First Stereoselective Synthesis of the Versatile Chiral Building Block (7a*R*)-5,6-Dihydro-7a-methyl-1*H*-indene-2,7(4*H*,7a*H*)-dione

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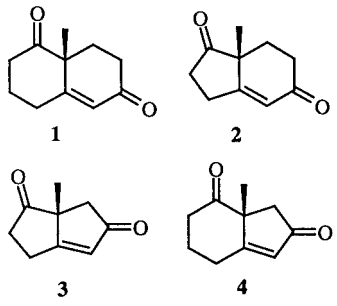
Abstract: A versatile chiral building block (7a*R*)-5,6-dihydro-7a-methyl-1*H*-indene-2,7(4*H*,7a*H*)-dione (**4**) was firstly enantiomerically synthesized from the microbial transformation ketol product **6** in 61.3% overall yield and over 96% ee.

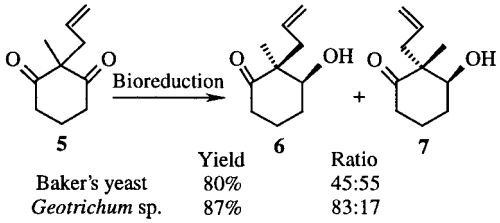
Key words: asymmetric synthesis, microbial reduction, ketols, bicyclic compounds, Wieland–Miescher ketone analog

Enantiomerically pure bicyclic enones **1**–**4** may constitute useful building blocks for elaboration of synthetic strategies in the field of steroids, terpenes and related molecules.[1–4] The enantiomerically enriched forms of Wieland–Miescher ketone **1** and Hajos–Parrish ketone **2** could be efficiently prepared by asymmetric cyclization of their prochiral precursors, 2-methyl-2-(3-oxobutyl)-1,3-cyclohexanedione and 2-methyl-2-(3-oxobutyl)-1,3-cyclopentanedione, respectively, by the use of proline-enamine[2] or catalytic antibody.[3] However, ring construction of the fused cyclopentenones **3** and **4** from their corresponding 1,3-cycloalkanedione derivatives using the standard aldol condition is quite difficult.[4] Though optically active **3** has been asymmetrically synthesized by Trost[5] and Brooks,[6] preparation of the enantiomerically enriched form of **4** has not been reported so far, to our knowledge.[7] In this paper, we present the first enantioselective synthesis of the versatile building block **4** from the ketol **6**.



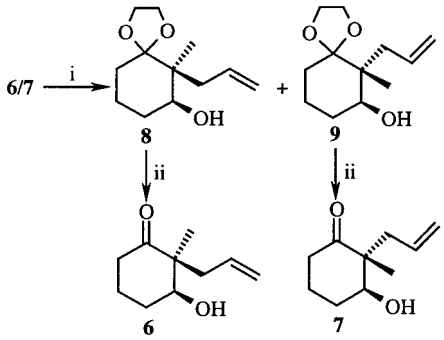
1 **2** **3** **4**

Enantiomerically pure ketol **6** could be prepared by microorganism asymmetric reduction of the corresponding prochiral diketone **5**,[8] or by enzymatic hydrolysis of prochiral dienol diacetate of **5** developed recently by Renouf et al.[9] Though baker's yeast mediated asymmetric reduction of ketones has been widely used to obtain chiral building blocks since it is cheap, versatile, and easy to perform,[10] baker's yeast reduction of **5** provided a nearly equivalent mixture of diastereomeric ketols **6** and **7**.[8] However, the highly reductive fungus, *Geotrichum* sp.,[11] could reduce the diketone **5** more efficiently to give the ketols **6** and **7** in the ratio of 83:17 (Scheme 1).



Scheme 1

Because the two diastereomeric ketols **6** and **7** could not be separated efficiently by silica gel chromatography, the mixture obtained from microbial reduction of **5** was converted to the corresponding acetals **8** and **9**, which could be separated efficiently by chromatography. After treatment with 3 M HCl, compounds **8** and **9** were converted to ketols **6** and **7** in nearly optically pure forms, respectively (Scheme 2).

