Stereoselective Reduction of β-Hydroxy α-Ketoesters: A Concise Synthesis of *anti*-α,β-Dihydroxy Esters

Mingyi Liao, Wengang Yao, Jianbo Wang*

Key Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, Department of Chemical Biology, College of Chemistry, Peking University, Beijing 100871, P. R. of China
E-mail: wangjb@pku.edu.cn

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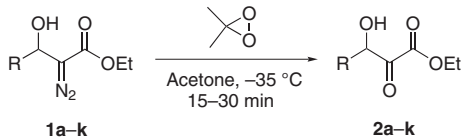
Abstract: A new synthetic route to *anti*-α,β-dihydroxy esters has been developed. The new method consists of three steps starting from an aldehyde: the nucleophilic condensation with ethyl diazoacetate, oxidation with dimethyldioxirane, and stereoselective reduction with $NaBH_4$.

Key words: diazo compounds, diols, nucleophilic addition, oxidation, reduction, diastereoselectivity

The reduction of α-ketols is an important approach leading to 1,2-diol compounds. The stereoselectivity of the reduction has been extensively studied over the past decades, and it has been well known that the stereochemical process is controlled by either the Cram model or by the chelation model.[1,2] However, to our knowledge, there has been no report on the reduction of β-hydroxy α-ketoesters. The reaction will give α,β-dihydroxy carbonyl compounds, which are important as natural product fragments and also as building blocks in organic synthesis. Although the structure is quite simple, the stereoselective synthesis of this type of compounds is limited. The most common approach is the dihydroxylation of the α,β-unsaturated carbonyl compounds.[3] Since the easily available α,β-unsaturated carbonyl compounds usually have *trans* configuration, the dihydroxylation method therefore in most cases gave only *syn*-α,β-dihydroxy carbonyl compounds. To the best of our knowledge, the only general method to prepare *anti* α,β-dihydroxy carbonyl compounds with high diastereoselectivity is the aldol condensation of glycolate enolate with aldehydes.[4,5] Here we report a concise and highly diastereoselective approach to this type of compounds through the reduction of easily available β-hydroxy α-ketoesters.

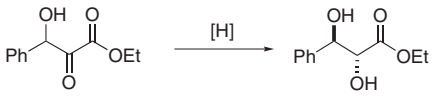


1a–k **2a–k**

Scheme 1

The β-hydroxy α-ketoesters **2a–k** were prepared by the oxidation of the β-hydroxy α-diazoesters **1a–k** with dimethyldioxirane (DMD).[6] The β-hydroxy α-diazoesters **1a–k** were easily obtained by the nucleophilic condensation of aldehydes with ethyl diazoacetate.[7] The oxidation of **1a–k** with dimethyldioxirane (DMD) in acetone is highly efficient (–35 °C, 15–30 min, Scheme 1). The β-hydroxy α-ketoesters **2a–k** thus obtained were found to be unstable in silica gel column. Consequently, they were used in the subsequent reduction without further purification.

Table 1 Reduction of α-Ketoesters **2a**



2a (±)-**3a**

Entry	Hydride	Solvent	Temp (°C)	dr[a]
1	$NaBH_4$	EtOH	–35	88:12
2	$NaBH_4$	EtOH	–78	89:11
3	$NaBH_4$	EtOH	–98	90:10
4	$NaBH(OAc)_3$	EtOH	–78	76:24
5	$NaBH_3(CN)$	EtOH	–78	73:27
6	$Zn(BH_4)_2$	THF	–35	87:13
7	$NaBH_4$	THF	–78	91:9
8	$NaBH_4$	CH_2Cl_2	–78 to r.t.[b]	>95:5
9	$NaBH_4$	CH_2Cl_2	–41	90:10
10	$NaBH_4$	CH_2Cl_2	–63 to r.t.	>95:5

[a] The diastereomeric ratio was determined by the ^1H NMR (300 MHz) of the crude product.
[b] The reduction at –78 °C was very slow.

