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

Alan H. Cherney, Nathaniel T. Kadunce, Sarah E. Reisman*

The Warren and Katharine Schlinger Laboratory of Chemistry and Chemical Engineering
Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena,
California 91125
reisman@caltech.edu

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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$_2$Cl$_2$), 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. $^1$H 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$_3$ ($^1$H, $\delta$ = 7.26) or acetone ($^1$H, $\delta$ = 2.05), and CDCl$_3$ ($^{13}$C, $\delta$ = 77.0) or acetone ($^{13}$C, $\delta$ = 29.8). Data for $^1$H NMR spectra are reported as follows: chemical shift (\(\delta\) 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 H2O (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 (MgSO4), 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

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3. Substrate Preparation.

A. General Procedure 2: Acid Chloride Synthesis

A flask was charged with the appropriate carboxylic acid (1.0 equiv) and CH2Cl2 (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$_2$Cl$_2$ (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, $J$ = 48.79, 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) calcd 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-butyldimethylsilox)ethyl]benzene (2m).

To a flask was added 2-chloro-2-phenylethanol (8.5 mmol, 1.0 equiv) and CH$_2$Cl$_2$ (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$_2$Cl$_2$ (2 X 20 mL). The combined organic layers were washed with brine (1 X 20 mL) and dried (Na$_2$SO$_4$), 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, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) 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 NiCl₂(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 H₂O (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 (MgSO₄), 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 CO₂, λ = 210 nm): \( t_R \) (minor) = 9.2 min, \( t_R \) (major) = 9.8 min. \( [\alpha]_D^{25} = -102.3^\circ \) (c = 1.10, CHCl₃); ¹H NMR (500 MHz, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) 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% ee 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 CO₂, λ = 210 nm): tᵣ (minor) = 9.0 min, tᵣ (major) = 9.8 min. [α]ᵣ²⁵ = −84.9° (c = 1.37, CHCl₃); ¹H NMR (500 MHz, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) 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, 1714, 1612, 1584, 1513, 1453, 1300, 1246, 1178, 1036, 825 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 CO₂, λ = 210 nm): tᵣ (minor) = 9.1 min, tᵣ (major) = 9.9 min. [α]ᵣ²⁵ = −90.4° (c = 1.46, CHCl₃); ¹H NMR (500 MHz, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) 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 CO₂, λ = 210 nm): tᵣ (minor) = 5.3 min, tᵣ (major) = 5.7 min. [α]ᵣ²⁵ = −72.3° (c = 0.56, CHCl₃); ¹H NMR (500 MHz, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) 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)-L.I (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 CO₂, λ = 210 nm): \( t_R \) (minor) = 6.8 min, \( t_R \) (major) = 7.4 min. \([\alpha]_D^{25} = -77.2^\circ \left( c = 1.22, \text{ CHCl}_3 \right)\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \( \delta \) 7.10 (d, \( J = 8.3 \text{ Hz}, 2\text{H} \)), 6.98 (d, \( J = 8.0 \text{ Hz}, 2\text{H} \)), 6.83 (d, \( J = 9.0 \text{ Hz}, 2\text{H} \)), 6.76 (d, \( J = 9.0 \text{ Hz}, 2\text{H} \)), 3.79 (s, 3\text{H}), 3.77 (s, 3\text{H}), 3.67 (q, \( J = 7.0 \text{ Hz}, 1\text{H} \)). \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \( \delta \) 210.3, 158.7, 157.9, 133.1, 133.4, 129.2, 129.0, 113.8, 55.2, 55.23, 52.3, 42.7, 29.1, 17.3; FTIR (NaCl, thin film): 2930, 2834, 1713, 1611, 1582, 1512, 1463, 1301, 12, 246, 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 CO₂, λ = 210 nm): \( t_R \) (minor) = 19.6 min, \( t_R \) (major) = 20.6 min. \([\alpha]_D^{25} = -64.1^\circ \left( c = 0.79, \text{ CHCl}_3 \right)\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \( \delta \) 7.25 (d, \( J = 8.8 \text{ Hz}, 2\text{H} \)), 7.06 (d, \( J = 8.8 \text{ Hz}, 2\text{H} \)), 6.97 (d, \( J = 8.8 \text{ Hz}, 2\text{H} \)), 6.76 (d, \( J = 8.4 \text{ Hz}, 2\text{H} \)), 3.77 (s, 3\text{H}), 3.67 (q, \( J = 7.0 \text{ Hz}, 1\text{H} \)), 2.83 – 2.55 (m, 4\text{H}), 1.34 (d, \( J = 7.0 \text{ Hz}, 3\text{H} \)); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \( \delta \) 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 CO₂, λ = 210 nm): \( t_R \) (minor) = 25.4 min, \( t_R \) (major) = 27.0 min. \([\alpha]_D^{25} = -53.5^\circ \left( c = 1.44, \text{ CHCl}_3 \right)\); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \( \delta \) 7.41 (d, \( J = 8.6 \text{ Hz}, 2\text{H} \)), 7.01 (d, \( J = 8.4 \text{ Hz}, 2\text{H} \)), 6.97 (d, \( J = 8.6 \text{ Hz}, 2\text{H} \)), 6.76 (d, \( J = 9.2 \text{ Hz}, 2\text{H} \)), 3.77 (s, 3\text{H}),
3.65 (q, J = 7.0 Hz, 1H), 2.83 – 2.55 (m, 4H), 1.34 (d, J = 7.0 Hz, 3H); \(^{13}\text{C}\) NMR (126 MHz, CDCl\(_3\)) \(\delta\) 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 \(\text{cm}^{-1}\); HRMS (MM) calc’d for M\(^+\) 346.0563, found 346.0463.

