Cationic Rhodium(I)/BINAP Complex-Catalyzed Isomerization of Secondary Propargylic Alcohols to α,β -Enones

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I. General

Anhydrous CH_2Cl_2 (No. 27,099-7), anhydrous $(\text{CH}_2\text{Cl})_2$ (No. 28,450-5), and anhydrous acetone (No. 27,072-5) were obtained from Aldrich and used as received. Tol-BINAP, H8-BINAP, and Segphos were obtained from Takasago International Corporation. All other reagents were obtained from commercial sources and used as received. All reactions were carried out under an atmosphere of argon in oven-dried glassware with magnetic stirring, unless otherwise indicated.

II. Synthesis of Propargylic Alcohols

1-Phenylpent-1-yn-3-ol (**1a**).¹

n-BuLi (1.58 M in hexane, 11.5 mL, 18.2 mmol) was added to a stirred solution of phenylacetylene (1.9 g, 18.2 mmol) in THF (20 mL) at 0 °C, and the mixture was stirred at 0 °C for 30 min. To the resulting solution was added propionaldehyde (1.3 mL, 18.2 mmol) at 0 °C, and the mixture was gradually warmed to rt. The reaction was quenched by the addition of water and extracted with Et_2O . The organic layer was washed with brine, dried over Na_2SO_4 , and concentrated. The residue was purified by silica gel column chromatography (hexane:EtOAc = 8:1), which furnished 1-phenylpent-1-yn-3-ol **1a** (2.7 g, 16.6 mmol, 92% yield) as a pale yellow oil.

^1H NMR (CDCl_3 , 400 MHz) δ 7.37–7.46 (m, 2H), 7.26–7.37 (m, 3H), 4.50–4.60 (m, 1H), 1.98–2.05 (m, 1H), 1.75–1.88 (m, 2H), 1.08 (t, J = 7.6 Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 131.7, 128.3, 128.2, 122.6, 89.9, 84.9, 64.2, 30.9, 9.5.

4-Phenylbut-3-yn-2-ol (**1b**).²

The title compound was prepared as a yellow oil in 67% isolated yield from acetaldehyde according to the procedure of **1a**.

^1H NMR (CDCl_3 , 300 MHz) δ 7.35–7.50 (m, 2H), 7.25–7.40 (m, 3H), 4.70–4.85 (m,

1H), 1.90 (d, $J = 5.4$ Hz 1H) 1.55 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 131.6, 128.3, 128.2, 122.5, 90.9, 83.9, 58.8, 24.3.

1-Phenylhept-1-yn-3-ol (1c).¹

The title compound was prepared as a colorless solid in 91% isolated yield from valeraldehyde according to the procedure of **1a**.

^1H NMR (CDCl_3 , 300 MHz) δ 7.38–7.48 (m, 2H), 7.24–7.36 (m, 3H), 4.60 (q, $J = 6.3$ Hz, 1H), 1.90–2.00 (m, 1H) 1.70–1.88 (m, 2H), 1.30–1.58 (m, 4H), 0.94 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 131.6, 128.3, 128.2, 122.6, 90.2, 84.7, 62.9, 37.6, 27.3, 22.4, 14.0.

4-Methyl-1-phenylpent-1-yn-3-ol (1d).³

The title compound was obtained from Lancaster.

^1H NMR (CDCl_3 , 300 MHz) δ 7.39–7.48 (m, 2H), 7.25–7.36 (m, 3H), 4.40 (d, $J = 6.6$ Hz, 1H), 1.80–2.07 (m, 2H), 1.07 (t, $J = 6.6$ Hz, 6H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 131.7, 128.3, 128.2, 122.7, 88.9, 85.5, 68.3, 34.7, 18.2, 17.5.

4, 4-Dimethyl-1-phenylpent-1-yn-3-ol (1e).³

The title compound was prepared as a colorless solid in 99% isolated yield from trimethylacetaldehyde according to the procedure of **1a**.