First, α-ketoesters **2a** (R = Ph) was used as the substrate to optimize the reduction condition. When sodium borohydride was used as the reducing agent in EtOH, **2a** was reduced efficiently to give α,β-dihydroxy ester, but the diastereoselectivity is moderate (88:12) (Table 1, entry 1). The reduction also proceeded well at low temperature; however, the stereoselectivity almost remained the same. Moreover, it was found that diastereoselectivity could not

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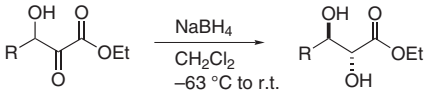
be improved by changing the reducing agent to $NaBH(OAc)_3$, $NaBH_3(CN)$ or $Zn(BH_4)_2$. The effect of the solvent was then examined. We were delighted to find that with CH_2Cl_2 as the solvent the stereoselectivity could be dramatically improved. ^1H NMR inspection of the crude product suggested that only one diastereoisomer was formed. The configuration of the main product was determined to be *anti* by comparing with the ^1H NMR spectrum of the *syn* product, which was prepared by the dihydroxylation of ethyl cinnamate.

The optimized reduction condition was then applied to the β-hydroxy α-ketoesters **2a–k**. As shown by the data in Table 2, all the α-ketoesters with β-aryl substituent gave excellent diastereoselectivities, regardless of the position of the substituent in the phenyl ring. When the β substituent is an alkyl group, the diastereoselectivity decreases to some extent (Table 2, entries 8–11).

Table 2 Diastereoselective Reduction of α-Ketoesters **2a–k** with $NaBH_4$ in CH_2Cl_2



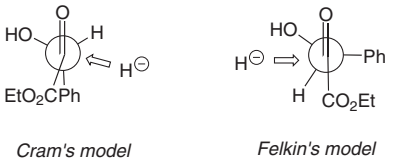
2a–k (±)-**3a–k**

Entry	α-Ketoester **2** (R =)	dr[a]	Yield (%)[b]
1	**2a**, C_6H_5	>95:5	75
2	**2b**, *o*-$CH_3C_6H_4$	>95:5	86
3	**2c**, *m*-BrC_6H_4	>95:5	78
4	**2d**, *m*-$CF_3C_6H_4$	>95:5	71
5	**2e**, *p*-FC_6H_4	>95:5	75
6	**2f**, *p*-$MeOC_6H_4$	>95:5	63
7	**2g**, *p*-PhC_6H_4	>95:5	62
8	**2h**, CH_3CH_2	93:7	60
9	**2i**, $CH_3(CH_2)_5$	92:8	80
10	**2j**, $(CH_3)_2CHCH_2$	85:15	69
11	**2k**, Cyclopentyl	85:15	74

[a] The diastereomeric ratio was determined by the ^1H NMR (300 MHz) of the crude product.
[b] Isolated yields for 2 steps.

The *anti* selectivity of the reduction can be rationalized by either Cram's rule[8] or Felkin's rule,[9] as shown in Figure 1. In Cram's model, the attacking hydride approaches from the small group (the hydrogen) side in the conformation, while in Felkin's model, the hydride attacks the carbonyl group from the direction between hydroxyl group and the hydrogen. Both models predict the *anti* isomer to be predominant.

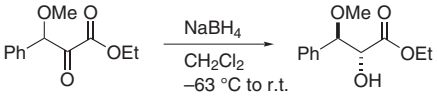
Another possibility in the stereochemical process is that the intramolecular hydrogen bonding may play a role. To determine whether the hydrogen bonding is responsible



Cram's model *Felkin's model*

Figure 1

for the observed stereoselectivity, β-methoxy α-ketoester was prepared and its reduction was investigated (Scheme 2). Under the identical reduction condition, the product was found to have a diastereomeric ratio of 88:12. This result is in favor of simple Cram's or Felkin's model in the stereochemical process, although it does not conclusively remove the possible role of hydrogen bonding in the stereochemical process.



4 90%; *dr* = 88:12 (±)-**5**

Scheme 2

In summary, it is found that the reduction of β-hydroxy α-ketoesters with $NaBH_4$ in CH_2Cl_2 can give good to excellent diastereoselectivity. Thus, the *anti* α,β-dihydroxy esters become easily available from aldehydes and ethyl diazoacetate in three steps.

