Supporting information for:

Highly Stereoselective Aldol Reactions of Titanium Enolates from Lactate-Derived Chiral Ketones

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1. Characterisation data for compounds 3, 5, and 6.

1. (2*S*,4*S*,5*R*)-5-Hydroxy-2-(*p*-methoxybenzyloxy)-4,6-dimethyl-3-heptanone (3a).

Colourless oil. R_f (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 13.3 min. $[\alpha]_D = -18.5$ ($c = 1.78$, CHCl₃). **IR** (film): ν 3519 (br), 2961, 2939, 1710, 1515, 1250, 1111 cm⁻¹. **¹H NMR** (300 MHz, CDCl₃) δ 7.29–7.26 (2H, m, ArH), 6.90–6.87 (2H, m, ArH), 4.50 (2H, s, ArCH₂O), 4.03 (1H, q, $J = 6.7$, CHOPMB), 3.81 (3H, s, OCH₃), 3.45 (1H, dd, $J = 8.2$, $J = 2.9$, CHOH), 3.22 (1H, qd, $J = 7.0$, $J = 2.9$, COCHCH₃), 1.72–1.60 (1H, m, CH₃CHCH₃), 1.35 (3H, d, $J = 6.7$, CH₃CHOPMB), 1.07 (3H, d, $J = 7.0$, COCHCH₃), 1.00 (3H, d, $J = 6.6$, CH₃CHCH₃), 0.85 (3H, d, $J = 6.9$, CH₃CHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 217.0, 159.4, 129.5, 113.9, 79.0, 76.4, 71.4, 55.3, 42.6, 30.7, 19.1, 19.0, 16.7, 9.3. **HRMS** (+FAB): m/z calcd. for C₁₇H₂₇O₄ [M+H]⁺: 295.1909. Found: 295.1915.

2. (2*S*,4*S*,5*R*)-5-Hydroxy-2-(*p*-methoxybenzyloxy)-4,7-dimethyl-3-octanone (3b).

Colourless oil. R_f (hexanes/EtOAc 80:20) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 15.5 min. $[\alpha]_D = -3.7$ ($c = 1.39$, CHCl₃). **IR** (film): ν 3504 (br), 2956, 2871, 1717, 1515, 1250, 1036 cm⁻¹. **¹H NMR** (300 MHz, CDCl₃) δ 7.29–7.26 (2H, m, ArH), 6.90–6.87 (2H, m, ArH), 4.50 (2H, s, ArCH₂O), 4.03 (1H, q, $J = 6.7$, CHOPMB), 3.93–3.91 (1H, m, CHOH), 3.81 (3H, s, OCH₃), 3.01 (1H, qd, $J = 7.0$, $J = 3.0$, COCHCH₃), 1.82–1.68 (1H, m, CH₃CHCH₃), 1.48–1.39 (1H, m, CH(OH)CH_xH_y), 1.35 (3H, d, $J = 6.7$, CH₃CHOPMB), 1.14–1.05 (1H, m, CH(OH)CH_xH_y), 1.08 (3H, d, $J = 7.0$, COCHCH₃), 0.91 (3H, d, $J = 6.7$, CH₃CHCH₃), 0.90 (3H, d, $J = 6.7$, CH₃CHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 216.7, 159.5, 129.5, 113.9, 79.1, 71.4, 69.3, 55.3, 45.6, 43.2, 24.6, 23.4, 21.9, 16.5, 9.9. **HRMS** (+FAB): m/z calcd. for C₁₈H₂₉O₄ [M+H]⁺: 309.2066. Found: 309.2079.

3. (2*S*,4*S*,5*R*)-5-Hydroxy-2-(*p*-methoxybenzyloxy)-4-methyl-3-heptanone (3c).

Colourless oil. R_f (hexanes/EtOAc 80:20) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 18.5 min. $[\alpha]_D = -13.8$ ($c = 1.11$, CHCl_3). **IR** (film): ν 3494 (br), 2966, 2937, 1711, 1515, 1250, 1036 cm^{-1} . **^1H NMR** (300 MHz, CDCl_3) δ 7.30–7.20 (2H, m, ArH), 6.95–6.85 (2H, m, ArH), 4.50 (2H, s, ArCH_2O), 4.03 (1H, q, $J = 6.8$, CHOPMB), 3.81 (3H, s, OCH_3), 3.81–3.70 (1H, m, CHOH), 3.07 (1H, qd, $J = 7.1$, $J = 3.0$, COCHCH_3), 1.60–1.30 (2H, m, CH_2CH_3), 1.35 (3H, d, $J = 6.8$, CH_3CHOPMB), 1.08 (3H, d, $J = 7.1$, COCHCH_3), 0.94 (3H, t, $J = 7.4$, CH_2CH_3). **^{13}C NMR** (75.4 MHz, CDCl_3) δ 216.7, 159.5, 129.5, 113.9, 79.2, 72.9, 71.5, 55.3, 44.9, 27.0, 16.6, 10.5, 9.9.

