

Nucleophilic Benzoylation Using a Mandelic Acid Dioxolanone as a Synthetic Equivalent of the Benzoyl Carbanion. Oxidative Decarboxylation of α -Hydroxyacids

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Abstract: The synthesis of alkyl aryl ketones using a mandelic acid dioxolanone as a synthetic equivalent (Umpolung) of the benzoyl carbanion is reported. The methodology involves alkylation of the mandelic acid dioxolanone, hydrolysis of the dioxolanone moiety in the alkylated products and oxidative decarboxylation of the resulting α -hydroxyacids. The last step is carried out in a catalytic aerobic way using a Co (III) complex in the presence of pivalaldehyde under very mild conditions.

Keywords: Alkylation, dioxolanone, decarboxylation, catalysts, cobalt, Umpolung.

Introduction

We have recently reported the use of methyl mandelate as a masked d^1 -synthon for nucleophilic benzoylation ("Umpolung") of the carbonyl group [1] in a synthesis of aryl alkyl ketones, which involves alkylation of methyl mandelate, hydrolysis of the ester group in the alkylated products and oxidative decarboxylation of the resulting α -hydroxyacids (Scheme 1). Because of the importance of the carbonyl group in organic chemistry, it is not surprising that several "Umpolung" methods for this group have been described, including the use of dithianes and dithiolanes [2], TosMIC [3], enol ethers [4], vinyl sulfides [5], α -aminonitriles [6], cyanohydrins [7], SAMP-hydrazones [8], or heterocycles

[9] among other. In this paper, we wish to report a related procedure which uses a different mandelic acid derivative, namely 2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (**3**) as a new unpoled synthon of the benzoyl carbanion leading to alkyl aryl ketones.

Results and Discussion

In our earlier work [1] we carried out the alkylation of the dianion of methyl mandelate (**2**) using 2 equiv. of LDA and 1 equiv. of an alkyl halide as it had been described in the literature [10]. Now, we have carried out the alkylation of a mandelic acid dioxolanone, namely 2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (**3**), which has the advantage of requiring only one equivalent of strong base (Scheme 1).

Dioxolanone **3** was readily prepared by acid catalysed condensation of mandelic acid (**1**) with acetone. The lithium enolate derived from **3** was alkylated with several organic halides (Table 1). The use of alkyl, allyl or benzyl chlorides as alkylating reagents was unsuccessful as the starting materials were recovered unreacted in all of the cases. Nevertheless the introduction of benzylic or allylic groups could be successfully achieved using the corresponding bromides as alkylating reagents [11], while the introduction of an alkyl group required the use of the corresponding iodide and the addition of 20% HMPA as cosolvent. Following this methodology we were able to prepare in good yields several α -alkylated dioxolanones **5** bearing primary, allyl or benzyl groups, and other additional functional groups such as a bromide or a carboxyester.