All reactions were performed under a nitrogen atmosphere in a flame-dried reaction flask, and the components were added via syringe. All solvents were distilled prior to use. CH_2Cl_2 was freshly distilled from CaH_2 before use. For chromatography, 100–200 mesh silica gel (Qindao, China) was employed. ^1H and ^{13}C NMR spectra were recorded at 300 MHz and 75 MHz, respectively, on a Varian Mercury 300 spectrometer. Chemical shifts are reported in ppm using TMS as internal standard. IR spectra were recorded on a Nicolet 5MX-S infrared spectrometer. Mass spectra were obtained on a VG ZAB-HS mass spectrometer.

Oxidation of β-Hydroxy α-Diazoesters 1a–k with DMD and the Subsequent Reduction of the β-Hydroxy α-Ketoesters 2a–k with $NaBH_4$; General Procedure

β-Hydroxy α-diazoesters **1a–k** (0.25 mmol) was dissolved in acetone (2 mL) and a solution of dimethyldioxirane in acetone (5 mL) was added to the solution at –35 °C. The mixture was allowed to stir for 15–30 min until the starting diazo compound was no longer present when monitored by TLC. Then the solvent was removed with rotary evaporator, and the residue was dissolved in CH_2Cl_2 (4 mL) and dried over anhyd Na_2SO_4. The drying agent was filtered off and washed with CH_2Cl_2. The solvent was removed by rotary evaporation to give crude β-hydroxy α-ketoesters **2a–k**, which were used in the following reaction without further purification.

The crude product was dissolved in CH_2Cl_2 (4 mL), then the solution was cooled to –63 °C and $NaBH_4$ (10 mg, 0.25 mmol) was added. The mixture was stirred for about 8 h, during which the reaction temperature rose to r.t. Then sat. solution of NH_4Cl was added, and the reaction mixture was stirred for an additional 30 min and was extracted with CH_2Cl_2 (4 × 10 mL). The combined organic layer was dried over anhyd Na_2SO_4 and concentrated under reduced pres-

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sure with rotary evaporator. The residue was subjected to silica gel chromatography (petroleum ether–$CHCl_3$–CH_3OH, 30:4:1) to afford the pure product.

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-phenylpropanoate (3a)[4a,10]
IR (film): 3399, 1731, 1208, 1100 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.14 (t, *J* = 7.2 Hz, 3 H), 3.30 (br s, 2 H), 4.10 (q, *J* = 7.2 Hz, 2 H), 4.48 (dd, *J* = 6.6, 4.2 Hz, 1 H), 5.02 (dd, *J* = 5.1, 4.2 Hz, 1 H), 7.26–7.36 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.86, 61.70, 74.80, 74.91, 126.35, 128.00, 128.14, 138.58, 171.92.

EI–MS: *m*/*z* (relative intensity) = 210 (6) [M^+], 119 (10), 107 (78), 104 (100), 91 (23), 76 (99), 29 (21).

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-(*o*-methylphenyl)propanoate (3b)
IR (film): 3438, 1732, 1210, 1090 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.08 (t, *J* = 7.2 Hz, 3 H), 2.35 (s, 3 H), 3.23 (br s, 1 H), 3.51 (br s, 1 H), 4.02–4.13 (m, 2 H), 4.40 (dd, *J* = 6.3, 4.5 Hz, 1 H), 5.22 (dd, *J* = 4.2, 4.5 Hz, 1 H), 7.12–7.22 (m, 3 H), 7.46–7.49 (m, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.75, 19.14, 61.65, 71.87, 73.71, 125.85, 126.02, 127.72, 130.24, 134.82, 137.06, 172.33.

EI–MS: *m*/*z* (relative intensity) = 224 (12) [M^+], 133 (9), 121 (100), 104 (60), 93 (53), 91 (36), 77 (35), 76 (56), 29 (12).

Anal. Calcd for $C_{12}H_{16}O_4$: C, 64.27; H, 7.19. Found: C, 64.12; H, 7.06.

