



Supporting Information

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Highly Enantioselective Catalytic Thiolysis of Prochiral Cyclic Dicarboxylic Anhydrides Utilizing a Bifunctional Chiral Sulfonamide

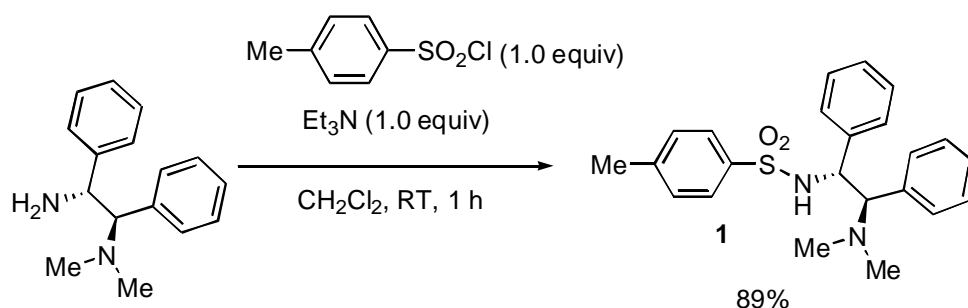
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General

All melting points were determined on a Yanagimoto micro melting point apparatus and uncorrected. IR spectra were recorded on a JASCO FT/IR-420 Infrared Fourier Transform Spectrometer. ¹H-NMR spectra were recorded on a JEOL-GSX400 (400 MHz) spectrometer. Chemical shifts are given in δ values (ppm) using tetramethylsilane (TMS) as an internal standard. EI-MS were recorded on a JEOL JMS SX-120A spectrometer. Elementary combustion analyses were determined by a Yanaco CHN CORDER MT-5. All reactions were monitored by TLC employing 0.25 mm silicagel plates (E. Merck 5715, 60F-254). Column chromatography was carried out on silica gel [Kanto Chemical 60N (63-210 mesh)]. Optical rotations were measured on a JASCO DIP-370 polarimeter. HPLC analyses were performed by using a JASCO (PU-980, UV-970, 807-IT) instrument. All reagents were used as purchased.

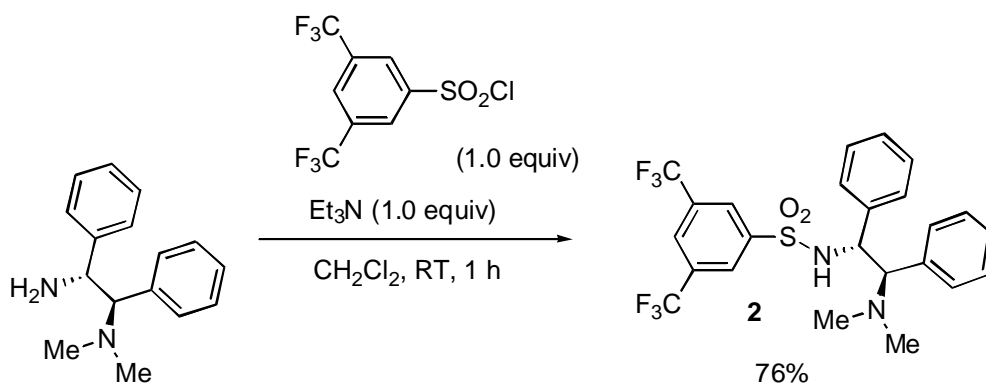
(1*R*,2*R*)-*N,N*-Dimethyl-*N'*-*p*-toluenesulfonyl-1,2-diphenyl-1,2-ethanediamine (1):

To a solution of (1*R*,2*R*)-*N,N*-dimethyl-1,2-diphenyl-1,2-ethanediamine^[1] (72 mg, 0.30 mmol) and *p*-toluenesulfonyl chloride (57 mg, 0.30 mmol) in CH₂Cl₂ (5 mL) was added Et₃N (42 μL, 0.30 mmol) at room temperature. The mixture was stirred at room temperature under Ar for 1 h. After addition of some water, the mixture was extracted with CHCl₃. The extract was dried over MgSO₄, filtrated, and then the filtrate was evaporated *in vacuo*. The residue was chromatographed on a silica gel column with CHCl₃-MeOH (25 : 1) to afford compound **2** (105 mg, 89% yield) as a white solid. mp 109-110 °C; [α]_D¹⁹ 53.0° (*c* 0.50, CHCl₃); ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.54-7.42 (3H, m), 7.25-6.86 (12H, m), 4.67-4.57 (1H, m), 3.77 (1H, d, *J* = 11.0 Hz), 2.31 (3H, s), 1.89 (6H, s); IR (KBr) 3087, 2967, 2938, 2865, 2832, 2786, 1596, 1497, 1454, 1369, 1311, 1149 cm⁻¹; HREI-MS calcd for C₂₃H₂₆N₂O₂S MW 394.1715, found *m/z* 394.1735 (M)⁺; Anal. Calcd for C₂₃H₂₆N₂O₂S: C, 70.02; H, 6.64; N, 7.10. Found: C, 69.74; H, 6.66; N, 7.02.