Scheme 1

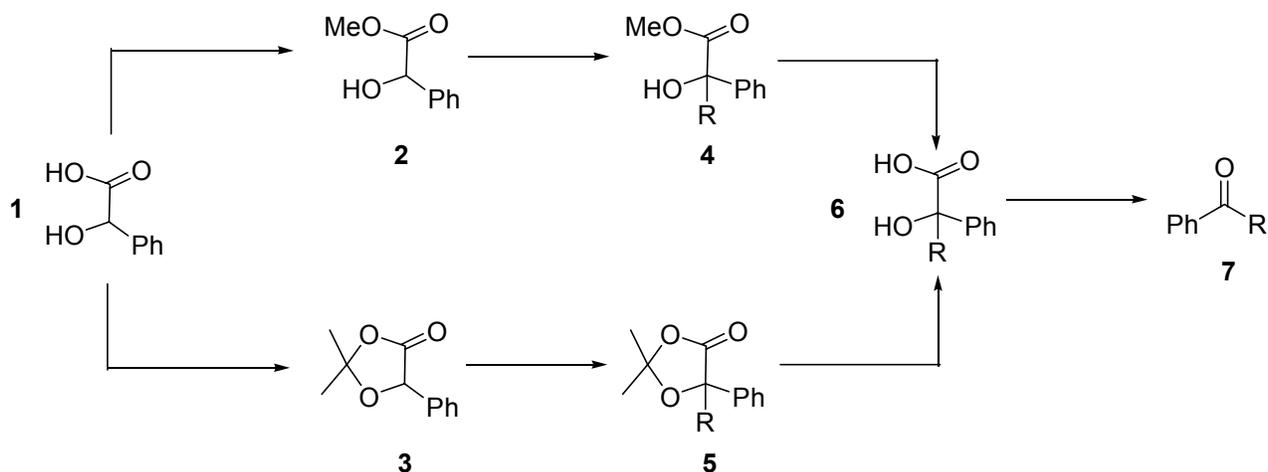


Table 1. Alkylation of 2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (**3**) and oxidative decarboxylation of α -alkylated mandelic acids (**6**).

Entry	Compound 5 ^a	Yield of 5 (%)	Compound 6 ^b	Hydrolysis time (h)	Ketone 7	Yield of 7 (%) ^c
a		76		6.0		87
b		65		2.0		95
c		62		2.5		75
d		65		3.0		93
e		75		3.0		94
f		52		7.0		97
g		55		5.5		81
h		77		9.5		50

^a Entries a-c required RI as alkylating reagent and the use of 20% of HMPA as cosolvent.

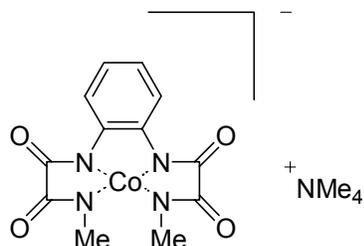
Entries d-h were carried out with RBr as alkylating reagent.

^b Nearly quantitative yields of the hydrolysis products **6** were obtained in all cases.

^c The reaction was carried out at room temperature; entries f-g at 0 °C and entry h at -47 °C.

Upon basic hydrolysis, the products **5** were transformed into the corresponding α -hydroxyacids **6** in almost quantitative yields. Finally, oxidative decarboxylation of **6** gave the corresponding alkyl aryl ketones **7**. This transformation was achieved using a catalytic procedure developed in our laboratory which employs molecular oxygen as terminal oxidant in the presence of pivalaldehyde as co-reductant and a catalytic amount of the Co(III) *ortho*-phenylene-*bis*(*N*'-methyloxamidate) complex [Co(III)-Me₂opba] (Figure 1) [12].

Figure 1



Conclusions

In summary, we have presented in this paper a procedure for the use of a mandelic acid derivative, namely 2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (**3**) as a new masked d¹-synthon for the benzoyl group (Umpolung), in which the key step is the aerobic oxidative decarboxylation with oxygen in the presence of pivalaldehyde as co-reductant and the Co(III)-Me₂opba complex as catalysts. This reaction is carried out under very mild conditions and it is compatible with the presence in the molecule of other oxidisable groups.

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Experimental

General

All melting points are uncorrected. Column chromatography was performed on silica gel 60 (Merck, 0.040-0.063 mm). Unless specified otherwise NMR experiments were run for CDCl₃ solutions at 300 MHz for ¹H- and at 75 MHz for ¹³C-NMR, and referenced to the solvent as internal standard. The carbon type was determined by DEPT experiments. Mass spectra were run by electron impact (70 eV) or by chemical ionisation using methane as ionising gas. Co(III)-Me₂opba complex was prepared according to the previously reported procedure [12].

Preparation of 2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (3)

Conc. H₂SO₄ (1.5 mL, 28.36 mmol) was added very slowly to a solution of mandelic acid (2 g, 13.16 mmol) in toluene (160 mL) and acetone (40 mL). The resulting solution was stirred at room temperature until the reaction was complete (24 hours). The reaction mixture was then poured into water (80 mL), extracted with ether (3 x 50 mL), washed with saturated solution of NaHCO₃ and brine, and dried over MgSO₄. After removal of the solvent under reduced pressure compound **3** (1.64 g, 65%) was obtained as an oil: ¹H-NMR δ 7.45-7.35 (5H, m), 5.38 (1H, m), 1.71 (3H, s), 1.66 (3H, s); ¹³C-NMR δ 171.7 (s), 134.7 (s), 129.2 (d), 129.0 (d), 126.7 (d), 111.2 (s), 76.1 (d), 27.5 (q), 26.4 (q); MS (EI) *m/z* 192 (M⁺, 9.5), 177 (19.6), 148 (35.9), 107 (100), 90 (25.2), 79 (46.8); HRMS (EI) *m/z* required for C₁₁H₁₂O₃ 192.0786, found 192.0796.