4. (2*S*,4*S*,5*S*)-5-Hydroxy-2-(*p*-methoxybenzyloxy)-4,6-dimethyl-6-hepten-3-one (3d).

Colourless oil. R_f (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 16.1 min. $[\alpha]_D = -11.4$ ($c = 2.27$, CHCl_3). **IR** (film): ν 3490 (br), 2981, 2939, 1717, 1613, 1515, 1457, 1250, 1111 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.28–7.26 (2H, m, ArH), 6.90–6.88 (2H, m, ArH), 5.07 (1H, br s, $\text{C}(\text{CH}_3)=\text{CH}_x\text{H}_y$), 4.94 (1H, br s, $\text{C}(\text{CH}_3)=\text{CH}_x\text{H}_y$), 4.50 (2H, s, ArCH_2O), 4.36 (1H, br s, CHOH), 4.05 (1H, q, $J = 6.8$, CHOPMB), 3.81 (3H, s, OCH_3), 3.22 (1H, qd, $J = 7.0$, $J = 3.5$, COCHCH_3), 1.68 (3H, br s, $\text{C}(\text{CH}_3)=\text{CH}_2$), 1.37 (3H, d, $J = 6.8$, CH_3CHOPMB), 1.03 (3H, d, $J = 7.0$, COCHCH_3). **^{13}C NMR** (75.4 MHz, CDCl_3) δ 216.2, 159.4, 143.7, 129.5, 129.4, 113.9, 111.9, 79.2, 74.0, 71.5, 55.3, 43.2, 19.3, 16.7, 9.6. **HRMS** (+FAB): m/z calcd. for $\text{C}_{17}\text{H}_{25}\text{O}_4$ $[\text{M}+\text{H}]^+$: 293.1753. Found: 293.1737.

5. (1*S*,2*S*,4*S*)-1-Hydroxy-4-(*p*-methoxybenzyloxy)-2-methyl-1-phenyl-3-pentanone (3e).

Colourless oil. R_f (hexanes/EtOAc 85:15) = 0.3. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 18.1 min. $[\alpha]_D = +1.3$ ($c = 1.55$, CHCl_3). **IR** (film): ν 3475 (br), 3033, 2937, 2838, 1717, 1515, 1248, 1034 cm^{-1} . **^1H NMR** (300 MHz, CDCl_3) δ 7.32–7.25 (5H, m, ArH), 7.22–7.19 (2H, m, ArH), 6.88–6.85 (2H, m, ArH), 4.97 (1H, d, $J = 4.7$, CHOH), 4.37 (1H, AB system, $J = 11.2$, $\text{ArCH}_x\text{H}_y\text{O}$), 4.24 (1H, AB system, $J = 11.2$, $\text{ArCH}_x\text{H}_y\text{O}$), 3.86 (1H, q, $J = 6.8$, CHOPMB), 3.79 (3H, s, OCH_3), 3.29 (1H, qd, $J = 7.0$, $J = 4.7$, COCHCH_3), 1.24 (3H, d, $J = 6.8$, CH_3CHOPMB), 1.09 (3H, d, $J = 7.0$, COCHCH_3). **^{13}C NMR** (75.4 MHz, CDCl_3) δ 215.9, 159.4, 141.8, 129.6, 129.4, 128.3, 127.5, 126.3, 113.8, 79.7, 73.8, 71.4, 55.3, 48.0, 16.4, 11.1. **HRMS** (+FAB): m/z calcd. for $\text{C}_{20}\text{H}_{25}\text{O}_4$ $[\text{M}+\text{H}]^+$: 329.1753. Found: 329.1763.

6. (1*S*,2*S*,4*S*)-1-(*p*-Chlorophenyl)-1-hydroxy-4-(*p*-methoxybenzyloxy)-2-methyl-3-pentanone (3f).