(1*R*,2*R*)-*N,N*-Dimethyl-*N'*-3,5-bis(trifluoromethyl)benzenesulfonyl-1,2-diphenyl-1,2-ethanediamine (2):

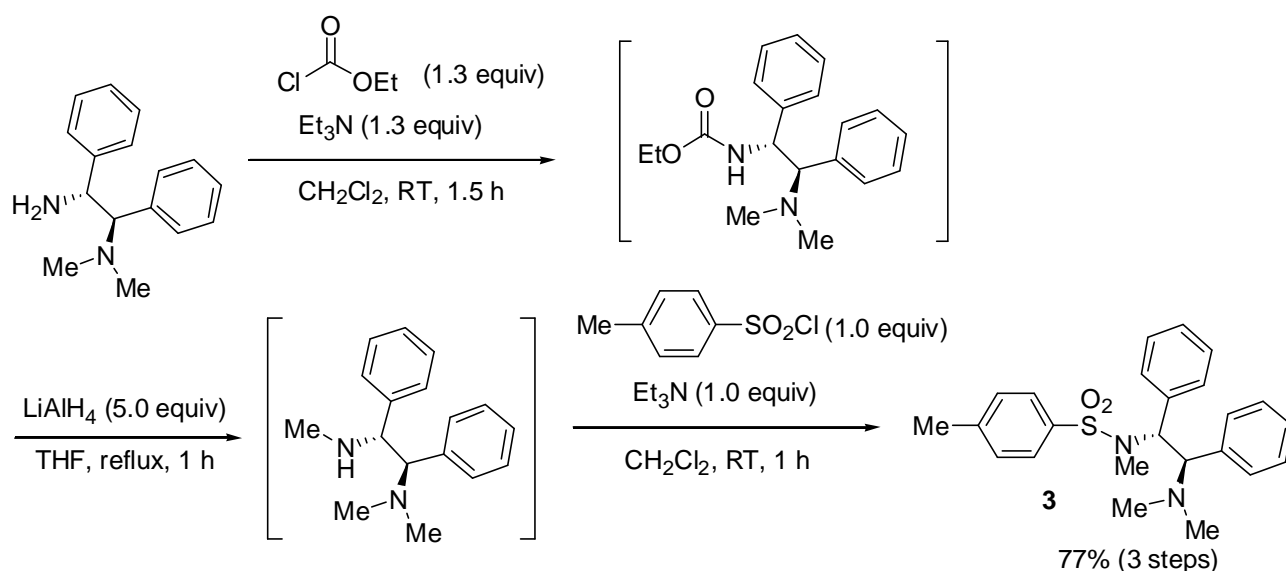
To a solution of (1*R*,2*R*)-*N,N*-dimethyl-1,2-diphenyl-1,2-ethanediamine (752 mg, 3.13 mmol) and 3,5-bis(trifluoromethyl)benzenesulfonyl chloride (978 mg, 3.13 mmol) in CH₂Cl₂ (30 mL) was added Et₃N (436 μL, 3.13 mmol) at room temperature. The mixture was stirred at room temperature under Ar for 1 h. After addition of some water, the mixture was extracted with CHCl₃. The extract was dried over MgSO₄, filtrated, and then the filtrate was evaporated *in vacuo*. The residue was chromatographed on a silica gel column with CHCl₃-MeOH (25 : 1) to afford compound **2** (1.23 g, 76% yield) as colorless needles (CHCl₃-*n*-hexane). mp 168-169 °C; [α]_D¹⁸ 131.5° (*c* 1.21, CHCl₃); ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.61 (1H, bs), 8.20 (1H, s), 8.05 (2H, s), 7.15-6.93 (7H, m), 6.84-6.73 (3H, m), 4.95 (1H, d, *J* = 11.2 Hz), 3.86 (1H, d, *J* = 11.2 Hz), 1.96 (6H, s); IR (KBr) 3301, 2975, 2942, 2869, 2836, 2794, 1454, 1365, 1280, 1180, 1157, 1133 cm⁻¹; HREI-MS calcd for C₂₄H₂₂F₆N₂O₂S MW 516.1306, found *m/z* 516.1331 (M)⁺; Anal. Calcd for C₂₄H₂₂F₆N₂O₂S: C, 55.81; H, 4.29; N, 5.42. Found: C, 55.61; H, 4.35; N, 5.37.