General procedure for alkylation of 2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (3)

A 1.6 M solution of *n*-BuLi in hexane (0.78 mL, 1.25 mmol) was added dropwise to a solution of diisopropylamine (0.175 mL, 1.25 mmol) in dry THF (0.8 mL) at 0 °C under argon. After 30 min, the solution was cooled to −78 °C and a solution of dioxolanone **3** (192 mg, 1 mmol) in THF (0.65 mL) was added. The resulting solution was stirred at −78 °C for 30 min and then the corresponding alkyl halide (1.25 mmol) in THF (0.39 mL) was added. The reaction mixture was stirred at this temperature for 2 hours and then at room temperature until the reaction was complete (2 hours). After this time, the reaction mixture was poured into aq. NH₄Cl (30 mL), extracted with ether (3 x 30 mL), washed with brine and dried over MgSO₄. After filtration and removal of the solvent under reduced pressure, the products **5** were obtained by column chromatography (elution with hexane-diethyl ether).

2,2,5-Trimethyl-5-phenyl-1,3-dioxolan-4-one (5a). An oil; ¹H-NMR δ 7.54 (2H, dd, J = 8.1, 1.2 Hz), 7.4-7.2 (3H, m), 1.67 (3H, s), 1.61 (3H, s), 1.37 (3H, s); ¹³C-NMR δ 172.7 (s), 139.8 (s), 127.4 (d), 127.0 (d), 123.5 (d), 109.1 (s), 79.5 (s), 28.2 (q), 27.4 (q), 26.6 (q); MS (EI) *m/z* 206 (M⁺, 6.7), 191 (7.8), 162 (83.5), 121 (36.1), 104 (100), 77 (31.8); HRMS (EI) *m/z* required for C₁₂H₁₄O₃ 206.0943, found 206.0949.

5-Dodecyl-2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (5b). An oil; ¹H-NMR δ 7.57 (2H, dd, J = 8.1, 1.2 Hz), 7.4-7.2 (3H, m), 1.89 (2H, m), 1.64 (3H, s), 1.36 (3H, s), 1.20 (20H, m), 0.83 (3H, t, J = 7.0 Hz); ¹³C-NMR δ 173.3 (s), 140.2 (s), 128.3 (d), 127.8 (d), 124.8 (d), 110.0 (s), 83.6 (s), 41.9 (t), 31.9 (t), 29.59 (t, two overlapped signals), 29.56 (t), 29.48 (t), 29.31 (t), 29.29 (t, two overlapped signals), 27.8 (q), 24.0 (t), 22.7 (t), 14.1 (q); MS (EI) *m/z* 360 (M⁺, 24.6), 316 (47.9), 275 (28.4), 191 (61.9), 163 (98.7), 105 (100), 77 (33.5); HRMS (EI) *m/z* required for C₂₃H₃₆O₃ 360.2664, found 360.2650.

2,2-Dimethyl-5-(3,7-dimethyl-6-octenyl)-5-phenyl-1,3-dioxolan-4-one (5c). An oil; ¹H-NMR δ 7.56 (2H, dd, J = 8.1, 1.2 Hz), 7.4-7.2 (3H, m), 5.00 (1H, m), 2.0-1.8 (4H, m), 1.64 (3H, s), 1.61 (3H, s),