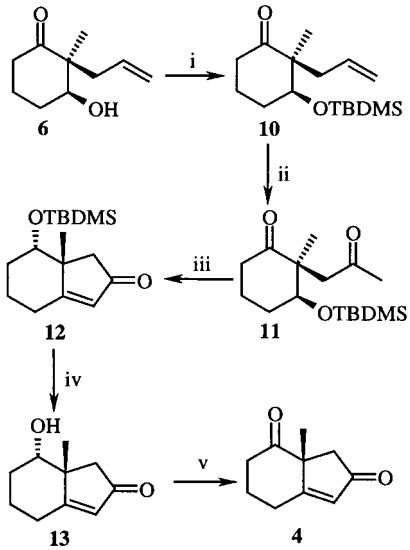


Reagents and conditions: i) Cat. *p*-TsOH, $HOCH_2CH_2OH$, $CH(OEt)_3$; ii) 3 M HCl.

Scheme 2

Considering that the hydroxy group would interfere with the subsequent transformations, it was efficiently protected as the *tert*-butyldimethylsilyl ether **10**. Wacker oxidation of the olefin provided the corresponding dione **11** with good yield (83%). Cyclization and dehydration of di-

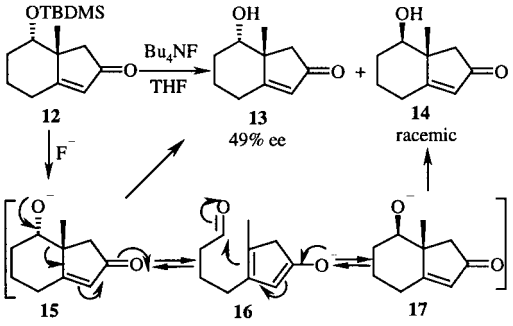
one **11** to bicyclic enone **12** was smoothly achieved in high yield (91%) by using potassium *tert*-butoxide in *tert*-butyl alcohol at room temperature. Deprotection of the silyl ether with aqueous HF gave the bicyclic ketol **13** efficiently (90%), which was subjected to Swern oxidation to provide the desired enedione **4** in 94% yield (Scheme 3). Therefore, (7a*R*)-5,6-dihydro-7a-methyl-1*H*-indene-2,7(4*H*,7a*H*)-dione (**4**) was enantiomerically synthesized, for the first time, from the microbial transformation ketol product **6** in 61.3% overall yield.



Reagents and conditions: i) TBDMSCl, DMF, imidazole, DMAP, 60 °C, 24 h, 96%; ii) $PdCl_2$, CuCl, DMF, H_2O, O_2, r.t., 30 h, 83%; iii) ^{t}BuOK, ^{t}BuOH, r.t., 4 h, 91%; iv) aq HF (40%), CH_3CN, r.t., 48 h, 90%; v) Swern oxidation, 94%.

Scheme 3

It is worth mentioning that the silyl ether **12** was initially treated with Bu_4NF and anhydrous THF in the deprotection step, and unexpectedly a mixture of the diastereomeric bicyclic ketol **13** and **14** was obtained in 3:1 ratio. Chiral HPLC analysis indicated that the major diastereoisomer **13** was in 49% ee, while the other **14** was in nearly racemic forms. This was thought to be a result of carbon-carbon bonds cleavage and reformation, under the deprotection conditions, through the intermediates **15** to **17** due to the unstable bicyclic ketol anion **15**. However, the ketol **13** obtained under the conditions was still in enantiomerically enriched form. It might be either due to absorption of moisture from outside or that some oxygen anion intermediate **15** still did not racemize when the reaction was worked up (Scheme 4). As a result, nearly optically pure **13** (>96% ee) determined by chiral HPLC analysis was provided as the sole diasteromeric product using aqueous HF as the deprotection conditions.



Scheme 4

All mps are uncorrected. IR spectra were recorded on a Shimadzu IR-440 spectrometer. EIMS were run on an HP-5989A mass spectrometer and high resolution mass spectra (HRMS) were recorded on a Finnigan MAT-4021 instrument or a Kratos Concept 1H Series Mass Spectromer. ^{1}H NMR spectra were recorded on a Bruker Am300 (300 MHz) or Bruker DRX-400 (400 MHz) spectrometer with TMS as the internal standard. Optical rotation were measured on a Perkin−Elmer 241 polarimeter. Chiral HPLC was carried out using Chiralpak AD column (0.46 cm x 25 cm) detected at UV 254 nm, flow-rate: 0.7 mL/min. TLC was carried out using HSG F_{254} silica gel plates and silica gel (200−400 mesh) was used for chromatography. Organic extracts were dried over anhyd. Na_2SO_4.