(1*R*,2*R*)-*N,N,N'*-Trimethyl-*N'*-*p*-toluenesulfonyl-1,2-diphenyl-1,2-ethanediamine (3):

To a solution of (1*R*,2*R*)-*N,N*-dimethyl-1,2-diphenyl-1,2-ethanediamine (201 mg, 0.837 mmol) and ethyl chlorocarbonate (103 μL, 1.09 mmol) in CH₂Cl₂ (15 mL) was added Et₃N (152 μL, 1.09 mmol) at room temperature. The mixture was stirred at room temperature under Ar for 1.5 h. After addition of some water, the mixture was extracted with CHCl₃. The extract was dried over MgSO₄, filtrated, and then the filtrate was evaporated *in vacuo*. The residue was dissolved in THF (10 mL), and LiAlH₄ (159 mg, 4.19 mmol) was added at room temperature, and then refluxed for 1 h. After addition of some water, the mixture was extracted with CHCl₃. The CHCl₃ extract was dried over

MgSO₄, filtrated, and then the filtrate was evaporated *in vacuo*. The residue was dissolved in CH₂Cl₂ (15 mL). To the solution were added *p*-toluenesulfonyl chloride (160 mg, 0.837 mmol) and Et₃N (117 μL, 0.837 mmol) at room temperature. The mixture was stirred at room temperature under Ar for 1 h and then treated with some water followed by extraction with CHCl₃. The CHCl₃ extract was dried over MgSO₄ and filtrated. The filtrate was evaporated *in vacuo* to give an oily residue. The residue was chromatographed on a silica gel column with AcOEt-*n*-hexane (1 : 2) to afford compound **3** (263 mg, 77% yield) as a colorless amorphous powder. $[\alpha]_D^{21} -30.2^\circ$ (*c* 1.24 CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.64-7.57 (2H, m), 7.22-7.00 (12H, m), 5.85 (1H, d, *J* = 12.0 Hz), 4.16 (1H, d, *J* = 12.0 Hz), 2.79 (3H, s), 2.34 (3H, s), 2.19 (6H, s); ¹³C NMR (100 MHz, CDCl₃) δ 142.3, 137.5, 136.8, 132.6, 129.4, 128.89, 128.8, 127.9, 127.5, 127.3, 127.2, 127.0, 67.1, 59.3, 40.9, 28.8, 21.4; IR (KBr) 3031, 2931, 2830, 2784, 2596, 1596, 1450, 1319, 1211, 1157 cm⁻¹; HREI-MS calcd for C₂₄H₂₈N₂O₂S MW 408.1872, found *m/z* 408.1857 (M)⁺; Anal. Calcd for C₂₄H₂₈N₂O₂S.



A typical procedure for the enantioselective catalytic thiolysis (Table 2, Entry 1):

To a solution of 3-phenylglutaric anhydride **6** (190 mg, 1.0 mmol) and chiral sulfonamide **2** (25.8 mg, 0.05 mmol) in Et₂O (10 mL) was added benzyl mercaptan (141 μL, 1.2 mmol) at room temperature. The mixture was stirred at room temperature under Ar for 20 h and then treated with 10% HCl followed by extraction with CHCl₃. The CHCl₃ extract was dried over MgSO₄ and filtrated. After evaporation of the filtrate *in vacuo*, the residue was dissolved in benzene-MeOH (7 : 2) (9 mL). To the solution was added an ethereal solution of TMSCHN₂ (2.0 M in Et₂O, 1 mL, 2.0 mmol). The mixture was stirred at room temperature for 15 min and then evaporated *in vacuo* to give an oily residue. Chromatographic purification of the residue on a silica gel column with AcOEt-*n*-hexane (1 : 4) afforded compound **8** (312 mg, 95% yield, 91% ee) as a white solid.