^1H NMR (CDCl_3 , 300 MHz) δ 7.38–7.48 (m, 2H), 7.24–7.36 (m, 3H), 4.23 (d, $J = 6.3$ Hz, 1H), 1.86 (d, $J = 6.3$ Hz, 1H) 1.07 (s, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 131.6, 128.22, 128.20, 122.7, 88.9, 85.6, 71.7, 36.0, 25.3.

1-*o*-Tolylpent-1-yn-3-ol (1h).

A solution of CBr₄ (22.2 g, 66.9 mmol) in CH₂Cl₂ (50 mL) was added dropwise to a solution of PPh₃ (35.1 g, 133.8 mmol) in CH₂Cl₂ (50 mL) at 0 °C. After stirring at 0 °C for 5 min, a solution of 2-methylbenzaldehyde (4.2 g, 33.5 mmol) was added dropwise to the reaction mixture at 0 °C. The resulting mixture was stirred at 0 °C for 20 min. The reaction was quenched by the addition of water and saturated aqueous NaHCO₃. The resulting mixture was extracted with CH₂Cl₂, dried over Na₂SO₄, and concentrated. The residue was purified by silica gel chromatography (hexane:EtOAc = 8:1), which furnished 1-(2,2-dibromovinyl)-2-methylbenzene (7.3 g, 26.6 mmol, 79% yield) as a pale yellow solid.

n-BuLi (1.58 M in hexane, 9.1 mL, 14.3 mmol) was added to a stirred solution of 1-(2,2-dibromovinyl)-2-methylbenzene (2.0 g, 7.2 mmol) in THF (20 mL) at –10 °C, and the resulting mixture was stirred at –10 °C for 30 min. To the resulting solution was added propionaldehyde (0.50 mL, 7.2 mmol) at –10 °C, and the mixture was gradually warmed to rt. The reaction was quenched by the addition of water and extracted with Et₂O. The organic layer was washed with brine, dried over Na₂SO₄, and concentrated. The residue was purified by silica gel column chromatography (hexane:EtOAc = 8:1), which furnished 1-*o*-tolylpent-1-yn-3-ol (1.2 g, 6.6 mmol, 92%) as a pale yellow oil.

IR (neat) 3300, 2950, 1015, 745 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 7.37–7.42 (m, 1H), 7.09–7.25 (m, 3H), 4.64 (q, *J* = 6.0 Hz, 1H), 2.43 (s, 3H), 1.93–2.00 (m, 1H), 1.78–1.90 (m, 2H), 1.09 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 140.0, 131.8, 129.3, 128.3, 125.4, 122.3, 93.8, 83.7, 64.3, 31.1, 20.8, 9.6; HRMS (EI) calcd for C₁₂H₁₄O [M]⁺ 174.1045, found 174.1011.

1-(4-Methoxyphenyl)pent-1-yn-3-ol (1f).

The title compound was prepared as a pale yellow oil in 89% isolated yield from 1-(2,2-dibromovinyl)-4-methoxybenzene and propionaldehyde according to the procedure of **1h**.

IR (neat) 3350, 2950, 1600, 1508, 1245, 1180, 1030, 835 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 7.34–7.40 (m, 2H), 6.80–6.86 (m, 2H), 4.54 (q, *J* = 6.3 Hz, 1H), 3.80 (s, 3H), 1.96–2.06 (m, 1H), 1.74–1.90 (m, 2H), 1.07 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 159.6, 133.1, 114.7, 113.8, 88.5, 84.8, 64.2, 55.2, 31.0, 9.5. HRMS (EI) calcd for C₁₂H₁₄O₂ [M]⁺ 190.0994, found 190.0970.

1-(4-Trifluoromethylphenyl)pent-1-yn-3-ol (1g).⁴

The title compound was prepared as a pale brown oil in 74% isolated yield from 1-(2,2-dibromovinyl)-4-trifluoromethylbenzene and propionaldehyde according to the procedure of **1h**.