\((R)-1-(4\text{-methoxyphenyl})-4-(4-(\text{trifluoromethyl})phenyl)\text{Pentan-3-one (3h)}\)

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\(_2\), \(\lambda\) = 210 nm): \(t_\text{R}\) (major) = 6.0 min, \(t_\text{R}\) (minor) = 7.3 min. [\(\alpha\)]\(_D\)\(^{25}\) = \(-50.8.0\) (c = 1.01, CHCl\(_3\)), \(^1\text{H}\) NMR (500 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\text{C}\) NMR (126 MHz, CDCl\(_3\)) \(\delta\) 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 \(\text{cm}^{-1}\); HRMS (MM) calc’d for M\(^+\) 336.1332, found 336.1342.

\((R)-1-(4\text{-methoxyphenyl})-4-(naphthalen-2-yl)\text{Pentan-3-one (3i)}\)

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\(_2\), \(\lambda\) = 210 nm): \(t_\text{R}\) (minor) = 10.7 min, \(t_\text{R}\) (major) = 11.3 min. [\(\alpha\)]\(_D\)\(^{25}\) = \(-100.4\) (c = 1.00, CHCl\(_3\)), \(^1\text{H}\) NMR (500 MHz, CDCl\(_3\)) \(\delta\) 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); \(^{13}\text{C}\) NMR (126 MHz, CDCl\(_3\)) \(\delta\) 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, 2937, 1713, 1611, 1583, 1511, 1455, 1374, 1300, 1245, 1178, 1035, 822, 750 \(\text{cm}^{-1}\); LRMS (ESI) calc’d for [M+H]\(^+\) 319.2, found 319.2.

\((R)-1-(4\text{-methoxyphenyl})-4-\text{Phenylhexan-3-one (3j)}\)

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 (OD, 2.5 mL/min, 5% IPA in CO₂, λ = 210 nm): \( t_R \) (minor) = 6.2 min, \( t_R \) (major) = 6.9 min. [\( \alpha \)]\(^D\) = -97.9° (c = 0.96, CHCl₃); \(^1\)H NMR (500 MHz, CDCl₃) 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, CDCl₃) 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 CO₂, λ = 210 nm): \( t_R \) (minor) = 18.1 min, \( t_R \) (major) = 19.4 min. [\( \alpha \)]\(^D\) = -79.7° (c = 1.85, CHCl₃); \(^1\)H NMR (500 MHz, CDCl₃) 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, CDCl₃) 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 CO₂, λ = 210 nm): \( t_R \) (major) = 4.5 min, \( t_R \) (minor) = 5.3 min. [\( \alpha \)]\(^D\) = -166.8° (c = 0.85, CHCl₃); \(^1\)H NMR (500 MHz, CDCl₃) 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, CDCl₃) 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-(tert-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 CO2, λ = 210 nm): tR (major) = 3.3 min, tR (minor) = 3.8 min. [α]D25 = −50.0° (c = 0.90, CHCl3); 1H NMR (500 MHz, CDCl3) 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), 3.73 (d, J = 5.7 Hz, 1H), 2.88 (dd, J = 9.7, 8.5 Hz, 1H), 2.54 (m, 4H), 2.09 – 1.93 (m, 3H), 1.74 – 1.63 (m, 1H), 1.37 – 1.14 (m, 2H); 13C NMR (126 MHz, CDCl3) 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, 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 CO2, λ = 210 nm): tR (major) = 10.9 min, tR (minor) = 11.9 min. [α]D25 = −90.9° (c = 0.47, CHCl3); 1H NMR (500 MHz, CDCl3) 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); 13C NMR (126 MHz, CDCl3) 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-1H-inden-1-yl)-3-(4-methoxyphenyl)Propan-1-one (3o)