Methyl (*S*)-5-(Benzylthio)-5-oxo-3-phenylpentanoate (**8**) (Table 2, Entry 1):

white solid; 95% yield; mp 34.5-35 °C; 91% ee; $[\alpha]_{\text{D}}^{20}$ 43.7° (*c* 1.10, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.34-7.07 (10H, m), 4.05 (2H, s), 3.77-3.67 (1H, m), 3.57 (3H, s), 2.96 (1H, dd, *J* = 15.1 and 7.1 Hz), 2.91 (1H, dd, *J* = 15.1 and 7.8 Hz), 2.73 (1H, dd, *J* = 15.6 and 6.8 Hz), 2.65 (1H, dd, *J* = 15.6 and 8.1 Hz); IR (KBr) 3054, 2946, 1735, 1685, 1492, 1423, 1365, 1257, 1203, 1160 cm⁻¹; HREI-MS calcd for C₁₉H₂₀O₃S MW 328.1133, found *m/z* 328.1130 (M)⁺; Anal. Calcd for C₁₉H₂₀O₃S: C, 69.48; H, 6.14. Found: C, 69.22; H, 6.12.

The ee of **8** was determined on a Daicel chiral OD-H column with *n*-hexane : isopropanol = 19 : 1, flow = 1 mL/min. The absolute configuration of **8** was determined by its chemical conversion to known ketoester **19**.^[2]

Chemical conversion of **8** to ketoester **19**:

To a solution of **8** (91% ee, 100 mg, 0.30 mmol) and Fe(acac)₃ (21.5 mg, 0.061 mmol) in THF (5 mL) was added EtMgBr (0.98 M in THF, 747 μL, 0.73 mmol) at -78 °C. The mixture was stirred at -78 °C under Ar for 4 h. 10% HCl was added to the reaction mixture, followed by extraction with CHCl₃, dried over MgSO₄, and filtrated. The filtrate was evaporated *in vacuo*. The residue was chromatographed on a silica gel column with AcOEt-*n*-hexane (1 : 4) to afford compound **19** (59 mg, 83% yield, 91% ee) as a white solid.

Methyl (*R*)-5-Oxo-3-phenylheptanoate (**19**):

white solid; 83% yield from **8**; mp 36-36.5°C; 91% ee; $[\alpha]_{\text{D}}^{18}$ 31.5° (*c* 0.75, C₆H₆) (*R* enantiomer: lit.^[2] $[\alpha]_{\text{D}}^{24}$ 35.3° (*c* 2-3, C₆H₆)); ¹H NMR (400 MHz, CDCl₃) δ 7.32-7.16 (5H, m), 3.72-3.65 (1H, m), 3.58 (3H, s), 2.82 (1H, dd, *J* = 16.6 and 7.1 Hz), 2.77 (1H, dd, *J* = 16.6 and 7.3 Hz), 2.69 (1H, dd, *J* = 15.3 and 7.2 Hz), 2.61 (1H, dd, *J* = 15.3 and 7.7 Hz), 2.41-2.23 (2H, m), 0.96 (3H, t, *J* = 7.3 Hz); IR (KBr) 3039, 2985, 2946, 2892, 1951, 1889, 1720, 1604, 1496, 1442, 1365 cm⁻¹; HREI-MS calcd for C₁₄H₁₈O₃ MW 234.1256, found *m/z* 234.1247 (M)⁺; Anal. Calcd for C₁₄H₁₈O₃: C, 71.77; H, 7.74. Found: C, 71.49; H, 7.72.

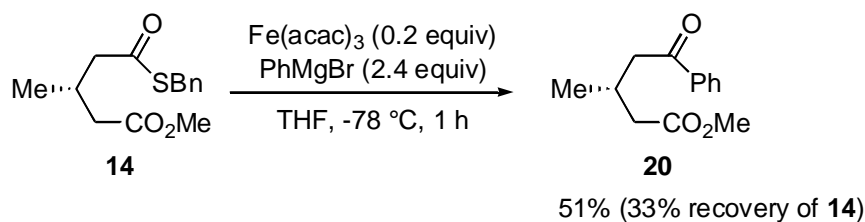
The ee of **19** was determined on a Daicel chiral OD-H column with *n*-hexane : isopropanol = 19 : 1, flow = 1 mL/min.

Methyl (*S*)-5-(Benzylthio)-3-methyl-5-oxopentanoate (**14**) (Table 2, Entry 2):

colorless oil; 87% yield; 91% ee; $[\alpha]_{\text{D}}^{20}$ 5.4° (*c* 1.24, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.34-7.19 (5H, m), 4.12 (2H, s), 3.66 (3H, s), 2.69-2.45 (3H, m), 2.39 (1H, dd, *J* = 15.4 and 5.9 Hz), 2.23 (1H, dd, *J* = 15.4 and 7.3 Hz), 1.01 (3H, d, *J* = 6.3 Hz); IR (neat) 2958, 2360, 1735, 1689, 1500, 1450, 1365 cm⁻¹; HREI-MS calcd for C₁₄H₁₈O₃S MW 266.0977, found *m/z* 266.1000 (M)⁺; Anal. Calcd for C₁₄H₁₈O₃S: C, 63.13; H, 6.81. Found: C, 63.05; H, 6.85.