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-(*m*-bromophenyl)propanoate (3c)
IR (film): 3395, 1732, 1209, 1101 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7.2 Hz, 3 H), 3.46 (br s, 2 H), 4.07–4.18 (m, 2 H), 4.45 (br s, 1 H), 5.01 (br s, 1 H), 7.17–7.51 (m, 4 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.93, 62.00, 74.34, 74.76, 122.31, 125.02, 129.56, 129.70, 130.98, 141.06, 171.75.

EI–MS: *m*/*z* (relative intensity) = 288 (4) [M^+], 259 (4), 215 (19), 185 (100), 104 (100), 76 (100), 29 (98).

Anal. Calcd for $C_{11}H_{13}O_4Br$: C, 45.70; H, 4.53. Found: C, 45.88; H, 4.60.

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-(*m*-trifluoromethylphenyl)propanoate (3d)
IR (film): 3405, 1734, 1328, 1170, 1123 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.11 (t, *J* = 7.2 Hz, 3 H), 3.57 (br s, 2 H), 4.10 (q, *J* = 7.2 Hz, 2 H), 4.49 (d, *J* = 3.7 Hz, 1 H), 5.11 (d, *J* = 3.7 Hz, 1 H), 7.43–7.62 (m, 4 H).

^{13}C NMR (75MHz, $CDCl_3$): δ = 13.70, 62.03, 74.43, 74.73, 122.23, 123.17, 123.22, 123.27, 123.32, 124.61, 124.66, 124.71, 124.76, 125.84, 128.57, 129.76, 130.23, 130.66, 139.79, 171.73.

EI–MS: *m*/*z* (relative intensity) = 259 (8) [M – 19]$^+$, 187 (14), 175 (41), 127 (43), 104 (99), 76 (100).

Anal. Calcd for $C_{12}H_{13}O_4F_3$: C, 51.80; H, 4.71. Found: C, 51.66; H, 4.75.

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-(*p*-fluorophenyl)propanoate (3e)
IR (film): 3417, 1733, 1221, 1106 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7.2 Hz, 3 H), 3.33 (br s, 2 H), 4.12 (q, *J* = 7.2 Hz, 2 H), 4.45 (dd, *J* = 6.3, 4.2 Hz, 1 H), 5.00 (dd, *J* = 4.2, 4.2 Hz, 1 H), 6.99–7.05 (m, 2 H), 7.27–7.33 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.93, 61.89, 74.28, 74.66, 114.90, 115.19, 128.07, 128.18, 134.38, 134.43, 160.81, 164.07, 171.84.

EI–MS: *m*/*z* (relative intensity) = 228 (5) [M^+], 209 (27), 155 (5), 137 (61), 125 (100), 109 (100), 104 (92), 97 (100), 95 (85), 77 (98), 76 (93), 29 (97).

Anal. Calcd for $C_{11}H_{13}O_4F$: C, 57.89; H, 5.74. Found: C, 58.05; H, 5.76.

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-(*p*-methoxyphenyl)propanoate (3f)
IR (film): 3426, 1732, 1248, 1107, 1028 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.18 (t, *J* = 7.2 Hz, 3 H), 3.20 (br s, 2 H), 3.78 (s, 3 H), 4.13 (q, *J* = 7.2 Hz, 2 H), 4.45 (d, *J* = 4.2 Hz, 1 H), 4.96 (d, *J* = 4.2 Hz, 1 H), 6.85 (d, *J* = 9.0 Hz, 2 H), 7.24 (d, *J* = 9.0 Hz, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.97, 55.17, 61.73, 74.49, 74.70, 113.54, 127.67, 130.67, 159.31, 171.99.

EI–MS: *m*/*z* (relative intensity) = 240 (7) [M^+], 137 (100), 109 (22), 77 (17), 29 (10).

Anal. Calcd for $C_{12}H_{16}O_5$: C, 59.99; H, 6.71. Found: C, 59.76; H, 6.70.

(±)-*anti*-Ethyl 2,3-Dihydroxy-3-(*p*-phenylphenyl)propanoate (3g)
IR (film): 3349, 1727, 1211, 1099 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7.2 Hz, 3 H), 3.39 (br s, 2 H), 4.12 (q, *J* = 7.2 Hz, 2 H), 4.51 (d, *J* = 3.6 Hz, 1 H), 5.07 (d, *J* = 3.6 Hz, 1 H), 7.33–7.58 (m, 9 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.92, 61.80, 74.75, 74.79, 126.86, 126.99, 127.30, 128.72, 137.63, 140.58, 140.84, 171.95.