1.52 (3H, s), 1.36 (3H, s), 1.35-1.10 (5H, m), 0.79 (3H, t, $J = 6.4$ Hz); $^{13}\text{C-NMR}$ δ 173.3 (s), 140.2 (s), 131.2 (s), 128.3 (d), 127.8 (d), 124.8 (d), 124.7 (d), 110.0 (s), 83.6 (s), 39.4 (t), 36.7 (t), 32.1 (d), 30.8 (t), 27.8 (q), 25.7 (q), 25.3 (t), 19.4 (q), 19.3 (q), 17.6 (q); MS (CI) m/z 331 ($\text{M}^+ + 1$, 0.5), 301 (8.1), 273 (100), 245 (15.9), 227 (32.0), 189 (17.7), 137 (13.1), 107 (8.7); HRMS (CI) m/z required for $\text{C}_{21}\text{H}_{31}\text{O}_3$ 331.2273, found 331.2263($\text{M}^+ + 1$).

5-Benzyl-2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (5d). M.p. 47-49 °C (from CH_2Cl_2); $^1\text{H-NMR}$ δ 7.63 (2H, dd, $J = 8.1, 1.2$ Hz), 7.35-7.22 (3H, m), 7.20-7.10 (5H, m), 3.31 (1H, d, $J = 13.9$ Hz), 2.98 (1H, d, $J = 13.9$ Hz), 1.27 (3H, s), 1.01 (3H, s); $^{13}\text{C-NMR}$ δ 172.5 (s), 140.0 (s), 135.0 (s), 131.0 (d), 128.4 (d), 128.0 (d), 127.1 (d), 124.8 (d), 110.4 (s), 84.3 (s), 47.7 (t), 27.9 (q), 27.0 (q); MS (EI) m/z 282 (M^+ , 6.4), 191 (96.8), 178 (16.6), 163 (23.1), 105 (100), 77 (45.9); HRMS (EI) m/z required for $\text{C}_{18}\text{H}_{18}\text{O}_3$ 282.1256, found 282.1243.

5-(p-Bromobenzyl)-2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (5e). M.p. 65-66 °C (from CH_2Cl_2); $^1\text{H-NMR}$ δ 7.65 (2H, dd, $J = 8.1, 1.2$ Hz), 7.40-7.30 (5H, m), 7.05 (2H, d, $J = 8.4$ Hz), 3.29 (1H, d, $J = 13.9$ Hz), 3.02 (1H, d, $J = 13.9$ Hz), 1.34 (3H, s), 1.19 (3H, s); $^{13}\text{C-NMR}$ δ 172.2 (s), 139.6 (s), 133.9 (s), 132.7 (d), 131.1 (d), 128.5 (d), 128.2 (d), 124.8 (d), 121.3 (s), 110.5 (s), 83.9 (s), 47.1 (t), 27.8 (q), 27.2 (q); MS (EI) m/z 362/360 (M^+ , 2.0/1.8), 277/275 (7.0/7.4), 191 (100), 178 (25.9), 163 (46.1), 105 (47.8), 77 (99.0); HRMS (EI) m/z required for $\text{C}_{18}\text{H}_{17}\text{O}_3\text{Br}$ 362.0341 / 360.0361, found 362.0382 / 360.0360.

5-Allyl-2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (5f). An oil; $^1\text{H-NMR}$ δ 7.62 (2H, dd, $J = 8.1, 1.2$ Hz), 7.40-7.25 (3H, m), 5.72 (1H, m), 5.15 (1H, brd, $J = 9.9$ Hz), 5.13 (1H, brd, $J = 17.4$ Hz), 2.70 (2H, m), 1.66 (3H, s), 1.40 (3H, s); $^{13}\text{C-NMR}$ δ 172.5 (s), 139.5 (s), 131.3 (d), 128.4 (d), 128.0 (d), 124.8 (d), 120.3 (t), 110.2 (s), 83.3 (s), 45.9 (t), 27.8 (q), 27.7 (q); MS (CI) m/z 233 ($\text{M}^+ + 1$, 0.8), 215 (2.3), 203 (5.0), 191 (4.5), 175 (100), 157 (7.9), 147 (12.0), 131(30.6) 129 (26.2), 105 (27.5); HRMS (CI) m/z required for $\text{C}_{14}\text{H}_{17}\text{O}_3$ 233.1178, found 233.1171 ($\text{M}^+ + 1$).