The ee of **14** was determined on a Daicel chiral OB-H column with *n*-hexane : isopropanol = 15 : 1,

flow = 0.7 mL/min. The absolute configuration of **14** was determined by its chemical conversion to known ketoester **20**.^[3]



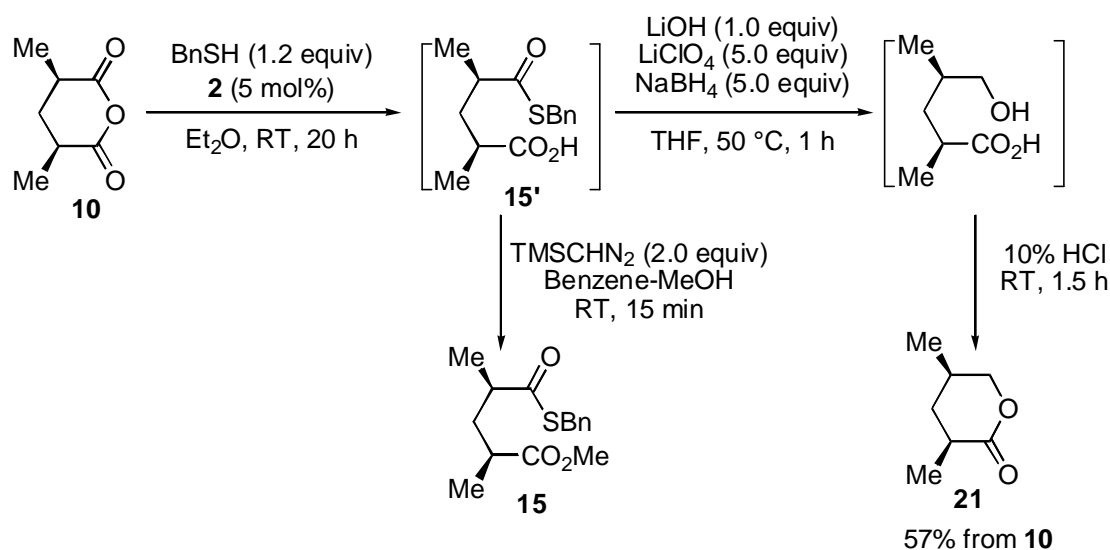
Methyl (*R*)-3-Methyl-5-oxophenylpentanoate (**20**):

colorless oil; 51% yield from **14**; $[\alpha]_{\text{D}}^{18}$ 3.14° (*c* 1.05, C₆H₆) (*S* enantiomer: lit.^[3] $[\alpha]_{\text{D}}^{25}$ -5.65° (*c* 1.24, C₆H₆)); ¹H NMR (400 MHz, CDCl₃) δ 8.01-7.91 (2H, m), 7.61-7.52 (1H, m), 7.51-7.41 (2H, m), 3.68 (3H, s), 3.11 (1H, dd, *J* = 16.4 and 5.9 Hz), 2.85 (1H, dd, *J* = 16.4 and 7.6 Hz), 2.73-2.61 (1H, m), 2.45 (1H, dd, *J* = 15.1 and 6.6 Hz), 2.33 (1H, dd, *J* = 15.1 and 7.1 Hz), 1.05 (3H, d, *J* = 6.8 Hz).

Methyl (*2S,4R*)-5-(Benzylthio)-2,4-dimethyl-5-oxopentanoate (**15**) (Table 2, Entry 3):

colorless oil; 100% yield; 90% ee; $[\alpha]_{\text{D}}^{20}$ -17.2° (*c* 1.05, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.33-7.20 (5H, m), 4.11 (2H, s), 3.66 (3H, s), 2.76-2.65 (1H, m), 2.56-2.45 (1H, m), 2.20-2.11 (1H, m), 1.54-1.44 (1H, m), 1.20 (3H, d, *J* = 7.1 Hz), 1.17 (3H, d, *J* = 7.1 Hz); IR (neat) 3023, 2969, 1735, 1689, 1604, 1450, 1373, 1319, 1187 cm⁻¹; HREI-MS calcd for C₁₅H₂₀O₃S MW 280.1133, found *m/z* 280.1118 (M)⁺; Anal. Calcd for C₁₅H₂₀O₃S: C, 64.26; H, 7.19. Found: C, 64.09; H, 7.15.