White solid. **mp** = 71–73 °C. **R_f** (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) **t_R** = 20.0 min. **[α]_D** = −7.3 (*c* = 1.58, CHCl₃). **IR** (film): ν 3467 (br), 2981, 2937, 1717, 1613, 1515, 1250, 1175, 1090 cm^{−1}. **¹H NMR** (300 MHz, CDCl₃) δ 7.32–7.20 (6H, m, ArH), 6.90–6.87 (2H, m, ArH), 4.98 (1H, dd, *J* = 4.2, *J* = 1.8, CHOH), 4.43 (1H, AB system, *J* = 11.3, ArCH₂H_yO), 4.37 (1H, AB system, *J* = 11.3, ArCH₂H_yO), 3.92 (1H, q, *J* = 6.9, CHOPMB), 3.81 (3H, s, OCH₃), 3.27 (1H, qd, *J* = 4.2, *J* = 7.0, COCHCH₃), 1.28 (3H, d, *J* = 6.9, CH₃CHOPMB), 1.04 (3H, d, *J* = 7.0, COCHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 216.1, 159.5, 140.2, 133.2, 129.5, 128.4, 127.6, 113.9, 79.4, 72.9, 71.5, 55.3, 47.6, 16.3, 10.5.

7. (2*S*,4*S*,5*R*)-2-Benzyloxy-5-hydroxy-4,6-dimethyl-3-heptanone (5a).

Colourless oil. **R_f** (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) **t_R** = 8.7 min. **[α]_D** = −16.0 (*c* = 1.06, CHCl₃). **IR** (film): ν 3512 (br), 2964, 2875, 1709, 1457, 1117 cm^{−1}. **¹H NMR** (500 MHz, CDCl₃) δ 7.40–7.30 (5H, m, ArH), 4.57 (2H, s, PhCH₂O), 4.07 (1H, q, *J* = 6.7, CHOBn), 3.47 (1H, dd, *J* = 8.3, *J* = 2.9, CHOH), 3.25 (1H, qd, *J* = 7.0, *J* = 2.9, COCHCH₃), 1.72–1.62 (1H, m, CH₃CHCH₃), 1.38 (3H, d, *J* = 6.7, CH₃CHOBn), 1.08 (3H, d, *J* = 7.0, COCHCH₃), 1.01 (3H, d, *J* = 6.5, CH₃CHCH₃), 0.86 (3H, d, *J* = 6.7, CH₃CHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 216.9, 137.5, 128.5, 127.9, 127.8, 79.4, 76.4, 71.8, 42.6, 30.8, 19.0, 19.0, 16.8. **HRMS** (+FAB): *m/z* calcd. for C₁₆H₂₅O₃ [M+H]⁺: 263.1804. Found: 263.1817.

8. (2*S*,4*S*,5*R*)-2-Benzyloxy-5-hydroxy-4,7-dimethyl-3-octanone (5b).

Colourless oil. **R_f** (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) **t_R** = 9.5 min. **[α]_D** = +0.9 (*c* = 1.46, CHCl₃). **IR** (film): ν 3490 (br), 2958, 2871, 1713, 1455, 1113 cm^{−1}. **¹H NMR** (500 MHz, CDCl₃) δ 7.37–7.32 (5H, m, ArH), 4.57 (2H, s, PhCH₂O), 4.07 (1H, q, *J* = 7.0, CHOBn), 3.94 (1H, ddd, *J* = 9.4, *J* = 4.0, *J* = 3.0, CHOH), 3.03 (1H, qd, *J* = 7.2, *J* = 3.0, COCHCH₃), 1.80–1.72 (1H, m, CH₃CHCH₃), 1.45 (1H, ddd, *J* = 13.7, *J* = 9.4, *J* = 5.3, CH(OH)CH₂H_y), 1.38 (3H, d, *J* = 7.0, CH₃CHOBn), 1.10 (3H, d, *J* = 7.2, COCHCH₃), 1.10 (1H, ddd, *J* = 13.7, *J* = 8.7, *J* = 4.0, CH(OH)CH₂H_y), 0.92 (3H, d, *J* = 6.7, CH₃CHCH₃), 0.90 (3H, d, *J* = 6.7, CH₃CHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 216.5, 137.4, 128.5, 127.9, 127.8, 79.5, 71.7, 69.3, 45.7, 43.1, 24.6, 23.4, 21.9, 16.5, 9.9. **HRMS** (+FAB): *m/z* calcd. for C₁₇H₂₇O₃ [M+H]⁺: 279.1960. Found: 279.1969.

9. (2S,4S,5R)-2-Benzyloxy-5-hydroxy-4-methyl-3-heptanone (5c).