^1H NMR (CDCl_3 , 300 MHz) δ 7.50–7.61 (m, 4H), 4.57 (q, J = 6.0 Hz, 1H), 2.08 (d, J = 5.7 Hz, 1H), 1.78–1.90 (m, 2H), 1.09 (t, J = 7.5 Hz, 3H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 131.9, 130.3, 129.9, 126.4, 125.6, 125.2 (q), 122.0, 92.4, 83.6, 64.1, 30.8, 9.5.

2-Deuterio-4-phenylbut-3-yn-2-ol (**1b-d**).

To a solution of 4-phenyl-3-butyn-2-ol (**1b**, 1.0 g, 6.8 mmol) in acetone (20 mL) was added Jones reagent (1.5 M, 5.1 mL, 7.6 mmol) at 0 °C. After stirring at 0 °C for 15 min, the reaction was quenched by the addition of 2-PrOH (6.2 mL) at 0 °C. The mixture was stirred at rt for 10 min and concentrated. The residue was diluted with water and extracted with Et_2O . The organic layer was concentrated and purified by silica gel chromatography (hexane:EtOAc = 6:1), which furnished 4-phenyl-3-butyn-2-ol (0.49 g, 3.4 mmol, 50% yield) as a colorless oil.

To a stirred mixture of NaBD_4 (70.0 mg, 1.7 mmol) in EtOH (20 mL) was added a EtOH (5.0 mL) solution of 4-phenylbut-3-yn-2-on (0.49 g, 3.4 mmol) at 0 °C. The resulting mixture was stirred at 0 °C for 1 h. The reaction was quenched with aqueous HCl (2 M, 10 mL), diluted with brine (80 mL), and extracted with CH_2Cl_2 . The organic layer was concentrated and purified by silica gel chromatography (hexane:EtOAc = 6:1), which furnished 2-deuterio-4-phenylbut-3-yn-2-ol (**1b-d**, 0.40 g, 2.7 mmol, 81% yield) as a colorless oil.

^1H NMR (CDCl_3 , 300 MHz) δ 7.40–7.46 (m, 2H), 7.27–7.35 (m, 3H), 1.96–2.05 (m, 1H), 1.55 (s, 3H); ^2H NMR (CDCl_3 , 61 MHz) δ 4.76 (s, 1H).

III. Isomerization of Secondary Propargylic Alcohols

General Procedure 1 (Table 2, entry 1). Under an Ar atmosphere, a CH_2Cl_2 (0.5 mL) solution of BINAP (15.6 mg, 0.025 mmol) was added to a CH_2Cl_2 (0.5 mL) solution of $[\text{Rh}(\text{cod})_2]\text{BF}_4$ (10.2 mg, 0.025 mmol) at rt, and the mixture was stirred for 5 min at rt. H_2 was introduced to the resulting solution in a Schlenk tube. After stirring for 0.5 h at rt, the resulting solution was concentrated to dryness and dissolved in $(\text{CH}_2\text{Cl}_2)_2$ (1.0 mL). To this solution was added a $(\text{CH}_2\text{Cl}_2)_2$ (0.5 mL) solution of 4-phenylbut-3-yn-2-ol (**1b**, 73.1 mg, 0.5 mmol), and washed remaining substrate away by using $(\text{CH}_2\text{Cl}_2)_2$ (0.5 mL). The solution was stirred at 80 °C for 1 h. The resulting solution was

concentrated and purified by silica gel column chromatography (Et₂O) to give 4-phenylbut-3-en-2-one (**2b**, 72.5 mg, 0.495mmol, 99% yield, *E:Z* = 100:0) as an orange solid.

(*E*)-4-Phenylbut-3-en-2-one (2b).⁵

Mp 35–37 °C; ¹H NMR (CDCl₃, 300 MHz) δ 7.48–7.60 (m, 3H), 7.30–7.48 (m, 3H), 6.72 (d, *J* = 15.9 Hz, 1H), 2.37 (s, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 198.4, 143.4, 134.3, 130.5, 128.9, 128.2, 127.1, 27.5.