5-(2-Bromoallyl)-2,2-dimethyl-5-phenyl-1,3-dioxolan-4-one (5g). An oil; $^1\text{H-NMR}$ δ 7.58 (2H, dd, $J = 8.1, 1.2$ Hz), 7.30-7.24 (3H, m), 5.61 (1H, s), 5.55 (1H, s), 3.14 (1H d, $J = 15.1$ Hz), 2.93 (1H, d, $J = 15.1$ Hz), 1.67 (3H, s), 1.36 (3H, s); $^{13}\text{C-NMR}$ δ 172.0 (s), 139.0 (s), 128.5 (d), 128.4 (d), 125.2 (s), 124.9 (d), 122.9 (t), 110.9 (s), 83.1 (s), 51.6 (t), 27.9 (q), 27.6 (q); MS (CI) m/z 283/281 ($\text{M}^+ - \text{C}_2\text{H}_5$, 3.5/3.9), 255/253 (65.8/70.1), 237/235 (26.7/27.8), 227/225 (22.8/24.0), 191 (64.3), 173 (75.7), 129 (63.8), 105 (100); HRMS (CI) m/z required for $\text{C}_{12}\text{H}_{10}\text{O}_3\text{Br}$ 2872.9793 / 280.9813, found 282.9757 / 280.9824,.

2,2-Dimethyl-5-(methoxycarbonylmethyl)-5-phenyl-1,3-dioxolan-4-one (5h). M.p. 105-107 °C (from CH_2Cl_2); $^1\text{H-NMR}$ δ 7.62 (2H, dd, $J = 8.1, 1.2$ Hz), 7.39-7.27 (3H, m), 3.69 (3H, s), 3.10 (1H, d, $J = 16.8$ Hz), 2.89 (1H, d, $J = 16.8$ Hz), 1.66 (3H, s), 1.35 (3H, s); $^{13}\text{C-NMR}$ δ 171.9 (s), 169.0 (s), 138.9

(s), 128.6 (d), 128.4 (d), 124.7 (d), 110.8 (s), 80.6 (s), 51.9 (q), 45.5 (t), 27.8 (q), 26.7 (q); MS (EI) m/z 264 (M^+ , 4.4), 220 (31.8), 205 (8.3), 179 (25.6), 133 (21.5), 105 (100), 77 (49.5); HRMS (EI) m/z required for $C_{14}H_{16}O_5$ 264.0998, found 264.1005.

General procedure for hydrolysis of α -alkylated dioxolanones **5**

The α -alkylated dioxolanones **5** (0.3 mmol) were treated with 5% ethanolic KOH (0.75 mL, 0.6 mmol) at room temperature until complete reaction of the starting material (as indicated by TLC). The solution was poured into ice and acidified with 1M HCl to pH \sim 2. The aqueous mixture was extracted with EtOAc (3 x 20 mL), the organic layers were washed with brine until neutral, dried, filtered and concentrated under reduced pressure to give the α -alkylated mandelic acids **6** in almost quantitative yield. For characterisation of compounds **6a**, **6b**, **6d** and **6f-6h** see ref [1].

2-Hydroxy-5,9-dimethyl-2-phenyl-8-decenoic acid (6c). M.p. 78-80 °C (from ethyl acetate); 1H -NMR δ 7.61 (2H, d, $J = 7.0$ Hz), 7.40-7.20 (3H, m), 5.06 (1H, m), 2.3-1.8 (4H, m), 1.67 (3H, s), 1.57 (3H, s), 1.45-1.10 (5H, m), 0.87 (3H, t, $J = 6.2$ Hz); ^{13}C -NMR δ 180.6 (s), 141.0 (s), 131.1 (s), 128.3 (d), 128.0 (d), 125.5 (d), 124.7 (d), 78.4 (s), 37.1 (t), 36.7 (t), 32.3 (d), 30.3 (t), 25.7 (q), 25.4 (t), 19.4 (q), 17.6 (q); MS (EI) m/z 290 (M^+ , 0.8), 272 (49.4), 245 (22.2), 133 (22.3); 110 (77.5), 105 (100), 69 (64.3); HRMS (EI) m/z required for $C_{18}H_{26}O_3$ 290.1882, found 290.1872.