The ee of **15** was determined on a Daicel chiral OB-H column with *n*-hexane : isopropanol = 15 : 1, flow = 1.0 mL/min. The absolute configuration of **15** was determined by chemical conversion of monocarboxylic acid **15'** to known lactone **21**.^[4]



Chemical conversion of **10** to known lactone **21** via monocarboxylic acid **15'**:

To a solution of anhydride **10** (55 mg, 0.387 mmol) and **2** (10 mg, 0.0193 mmol) in Et₂O (3.9 mL) was added benzyl mercaptan (55 μL, 0.464 mmol) at room temperature. The mixture was stirred at room temperature under Ar for 20 h. *Sat.* NaHCO₃ *aq.* was added to the reaction mixture and washed with Et₂O. The aqueous portion was acidified with 10% HCl, and extracted with CHCl₃. The extract was dried over MgSO₄, filtrated, and then the filtrate was evaporated *in vacuo*. The residue was dissolved in THF (12 mL), and LiOH (9.2 mg, 0.387 mmol) was added. After being stirred at 50 °C for 15 min, LiClO₄ (205 mg, 1.935 mmol) and NaBH₄ (73 mg, 1.935 mmol) were added. The mixture was stirred at 50 °C for 1 h. The reaction mixture was concentrated *in vacuo*. The residue was treated with 10% HCl (15 mL), and stirred at room temperature for 1.5 h followed by extraction with CHCl₃. The extract was dried over MgSO₄, filtrated, and the filtrate was evaporated *in vacuo*. The residue was chromatographed on a silica gel column with AcOEt-*n*-hexane (1 : 1) to afford compound **21** (28.4 mg, 57% yield from **10**).

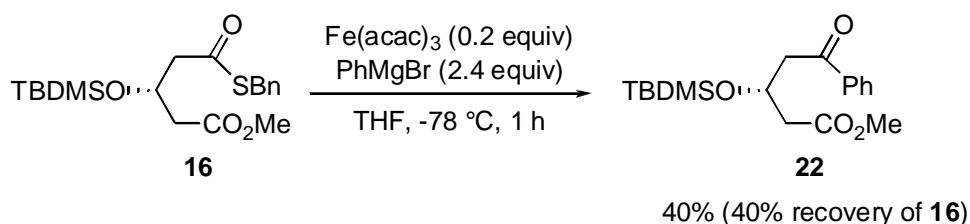
(2*S*,4*R*)-cis-2,4-Dimethyl-d-valerolactone (21):

white solid; 57% yield ; mp 33-36°C; [α]_D¹⁹ 41.2° (*c* 0.95, CHCl₃) (*2*R*,4*S** enantiomer: lit.^[4] [α]_D²⁵ -41.1° (*c* 5-10, CHCl₃)); ¹H NMR (400 MHz, CDCl₃) δ 4.36-4.29 (1H, m), 3.93-3.86 (1H, m), 2.59-2.48 (1H, m), 2.20-2.03 (2H, m), 1.32-1.19 (1H, m), 1.27 (3H, d, *J* = 6.6 Hz), 0.99 (3H, d, *J* = 6.6 Hz).

Methyl (S)-5-(Benzylthio)-3-(tert-butyldimethylsilyloxy)-5-oxopentanoate (16) (Table 2, Entry 4):

colorless oil; 88% yield; 93% ee; [α]_D²⁰ 16.6° (*c* 1.38, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.32-7.21 (5H, m), 4.62-4.53 (1H, m), 4.14 (1H, d, *J* = 13.7 Hz), 4.09 (1H, d, *J* = 13.7 Hz), 3.66 (3H, s), 2.83 (1H, dd, *J* = 14.9 and 6.6 Hz), 2.77 (1H, dd, *J* = 14.9 and 5.7 Hz), 2.56 (1H, dd, *J* = 15.1 and 5.9 Hz), 2.51 (1H, dd, *J* = 15.1 and 6.6 Hz), 0.83 (9H, s), 0.05 (3H, s), 0.04 (3H, s); IR (neat) 2946, 2857, 1739, 1689, 1442, 1369, 1319, 1253 cm⁻¹; HREI-MS calcd for C₁₉H₃₀O₄SSi MW 382.1634, found *m/z* 382.1642 (M)⁺; Anal. Calcd for C₁₉H₃₀O₄SSi: C, 59.65; H, 7.90. Found: C, 59.39; H, 7.85.

