rodríguez, C.; de Gonzalo, G.; Fraaije, M. W.; Gotor, V. *Tetrahedron: Asymmetry* **2007**, *18*, 1338.

[3] Lou, S.; Fu, G. C. *J. Am. Chem. Soc.* **2010**, *132*, 1264.


Sample Name:
NTK-II-86-DMBAmixedanhydride
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-86-DMBAmixedanhydride
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 12 2013
Sample #14, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
O
O
Me
MeO
Me
1b
9
8
7
6
5
4
3
2
1
ppm
1.14
2.03
2.09
2.15
3.47
2.19
2.19
6.00

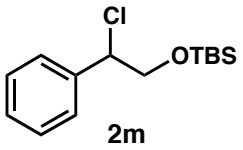

Sample Name:
NTK-II-86-DMBAmixedanhydride
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-86-DMBAmixedanhydride
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 12 2013
Sample #14, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
750 repetitions
OBSERVE C13, 125.6509007 MHz
DECOUPLE H1, 499.7074131 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min
O
O
Me
O
MeO
Me
1b
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
NTK-I-289-II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-I-289-II
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 26 2013

Sample #35, Operator: nkadunce

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



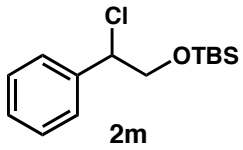
2m

Sample Name:
NTK-I-289-II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-I-289-II
FidFile: CARBON01

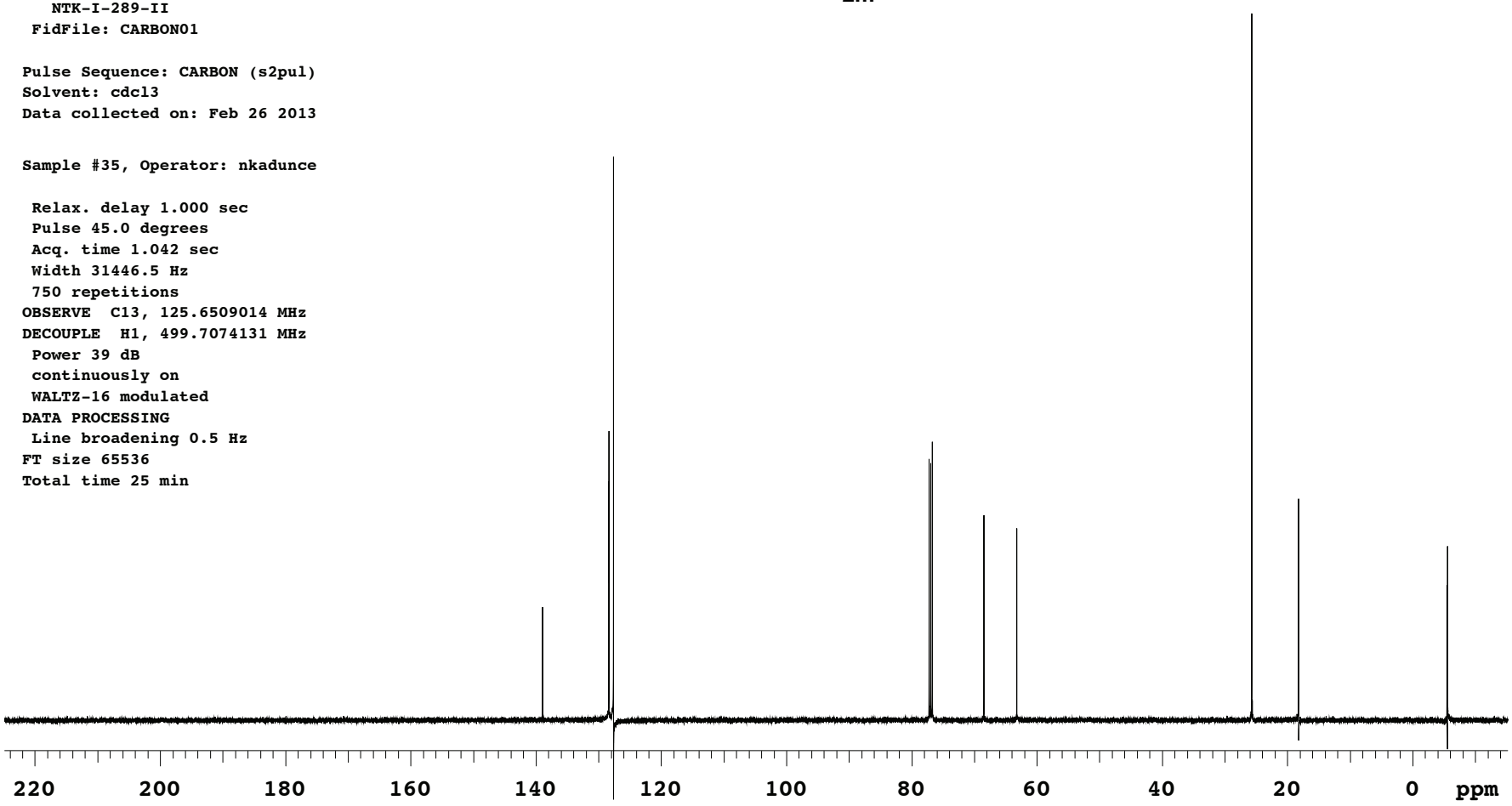
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 26 2013

Sample #35, Operator: nkadunce

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
750 repetitions
OBSERVE C13, 125.6509014 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min



2m

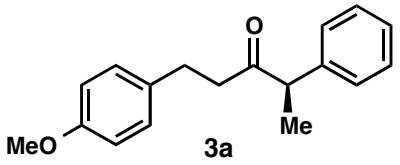


Sample Name:
ahc-3-55-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
ahc-3-55-1
FidFile: PROTON01

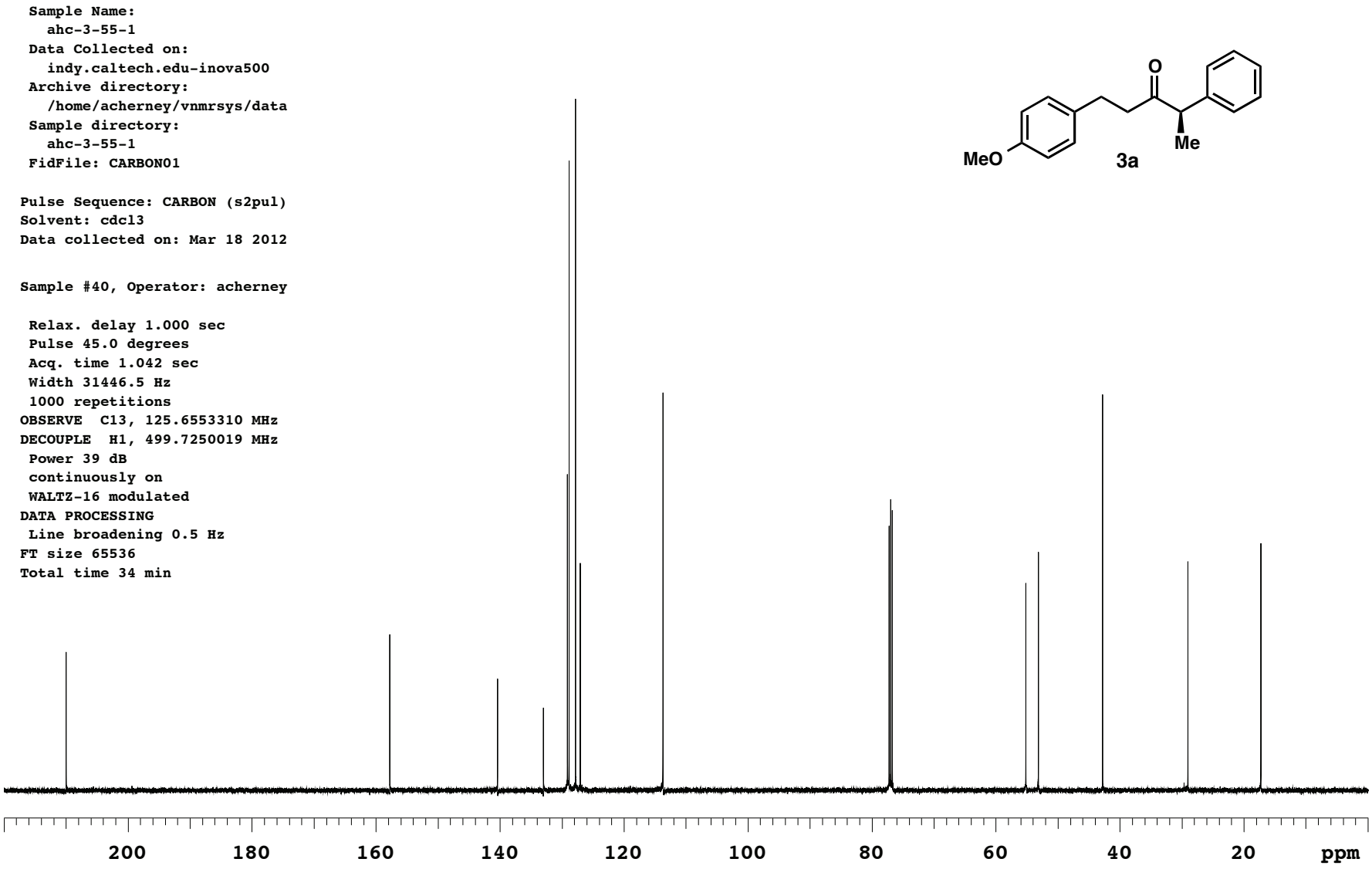
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 18 2012

Sample #40, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7225125 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min 52 sec



3a

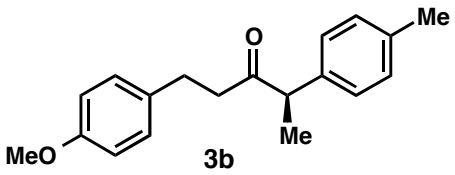

Sample Name:
ahc-3-55-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
ahc-3-55-1
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 18 2012
Sample #40, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6553310 MHz
DECOUPLE H1, 499.7250019 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
Me
MeO
3a
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-4-281-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-281-1
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 7 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



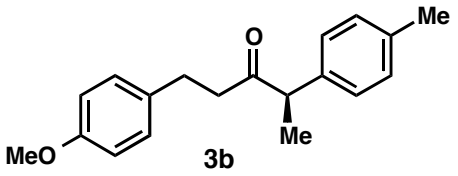
3b

Sample Name:
CH-ahc-4-281-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-281-1
FidFile: CARBON01

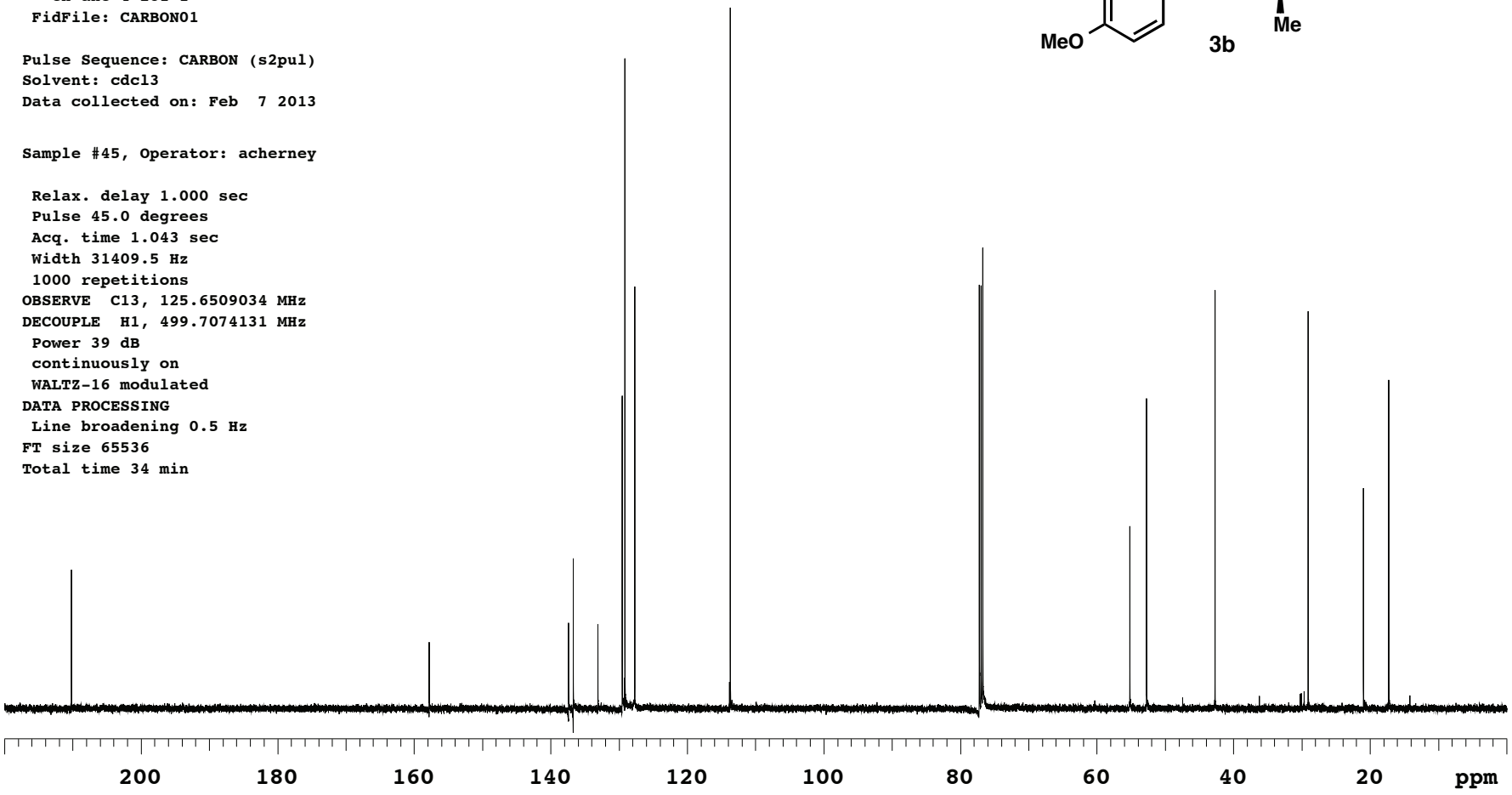
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 7 2013