Colourless oil. R_f (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 15.4 min. $[\alpha]_D = -12.0$ ($c = 1.25$, CHCl_3). **IR** (film): ν 3490 (br), 2975, 2937, 1715, 1457, 1104 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.40–7.30 (5H, m, ArH), 4.57 (2H, s, PhCH_2O), 4.07 (1H, q, $J = 6.8$, CHOBn), 3.80–3.70 (1H, m, CHOH), 3.09 (1H, qd, $J = 7.1$, $J = 3.1$, COCHCH_3), 1.60–1.35 (2H, CH_2CH_3), 1.37 (3H, d, $J = 6.8$, CH_3CHOBn), 1.09 (3H, d, $J = 7.1$, COCHCH_3), 0.95 (3H, t, $J = 7.4$, CH_2CH_3). **^{13}C NMR** (100.6 MHz, CDCl_3) δ 216.6, 137.5, 128.5, 128.0, 127.8, 79.6, 72.9, 71.8, 44.9, 27.0, 16.6, 10.4, 9.9.

10. (2S,4S,5S)-2-Benzyloxy-5-hydroxy-4,6-dimethyl-6-hepten-3-one (5d).

Colourless oil. R_f (hexanes/EtOAc 85:15) = 0.3. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 10.8 min. $[\alpha]_D = -12.7$ ($c = 1.07$, CHCl_3). **IR** (film): ν 3483 (br), 3033, 2983, 2937, 1715, 1654, 1455, 1119 cm^{-1} . **^1H NMR** (500 MHz, CDCl_3) δ 7.40–7.30 (5H, m, ArH), 5.07 (1H, br s, $\text{C}(\text{CH}_3)=\text{CH}_x\text{H}_y$), 4.94 (1H, br s, $\text{C}(\text{CH}_3)=\text{CH}_x\text{H}_y$), 4.57 (2H, s, PhCH_2O), 4.38–4.37 (1H, m, CHOH), 4.08 (1H, q, $J = 7.0$, CHOBn), 3.24 (1H, qd, $J = 7.0$, $J = 3.7$, COCHCH_3), 1.69 (3H, br s, $\text{C}(\text{CH}_3)=\text{CH}_2$), 1.40 (3H, d, $J = 7.0$, CH_3CHOBn), 1.05 (3H, d, $J = 7.0$, COCHCH_3). **^{13}C NMR** (75.4 MHz, CDCl_3) δ 215.9, 143.8, 137.5, 128.5, 127.9, 127.8, 112.0, 79.6, 74.0, 71.8, 43.2, 19.2, 16.7, 9.7. **HRMS** (+FAB): m/z calcd. for $\text{C}_{16}\text{H}_{23}\text{O}_3$ $[\text{M}+\text{H}]^+$: 263.1647. Found: 263.1657.

11. (1S,2S,4S)-4-Benzyloxy-1-hydroxy-2-methyl-1-phenyl-3-pentanone (5e).

White solid. **mp** = 56–57 °C. R_f (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 11.9 min. $[\alpha]_D = +6.9$ ($c = 1.37$, CHCl_3). **IR** (KBr): ν 3467 (br), 3033, 2983, 2936, 1717, 1455, 1121 cm^{-1} . **^1H NMR** (500 MHz, CDCl_3) δ 7.40–7.20 (10H, m, ArH), 4.97 (1H, d, $J = 5.0$, CHOH), 4.43 (1H, d, $J = 11.7$, $\text{PhCH}_x\text{H}_y\text{O}$), 4.29 (1H, d, $J = 11.7$, $\text{PhCH}_x\text{H}_y\text{O}$), 3.88 (1H, q, $J = 7.0$, CHOBn), 3.30 (1H, qd, $J = 7.0$, $J = 5.0$, COCHCH_3), 1.26 (3H, d, $J = 7.0$, CH_3CHOBn), 1.11 (3H, d, $J = 7.0$, COCHCH_3). **^{13}C NMR** (75.4 MHz, CDCl_3) δ 215.7, 141.8, 137.5, 128.4, 128.3, 127.8, 127.7, 127.6, 126.3, 80.1, 73.9, 71.7, 48.1, 16.4, 11.2. **HRMS** (+FAB): m/z calcd. for $\text{C}_{19}\text{H}_{23}\text{O}_3$ $[\text{M}+\text{H}]^+$: 299.1647. Found: 299.1636.

12. (1*S*,2*S*,4*S*)-4-Benzoyloxy-1-(*p*-chlorophenyl)-1-hydroxy-2-methyl-3-pentanone (5f).