(*E*)-1-Phenylpent-1-en-3-one (2a, *E:Z* = 100:0).⁶

Orange oil; ¹H NMR (CDCl₃, 300 MHz) δ 7.50–7.62 (m, 3H), 7.26–7.50 (m, 3H), 6.75 (d, *J* = 16.2 Hz, 1H), 2.71 (q, *J* = 7.2 Hz, 2H) 1.17 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 200.9, 142.2, 134.5, 130.3, 128.9, 128.2, 126.0, 34.0, 8.2.

(*E*)-1-Phenylhept-1-en-3-one (2c, *E:Z* = 100:0).⁷

Pale yellow solid; Mp 32–33 °C; ¹H NMR (CDCl₃, 300 MHz) δ 7.50–7.60 (m, 3H), 7.30–7.50 (m, 3H), 6.75 (d, *J* = 15.9 Hz, 1H), 2.67 (t, *J* = 7.2 Hz, 2H), 1.60–1.74 (m, 2H), 1.30–1.47 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 75 MHz) δ 200.5, 142.2, 134.5, 130.3, 128.8, 128.1, 126.1, 40.7, 26.5, 22.5, 14.0.

(*E*)-4-Methyl-1-phenylpent-1-en-3-one (2d, *E:Z* = 100:0).⁸

Orange oil; ¹H NMR (CDCl₃, 300 MHz) δ 7.50–7.60 (m, 3H), 7.30–7.50 (m, 3H), 6.82 (d, *J* = 16.2 Hz, 1H), 2.94 (septet, *J* = 6.9 Hz, 1H) 1.19 (d, *J* = 6.9 Hz, 6H); ¹³C NMR (CDCl₃, 75 MHz) δ 203.7, 153.7, 142.3, 134.6, 130.3, 128.8, 128.2, 124.4, 114.5, 55.6, 39.2, 18.4.

(E)-4, 4-Dimethyl-1-phenylpent-1-en-3-one (2e, E:Z = 100:0).⁹

Pale yellow oil; ¹H NMR (CDCl₃, 300 MHz) δ 7.69 (d, *J* = 15.6 Hz, 1H), 7.50–7.65 (m, 2H), 7.34–7.50 (m, 3H), 7.13 (d, *J* = 15.6 Hz, 1H), 1.20 (s, 9H); ¹³C NMR (CDCl₃, 75 MHz) δ 204.1, 142.8, 134.8, 130.1, 128.8, 128.2, 120.6, 43.2, 26.2.

(E)-1-(4-Methoxyphenyl)pent-1-en-3-one (2f, E:Z = 100:0).¹⁰

Orange solid; Mp 47–49 °C; ¹H NMR (CDCl₃, 400 MHz) δ 7.44–7.60 (m, 3H), 6.85–6.95 (m, 2H), 6.63 (d, *J* = 16.0 Hz, 1H), 3.84 (s, 3H), 2.68 (q, *J* = 7.2 Hz, 2H), 1.16 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 200.7, 161.3, 141.8, 129.7, 127.0, 123.7, 114.2, 55.3, 33.9, 8.4.

(E)-1-(4-Trifluoromethylphenyl)pent-1-en-3-one (2g, E:Z = 100:0).

Pale brown solid; Mp 62–64 °C; IR (neat) 2950, 1660, 1320, 1160, 1120, 985, 840 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 7.65 (s, 4H), 7.57 (d, *J* = 16.2 Hz, 1H), 6.81 (d, *J* = 16.2 Hz, 1H), 2.72 (q, *J* = 7.5 Hz, 2H), 1.18 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 200.2, 140.0, 137.9, 131.8, 128.2, 127.9, 125.7 (q), 122.3, 34.4, 8.1; HRMS (EI) calcd for C₁₂H₁₁F₃O [M]⁺ 228.0762, found 228.0736.