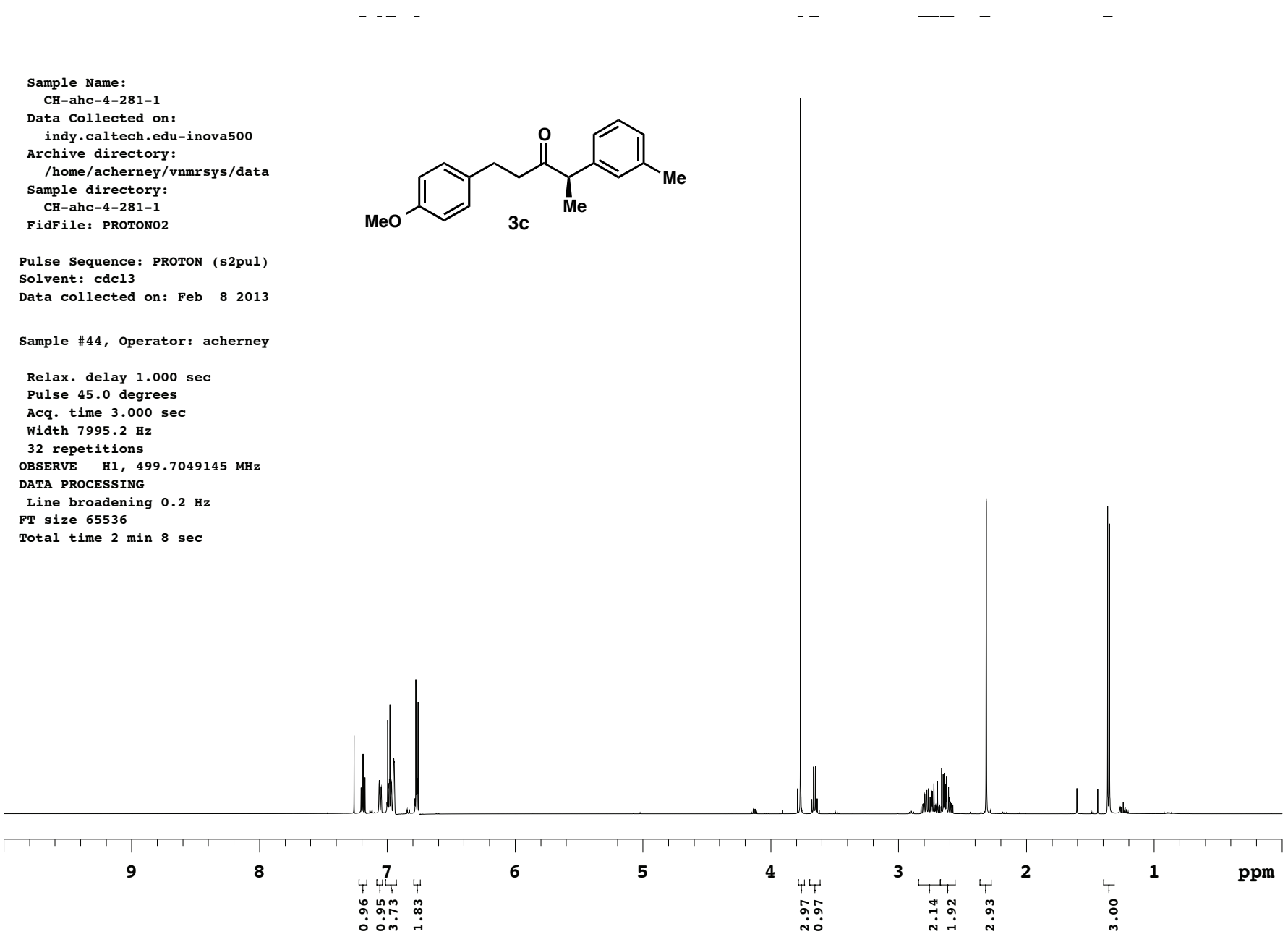
Sample #45, Operator: acherney

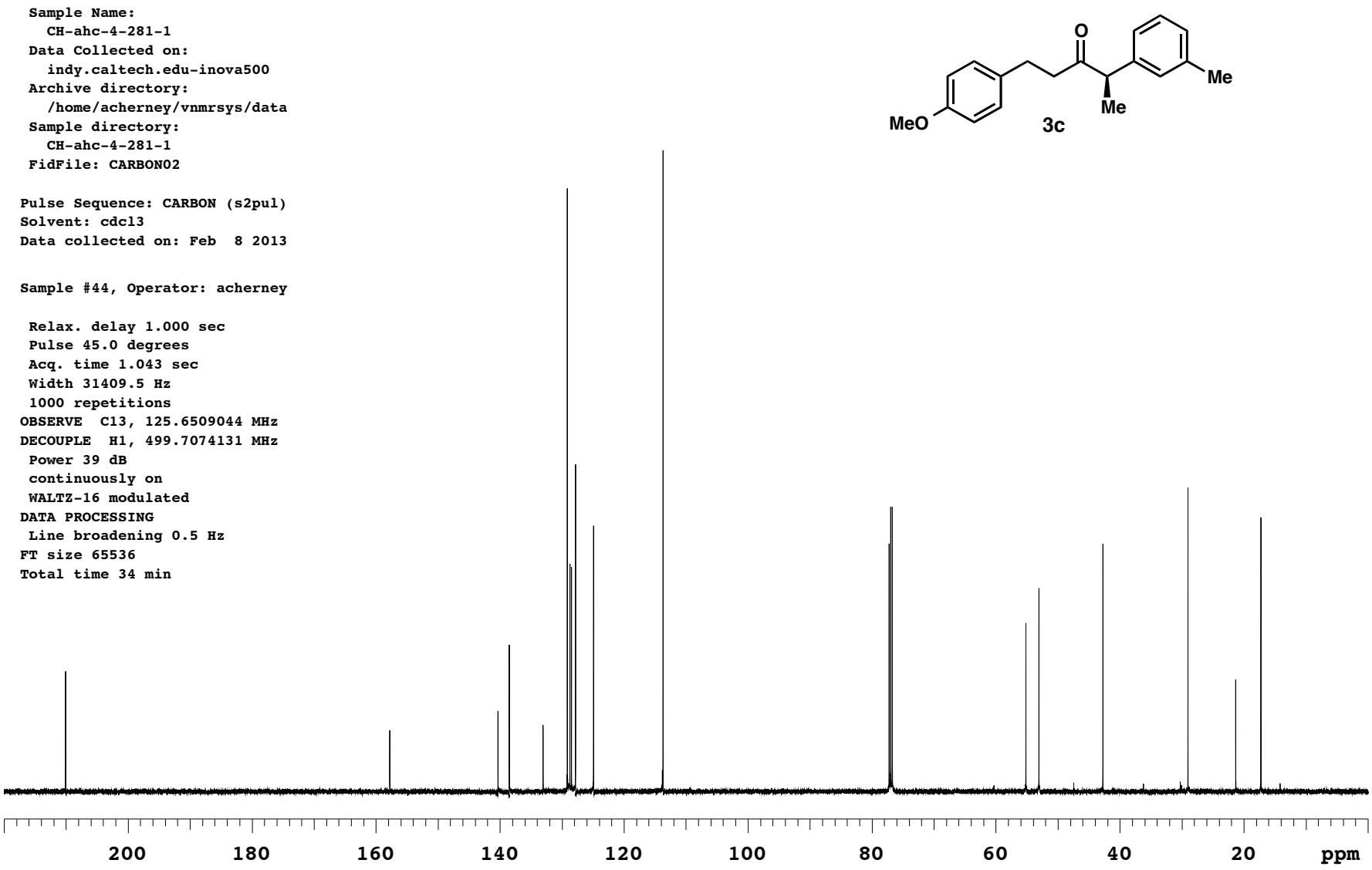
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509034 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

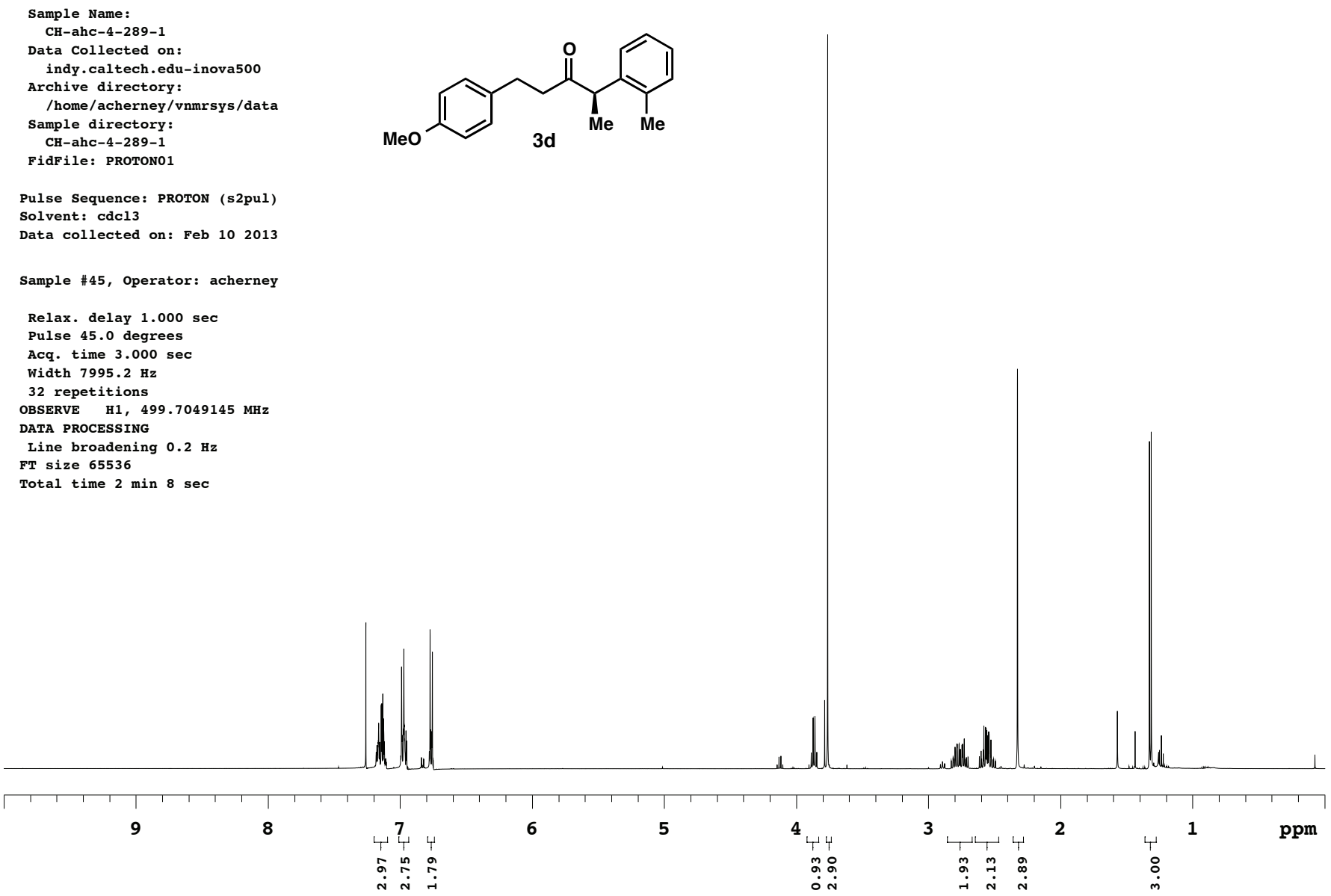


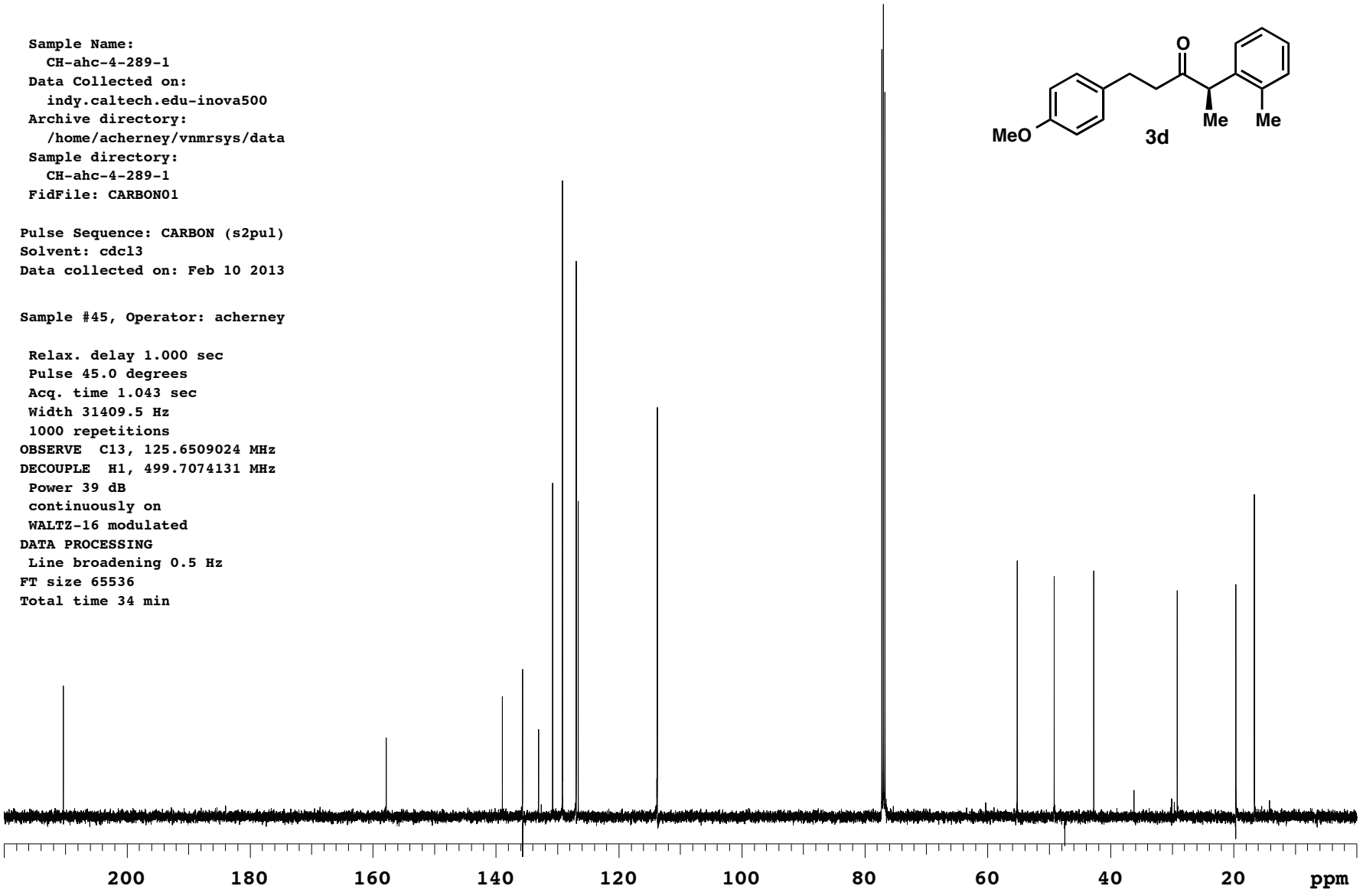
3b




Sample Name:
CH-ahc-4-281-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-281-1
FidFile: PROTON02
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 8 2013
Sample #44, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
Me
Me
MeO
3c
9
8
7
6
5
4
3
2
1
ppm
0.96
0.95
3.73
1.83
2.97
0.97
2.14
1.92
2.93
3.00


Sample Name:
CH-ahc-4-281-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-281-1
FidFile: CARBON02
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 8 2013
Sample #44, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509044 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
Me
Me
MeO
3c
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
CH-ahc-4-289-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-289-1
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
MeO
Me
Me
3d
9
8
7
6
5
4
3
2
1
ppm
2.97
2.75
1.79
0.93
2.90
1.93
2.13
2.89
3.00

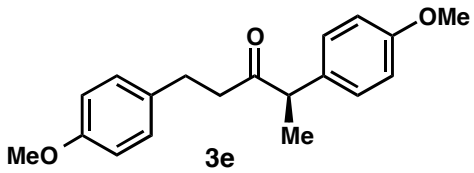

Sample Name:
CH-ahc-4-289-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-289-1
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509024 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
MeO
Me
Me
3d
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-5-53-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-53-2
FidFile: PROTON01

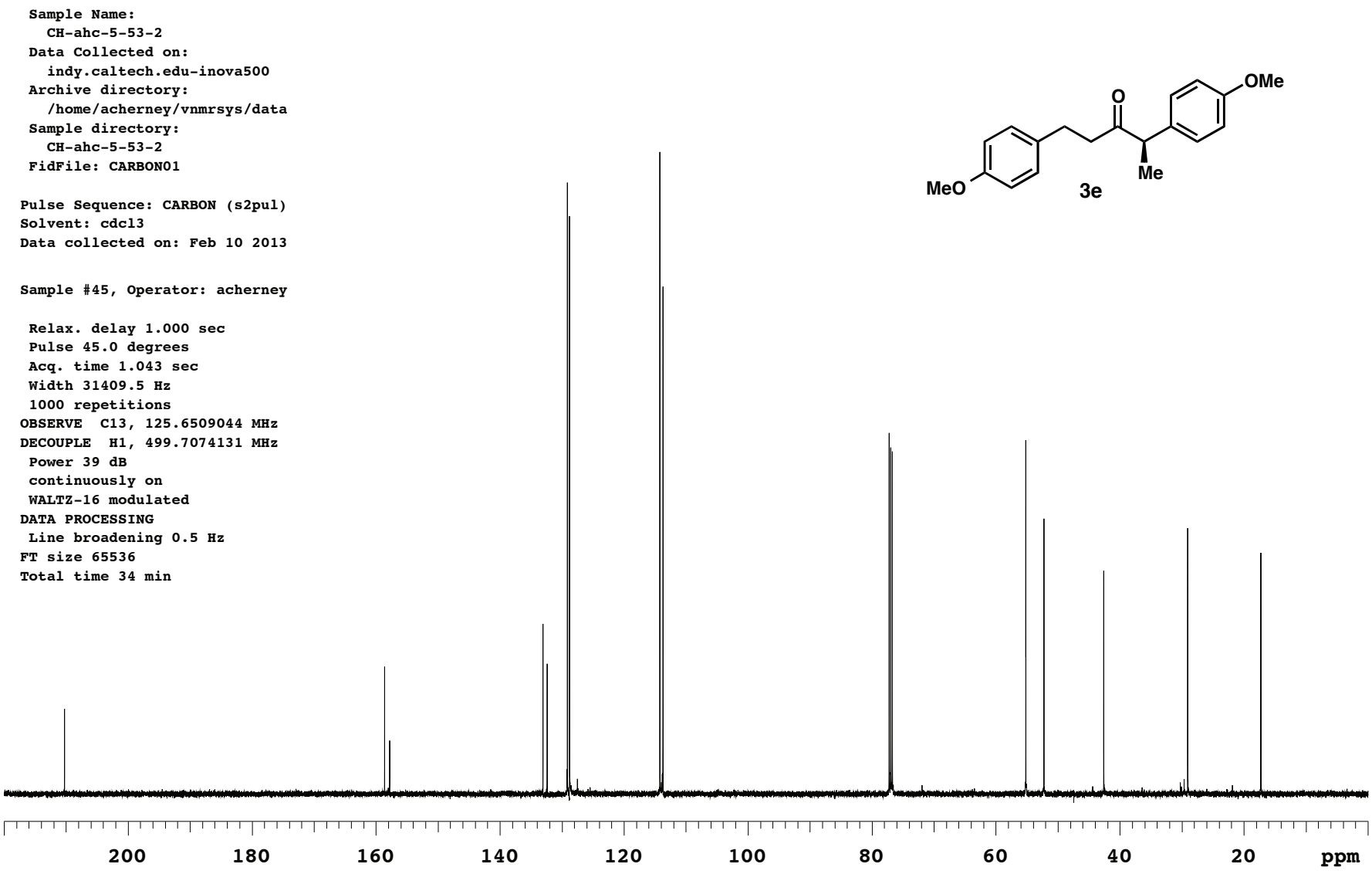
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013