Prepared from 1-chloro-2,3-dihydro-1H-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 CO2, λ = 210 nm): tR (minor) = 7.9 min, tR...
(major) = 8.9 min. [α]D25 = 11.3° (c = 0.179, CHCl3); 1H NMR (500 MHz, CDCl3) 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); 13C NMR (126 MHz, CDCl3) 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, 1759, 1709, 1611, 1583, 1513, 1458, 1300, 1247, 1178, 1036, 826, 755 cm⁻¹; LRMS (ESI) calc’d for [M+H]⁺ 281.1, 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 CO2, λ = 210 nm): tR (minor) = 1.8 min, tR (major) = 2.0 min. [α]D25 = −225.9° (c = 0.57, CHCl3); 1H NMR (500 MHz, CDCl3) 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); 13C NMR (126 MHz, CDCl3) 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 CO2, λ = 210 nm): tR (minor) = 2.2 min, tR (major) = 2.7 min. [α]D25 = −205.8° (c = 0.92, CHCl3); 1H NMR (500 MHz, CDCl3) 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); 13C NMR (126 MHz, CDCl3) 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.

(2R,5S)-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). [α]D25 = −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, 2968, 2930, 1714, 1601, 1494, 1452, 1374, 1125, 1069, 1029, 759 cm$^{-1}$; LRMS (ESI) calc’d for [M+H]$^+$ 253.2, found 253.2.

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

Prepared from (1-chloroethyl)benzene (2a, 0.20 mmol) and (S)-3-phenylbutyryl chloride (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). [α]$_D^{25}$ = 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. [α]$_D^{25}$ = -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 CO₂, λ = 210 nm): tᵣ (minor) = 4.9 min, tᵣ (major) = 5.3 min. [α]ᵣ²⁵ = −146.8° (c = 0.85, CHCl₃); ¹H NMR (500 MHz, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) δ 210.4, 173.4, 140.6, 128.9, 127.8, 127.1, 60.2, 53.0, 40.5, 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 CO₂, λ = 210 nm): tᵣ (minor) = 5.8 min, tᵣ (major) = 6.5 min. [α]ᵣ²⁵ = −163.3° (c = 0.78, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 7.36 – 7.30 (m, 2H), 7.29 – 7.24 (m, 1H), 7.23 – 7.18 (m, 2H), 3.74 (q, J = 7.0 Hz, 1H), 3.45 (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); ¹³C NMR (126 MHz, CDCl₃) 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, 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 CO₂, λ = 210 nm): tᵣ (minor) = 7.3 min, tᵣ (major) = 8.1 min. [α]ᵣ²⁵ = −146.8° (c = 1.57, CHCl₃); ¹H NMR (500 MHz, CDCl₃) 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); ¹³C NMR (126 MHz, CDCl₃) 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.
(3R,5R,8R,9S,10S,12S,13R,14S,17R)-10,13-Dimethyl-17-((2R,6S)-5-oxo-6-phenylheptan-2-yl)hexadecahydro-1H-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. NaHCO₃ (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). [α]D²⁵ = 146.0° (c = 2.05, CHCl₃); ¹H NMR (500 MHz, Acetone-d₆) 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); ¹³C NMR (126 MHz, CDCl₃) 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+H₂O]⁺ 582.4, found 582.4.
**5. SFC Traces of Racemic and Enantioenriched Ketone Products**