Bioreduction of 2-Methyl-2-(2-propenyl)cyclohexane-1,3-dione (5)

Wet mycelium of *Geotrichum* sp. (15 g) was suspended in 5% glucose solution (50 mL) and the dione **5** (1 mmol) was added slowly. The mixture was shaken at 30 °C. After completion of the biotransformation (15 h), the mycelium was filtered out and washed with EtOAc. The filtrate was saturated with NaCl and extracted with EtOAc (4 x 50 mL). The combined extracts were washed with brine, dried, filtered, and evaporated under reduced pressure. The residue was purified by chromatography to provide a mixture of the ketols **6** and **7** (87%) in the ratio of 83:17, determined by GC-MS analysis.

Ethylene Acetals 8 and 9

A solution of **6**/**7** (1.5 g, 9 mmol), ethylene glycol (3 g, 48 mmol), triethyl orthoformate (4.8 g, 18 mmol), and *p*-toluenesulfonic acid (200 mg, 1.2 mmol) was stirred at r.t. for 20 h. Sat. $NaHCO_3$ (40 mL) was added and the mixture was extracted with Et_2O (3 x 50 mL). The organic layers were combined and washed with brine, dried, filtered, and evaporated under reduced pressure. The residue was purified by silica gel chromatography (petroleum ether/EtOAc, 20:1) to provide **8** (1.50 g, 81%) and **9** (0.35 g, 16%).

(1*S*,2*S*)-3-(1,3-Dioxolan-2-yl)-2-methyl-2-(2-propenyl)cyclohexan-1-ol (8)

Colorless oil; $[\alpha]_D^{21}$ +4.7 (*c* 1.0, $CHCl_3$).

IR (film): ν_{max} = 3500, 2920, 1640, 1450, 1410, 1120, 1070, 910 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 5.98−5.83 (m, 1H), 5.12−5.05 (m, 2H), 4.04−3.87 (m, 4H), 3.61 (s, 1H), 3.20 (br s, 1H), 2.54 (dd, 1H, *J* = 13.2, 6.8 Hz), 2.01 (dd, 1H, *J* = 13.2, 8.1 Hz), 1.90−1.51 (m, 6H), 1.01 (s, 3H).

EIMS: *m/z* (rel. intensity) = 212 (M^+, 0.6), 194 (4.3), 108 (51.1), 99 (100), 86 (72.3), 55 (33.8), 43 (34.2), 41 (46).

Anal. Calcd. for $C_{12}H_{20}O_3$: C, 67.89; H, 9.50. Found: C, 67.48; H, 9.72.

(1*S*,2*R*)-3-(1,3-Dioxolan-2-yl)-2-methyl-2-(2-propenyl)cyclohexan-1-ol (9)

Colorless oil; $[\alpha]_D^{21}$ +22.8 (*c* 0.9, $CHCl_3$).

IR (film): ν_{max} = 3400, 2920, 1700, 1640, 1450, 1180, 1120, 1050, 950, 910 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 6.00−5.85 (m, 1H), 5.15−5.00 (m, 2H), 4.05−3.85 (m, 4H), 3.72 (dd, 1H, *J* = 5.1, 2.4 Hz), 2.90 (br s, 1H), 2.45−2.25 (m, 2H), 1.80−1.50 (m, 6H), 1.03 (s, 3H).

EIMS: *m/z* (rel. intensity) = 212 (M^+, 1.9), 195 (12), 108 (42), 99 (100), 86 (46.4), 55 (43.2), 43 (51.7), 41 (77.5).

Anal. Calcd. for $C_{12}H_{20}O_3$: C, 67.89; H, 9.50. Found: C, 67.58; H, 9.75.

(2*S*,3*S*)-3-Hydroxy-2-methyl-2-(2-propenyl)cyclohexan-1-one (6)

A mixture of the ethylene acetal **8** (1.4 g, 6.6 mmol) and 3 M HCl (40 mL) was stirred at r.t. for 10 h. The reaction mixture was extracted with CH_2Cl_2 (3 x 50 mL). The organic layers were combined and washed with sat. brine, dried, filtered, and condensed. The residue was purified by chromatography on silica gel (petroleum ether/EtOAc, 5:1) to provide the ketol **6** (1.1 g, 98%), whose spectral data were identical with those reported.[8]

(2*S*,3*S*)-3-[(*tert*-Butyldimethylsilyl)oxy]-2-methyl-2-(2-propenyl)cyclohexan-1-one (10)

A mixture of ketol **6** (770 mg, 4.6 mmol), DMF (5 mL), imidazole (1 g, 15 mmol), 4-(dimethylamino)pyridine (130 mg, 1 mmol), and *tert*-butyldimethylsilyl chloride (1.12 g, 7.5 mmol) was stirred under N_2 at 60 °C for 24 h. After cooling the mixture, Et_2O (150 mL) was added. The organic layer was washed with sat. brine, dried, filtered, and evaporated under reduced pressure. The residue was purified by chromatography over silica gel (petroleum ether/EtOAc, 10:1) to provide silyl ether **10** (1.25 g, 96%).