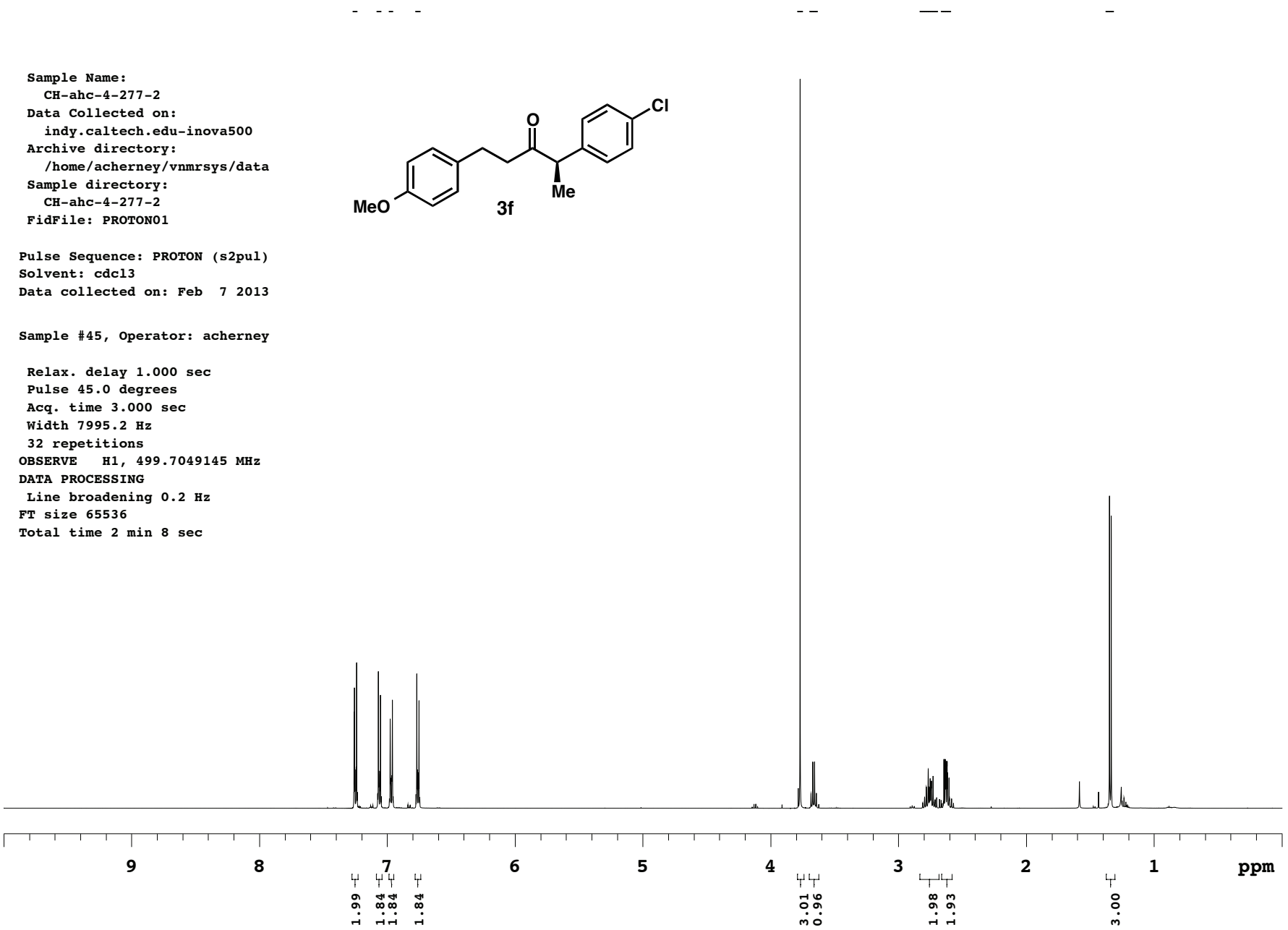
Sample #45, Operator: acherney

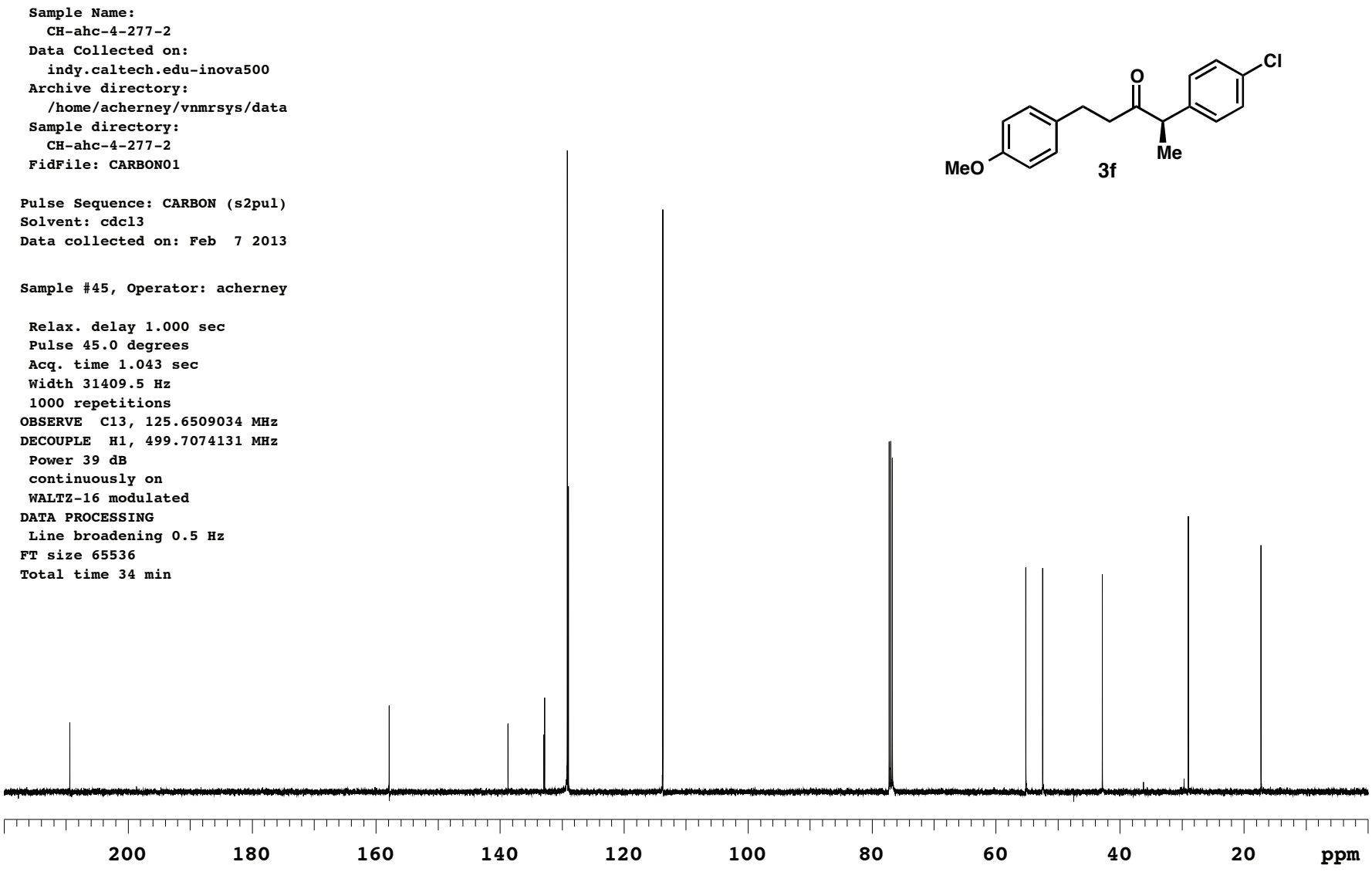
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



3e


Sample Name:
CH-ahc-5-53-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-53-2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509044 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
OMe
MeO
Me
3e
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
CH-ahc-4-277-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-277-2
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 7 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
Cl
MeO
Me
3f
9
8
7
6
5
4
3
2
1
ppm
1.99
1.84
1.84
1.84
3.01
0.96
1.98
1.93
3.00

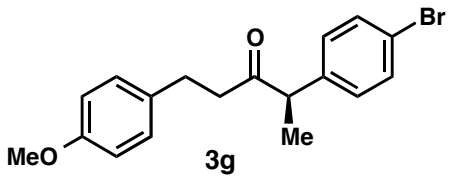

Sample Name:
CH-ahc-4-277-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-277-2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 7 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509034 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
Cl
MeO
Me
3f
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-5-17-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-17-2
FidFile: PROTON01

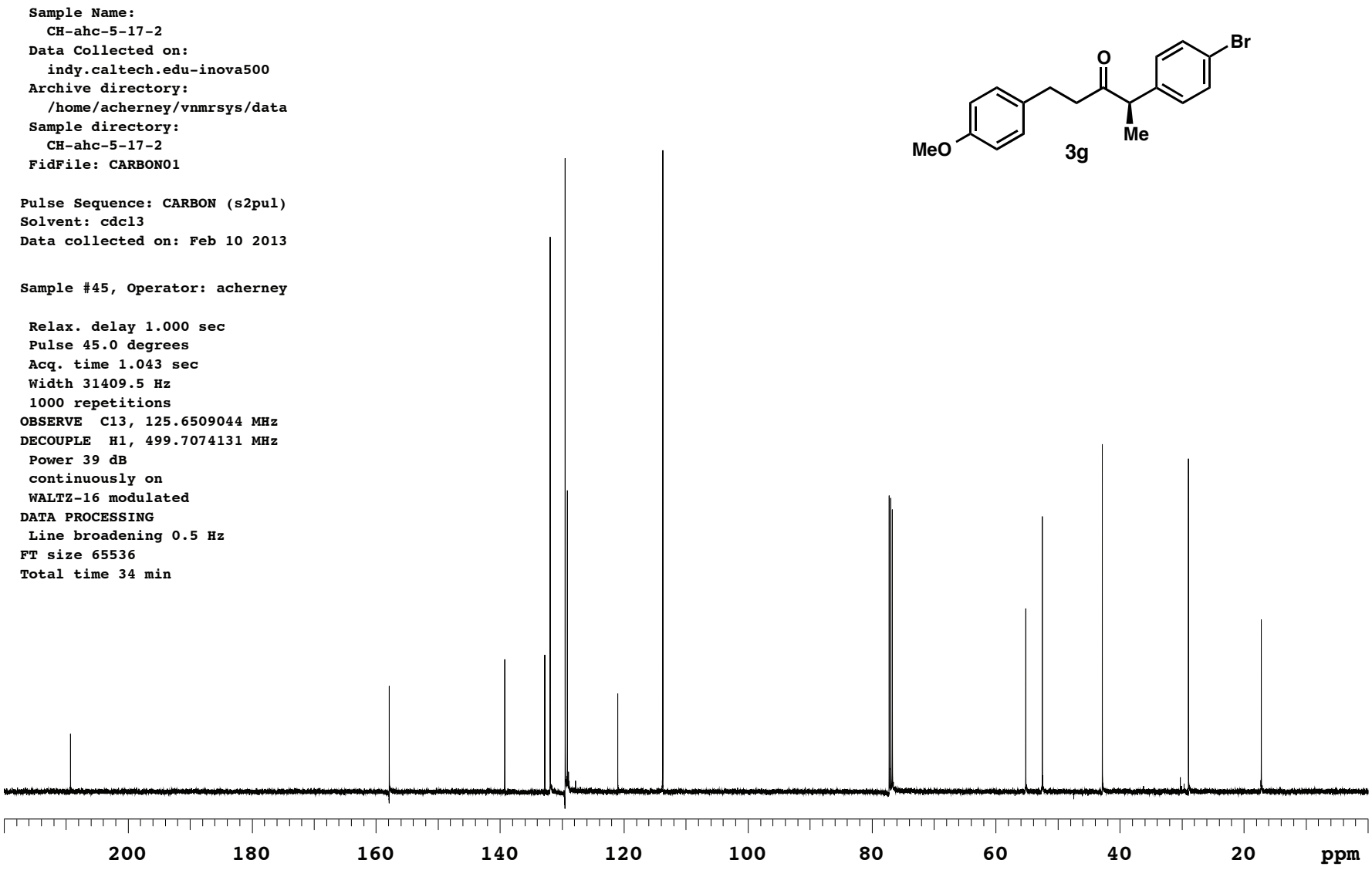
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



3g

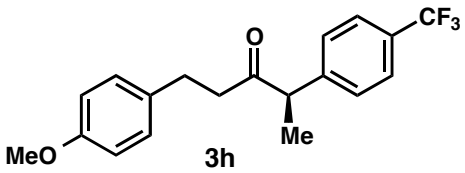

Sample Name:
CH-ahc-5-17-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-17-2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509044 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
Br
MeO
Me
3g
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-5-61-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-61-2
FidFile: PROTON01

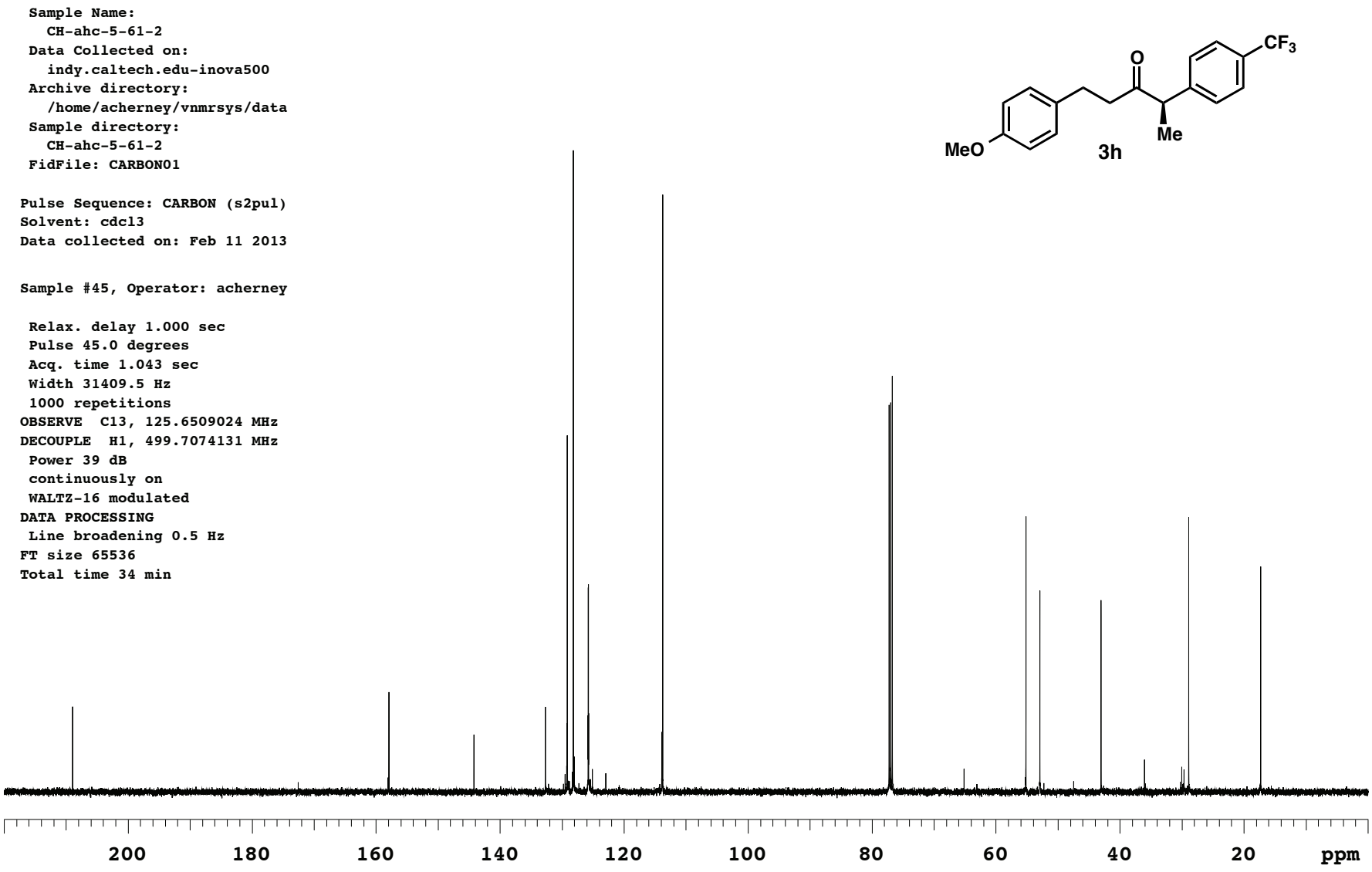
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 11 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



3h

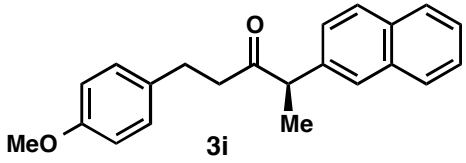

Sample Name:
CH-ahc-5-61-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-61-2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 11 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509024 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
CF3
Me
MeO
3h
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
NTK-II-NaphthylXtals
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-NaphthylXtals
FidFile: PROTON01

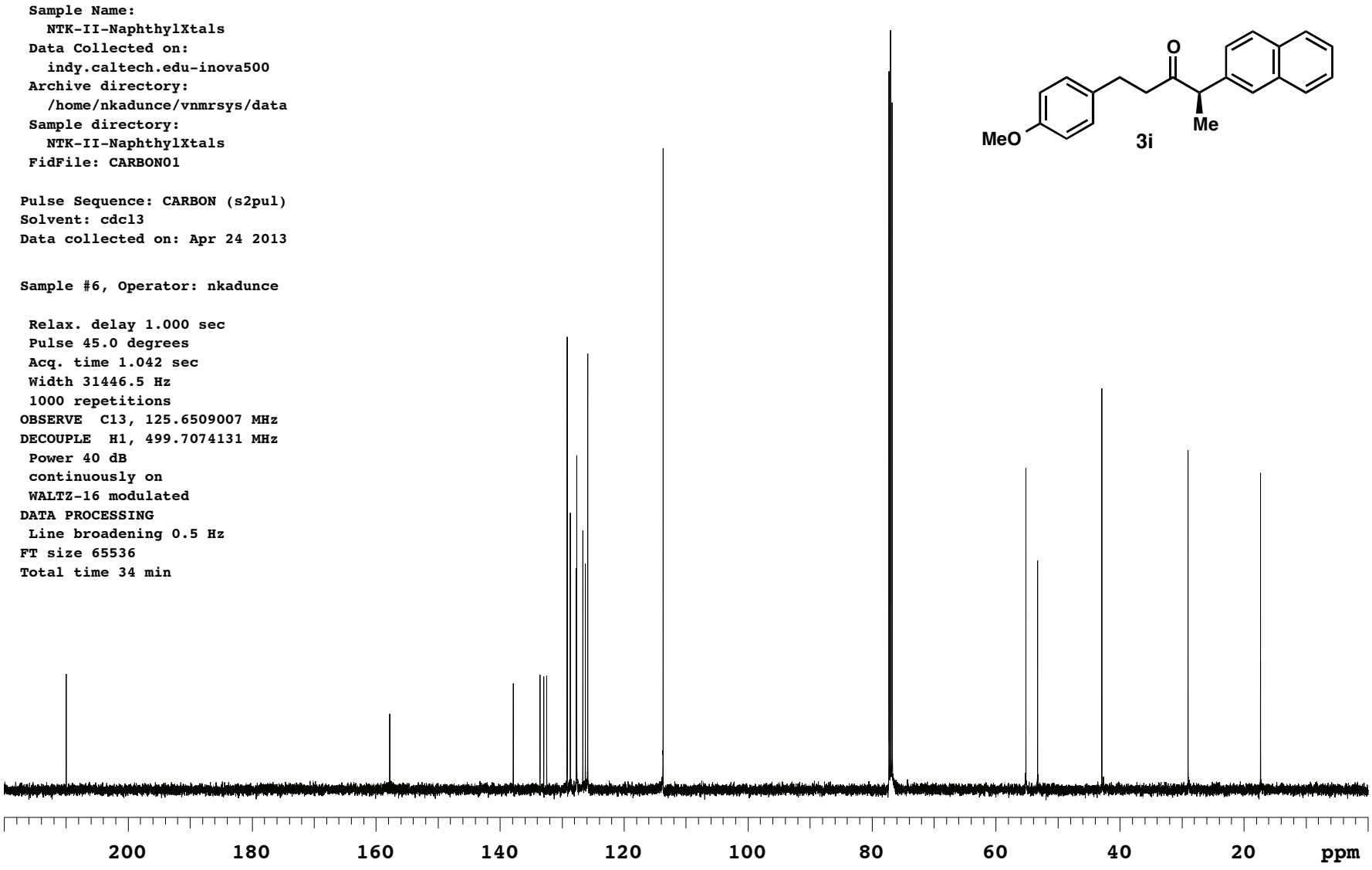
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 24 2013

Sample #6, Operator: nkadunce

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 17 sec



3i

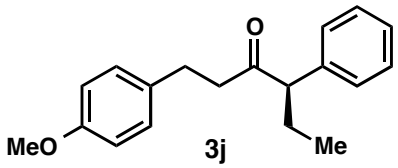

Sample Name:
NTK-II-NaphthylXtals
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-NaphthylXtals
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 24 2013
Sample #6, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509007 MHz
DECOUPLE H1, 499.7074131 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
MeO
Me
3i
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-4-285-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-285-2
FidFile: PROTON01