3a (Table 2, entry 1): racemic

![Graph of peak retention times for racemic 3a](image)

<p>| Peak RetTime Type Width Area Height Area % |</p>
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<td>1.13526e4</td>
<td>711.29791</td>
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</table>

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

![Graph of peak retention times for enantioenriched 3a](image)

<p>| Peak RetTime Type Width Area Height Area % |</p>
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3b (Table 2, entry 2): racemic

![Graph of 3b (racemic)]

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

![Graph of 3b (93% ee)]

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3c (Table 2, entry 3): racemic

![Graph 1](Image)

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

![Graph 2](Image)

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3d (Table 2, entry 4): racemic

![Racemic 3d Chromatogram](image)

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

![Enantioenriched 3d Chromatogram](image)

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3e (Table 2, entry 5): racemic

![DAD1, Sig=210.8 Ref=360.100 (AHC/AHC 2012-08-09 08-57-05/AHC-4-59-5-S3C6-10.D)](image)

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

![DAD1, Sig=210.8 Ref=360.100 (AHC/AHC 2012-08-09 08-57-05/AHC-4-59-5-S3C6-10.D)](image)

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3f (Table 2, entry 6): racemic

![Graph showing peak retention times and areas for 3f (racemic)]

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

![Graph showing peak retention times and areas for 3f (91% ee)]

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3g (Table 2, entry 7): racemic

![Graph of 3g racemic](image)

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

![Graph of 3g ee](image)

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**3h (Table 2, entry 8):** racemic

![Graph of 3h](image)

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

![Graph of 3h with 82% ee](image)

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3i (Table 2, entry 9): racemic

![Diagram of 3i](image1)

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

![Diagram of 3i](image2)

| Peak RetTime | Type  | Width | Area       | Height       | Area %
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3j (Table 2, entry 10): racemic

![Graph of racemic compound 3j](image1)

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<td>5341.29736</td>
<td>388.53415</td>
<td>49.8184%</td>
</tr>
<tr>
<td>2</td>
<td>6.837 MM</td>
<td>0.2540</td>
<td>5380.24316</td>
<td>353.03430</td>
<td>50.1816%</td>
</tr>
</tbody>
</table>

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

![Graph of enantioenriched compound 3j](image2)

<table>
<thead>
<tr>
<th>#</th>
<th>Ret Time [min]</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.179 MM</td>
<td>0.1983</td>
<td>422.02435</td>
<td>35.47295</td>
<td>3.1063%</td>
</tr>
<tr>
<td>2</td>
<td>6.898 MM</td>
<td>0.2556</td>
<td>1.31640e4</td>
<td>858.35632</td>
<td>96.8937%</td>
</tr>
</tbody>
</table>
### 3k (Table 2, entry 11): racemic

![Graph showing peak retention times and areas for 3k](image)

<table>
<thead>
<tr>
<th>#</th>
<th>[min]</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>17.749</td>
<td>MM</td>
<td>0.6673</td>
<td>2.53780e4</td>
<td>633.81763</td>
</tr>
<tr>
<td>2</td>
<td>19.022</td>
<td>MM</td>
<td>0.8126</td>
<td>2.67237e4</td>
<td>548.09839</td>
</tr>
</tbody>
</table>

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

![Graph showing peak retention times and areas for 3k](image)

<table>
<thead>
<tr>
<th>#</th>
<th>[min]</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.118</td>
<td>MM</td>
<td>0.5944</td>
<td>580.79498</td>
<td>16.28630</td>
</tr>
<tr>
<td>2</td>
<td>19.381</td>
<td>MM</td>
<td>0.7154</td>
<td>1.27287e4</td>
<td>296.55020</td>
</tr>
</tbody>
</table>
3l (Table 2, entry 12): racemic

![Graph of racemic 3l](image)

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>4.522 MM</td>
<td>0.1239</td>
<td>1.06024e4</td>
<td>1426.70251</td>
<td>49.4425</td>
</tr>
<tr>
<td>2</td>
<td>5.244 MM</td>
<td>0.1721</td>
<td>1.08415e4</td>
<td>1050.01685</td>
<td>50.5575</td>
</tr>
</tbody>
</table>

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

![Graph of 92% ee 3l](image)