Colorless oil; $[\alpha]_D^{25}$ +54 (*c* 1.0, $CHCl_3$).

IR (film): ν_{max} = 2980, 1730, 1480, 1390, 1280, 1110, 1030, 870, 800 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 5.80−5.60 (m, 1H), 5.07−5.00 (m, 2H), 3.70 (dd, 1H, *J* = 8.1, 4.0 Hz), 2.50 (dd, 1H, *J* = 14.3, 7.8 Hz), 2.39−2.30 (m, 3H), 2.04−1.80 (m, 3H), 1.63−1.58 (m, 2H), 1.09 (s, 3H), 0.9 (s, 9H), 0.07 (s, 6H).

EIMS: *m/z* (rel. intensity) = 265 (1.22), 225 (16.3), 183 (28.8), 133 (25.2), 75 (100), 73 (50.8), 57 (38.3), 41 (74).

Anal. Calcd. for $C_{16}H_{30}O_2Si$: C, 68.03; H, 10.70. Found: C, 67.83; H, 10.73.

(2*S*,3*S*)-3-[(*tert*-Butyldimethylsilyl)oxy]-2-methyl-2-(2-oxopropyl)cyclohexan-1-one (11)

To a solution of **10** (1.25 g, 4.5 mmol) in DMF (3 mL) was added palladium chloride (60 mg, 0.33 mmol), cuprous chloride (450 mg, 4.5 mmol) and H_2O (0.5 mL). The reaction mixture was stirred under an O_2 atm at r.t. for 30 h. EtOAc (100 mL) was added to the reaction mixture and filtered. The filtrate was washed with sat. brine, dried, filtered, and evaporated under reduced pressure. The obtained residue was purified by chromatography over silica gel (petroleum ether/EtOAc, 10:1) to afford **11** (1.1 g, 83%).

Colorless oil; $[\alpha]_D^{25}$ +2.3 (*c* 2.0, $CHCl_3$).

IR (film): ν_{max} = 2950, 1710, 1460, 1360, 1250, 1080, 1020, 840, 780 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 4.14 (d, 1H, *J* = 4.9 Hz), 2.90 and 2.67 (AB, 2H, *J* = 17.8 Hz), 2.55−2.40 (m, 1H), 2.30−2.23 (m, 1H), 2.10 (s, 3H), 2.10−1.90 (m, 2H), 1.75−1.60 (m, 2H), 1.25 (s, 3H), 0.83 (s, 9H), 0.01 (s, 3H), 0.05 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 213.11, 207.83, 77.05, 60.38, 53.89, 45.18, 37.13, 31.61, 28.40, 25.80, 20.82, 20.35, 18.06, 14.21, 4.33, 5.14.

Anal. Calcd. for $C_{16}H_{30}O_3Si$: C, 64.38; H, 10.13. Found: C, 64.51; H, 10.43.

(7*S*,7a*R*)-7-[(*tert*-Butyldimethylsilyl)oxy]-5,6-dihydro-7a-methyl-1*H*-inden-2(4*H*,7a*H*)-one (12)

To a solution of the dione **11** (850 mg, 2.85 mmol) in *tert*-butyl alcohol (20 mL) was added potassium *tert*-butoxide (320 mg, 2.85 mmol), and the reaction mixture was stirred under N_2 for 4 h. The solution was then mediated to pH 7.0 with 0.5 M HCl at 0 °C, and evaporated under reduced pressure to remove the alcohol. The residue was dissolved in EtOAc (150 mL) and washed with sat. brine, dried, filtered, and condensed. The crude product was purified by chromatography over silica gel (petroleum ether/EtOAc, 5:1) to provide **12** (725 mg, 91%) as a white solid, mp: 81−82 °C.

$[\alpha]_D^{21}$ +121 (*c* 1.0, $CHCl_3$).