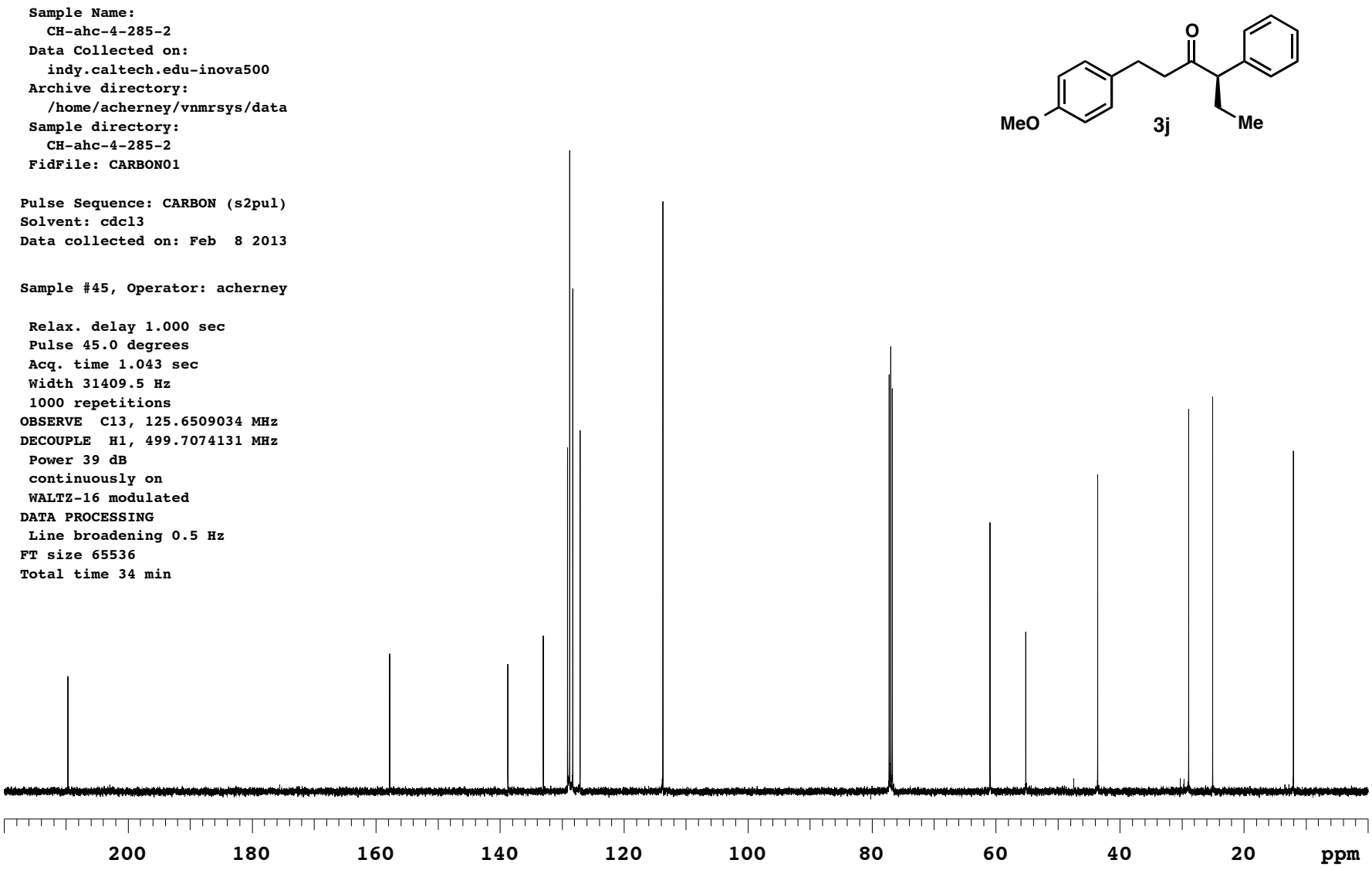
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 8 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



3j

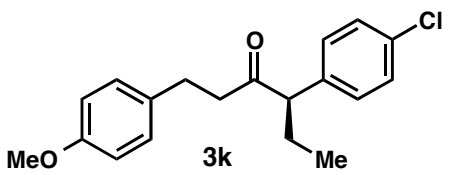

Sample Name:
CH-ahc-4-285-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-285-2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 8 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509034 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
MeO
3j
Me
200
180
160
140
120
100
80
60
40
20
ppm

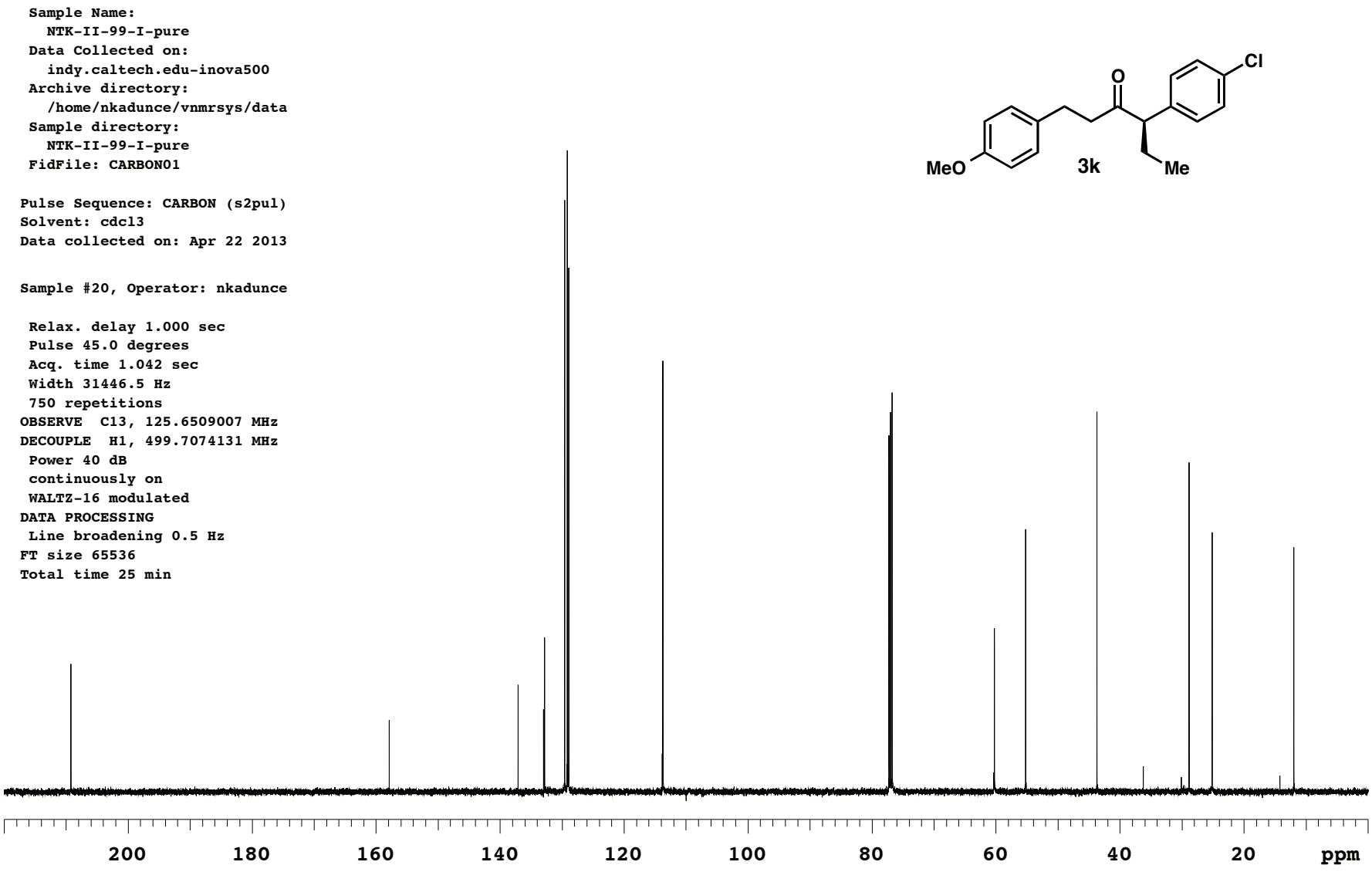
Sample Name:
NTK-II-99-I-pure
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-99-I-pure
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 22 2013

Sample #20, Operator: nkadunce

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



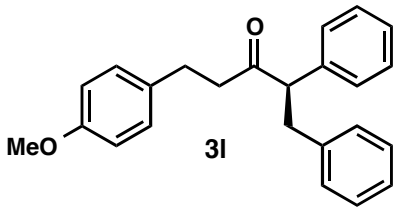

Sample Name:
NTK-II-99-I-pure
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-99-I-pure
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 22 2013
Sample #20, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
750 repetitions
OBSERVE C13, 125.6509007 MHz
DECOUPLE H1, 499.7074131 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min
O
Cl
MeO
3k
Me
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-4-277-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-277-1
FidFile: PROTON01

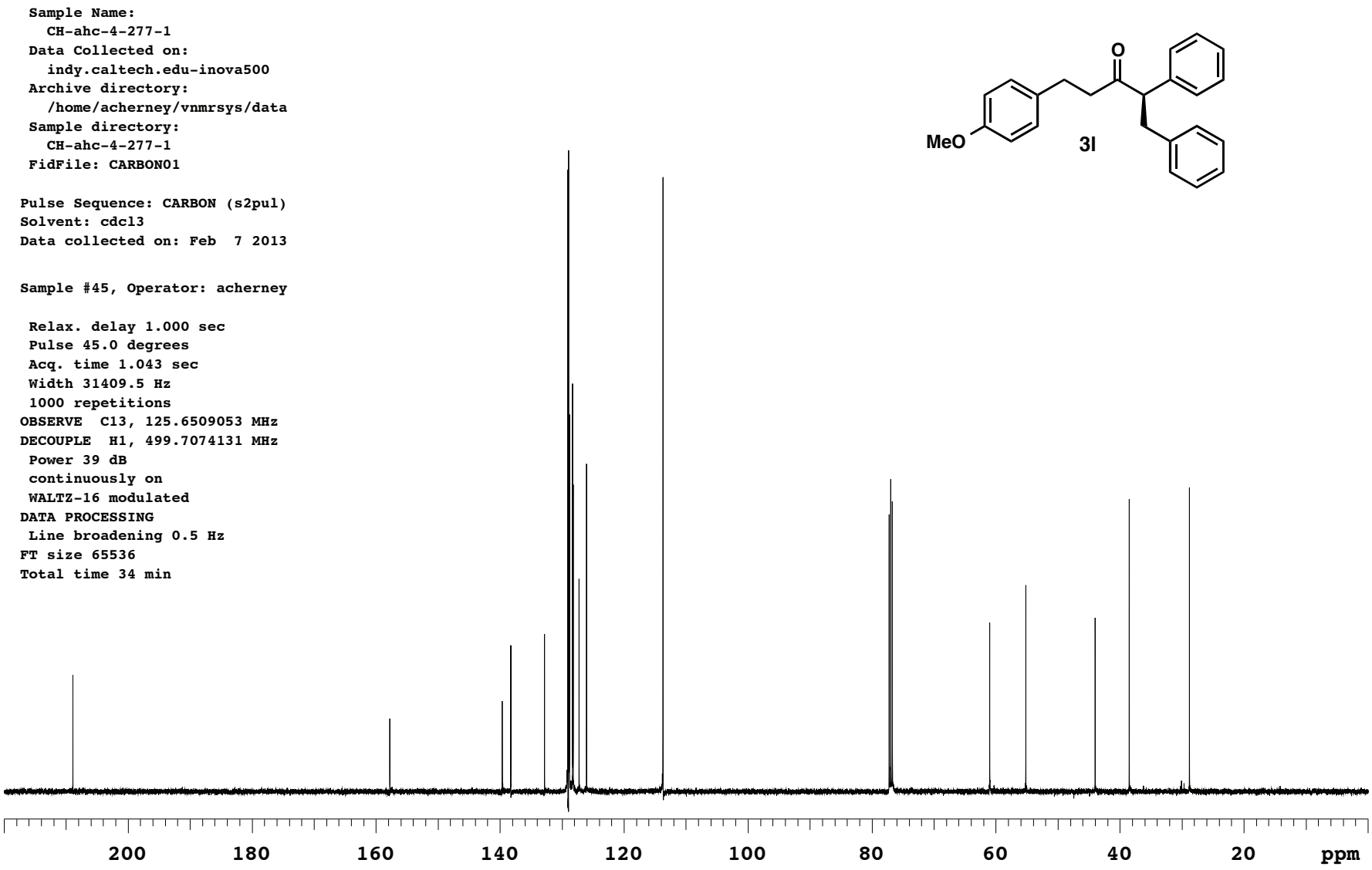
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 7 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



3l

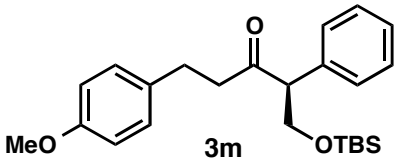

Sample Name:
CH-ahc-4-277-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-4-277-1
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 7 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509053 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
MeO
3l
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-5-91-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-91-1
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



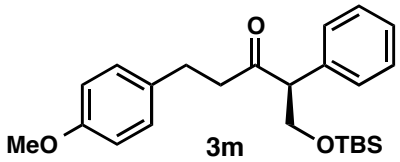
3m

Sample Name:
CH-ahc-5-91-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-91-1
FidFile: CARBON01

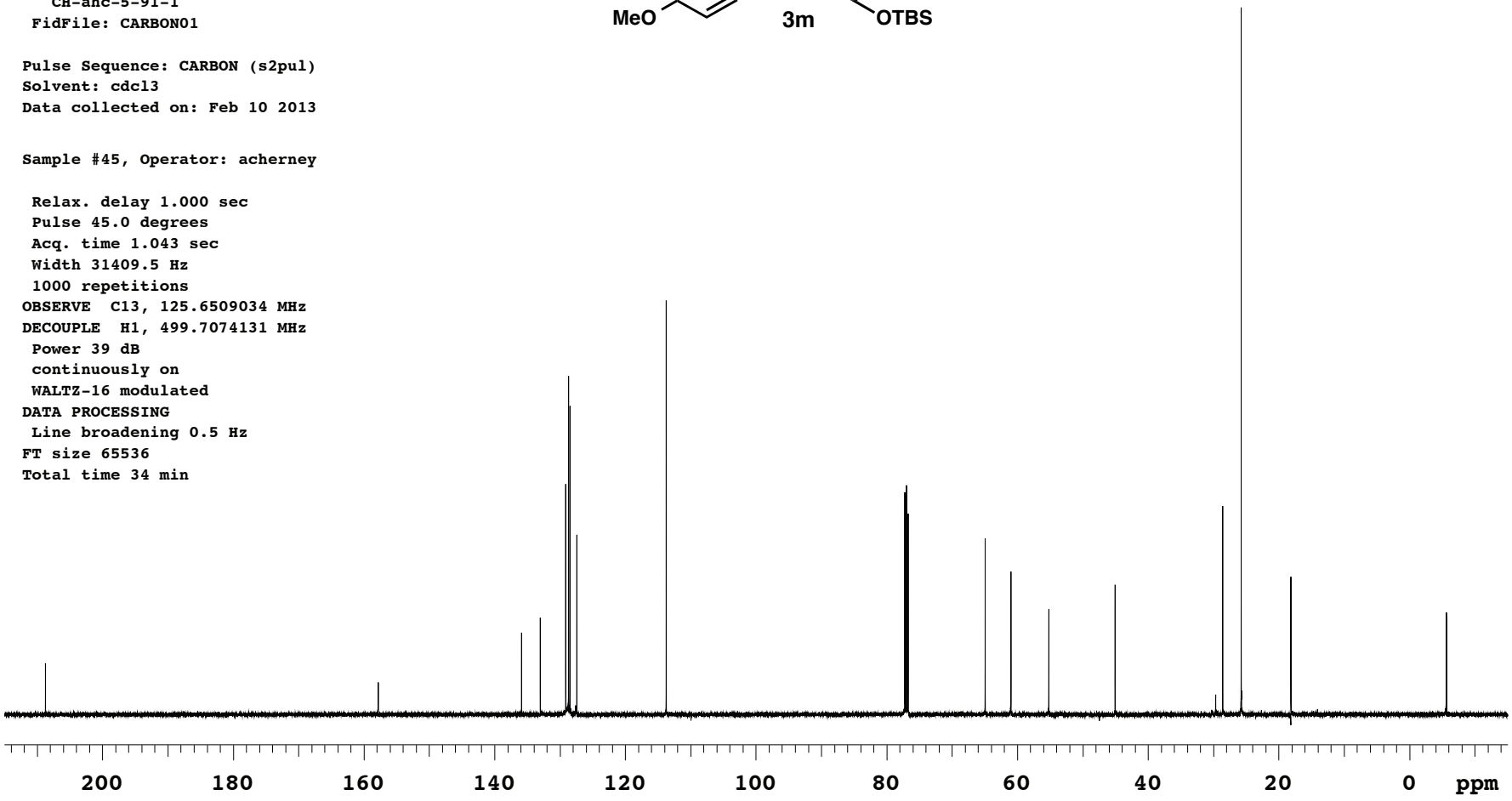
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 10 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509034 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



3m

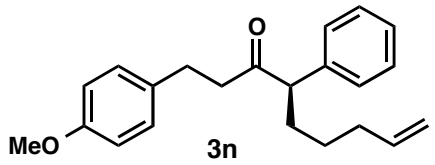


Sample Name:
CH-ahc-5-33-4
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-33-4
FidFile: PROTON01