(E)-1-*o*-Tolylpent-1-en-3-one (2h, E:Z = 67:33).

Orange oil; IR (neat) 2925, 1660, 1600, 1120, 1040, 980, 745 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) *E* isomer: δ 7.86 (d, *J* = 15.6 Hz, 1H), 7.54–7.60 (m, 1H), 7.10–7.37 (m, 3H), 6.67 (d, *J* = 15.6 Hz, 1H), 2.70 (q, *J* = 7.2 Hz, 2H), 2.25 (s, 3H), 1.18 (t, *J* = 7.2 Hz, 3H); *Z* isomer: 7.10–7.37 (m, 4H), 7.05 (d, *J* = 12.4 Hz, 1H), 6.22 (d, *J* = 12.4 Hz, 1H), 2.30 (s, 3H), 2.30 (q, *J* = 7.2 Hz, 2H), 0.96 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, 100

MHz) δ 203.8, 200.7, 139.5, 139.1, 137.7, 135.6, 135.3, 133.4, 130.7, 129.9, 129.8, 129.7, 128.8, 128.5, 126.8, 126.2, 126.1, 125.5, 36.1, 34.3, 19.9, 19.8, 8.3, 8.1; HRMS (GC–MS, EI) *E* isomer: calcd for C₁₂H₁₄O [M]⁺ 174.1045, found 174.1028; *Z* isomer: calcd for C₁₂H₁₄O [M]⁺ 174.1045 found 174.1011.

(*E*)-1-Deuterio-1-phenylbut-1-en-3-one (2b-*d*).

¹H NMR (CDCl₃, 300 MHz) δ 7.50–7.57 (m, 2.25H), 7.35–7.44 (m, 3H), 6.72 (t, *J* = 2.4 Hz, 0.75H), 2.39 (s, 3H); ²H NMR (CDCl₃, 61 MHz) δ 7.35–7.68 (m, 0.75D), 6.60–6.85 (m, 0.25D).

IV. Kinetic Resolution of Secondary Propargylic Alcohols

General Procedure 2 (Table 4, entry 3). Under an Ar atmosphere, AgOTf (6.4 mg, 0.025 mmol) and [Rh(cod)Cl]₂ (6.2 mg, 0.0125 mmol) were dissolved in acetone (1.0 mL) and the mixture was stirred at rt for 5 min. An acetone (0.5 mL) solution of (*R*)-BINAP (15.6 mg, 0.025 mmol) was added and the mixture was stirred at rt for 5 min. The resulting solution was concentrated to dryness and dissolved in CH₂Cl₂ (0.5 mL). H₂ was introduced to the resulting solution in a Schlenk tube. After stirring at rt for 0.5 h, the resulting solution was concentrated to dryness and dissolved in CH₂Cl₂ (1.0 mL). To this solution was added a CH₂Cl₂ (0.5 mL) solution of 1-phenylhept-1-yn-3-ol (**1c**, 94.1 mg, 0.500 mmol), and washed remaining substrate away by using CH₂Cl₂ (0.5 mL). The solution was stirred at 25 °C for 14 h. The conversion of the reaction was determined to be 59.8% by ¹H NMR using 1,4-dimethoxybenzene as an internal standard. The solution was concentrated, and purified by preparative TLC (hexanes:EtOAc = 4:1), which furnished (*E*)-1-phenylhept-1-en-3-one (**2c**, 50.1 mg, 0.270 mmol, 53% isolated yield) and (+)-1-phenylhept-1-yn-3-ol [(+)-**1c**, 33.8 mg, 0.180 mmol, 36% isolated yield, [α]_D²⁵ = +1.29° (CHCl₃, *c* = 1.69, 81.5% ee)]. Selectivity factor = 8.2. CHIRALPAK OD-H, hexane:2-PrOH = 95:5, 1.0 mL/min, retention times: 8.3 min (minor isomer) and 27.7 min (major isomer).