IR (KBr): ν_{max} = 2950, 1700, 1630, 1470, 1280, 1090, 1030, 850, 790 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 5.71 (d, 1H, *J* = 1.5 Hz), 3.74 (s, 1H), 2.53 and 1.81 (AB, 2H, *J* = 18.0 Hz), 2.53−2.47 (m, 1H), 2.35−2.24 (m, 1H), 1.92−1.51 (m, 4H), 1.16 (s, 3H), 0.72 (s, 9H), 0.07 (s, 3H), 0.09 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.06, 184.52, 127.84, 74.50, 48.38, 46.48, 28.34, 27.02, 25.54, 23.78, 21.02, 17.80, 4.46, 5.48.

Anal. Calcd. for $C_{16}H_{28}O_2Si$: C, 68.52; H, 10.06. Found: C, 68.38; H, 10.30.

(7*S*,7a*R*)-5,6-Dihydro-7-hydroxy-7a-methyl-1*H*-inden-2(4*H*,7a*H*)-one (13)

A solution of silyl ether **12** (140 mg, 0.5 mmol) in MeCN (2 mL) and 40% of aqueous HF (70 μL, 1.5 mmol) was stirred at r.t. for two days. The solution was mediated to neutral with sat. $NaHCO_3$ and extracted with EtOAc (3 x 15 mL). The combined organic layers were dried, filtered, condensed, and the obtained residue was purified over silica gel (petroleum ether/EtOAc, 1:1) to provide **13** (75 mg, 90%) as a white solid, mp: 139−140 °C; 96.2% ee; $[\alpha]_D^{24}$ +64 (*c* 1.0, $CHCl_3$). The enantiomeric purity was determined by HPLC analysis using Chiralpak AD column (eluent, hexane/*i*-PrOH, 95:5), detected at UV_{254}.

IR (KBr): ν_{max} = 3450, 2940, 1680, 1610, 1410, 1290, 1230, 1140, 1055, 980, 890 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 5.89 (d, 1H, *J* = 1.6 Hz), 3.92−3.90 (m, 1H), 2.84 and 2.00 (AB, 2H, *J* = 18.4 Hz), 2.69−2.63 (m, 1H), 2.49−2.38 (m, 1H), 2.20 (br s, 1H), 2.10−1.96 (m, 1H), 1.88−1.74 (m, 3H), 1.29 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 209.31, 185.45, 128.18, 73.49, 48.21, 46.13, 28.18, 27.07, 24.10, 20.94.

EIMS: *m/z* (rel. intensity) = 166 (M^+, 20), 148 (19), 138 (30.6), 123 (47.9), 110 (100), 109 (51), 95 (61.1), 79 (62.3), 67 (35.28), 41 (37.21).

HRMS: calcd. for $(C_{10}H_{14}O_2)^+$: 166.0994. Found: 166.0990.

(7a*R*)-5,6-Dihydro-7a-methyl-1*H*-indene-2,7(4*H*,7a*H*)-dione (4)

To a solution of oxalyl chloride (0.14 mL, 1.64 mmol) in CH_2Cl_2 (3 mL) was added dropwise at −78 °C DMSO (0.3 g, 3.79 mmol). After stirring for 0.5 h, a solution of **13** (220 mg, 1.33 mmol) in CH_2Cl_2 (3 mL) was added, and the mixture was stirred at −60 °C for 2 h. Then a solution of Et_3N (3 mL) and CH_2Cl_2 (5 mL) was added. The mixture was warmed slowly to r.t. and poured into cooled H_2O (20 mL), extracted with CH_2Cl_2 (3 x 30 mL). The organic layers were combined and washed with brine, dried, filtered, and condensed. The residue was purified by chromatography over silica gel (petroleum ether/EtOAc, 2:1) to afford the desired **4** (205 mg, 94%).

$[\alpha]_D^{20}$ +47.3 (*c* 0.42, $CHCl_3$).

IR (film): ν_{max} = 2962, 1712, 1621 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 5.72 (d, 1H, *J* = 1.5 Hz), 3.10 and 2.05 (AB, 2H, *J* = 18.9 Hz), 2.84−2.61 (m, 3H), 2.39−2.31 (m, 1H), 2.25−2.15 (m, 1H), 1.73−1.56 (m, 1H), 1.45 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 209.28, 205.83, 181.95, 127.05, 58.63, 43.63, 36.77, 26.20, 25.58, 24.76.

EIMS: *m/z* (rel. intensity) = 164 (M^+, 100), 136 (62.5), 121 (74.9), 108 (42.7), 93 (31.1), 80 (60.9), 79 (79.5), 77 (27.5).

HRMS: calcd. for $(C_{10}H_{12}O_2)^+$: 164.08373. Found: 164.08428.

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