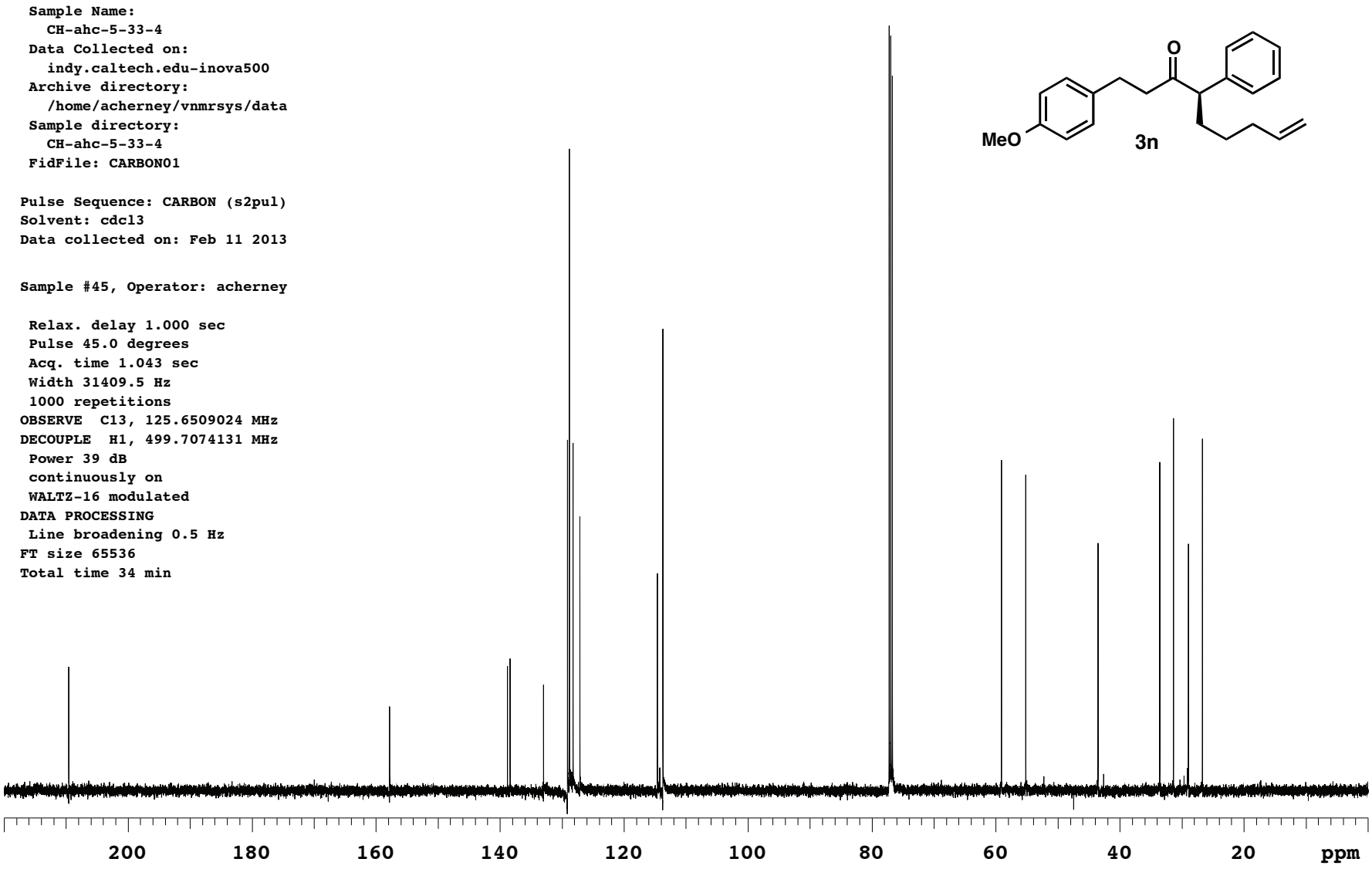
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 11 2013

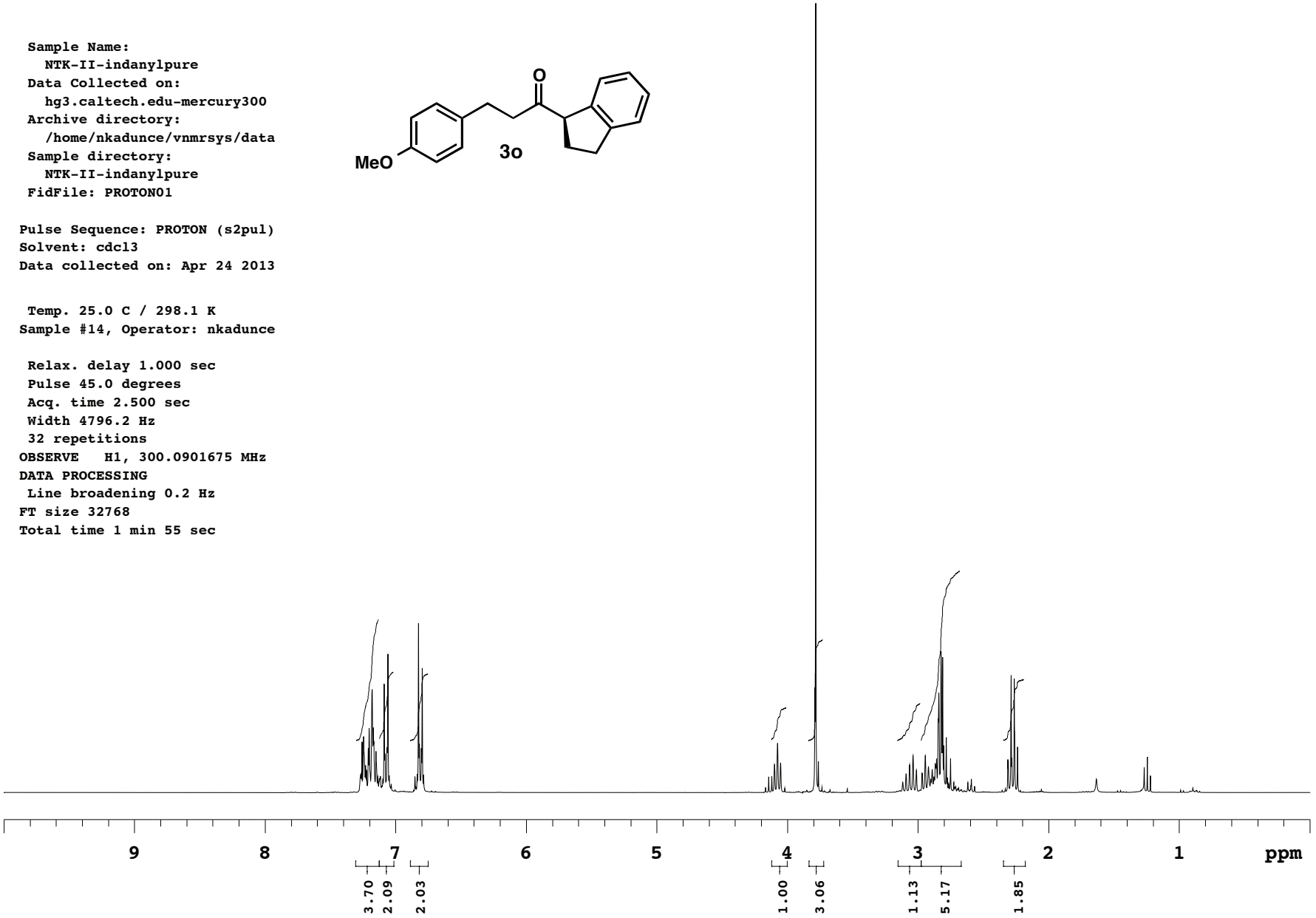
Sample #45, Operator: acherney

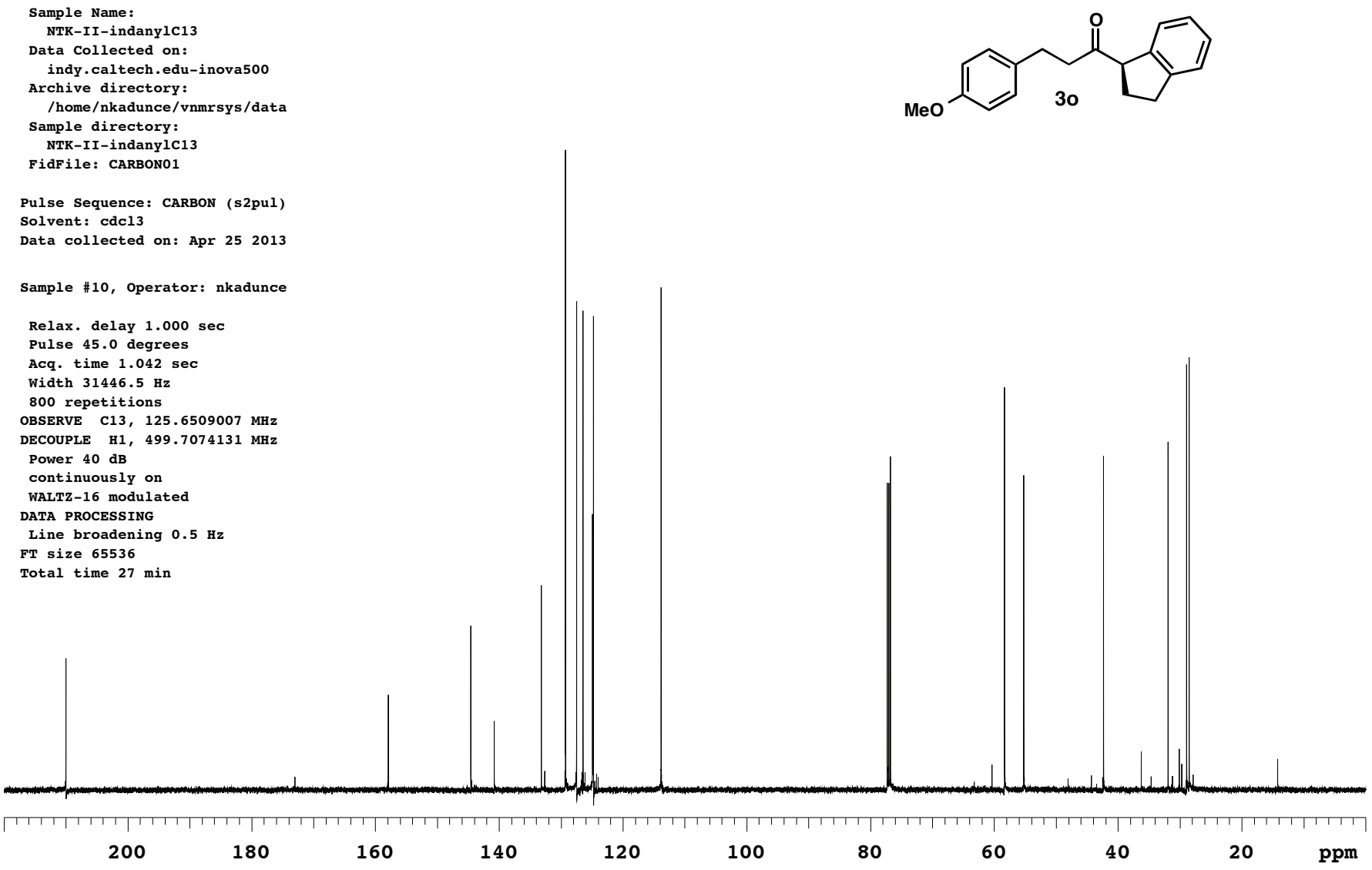
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 7995.2 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec

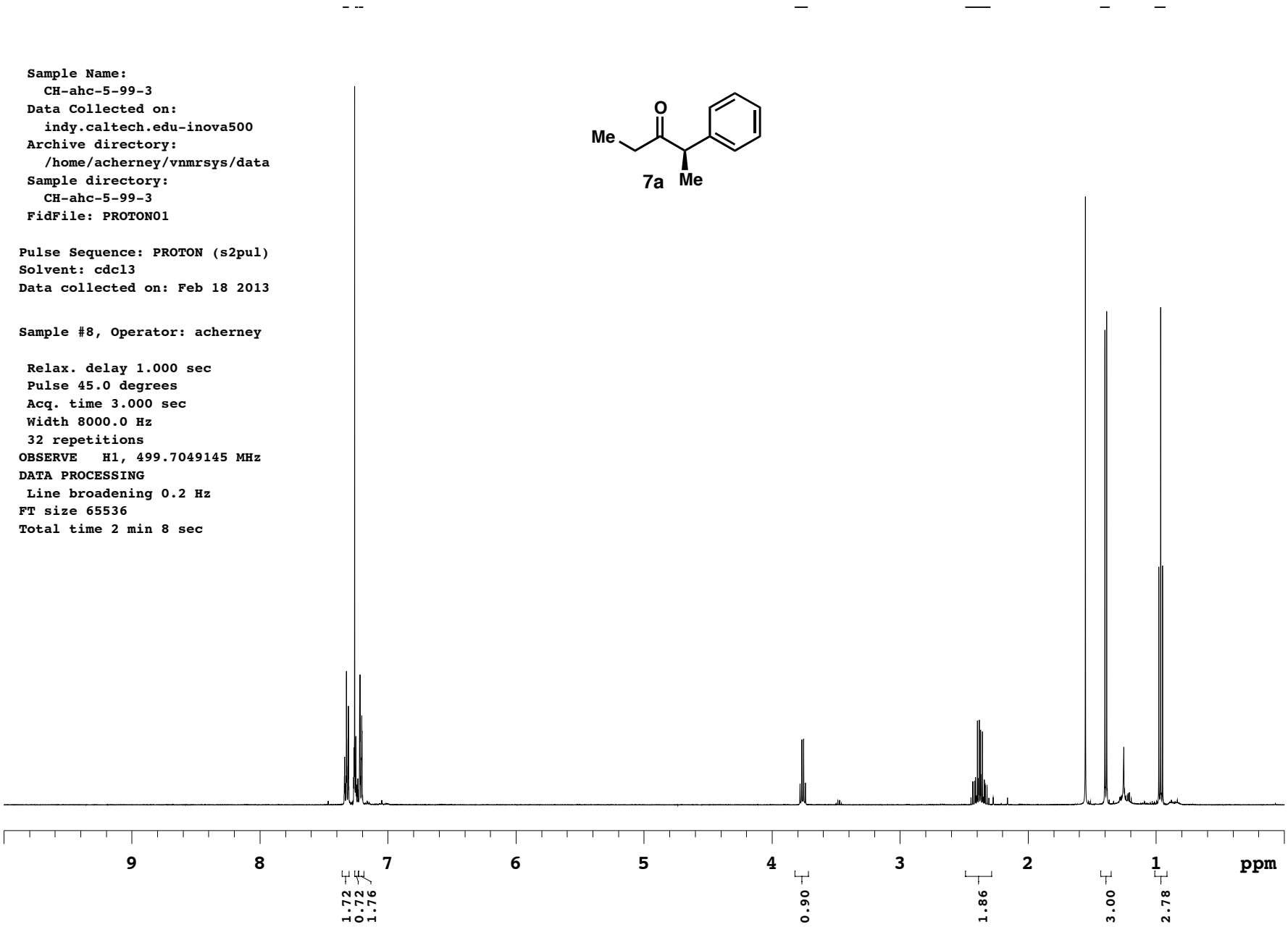


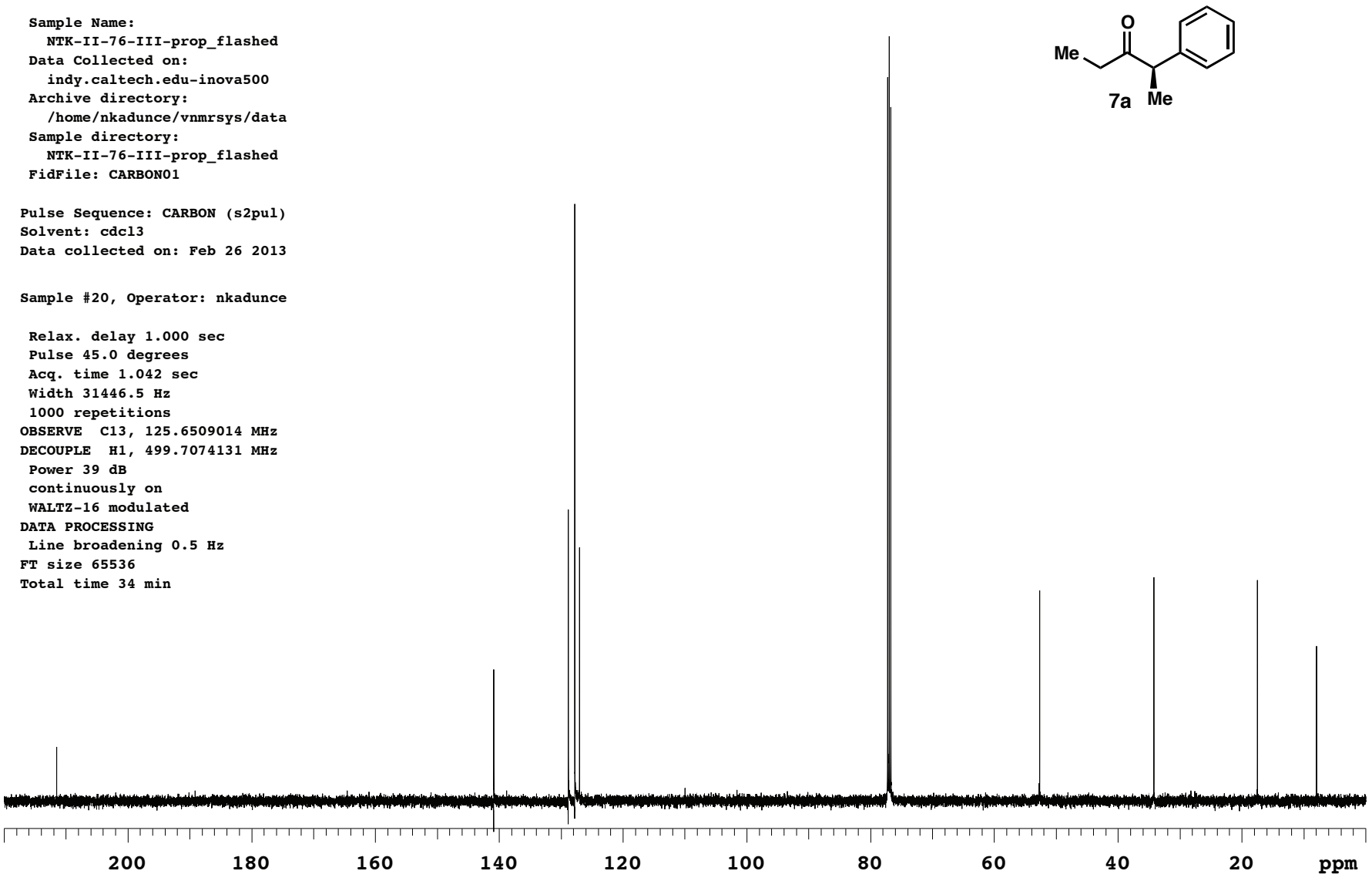
3n

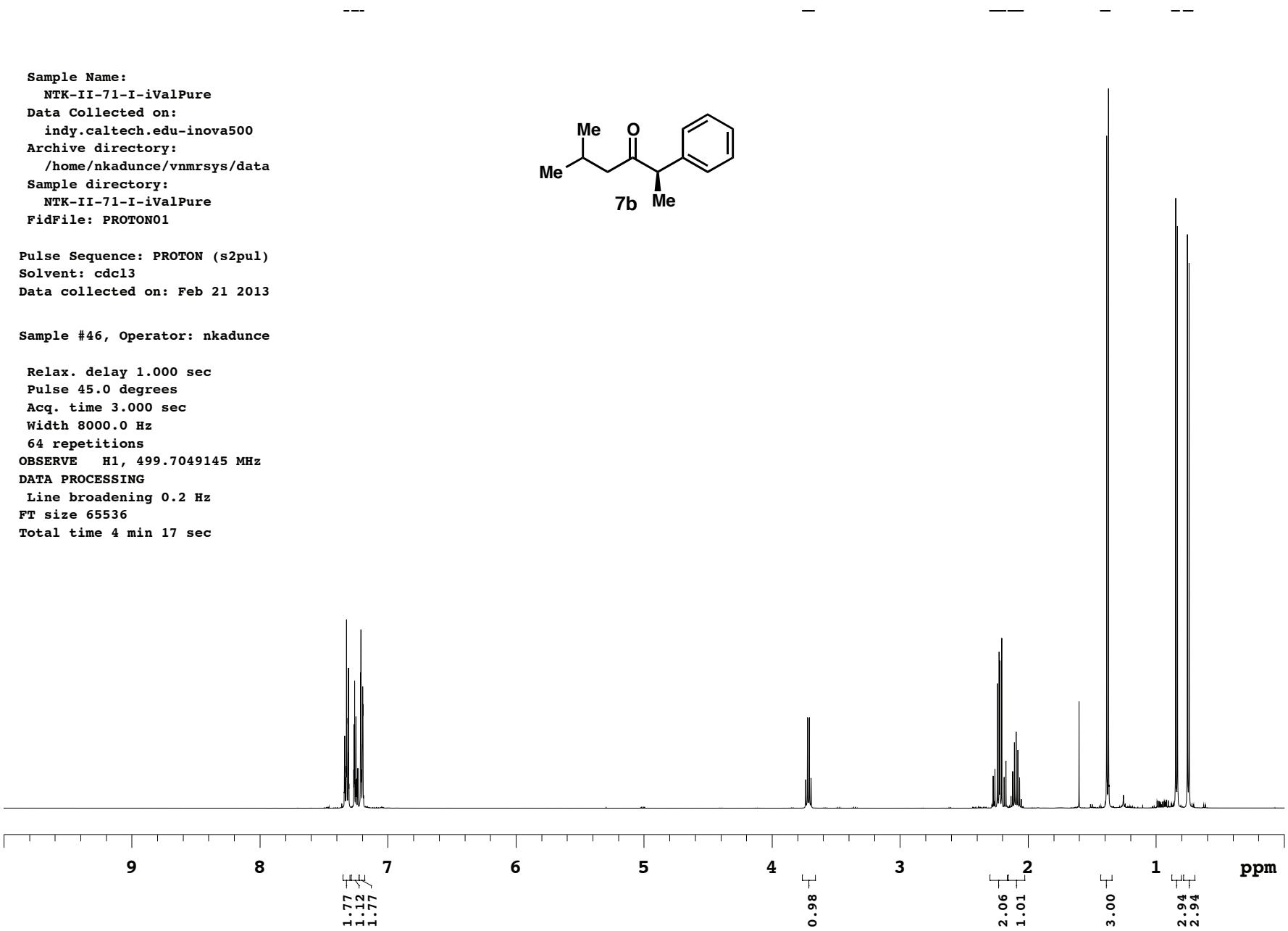

Sample Name:
CH-ahc-5-33-4
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-33-4
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 11 2013
Sample #45, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Width 31409.5 Hz
1000 repetitions
OBSERVE C13, 125.6509024 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
MeO
3n
200
180
160
140
120
100
80
60
40
20
ppm

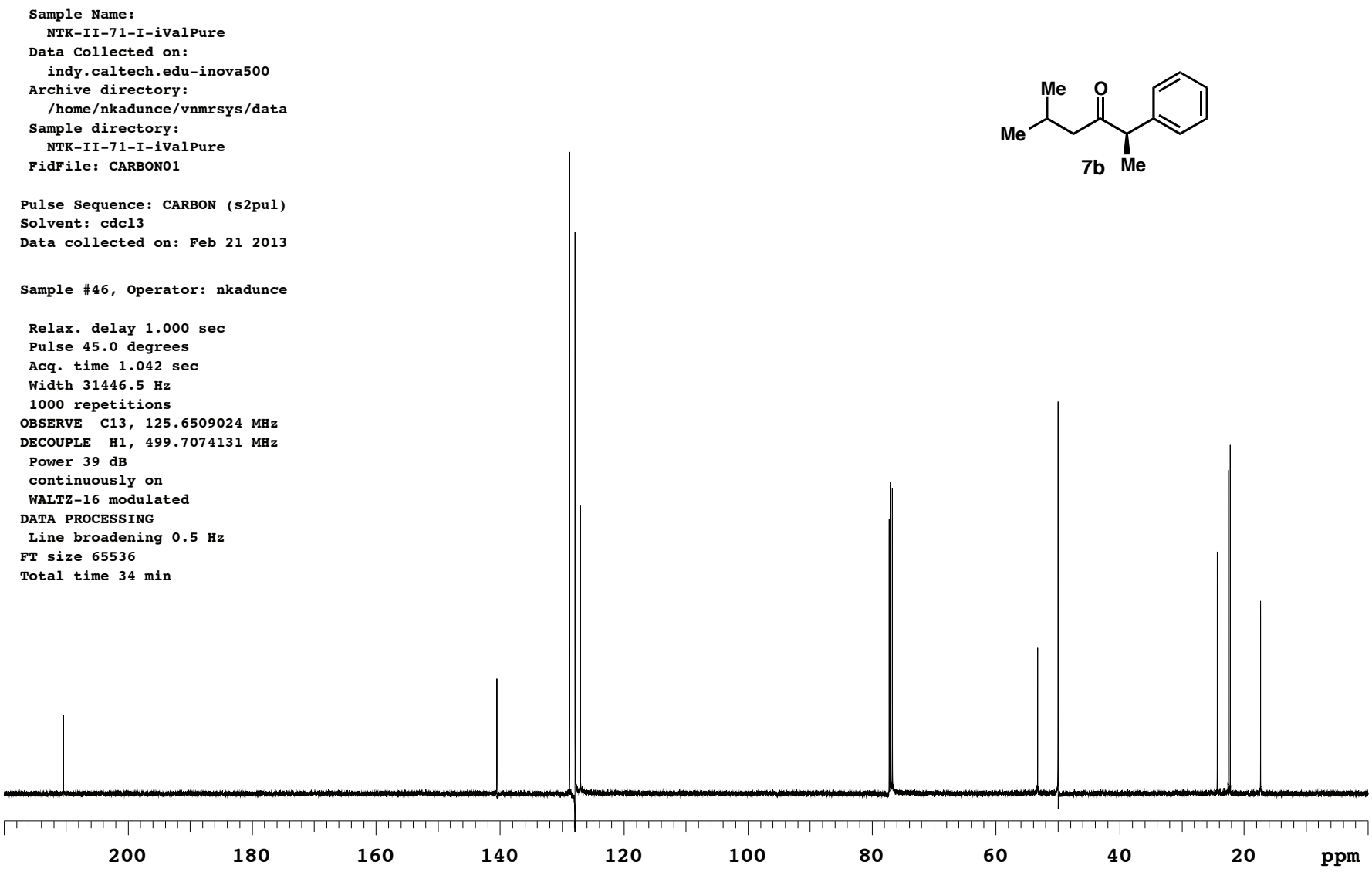

Sample Name:
NTK-II-indanylpure
Data Collected on:
hg3.caltech.edu-mercury300
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-indanylpure
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Apr 24 2013
Temp. 25.0 C / 298.1 K
Sample #14, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 4796.2 Hz
32 repetitions
OBSERVE H1, 300.0901675 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min 55 sec
O
MeO
3o
9
8
7
6
5
4
3
2
1
ppm
3.70
2.09
2.03
1.00
3.06
1.13
5.17
1.85

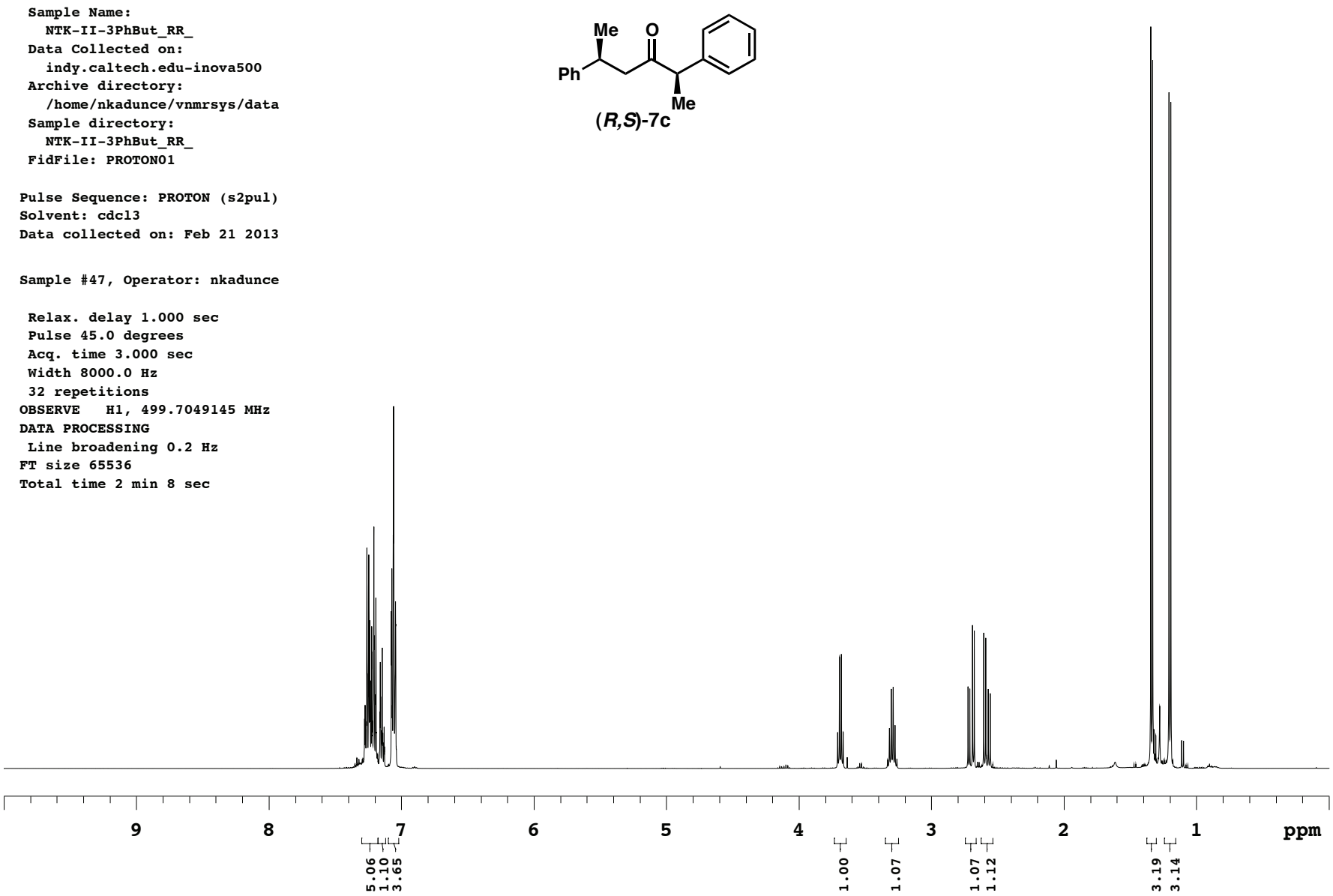

Sample Name:
NTK-II-indanylC13
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-indanylC13
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 25 2013
Sample #10, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
800 repetitions
OBSERVE C13, 125.6509007 MHz
DECOUPLE H1, 499.7074131 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 27 min
O
MeO
3o
200
180
160
140
120
100
80
60
40
20
ppm

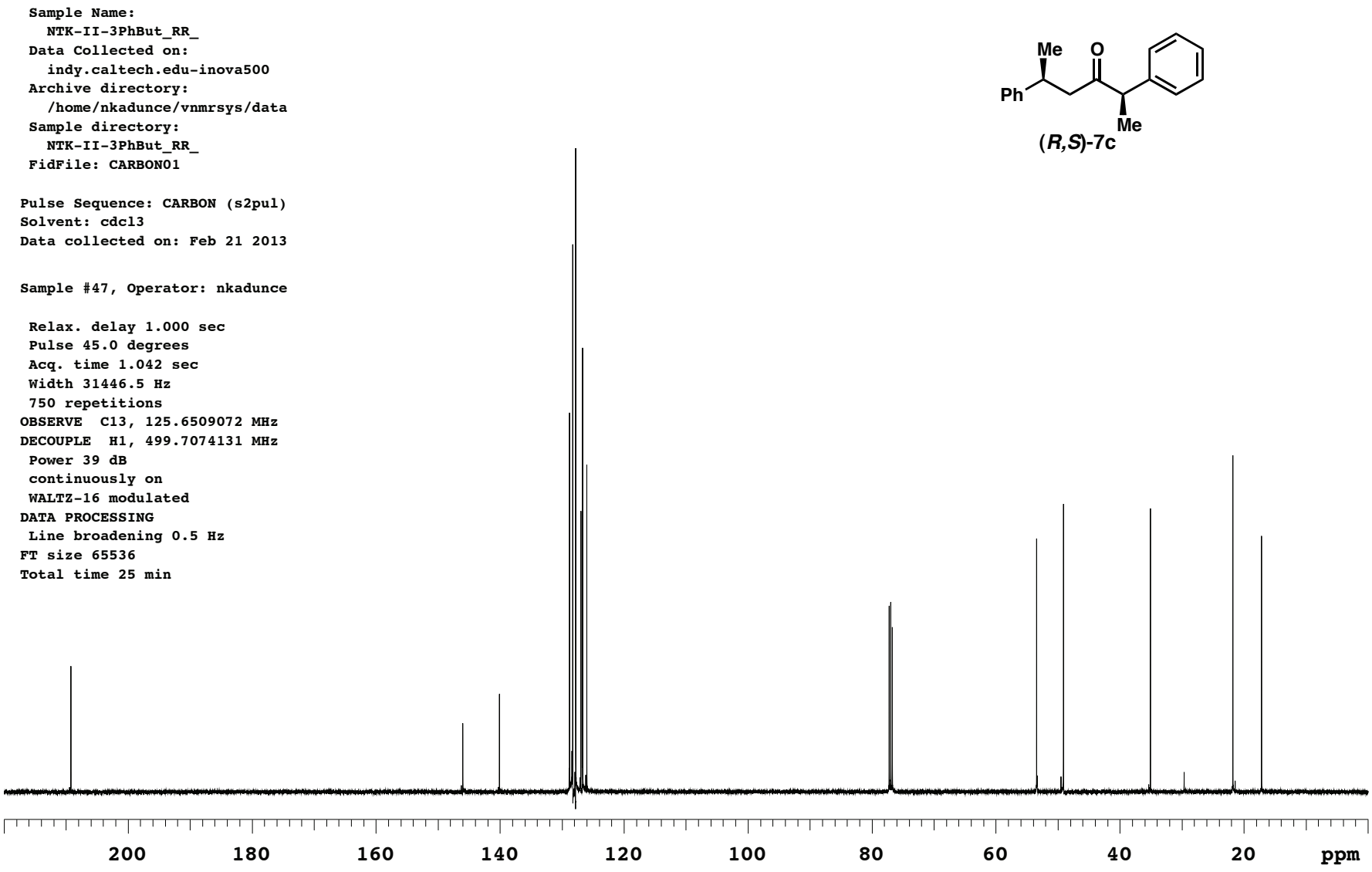

Sample Name:
CH-ahc-5-99-3
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-99-3
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 18 2013
Sample #8, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
Me
7a
Me
9
8
7
6
5
4
3
2
1
ppm
1.72
0.72
1.76
0.90
1.86
3.00
2.78


Sample Name:
NTK-II-76-III-prop_flashed
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-76-III-prop_flashed
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 26 2013
Sample #20, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509014 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
Me
Me
7a
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
NTK-II-71-I-iValPure
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-71-I-iValPure
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013
Sample #46, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
64 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 4 min 17 sec
Me
O
Me
7b
Me
9
8
7
6
5
4
3
2
1
ppm
1.77
1.12
1.77
0.98
2.06
1.01
3.00
2.94
2.94


Sample Name:
NTK-II-71-I-iValPure
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-71-I-iValPure
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013
Sample #46, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509024 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Me
O
Me
7b
Me
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
NTK-II-3PhBut_RR_
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-3PhBut_RR_
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013
Sample #47, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
Me
O
Ph
Me
(R,S)-7c
9
8
7
6
5
4
3
2
1
ppm
5.06
1.10
3.65
1.00
1.07
1.07
1.12
3.19
3.14

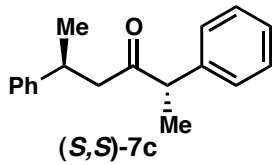

Sample Name:
NTK-II-3PhBut_RR_
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-3PhBut_RR_
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013
Sample #47, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
750 repetitions
OBSERVE C13, 125.6509072 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min
Me
O
Ph
Me
(R,S)-7c
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
NTK-II-3PhBut_SS_
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-3PhBut_SS_
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013

Sample #48, Operator: nkadunce

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



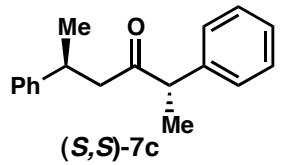
(*S*,*S*)-7c

Sample Name:
NTK-II-3PhBut_SS_
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-3PhBut_SS_
FidFile: CARBON01