Table 4, entry 1. (*S*)-4-Phenylbut-3-yn-2-ol [(*S*)-**1b**, [α]_D²⁵ = −19.6° (Et₂O, *c* 1.04, 42.4% ee); lit.² [α]_D²⁵ = −33.9° (Et₂O, *c* 1.1, 97% ee)] was obtained for 14 h at 57.5% conversion. Selectivity factor = 2.8. CHIRALPAK OD-H, hexane:2-PrOH = 90:10, 1.0 mL/min, retention times: 7.2 min (minor isomer) and 14.4 min (major isomer).

Table 4, entry 2. (*S*)-1-Phenylpent-1-yn-3-ol [(*S*)-**1a**, [α]_D²⁵ = −15.0° (Et₂O, *c* 1.49, 76.0% ee); lit.¹ [α]_D²⁵ = +19.2° (Et₂O, *c* 1.24, 94% ee)] was obtained for 72 h at 58.5% conversion. Selectivity factor = 7.3. CHIRALPAK OD-H, hexane:2-PrOH = 95:5, 1.0 mL/min, retention times: 8.8 min (minor isomer) and 24.4 min (major isomer).

Table 4, entry 4. (*S*)-4-Methyl-1-phenylpent-1-yn-3-ol [(*S*)-**1d**, [α]_D²⁵ = −0.28°

(CHCl₃, *c* 1.72, 58.1% ee); lit.³ [α]_D²⁵ = +3.2° (CHCl₃, *c* 6.8, 90% ee)] was obtained for 28 h at 51.2% conversion. Selectivity factor = 6.1. CHIRALPAK OD-H, hexane:2-PrOH = 95:5, 1.0 mL/min, retention times: 7.6 min (minor isomer) and 17.2 min (major isomer).

Table 4, entry 5. (*S*)-4,4-Dimethyl-1-phenylpent-1-yn-3-ol [(*S*)-**1e**, [α]_D²⁵ = −0.23° (CHCl₃, *c* 1.73, 61.7% ee); lit.³ [α]_D²⁵ = +2.4° (CHCl₃, *c* 4.0, 94% ee)] was obtained for 38 h at 58.2% conversion. Selectivity factor = 4.7. CHIRALPAK OD-H, hexane:2-PrOH = 95:5, 1.0 mL/min, retention times: 8.7 min (minor isomer) and 12.8 min (major isomer); lit.³ CHIRALPAK OD, hexane:2-PrOH = 90:10, retention times: 7.4 min (*R* isomer) and 10.1 min (*S* isomer).

Table 4, entry 6. (−)-1-(4-Methoxyphenyl)pent-1-yn-3-ol [(−)-**1f**, [α]_D²⁵ = −1.15° (CHCl₃, *c* 1.74, 62.3% ee)] was obtained for 45 h at 54.5% conversion. Selectivity factor = 5.8. CHIRALPAK OD-H, hexane:2-PrOH = 95:5, 1.0 mL/min, retention times: 11.5 min (minor isomer) and 40.8 min (major isomer).

Table 4, entry 7. (−)-1-(4-Trifluoromethylphenyl)pent-1-yn-3-ol [(−)-**1g**, [α]_D²⁵ = −1.05° (CHCl₃, *c* 1.76, 78.0% ee)] was obtained for 64 h at 60.0% conversion. Selectivity factor = 7.2. CHIRALPAK OD-H, hexane:2-PrOH = 99:1, 1.0 mL/min, retention times: 19.7 min (minor isomer) and 22.6 min (major isomer).

Table 4, entry 8. (−)-1-*o*-Tolylpent-1-yn-3-ol [(−)-**1h**, [α]_D²⁵ = −3.07° (CHCl₃, *c* 1.79, 80.0% ee)] was obtained for 42 h at 54.9% conversion. Selectivity factor = 11.5. CHIRALPAK OD-H, hexane:2-PrOH = 95:5, 1.0 mL/min, retention times: 8.9 min (minor isomer) and 15.8 min (major isomer).

V. References

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