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>4.528 MM</td>
<td>0.1377</td>
<td>1.34978e4</td>
<td>1634.09204</td>
<td>95.9039</td>
</tr>
<tr>
<td>2</td>
<td>5.330 MM</td>
<td>0.1513</td>
<td>576.49988</td>
<td>63.49461</td>
<td>4.0961</td>
</tr>
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</table>
3m (Table 2, entry 13): racemic

![Graph of racemic 3m]

<table>
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<tr>
<th>#</th>
<th>Ret Time [min]</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.308</td>
<td>0.1105</td>
<td>3324.00073</td>
<td>501.56870</td>
<td>49.8322</td>
</tr>
<tr>
<td>2</td>
<td>3.723</td>
<td>0.1365</td>
<td>3346.39185</td>
<td>408.55920</td>
<td>50.1678</td>
</tr>
</tbody>
</table>

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

![Graph of 89% ee 3m]

<table>
<thead>
<tr>
<th>#</th>
<th>Ret Time [min]</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.287</td>
<td>0.1129</td>
<td>5205.23877</td>
<td>768.54687</td>
<td>94.5053</td>
</tr>
<tr>
<td>2</td>
<td>3.754</td>
<td>0.1313</td>
<td>302.64224</td>
<td>38.40394</td>
<td>5.4947</td>
</tr>
</tbody>
</table>
3n (Table 2, entry 14): racemic

![Graph showing chromatogram of 3n](image)

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>10.925 MM</td>
<td>0.3357</td>
<td>1403.79077</td>
<td>69.69751</td>
</tr>
<tr>
<td>2</td>
<td>11.946 MM</td>
<td>0.4028</td>
<td>197.63620</td>
<td>8.17804</td>
</tr>
</tbody>
</table>

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

![Graph showing chromatogram of 3n](image)

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>10.911 MM</td>
<td>0.3519</td>
<td>4747.33496</td>
<td>224.86562</td>
</tr>
<tr>
<td>2</td>
<td>11.946 MM</td>
<td>0.4028</td>
<td>197.63620</td>
<td>8.17804</td>
</tr>
</tbody>
</table>
3o (Table 2, entry 15): racemic

![Diagram of racemic 3o]

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>8.651</td>
<td>0.3179</td>
<td>9735.89941</td>
<td>510.35568</td>
</tr>
<tr>
<td>2</td>
<td>9.651</td>
<td>0.3663</td>
<td>1.01652e4</td>
<td>462.52780</td>
</tr>
</tbody>
</table>

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

![Diagram of 78% ee 3o]

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>7.943</td>
<td>0.2586</td>
<td>1696.82703</td>
<td>109.37256</td>
</tr>
<tr>
<td>2</td>
<td>8.880</td>
<td>0.3030</td>
<td>1.37415e4</td>
<td>755.81702</td>
</tr>
</tbody>
</table>
7a (Table 3): racemic

![Graph of racemic compound]

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.882 MM</td>
<td>0.0607</td>
<td>468.89008</td>
<td>128.69202</td>
<td>51.9944</td>
</tr>
<tr>
<td>2</td>
<td>2.020 MM</td>
<td>0.0587</td>
<td>432.91794</td>
<td>122.82136</td>
<td>48.0056</td>
</tr>
</tbody>
</table>

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

![Graph of enantioenriched compound]

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.848 MM</td>
<td>0.0672</td>
<td>502.57666</td>
<td>124.59120</td>
<td>4.5163</td>
</tr>
<tr>
<td>2</td>
<td>1.973 MM</td>
<td>0.0923</td>
<td>1.06255e4</td>
<td>1918.92407</td>
<td>95.4837</td>
</tr>
</tbody>
</table>
7b (Table 3): racemic

![Graph of peak retention time and area](image1)

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Width</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.147</td>
<td>MM</td>
<td>0.1035</td>
<td>3200.22705</td>
<td>515.47467</td>
</tr>
<tr>
<td>2</td>
<td>2.651</td>
<td>MM</td>
<td>0.1261</td>
<td>3176.07007</td>
<td>419.70117</td>
</tr>
</tbody>
</table>

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

![Graph of peak retention time and area](image2)