The ee of **16** was determined on a Daicel chiral OD-H column with *n*-hexane : isopropanol = 19 : 1, flow = 0.5 mL/min. The absolute configuration of **16** was determined by its chemical conversion to known ketoester **22**.^[5]



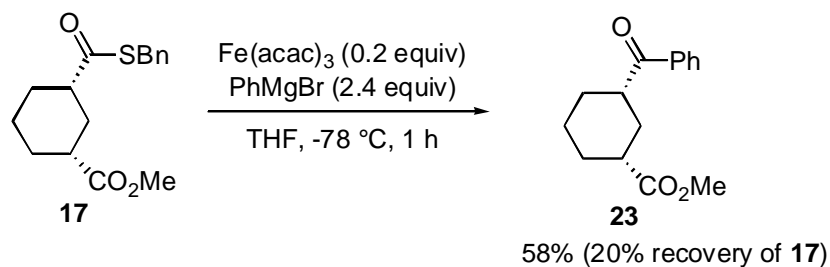
Methyl (*R*)-3-(*tert*-Butyldimethylsilyloxy)-5-oxo-5-phenylpentanoate (22**):**

colorless oil; 40% yield from **16**; $[\alpha]_{\text{D}}^{19}$ 10.4° (*c* 1.50, CHCl₃) (*S* enantiomer: lit.^[5] $[\alpha]_{\text{D}}^{25}$ -7.7° (*c* 0.99, CHCl₃)); ¹H NMR (400 MHz, CDCl₃) δ 7.99-7.93 (2H, m), 7.60-7.54 (1H, m), 7.50-7.42 (2H, m), 4.80-4.72 (1H, m), 3.69 (3H, s), 3.29 (1H, dd, *J* = 16.2 and 6.6 Hz), 3.17 (1H, dd, *J* = 16.2 and 6.0 Hz), 2.66 (1H, dd, *J* = 14.7 and 5.7 Hz), 2.56 (1H, dd, *J* = 14.7 and 6.3 Hz), 0.80 (9H, s), 0.07 (3H, s), -0.02 (3H, s).

Methyl (*1R,3S*)-3-(Benzylthiocarbonyl)cyclohexanecarboxylate (17**) (Table 2, Entry 5):**

colorless oil; 90% yield; 98% ee; $[\alpha]_{\text{D}}^{20}$ -22.6° (*c* 1.42, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.35-7.20 (5H, m), 4.11 (2H, s), 3.66 (3H, s), 2.58-2.48 (1H, m), 2.38-2.28 (1H, m), 2.26-2.18 (1H, m), 2.03-1.85 (3H, m), 1.68-1.56 (1H, m), 1.49-1.24 (3H, m); IR (neat) 3023, 2938, 2861, 1735, 1681, 1604, 1496, 1450, 1373, 1303, 1249, 1203 cm⁻¹; HREI-MS calcd for C₁₆H₂₀O₃S MW 292.1133, found *m/z* 292.1118 (M)⁺; Anal. Calcd for C₁₆H₂₀O₃S: C, 65.72; H, 6.89. Found: C, 65.42; H, 6.90.

The ee of **17** was determined on a Daicel chiral AD-H column with *n*-hexane : isopropanol = 19 : 1, flow = 0.5 mL/min. The absolute configuration of **17** was determined by its chemical conversion to known ketoester **23**.^[5]