White solid. R_f (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99.5:0.5) t_R = 24.5 min. $[\alpha]_D = -2.0$ ($c = 1.07$, CHCl₃). **IR** (KBr): ν 3467 (br), 3033, 2983, 2937, 1713, 1493, 1455, 1092 cm⁻¹. **¹H NMR** (300 MHz, CDCl₃) δ 7.40–7.20 (9H, m, ArH), 4.98 (1H, dd, $J = 4.2$, $J = 1.8$, CHOH), 4.49 (1H, AB system, $J = 11.6$, PhCH_xH_yO), 4.42 (1H, AB system, $J = 11.6$, PhCH_xH_yO), 3.93 (1H, q, $J = 6.9$, CHOBn), 3.29 (1H, qd, $J = 7.1$, $J = 4.2$, COCHCH₃), 1.29 (3H, d, $J = 6.9$, CH₃CHOBn), 1.05 (3H, d, $J = 7.1$, COCHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 215.9, 140.2, 137.3, 133.2, 128.5, 128.4, 128.0, 127.8, 127.6, 79.8, 72.9, 71.8, 47.7, 16.3, 10.7. **HRMS** (+FAB): m/z calcd. for C₁₉H₂₂ClO₃ [M+H]⁺: 333.1257. Found: 333.1262.

13. (2*S*,4*R*,5*R*)-2-Benzoyloxy-5-hydroxy-4,6-dimethyl-6-hepten-3-one (6d).

Colourless oil. R_f (hexanes/EtOAc 85:15) = 0.3. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 9.4 min. $[\alpha]_D = -27.1$ ($c = 1.37$, CHCl₃). **IR** (film): ν 3490 (br), 3033, 2983, 2939, 1717, 1654, 1457, 1121 cm⁻¹. **¹H NMR** (500 MHz, CDCl₃) δ 7.40–7.30 (5H, m, ArH), 5.09–5.08 (1H, m, C(CH₃)=CH_xH_y), 4.95–4.94 (1H, m, C(CH₃)=CH_xH_y), 4.57 (2H, s, PhCH₂O), 4.37 (1H, br s, CHOH), 4.09 (1H, q, $J = 7.0$, CHOBn), 3.20 (1H, qd, $J = 7.0$, $J = 3.3$, COCHCH₃), 1.65 (3H, br s, C(CH₃)=CH₂), 1.40 (3H, d, $J = 7.0$, CH₃CHOBn), 1.06 (3H, d, $J = 7.0$, COCHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 216.4, 143.6, 137.4, 128.5, 127.9, 127.7, 111.8, 79.5, 73.6, 71.7, 43.1, 19.3, 17.1, 9.8. **HRMS** (+FAB): m/z calcd. for C₁₆H₂₃O₃ [M+H]⁺: 263.1647. Found: 263.1644.

14. (1*R*,2*R*,4*S*)-4-Benzoyloxy-1-hydroxy-2-methyl-1-phenyl-3-pentanone (6e).

White oil. R_f (hexanes/EtOAc 75:25) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 8.7 min. $[\alpha]_D = -33.2$ ($c = 1.93$, CHCl₃). **IR** (film): ν 3475 (br), 3033, 2988, 2937, 1717, 1457, 1109 cm⁻¹. **¹H NMR** (250 MHz, CDCl₃) δ 7.36–7.25 (10H, m, ArH), 5.01 (1H, d, $J = 4.5$, CHOH), 4.49 (1H, AB system, $J = 11.7$, PhCH_xH_yO), 4.43 (1H, AB system, $J = 11.7$, PhCH_xH_yO), 3.93 (1H, q, $J = 6.9$, CHOBn), 3.24 (1H, qd, $J = 7.2$, $J = 4.5$, COCHCH₃), 1.24 (3H, d, $J = 6.9$, CH₃CHOBn), 1.10 (3H, d, $J = 7.2$, COCHCH₃). **¹³C NMR** (75.4 MHz, CDCl₃) δ 216.3, 141.8, 137.4, 128.4, 128.2, 127.9, 127.7, 127.3, 126.0, 79.5, 73.2, 71.6, 48.2, 16.9, 11.1. **HRMS** (+FAB): m/z calcd. for C₁₉H₂₃O₃ [M+H]⁺: 299.1647. Found: 299.1655.