3-(p-Bromophenyl)-2-hydroxy-2-phenylpropanoic acid (6e). M.p. 210-212 °C (from CH_2Cl_2); 1H -NMR (DMSO- d_6) δ 7.62 (2H, d, $J = 7.2$ Hz), 7.40-7.25 (5H, m), 7.16 (2H, d, $J = 7.2$ Hz) 3.44 (1H, d, $J = 13.6$ Hz), 3.19 (1H, d, $J = 13.6$ Hz); ^{13}C -NMR (DMSO- d_6) δ 175.0 (s), 142.4 (s), 136.2 (s), 132.7 (d), 130.1 (d), 127.7 (d), 127.1 (d), 125.6 (d), 119.4 (s), 77.8 (s), 44.2 (t); MS (EI) m/z 304/302 (M^+ - H_2O , 4.0/3.8), 178 (9.3), 171 (12.3), 169 (13.0), 149 (16.5), 105 (100), 77 (28.8); HRMS (EI) m/z required for $C_{15}H_{11}BrO_2$ 303.9922 / 301.9942, found 303.9919 / 301.9937.

General procedure for catalytic aerobic decarboxylation of α -hydroxyacids **6**

Co (III)- Me_2opba complex (3.3 mg, 7.7×10^{-3} mmol) and pivalaldehyde (46 μ L, 0.39 mmol) were added to a stirred solution of alkylated α -hydroxyacids **6** (0.13 mmol) in acetonitrile (0.5 mL) under a dioxygen atmosphere. The mixture was stirred at the indicated temperature until consumption of the starting α -hydroxyacid, as indicated by TLC. The reaction products **7** were purified by flash chromatography. For characterisation of compounds **7a**, **7b**, **7d** and **7f-7h** see ref [1].

3,7-Dimethyl-6-octenyl phenyl ketone (7c). An oil; 1H -NMR δ 7.94 (2H, dd, $J = 8.1$ and 1.2 Hz), 7.53 (1H, tt, $J = 8.1$ and 1.2 Hz) 7.43 (2H, td, $J = 8.1$ and 1.2 Hz), 5.08 (1H, m), 2.94 (2H, m), 1.98 (2H, m), 1.75 (2H, m), 1.66 (3H, s), 1.58 (3H, s), 1.50 (2H, m), 1.35 (1H, m), 0.92 (3H, d, $J = 6.2$ Hz); ^{13}C -NMR δ 200.8 (s), 137.0 (s), 132.8 (d), 131.2 (s), 128.5 (d), 128.0 (d), 124.7 (d), 36.9 (t), 36.3 (t),

32.2 (d), 31.3 (t), 25.7 (q), 25.5 (t), 19.4 (q), 17.6 (q); MS (EI) m/z 244 (M^+ , 58.8), 201 (6.9), 173 (18.5), 133 (78.9); 122 (77.5), 120 (71.4); 105 (100), 77 (46.6); HRMS (EI) m/z required for $C_{17}H_{24}O$ 244.1827, found 244.1829.

p-Bromobenzyl phenyl ketone (7e). M.p. 136-138 °C (from CH_2Cl_2); 1H -NMR δ 8.00 (2H, dd, $J = 8.1$, 1.2 Hz), 7.65-7.40 (5H, m), 7.15 (2H, d, $J = 8.4$ Hz), 4.24 (2H, s); ^{13}C -NMR δ 197.2 (s), 136.6 (s), 135.3 (s), 133.6 (d), 132.0 (d), 131.5 (d), 129.0 (d), 128.8 (d), 121.2 (s), 45.0 (t); MS (EI) m/z 276/274 (M^+ , 9.3/9.8), 185/183 (15.8/15.0), 171 (9.7), 165 (5.0), 157 (5.8), 105 (73.8), 90 (22.4), 77 (100); HRMS (EI) m/z required for $C_{14}H_{11}OBr$ 275.9973 / 273.9993, found 275.9963 / 273.9982.

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Samples Availability: Available from the authors.