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Width</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.186</td>
<td>MM</td>
<td>0.1493</td>
<td>1238.74841</td>
<td>138.30533</td>
</tr>
<tr>
<td>2</td>
<td>2.675</td>
<td>MM</td>
<td>0.1852</td>
<td>2.06659e4</td>
<td>1859.45630</td>
</tr>
</tbody>
</table>
7d (Table 3): racemic

![Graph of racemic compound]

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>5.415 MM</td>
<td>0.1596</td>
<td>1.31741e4</td>
<td>1375.35034</td>
</tr>
<tr>
<td>2</td>
<td>5.880 MM</td>
<td>0.1802</td>
<td>1.31521e4</td>
<td>1216.19214</td>
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</tbody>
</table>

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

![Graph of enantioenriched compound]

<table>
<thead>
<tr>
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<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
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</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>5.371 MM</td>
<td>0.1661</td>
<td>412.66797</td>
<td>41.40784</td>
</tr>
<tr>
<td>2</td>
<td>5.833 MM</td>
<td>0.1703</td>
<td>5213.12402</td>
<td>510.12839</td>
</tr>
</tbody>
</table>
7e (Table 3): racemic

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

![Diagram of racemic 7f](image)

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>5.744</td>
<td>0.1945</td>
<td>8898.86133</td>
<td>762.63263</td>
<td>49.4914</td>
</tr>
<tr>
<td>2</td>
<td>6.413</td>
<td>0.2244</td>
<td>9081.74902</td>
<td>674.62744</td>
<td>50.5086</td>
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</table>

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

![Diagram of enantioenriched 7f](image)

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>5.798</td>
<td>0.1418</td>
<td>69.75076</td>
<td>8.19656</td>
<td>4.0504</td>
</tr>
<tr>
<td>2</td>
<td>6.465</td>
<td>0.1542</td>
<td>1652.33777</td>
<td>178.58195</td>
<td>95.9496</td>
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</table>
7g (Table 3): racemic

![Graph](image)

<table>
<thead>
<tr>
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<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>7.107</td>
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<td>7.963</td>
<td>0.2795</td>
<td>8043.45215</td>
<td>479.56842</td>
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</tbody>
</table>

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

![Graph](image)

<table>
<thead>
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<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>7.295</td>
<td>0.2652</td>
<td>355.56323</td>
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</tr>
<tr>
<td></td>
<td>2</td>
<td>8.144</td>
<td>0.2027</td>
<td>4556.22852</td>
<td>374.71005</td>
</tr>
</tbody>
</table>
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: $[\alpha]_D^{25} = -225.9^\circ$ (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

Sample Name: MTK-II-86-DMBAmixedanhydride
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/nkadunce/vnmrsys/data
Sample directory: MTK-II-86-DMBAmixedanhydride
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
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
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 (s2pu1)
Solvent: dcl3
Data collected on: Apr 12 2013

Sample #14, Operator: nkadunc

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, 499.7047131 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min
Sample Name:
MTK-I-289-II
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/nkadunce/vnmrsys/data
Sample directory:
MTK-I-289-II
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
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
J2 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-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: cdc13
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
WALTS-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 25 min
Sample Name:
  ahs-3-55-1
Data Collected on:
  indy.caltech.edu-inova500
Archive directory:
  /home/acherney/vnmrsys/data
Sample directory:
  ahs-3-55-1
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Mar 18 2012

Sample #40, Operator: archerney

  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
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: cdc13
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, 125.6555310 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
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 (s2p1)
Solvent: cdc13
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
J2 repetitions
OBSERVE  H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
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: cdc13
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
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: cdc13
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
Sample Name: CH-ahc-4-281-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vmsys/data
Sample directory:
/CH-ahc-4-281-1
FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
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 31609.5 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
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: cdc13
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
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: cdc13
Data collected on: Feb 10 2013

Sample #45, Operator: acherney

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.043 sec
Height 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
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
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: cdc13
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, 125.6509014 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
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
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: cdc13
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
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: cdc13
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

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S53
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: dcl3
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
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: cdc13
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
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: cdc13
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
Sample Name:  
MTK-II-NaphthylXtals  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/nkadunce/vnmrsys/data  
Sample directory:  
MTK-II-NaphthylXtals  
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)  
Solvent: cdc13  
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 85536  
Total time 4 min 17 sec
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: cdc13
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
  WALTS-16 modulated
  DATA PROCESSING
  Line broadening 0.5 Hz
  FT size 65536
  Total time 34 min
Sample Name:  CH-ahc-4-285-2
Data Collected on:  indy.caltech.edu-inova500
Archive directory:  /home/acherney/vnmrsyndata
Sample directory:  CH-ahc-4-285-2
FidFile:  PROTON01