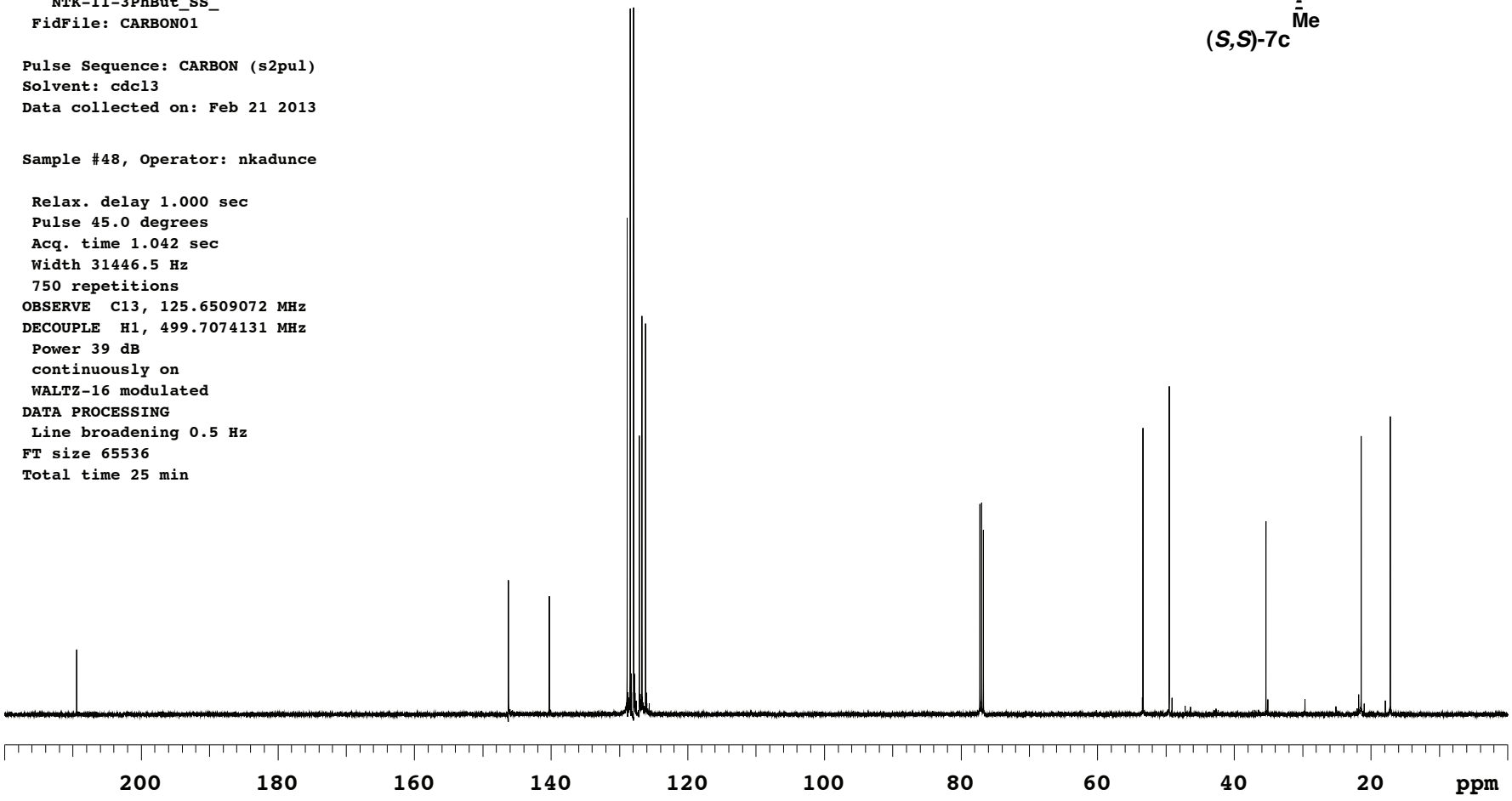
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013

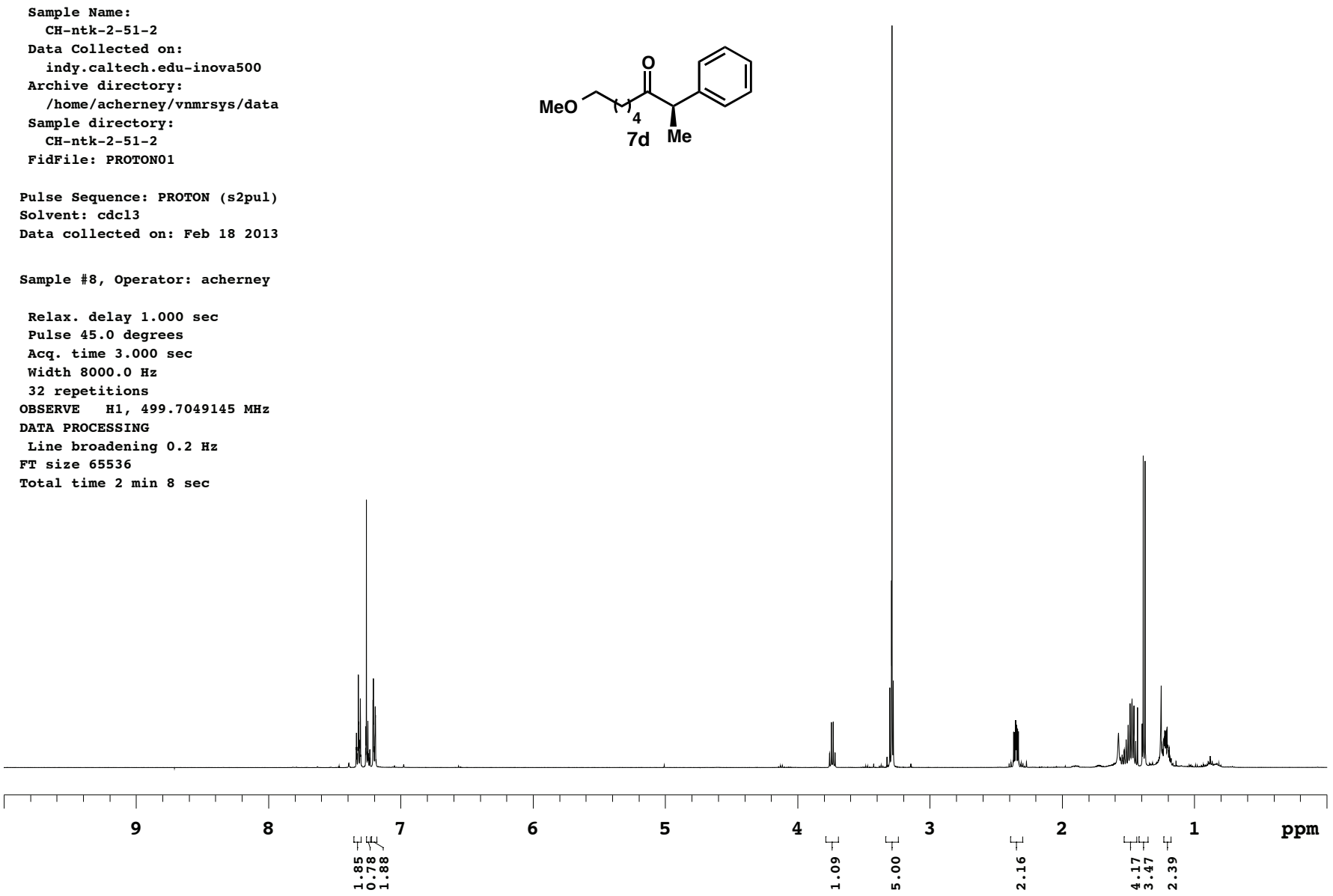
Sample #48, Operator: nkadunce

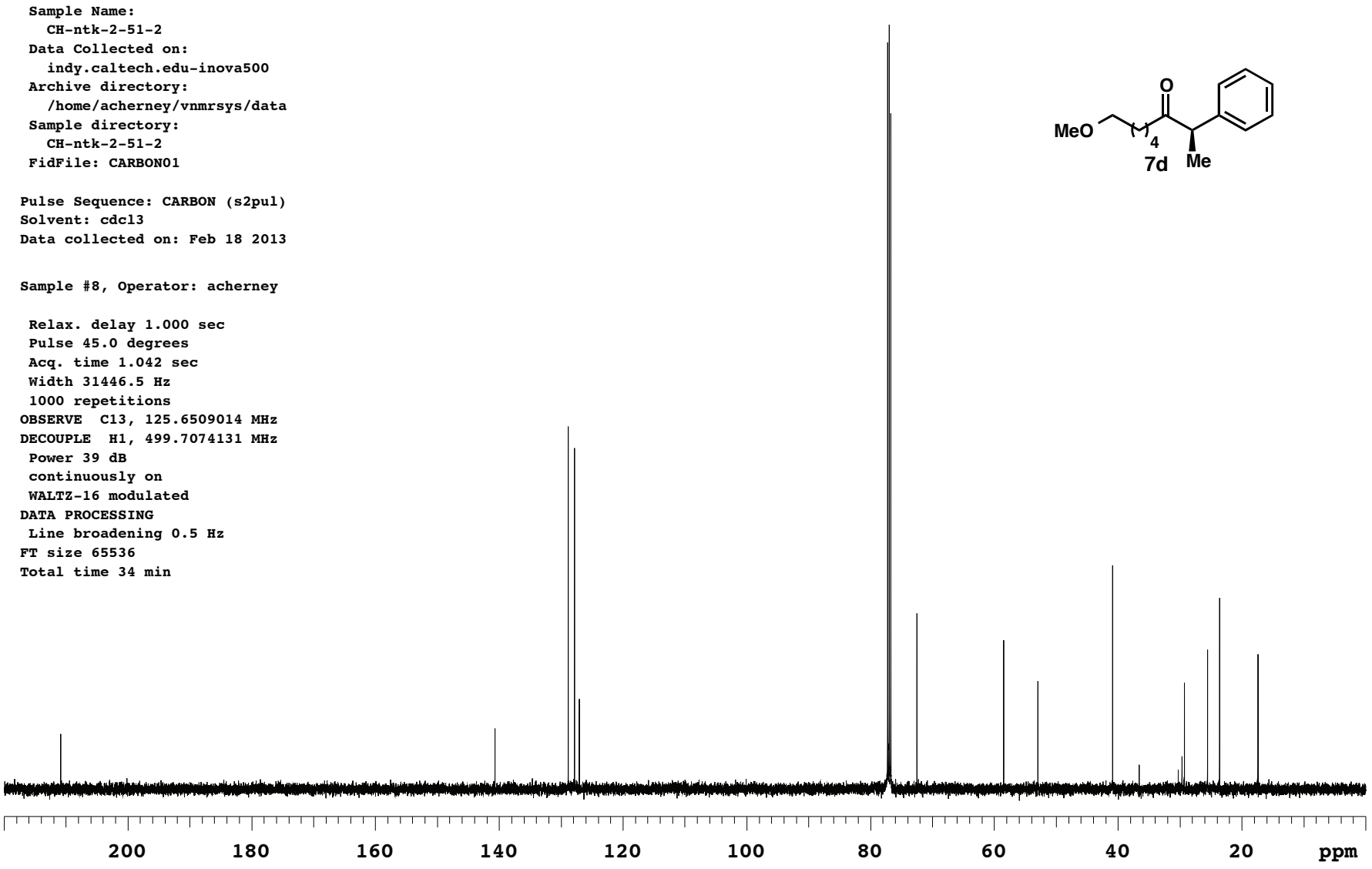
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
750 repetitions
OBSERVE C13, 125.6509072 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min



(*S,S*)-7c




Sample Name:
CH-ntk-2-51-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ntk-2-51-2
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 18 2013
Sample #8, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
MeO
4
Me
7d
9
8
7
6
5
4
3
2
1
ppm
1.85
0.78
1.88
1.09
5.00
2.16
4.17
3.47
2.39

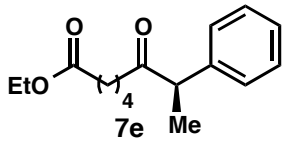

Sample Name:
CH-ntk-2-51-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ntk-2-51-2
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 18 2013
Sample #8, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509014 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
MeO
4
7d
Me
O
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
CH-ahc-5-93-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/autouser/vnmrsys/data
Sample directory:
CH-ahc-5-93-1
FidFile: PROTON01

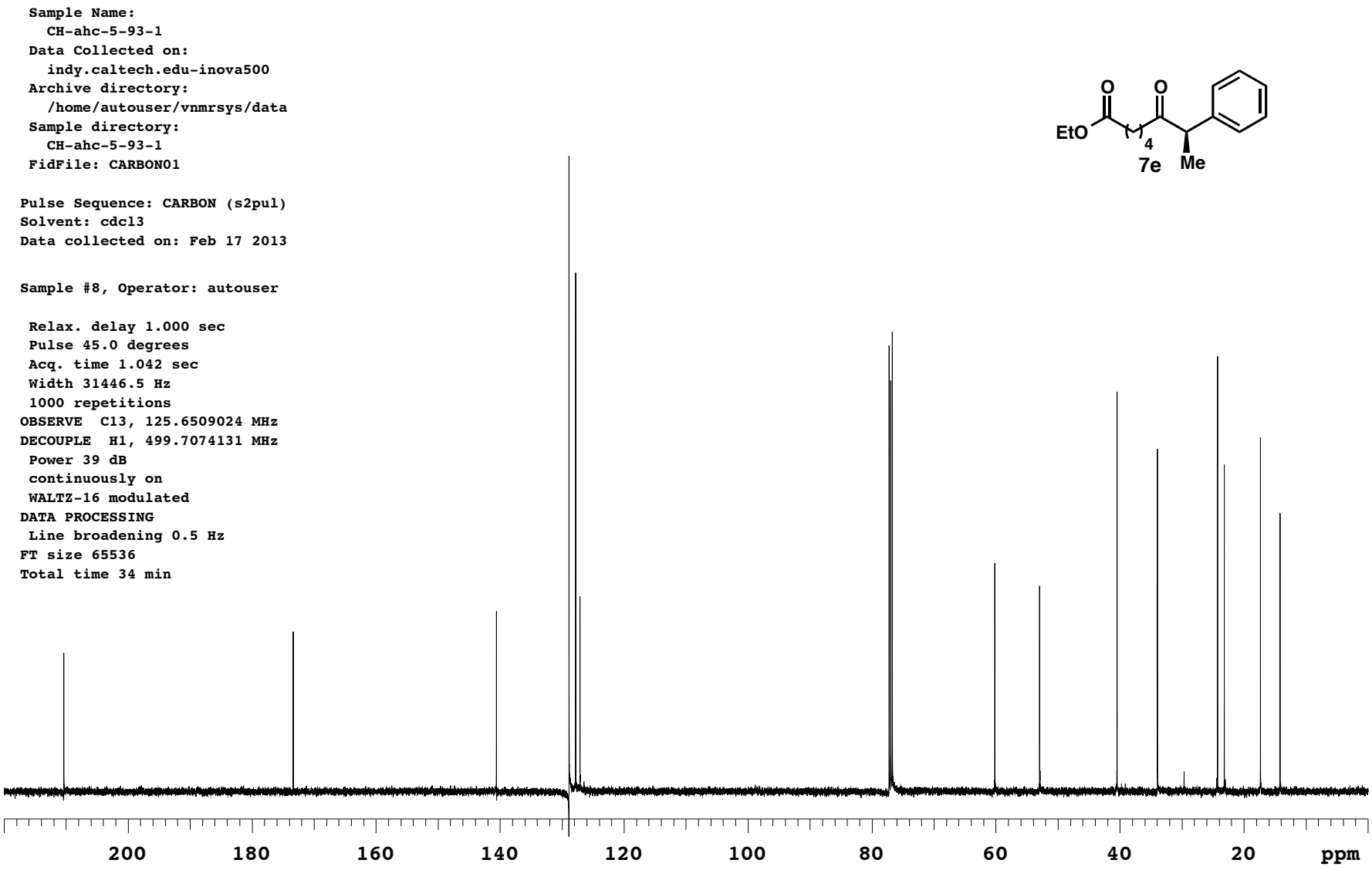
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 17 2013

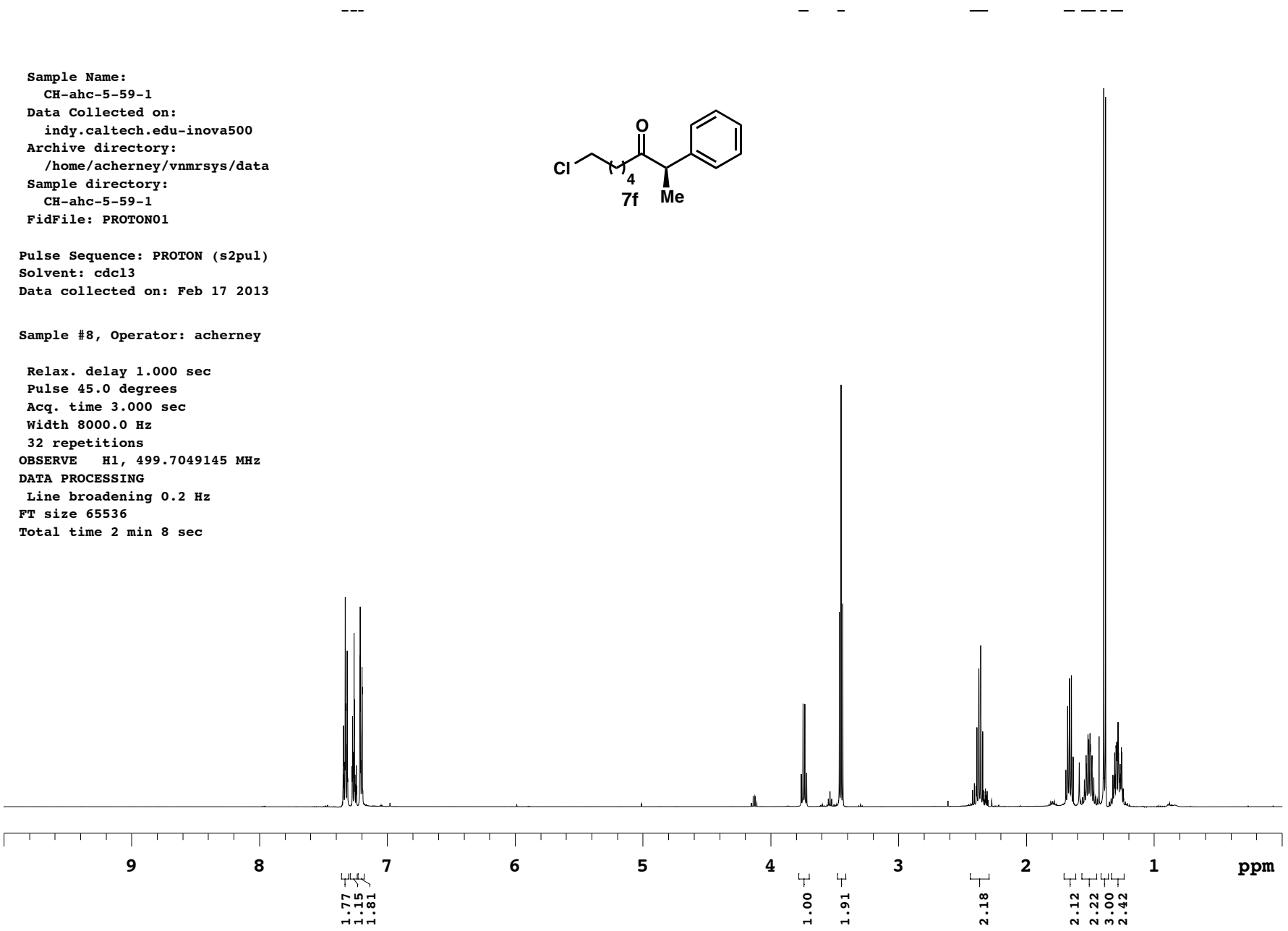
Sample #8, Operator: autouser

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



7e


Sample Name:
CH-ahc-5-93-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/autouser/vnmrsys/data
Sample directory:
CH-ahc-5-93-1
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 17 2013
Sample #8, Operator: autouser
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509024 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
O
O
EtO
4
7e
Me
200
180
160
140
120
100
80
60
40
20
ppm