15. (1*R*,2*R*,4*S*)-4-Benzoyloxy-1-(*p*-chlorophenyl)-1-hydroxy-2-methyl-3-pentanone (6f)

White oil. R_f (hexanes/EtOAc 85:15) = 0.2. **HPLC** (hexanes/*i*-PrOH 99:1) t_R = 10.9 min. $[\alpha]_D = -34.0$ ($c = 1.29$, CHCl_3). **IR** (film): ν 3473 (br), 3033, 2983, 2935, 1713, 1493, 1455, 1090 cm^{-1} . **^1H NMR** (400 MHz, CDCl_3) δ 7.38–7.19 (9H, m, ArH), 4.99 (1H, br s, CHOH), 4.50 (2H, s, PhCH_2O), 3.97 (1H, q, $J = 6.8$, CHOBn), 3.21 (1H, qd, $J = 7.0$, $J = 4.1$, COCHCH₃), 1.30 (3H, d, $J = 6.8$, CH₃CHOBn), 1.06 (3H, d, $J = 7.0$, COCHCH₃). **^{13}C NMR** (100.6 MHz, CDCl_3) δ 216.5, 140.2, 137.3, 133.1, 128.6, 128.4, 128.0, 127.8, 127.4, 79.6, 72.5, 71.7, 47.8, 17.1, 10.7.

2. ^1H NMR copies.

3a

^1H NMR (300 MHz, CDCl_3)

3d

^1H NMR (400 MHz, CDCl_3)

3e

¹H NMR (300 MHz, CDCl₃)

5a

^1H NMR (500 MHz, CDCl_3)

5e

¹H NMR (500 MHz, CDCl₃)

3. Crystal data for 3f and 5f.

1. (1S,2S,4S)-1-(p-Chlorophenyl)-1-hydroxy-4-(p-methoxybenzyloxy)-2-methyl-3-pentanone (3f)

Empirical formula	C ₂₀ H ₂₃ ClO ₄
Formula weight	362.83
Temperature	571 (2) K
Wavelength	0.71069 Å
Crystal system, space group	Monoclinic, P2 ₁
Unit cell dimensions	a = 11.069 (4) Å α = 90 ° b = 8.021 (8) Å β = 116.87 (2) ° c = 12.096 (2) Å γ = 90 °
Volume	958.0 (10) Å ³
Z, Calculated density	2, 1.258 Mg / m ³
Absorption coefficient	0.220 mm ⁻¹
F(000)	384
Crystal size	0.1 x 0.1 x 0.2 mm
Theta range for data collection	2.06 to 29.96 °
Index ranges	-15 ≤ h ≤ 13, -3 ≤ k ≤ 11, 0 ≤ l ≤ 17
Reflections collected / unique	3087 / 2956 [R(int) = 0.0253]
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2956 / 1 / 268
Goodness-of-fit on F ²	1.041
Final R indices [I > 2σ(I)]	R1 = 0.0580, wR2 = 0.1398
R indices (all data)	R1 = 0.1006, wR2 = 0.1596
Absolute structure parameter	0.08 (14)
Largest diff. peak and hole	0.348 and -0.282 e.Å ⁻³

2. (1*S*,2*S*,4*S*)-4-Benzoyloxy-1-(*p*-chlorophenyl)-1-hydroxy-2-methyl-3-pentanone (5f)

Empirical formula	C ₁₉ H ₂₁ ClO ₃
Formula weight	332.81
Temperature	293 (2) K
Wavelength	0.71069 Å
Crystal system, space group	Orthorhombic, P2 ₁ 2 ₁ 2 ₁
Unit cell dimensions	a = 8.3420 (10) Å α = 90 ° b = 10.8600 (10) Å β = 90 ° c = 19.3020 (10) Å γ = 90 °
Volume	1748.6 (3) Å ³
Z, Calculated density	4, 1.264 Mg / m ³
Absorption coefficient	0.230 mm ⁻¹
F(000)	704
Crystal size	0.1 x 0.1 x 0.2 mm
Theta range for data collection	2.66 to 24.88 °
Index ranges	0 ≤ h ≤ 9, 0 ≤ k ≤ 10, 0 ≤ l ≤ 22
Reflections collected / unique	7573 / 1490 [R(int) = 0.0437]
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1490 / 0 / 293
Goodness-of-fit on F ²	1.077
Final R indices [I > 2σ(I)]	R1 = 0.0417, wR2 = 0.1043
R indices (all data)	R1 = 0.0666, wR2 = 0.1147
Absolute structure parameter	0.34 (18)
Extinction coefficient	0.011 (3)
Largest diff. peak and hole	0.155 and -0.171 e.Å ⁻³