EI–MS: *m*/*z* (relative intensity) = 286 (6) [M^+], 183 (100), 155 (29), 115 (4), 102 (13), 76 (16), 29 (7).

Anal. Calcd for $C_{17}H_{18}O_4$: C, 71.31; H, 6.34. Found: C, 71.29; H, 6.32.

(±)-*anti*-Ethyl 2,3-Dihydroxypentanoate (3h)
IR (film): 3397, 1735, 1209, 1125 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.01 (t, *J* = 7.2 Hz, 3 H), 1.32 (t, *J* = 7.2 Hz, 3 H), 1.48–1.61 (m, 2 H), 2.35 (d, *J* = 6.6 Hz, 1 H), 3.17 (d, *J* = 5.7 Hz, 1 H), 3.74–3.82 (m, 1 H), 4.21–4.32 (m, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ =10.24, 14.14, 24.78, 61.96, 73.62, 74.69, 172.83.

EI–MS: *m*/*z* (relative intensity) = 163 (0.3) [M + 1]$^+$, 133 (4), 104 (78), 89 (16), 76 (100), 59 (35), 29 (39).

Anal. Calcd for $C_7H_{14}O_4$: C, 51.84; H, 8.70. Found: C, 51.59; H, 8.95.

(±)-*anti*-Ethyl 2,3-Dihydroxynonanoate (3i)
IR (film): 3403, 1736, 1208, 1084 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.6 Hz, 3 H), 1.28–1.60 (m, 13 H), 2.58 (br s, 1 H), 3.30 (br s, 1 H), 3.83–3.88 (m, 1 H), 4.20–4.33 (m, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.02, 14.13, 22.54, 25.65, 29.10, 31.65, 61.86, 73.22, 73.96, 172.80.

EI–MS: *m*/*z* (relative intensity) = 219 (3) [M + 1]$^+$, 145 (9), 133 (3), 104 (100), 76 (99), 55 (38), 29 (34).

Anal. Calcd for $C_{11}H_{22}O_4$: C, 60.52; H, 10.16. Found: C, 60.47; H, 9.67.

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(±)-*anti*-Ethyl 2,3-Dihydroxy-5-methylhexanoate (3j)
IR (film): 3377, 1734, 1211, 1079 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 0.93 (dd, *J* = 10.8, 6.6 Hz, 6 H), 1.08–1.17 (m, 1 H), 1.32 (t, *J* = 7.2 Hz, 3 H), 1.48–1.58 (m, 1 H), 1.78–1.87 (m, 1 H), 2.60 (br s, 1 H), 3.42 (br s, 1 H), 3.96 (d, *J* = 4.5 Hz, 1 H), 4.19–4.37 (m, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.13, 21.57, 23.51, 24.32, 40.45, 61.81, 71.24, 74.23, 172.72.

EI–MS: *m*/*z* (relative intensity) = 191 (0.2) $[M + 1]^+$, 117 (7), 104 (100), 87 (6), 76 (99), 43 (33), 29 (26).

Anal. Calcd for $C_9H_{18}O_4$: C, 56.82; H, 9.54. Found: C, 56.59; H, 9.35.

(±)-*anti*-Ethyl 3-Cyclopentyl-2,3-dihydroxy propanoate (3k)
IR (film): 3403, 1734, 1207, 1090 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.17–1.92 (m, 11 H), 1.99–2.10 (m, 1 H), 2.57 (br s, 1 H), 3.36 (dd, *J* = 6.0, 6.0 Hz, 1 H), 3.61–3.67 (m, 1 H), 4.22–4.31 (m, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.12, 25.13, 25.52, 28.91, 29.84, 41.85, 61.80, 73.35, 78.16, 172.86.

EI–MS: *m*/*z* (relative intensity) = 133 (3), 104 (100), 81 (34), 76 (81), 41 (37), 29 (31).

Anal. Calcd for $C_{10}H_{18}O_4$: C, 59.39; H, 8.97. Found: C, 59.48; H, 8.73.

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