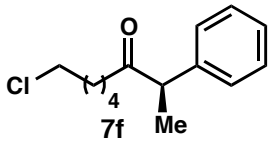

Sample Name:
CH-ahc-5-59-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-59-1
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 17 2013
Sample #8, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
Cl
4
7f
Me
9
8
7
6
5
4
3
2
1
ppm
1.77
1.15
1.81
1.00
1.91
2.18
2.12
2.22
3.00
2.42

Sample Name:
CH-ahc-5-59-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-59-1
FidFile: CARBON01

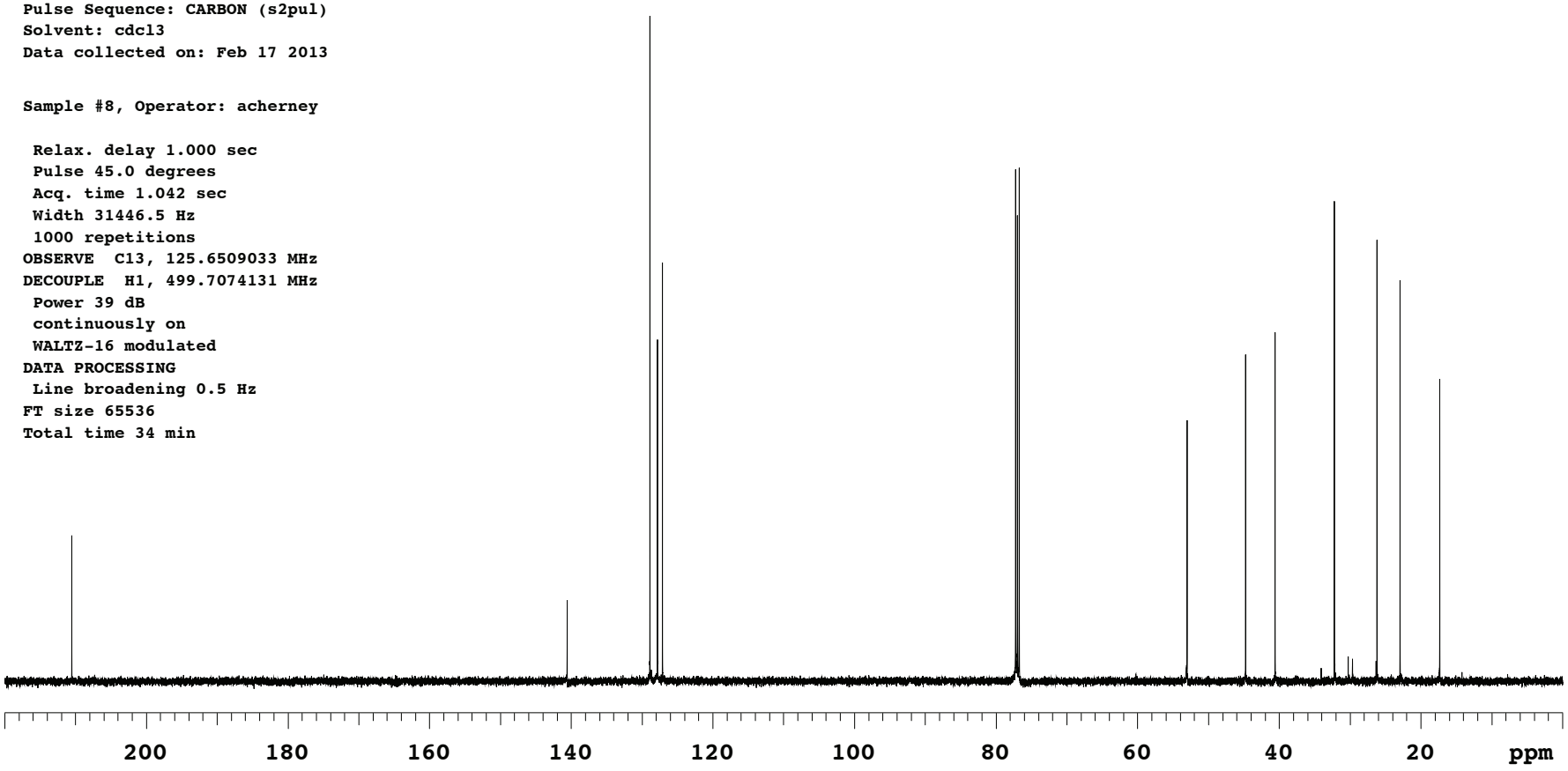
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 17 2013

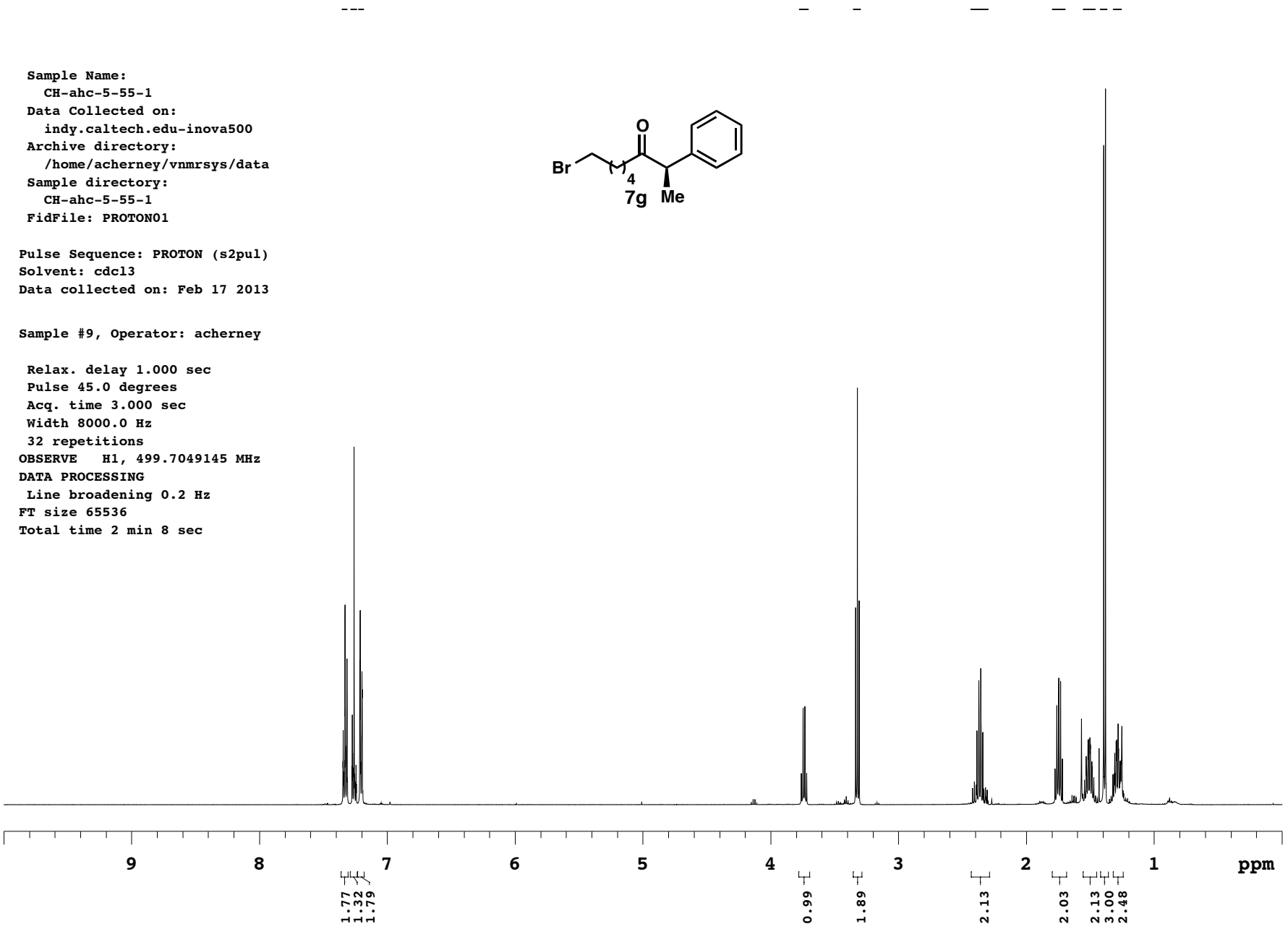
Sample #8, Operator: acherney

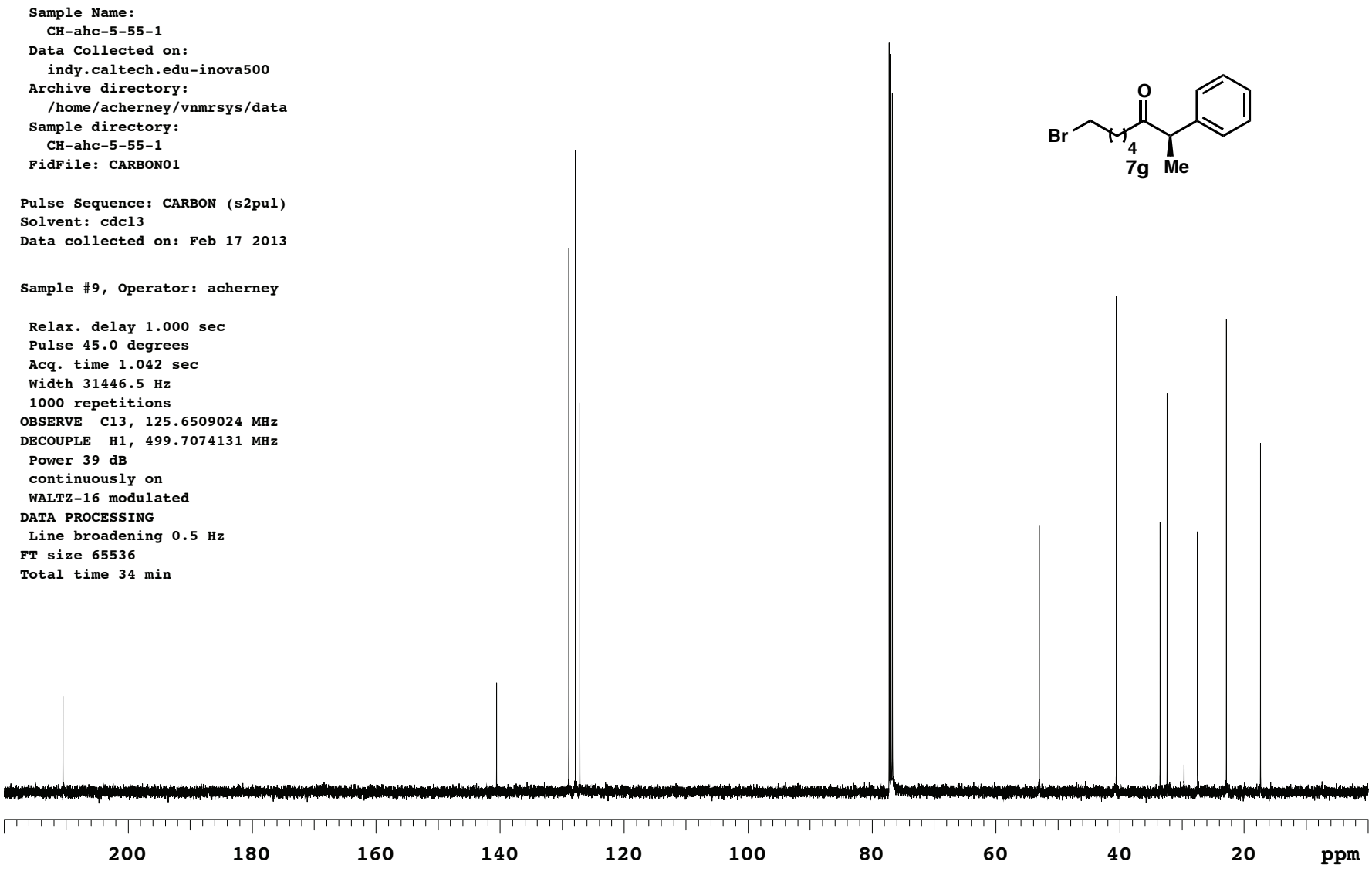
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509033 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min



7f




Sample Name:
CH-ahc-5-55-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-55-1
FidFile: PROTON01
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 17 2013
Sample #9, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
O
Br
4
7g
Me
9
8
7
6
5
4
3
2
1
ppm
1.77
1.32
1.79
0.99
1.89
2.13
2.03
2.13
3.00
2.48

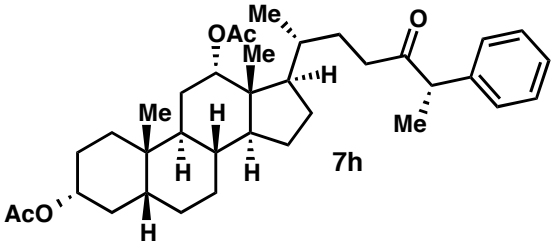

Sample Name:
CH-ahc-5-55-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmrsys/data
Sample directory:
CH-ahc-5-55-1
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 17 2013
Sample #9, Operator: acherney
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
1000 repetitions
OBSERVE C13, 125.6509024 MHz
DECOUPLE H1, 499.7074131 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
Br
O
4
7g
Me
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name:
NTK-II-steroid_dry
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-steroid_dry
FidFile: PROTON01

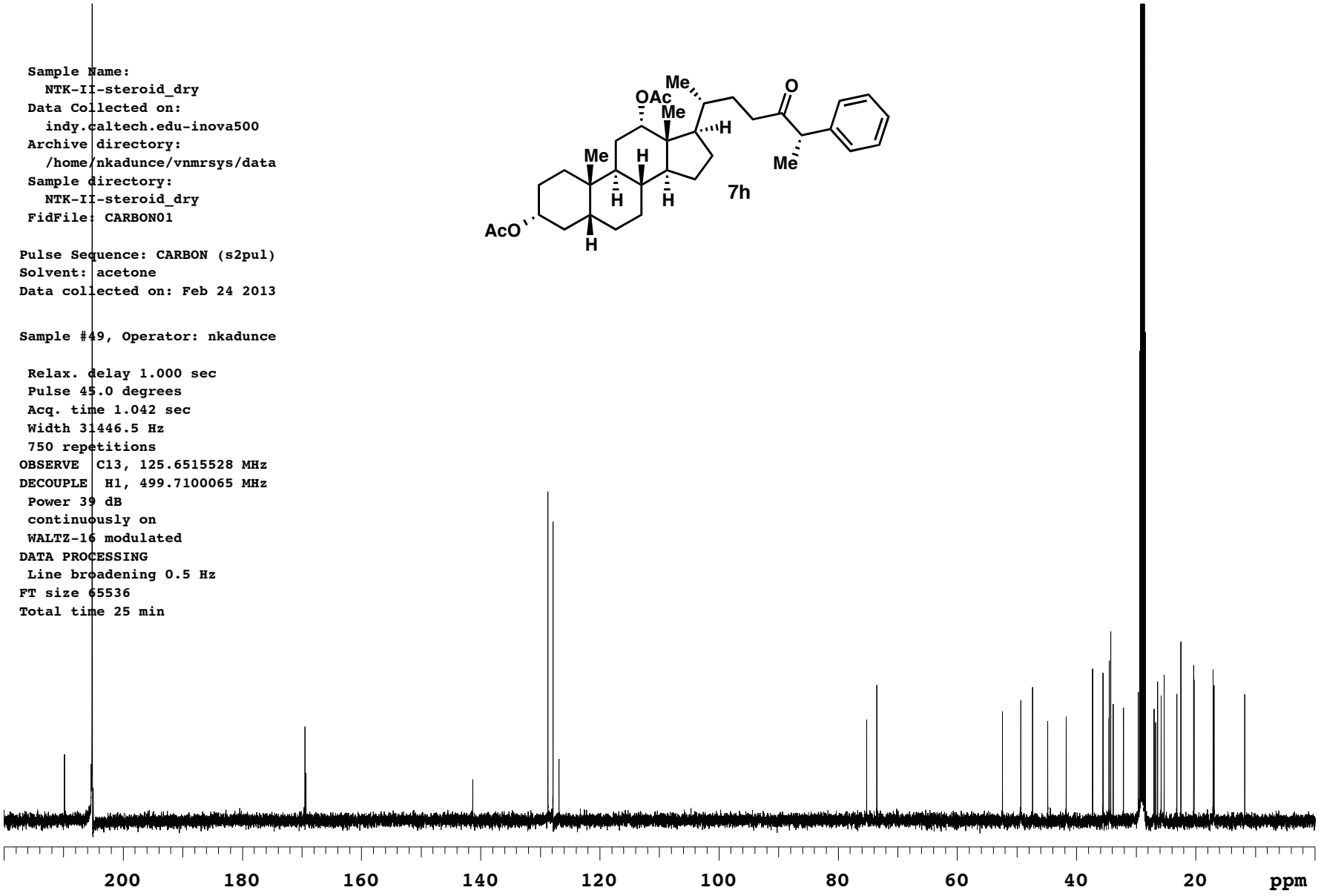
Pulse Sequence: PROTON (s2pul)
Solvent: acetone
Data collected on: Feb 24 2013

Sample #49, Operator: nkadunce

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7075080 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec



7h


Sample Name:
NTK-II-steroid_dry
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
NTK-II-steroid_dry
FidFile: CARBON01
Pulse Sequence: CARBON (s2pul)
Solvent: acetone
Data collected on: Feb 24 2013
Sample #49, Operator: nkadunce
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.042 sec
Width 31446.5 Hz
750 repetitions
OBSERVE C13, 125.6515528 MHz
DECOUPLE H1, 499.7100065 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min
Me
OAc
Me
O
H
Me
H
Me
H
H
AcO
H
7h
200
180
160
140
120
100
80
60
40
20
ppm