**Methyl (*1S,3R*)-3-Benzoylcyclohexanecarboxylate (**23**):**

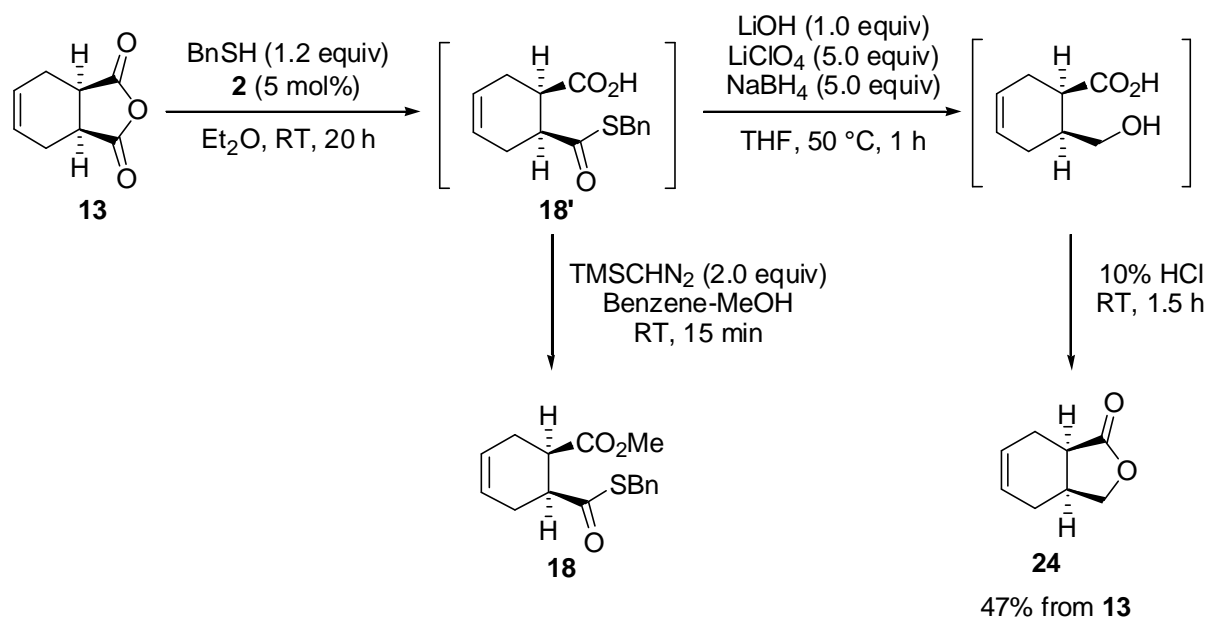
colorless oil; 58% yield from **17**; $[\alpha]_{\text{D}}^{18}$ -26.3° (*c* 1.60, CHCl₃) (*1S,3R* enantiomer: lit.^[5] $[\alpha]_{\text{D}}^{25}$ 22.4° (*c* 1.13, CHCl₃)); ¹H NMR (400 MHz, CDCl₃) δ 7.98-7.84 (2H, m), 7.60-7.38 (3H, m), 3.67 (3H, s), 3.36-3.23 (1H, m), 2.54-2.39 (1H, m), 2.21-1.84 (4H, m), 1.74-1.57 (1H, m), 1.53-1.34 (3H, m).

Methyl (*1R,6S*)-6-(Benzylthiocarbonyl)cyclohex-3-enecarboxylate (18**) (Table 2, Entry 6):**

colorless oil; 90% yield; 83% ee; $[\alpha]_{\text{D}}^{20}$ -15.6° (*c* 1.17, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 7.32-7.19 (5H, m), 5.74-5.64 (2H, m), 4.16 (1H, d, *J* = 13.7 Hz), 4.10 (1H, d, *J* = 13.7 Hz), 3.65 (3H, s), 3.22-3.15 (1H, m), 3.10-3.03 (1H, m), 2.66-2.31 (4H, m); IR (neat) 3031, 2923, 2846, 1735, 1681, 1604, 1496, 1442, 1342, 1288, 1203 cm⁻¹; HREI-MS calcd for C₁₆H₁₈O₃S MW 290.0977, found *m/z* 290.0968 (M)⁺; Anal. Calcd for C₁₆H₁₈O₃S: C, 66.18; H, 6.25. Found: C, 65.94; H, 6.29.

The ee of **18** was determined on a Daicel chiral OD-H column with *n*-hexane : isopropanol = 19 :

1, flow = 1.0 mL/min. The absolute configuration of **18** was determined by chemical conversion of **13** to known lactone **24**^[6] via monocarboxylic acid **18'**.



(1S,6R)-8-Oxabicyclo[4.3.1]non-3-en-7-one (24):

colorless oil; 47% yield from **13**; $[\alpha]_D^{19}$ 41.8° (*c* 1.40, CHCl₃) (*1R,6S* enantiomer: lit.^[6] $[\alpha]_D^{25}$ -67.1° (*c* 1.00, CHCl₃)); ¹H NMR (400 MHz, CDCl₃) δ 5.84-5.66 (2H, m), 4.33 (1H, dd, *J* = 8.8 and 5.1 Hz), 4.04 (1H, dd, *J* = 8.8 and 1.7 Hz), 2.87-2.19 (5H, m), 1.98-1.80 (1H, m).

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[2] D. Enders, K. Paradopoulos, *Tetrahedron Lett.* **1983**, *24*, 4967-4970.

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