Pulse Sequence:  PROTON (s2pul)
Solvent:  cdc13
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
J2 repetitions
OBSERVE  H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
Sample Name:
CH-ahc-4-285-2
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/acherney/vnmsys/data
Sample directory:
CH-ahc-4-285-2
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
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 3149.5 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
Sample Name: NTK-II-99-1-pure
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/nkadunce/vnmrsys/data
Sample directory: NTK-II-99-1-pure
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
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:  
MTK-II-99-I-pure 
Data Collected on: indy.caltech.edu-inova500 
Archive directory: /home/nkadunce/vnmrsys/data 
Sample directory: 
MTK-II-99-I-pure 
FidFile: CARBON01 

Pulse Sequence: CARBON (s2pul) 
Solvent: cdc13 
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
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
J2 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
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: cdc13
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
WALTS-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min

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: cdc13
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
WALTS-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 34 min
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: cdc13
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
J2 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
Sample Name: CH-ahc-5-91-1
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/acherney/vmsrsys/data
Sample directory: CH-ahc-5-91-1
FidFile: CARB0N01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
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
Sample Name: CH-abc-5-33-4
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/acherney/vnmrsys/data
Sample directory: CH-abc-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
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: cdc13  
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
Sample Name: MTK-II-indanylpure
Data Collected on: hp3.caltech.edu-mercury300
Archive directory: /home/nkadunce/vnmrsys/data
Sample directory: MTK-II-indanylpure
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
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
Sample Name: MTK-II-indanylC13
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/nkadunce/vmrsys/data
Sample directory: MTK-II-indanylC13
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
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
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: cdc13
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
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: cchl3
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
Sample Name:
MTK-II-71-I-ValPure

Data Collected on:
indy.caltech.edu-inova500

Archive directory:
/home/nkadunce/vnmrsys/data

Sample directory:
MTK-II-71-I-ValPure

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
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: cdc13
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
Sample Name:  NTK-II-3PhBut_RR  
Data Collected on:  indy.caltech.edu-inova500  
Archive directory:  
/home/nkadunce/vrmsys/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
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 (s2pol)
Solvent: cdc13
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
Sample Name: NTK-II-3PhBut_SS
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/akadunce/vnmrsys/data
Sample directory: NTK-II-3PhBut_SS
FidFile: PROTON01

Pulse Sequence: PROTON (2pul)
Solvent: cdcl3
Data collected on: Feb 21 2013

Sample #48, Operator: akadunce

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-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: dcl3
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
Sample Name:
CH-ntk-2-51-2
Data Collected on:
indty.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
J2 repetitions
OBSERVE H1, 499.7049145 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 min 8 sec
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 (s2pu)
Solvent: cdc13
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
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: cdc13
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
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
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  
J2 repetitions  
OBSERVE H1, 499.7049145 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 2 min 8 sec
Sample Name:
CH-ahc-5-59-1
Data Collected on:
indy.caltech.edu-inova500
Archive directory:
/home/cherney/vnmrsys/data
Sample directory:
CH-ahc-5-59-1
FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)
Solvent: cdc13
Data collected on: Feb 17 2013

Sample #8, Operator: cherney

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
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: dcl3
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
Sample Name: CH-ahe-5-55-1
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/acherney/vmrsys/data
Sample directory: CH-ahe-5-55-1
FidFile: CARBON01

Pulse Sequence: CARBON (s2pol)
Solvent: cdc13
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
Sample Name: MTK-II-steroid_dry
Data Collected on: indy.caltech.edu-inova500
Archive directory: /home/nkadunce/vnmrsys/data
Sample directory: MTK-II-steroid_dry
FidFile: PROTON01

Pulse Sequence: PROTON (s2p1)
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
Sample Name:  MTK-II-steroid_dry
Data Collected on: indy.caltech.edu-inova500
Archive directory:  /home/nkadunce/vnmrsys/data
Sample directory:  MTK-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