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Indirect Electrochemical Oxidation of Aryl Alkyl Ketones Mediated by NaI–NaOH System: Facile and Effective Way to α-Hydroxyketals

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Abstract—Indirect electrochemical oxidation of aryl alkyl ketones in methanol in an undivided cell in the presence of sodium iodide–sodium hydroxide leads to the corresponding α-hydroxyketals in 75–85% substance yield (70–75% current yield).

The oxidation of ketones is a method for preparing carboxylic acids and their derivatives, bifunctional compounds such as α-hydroxyketones, diketones and other useful intermediates in organic synthesis.[1] The formation of adipic acid from cyclohexanone is an important industrial process. α-Hydroxyketones are significant 'building blocks' in the construction of natural products and fine chemicals.[2,3] In the case of aryl alkyl ketones, the corresponding α-hydroxyketones and α-hydroxyketals are convenient compounds for synthesis of the pharmacologically active 2-arylalkanoic acids.[4,5]

The advance of electrooxidation in recent years has provided organic chemists with a new versatile synthetic device of great promise.[6] Despite the long history of the electroorganic chemistry, most of the electroorganic reactions which could provide product-selectivity have been developed only within last twenty years, and research on various applications has spread gradually to cover many areas of fundamental and industrial organic chemistry.

However, in the case of the electrochemical oxidation of ketones, only rare examples of the procedures which could ensure product-selectivity are known.

The direct electrochemical oxidation of ketones led to the formation of a mixture of acids, saturated and unsaturated hydrocarbons, carbon monoxide and dioxide.[7–10] Remote non selective oxidative functionalization of aliphatic ketones was observed when electrooxidation was carried out in acetonitrile or trifluoroacetic acid as a result of the subsequent transformation of the initially produced cation radical $R^1R^2C{=}O^{+\cdot}$.[11,12]

In some oxidative transformations of ketones, such as in the haloform reaction, the α-halogenation of ketones is an important step.[13] So for certain cases, the selective indirect electrooxidation of ketones with the electrochemically generated halides is also possible. Thus, the electrocatalytic variant of the haloform reaction—the procedure to prepare carboxylic acid esters by the electrooxidation of methyl alkyl and methyl aryl ketones in methanol in the presence of alkali metal bromides is well known.[14] Aryl alkyl ketones electrolysed in methyl orthoformate in the presence of iodides give rise to methyl 2-arylalkanoates.[15]

The mediatory system NaI–NaOH for the effective indirect electrochemical oxidation of carbonyl compounds is also known. It was employed for the indirect electrochemical oxidation of aldehydes[16] and cyclic ketones.[16,17]

Continuing our studies on the electrooxidation of ketones,[18–20] we have recently accomplished the indirect electrochemical oxidation of aliphatic ketones **1a–e** into unsaturated conjugated esters **2a–f** in methanol in an undivided cell in the presence of the NaI–NaOH system[21] (Scheme 1).

Now we wish to report our results on the indirect electrochemical oxidation of aryl alkyl ketones **3a–f** into α-hydroxyketals **4a–f** in methanol in an undivided cell in the presence of the mediatory system NaI–NaOH (Table 1) (Scheme 2).

Keywords: electrochemical reactions; aryl alkyl ketones; α-hydroxyketals.
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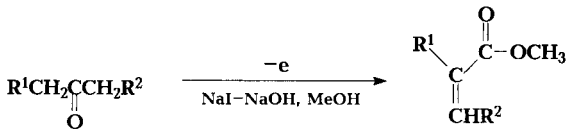


1a–e

2a–f

a $R^1 = R^2 = Et$
b $R^1 = R^2 = n\text{-}Pr$
c $R^1 = R^2 = (CH_2)_4CH_3$
d $R^1 = R^2 = (CH_2)_6CH_3$
e $R^1 = R^2 = CH(CH_3)CH_2CH_3$

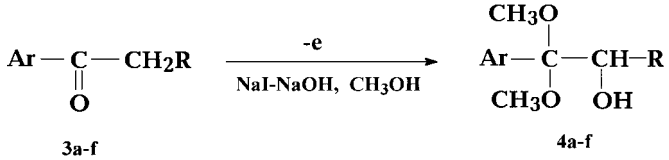
a $R^1 = R^2 = Et$
b $R^1 = R^2 = n\text{-}Pr$
c $R^1 = R_2 = (CH_2)_4CH_3$
d $R^1 = R_2 = (CH_2)_6CH_3$
e $R^1 = Me$, $R^2 = CH(CH_3)CH_2CH_3$
f $R^1 = CH(CH_3)CH_2CH_3$, $R^2 = Me$

Scheme 1.

Table 1. Electrooxidation of aryl alkyl ketones **3a–f** (8 mmol of ketone, 10 mmol of NaI, 1 mmol of NaOH in 20 ml MeOH, Fe-cathode, C-anode, undivided cell, 30°C; conversion of **3a–f** 98–100%)

N	Ketone	Current density mA/cm^2	Electricity passed, F/mol	Product, yield [%][a]	Current yield [%]
1	**3a**[b]	100	2.3	**4a**, 52	45
2	**3a**	100	2.3	**4a**, 64	56
3	**3a**	200	2.3	**4a**, 82 (75)	71
4	**3a**[b]	200	2.3	**4a**, 68	59
5	**3a**[c]	200	2.3	**4a**, 80 (72)	70
6	**3a**	300	2.3	**4a**, 78	68
7	**3a**	400	2.3	**4a**, 65	57
8	**3a**[d]	200	3.5	**4a**, 79	45
9	**3a**[e]	200	4.5	**4a**, 83	37
10	**3b**	200	2.3	**4b**, 86 (77)	75
11	**3c**	200	2.3	**4c**, 75 (70)	65
12	**3d**	200	2.3	**4d**, 84 (76)	73
13	**3e**	200	2.3	**4e**, 78 (72)	68
14	**3f**	200	2.3	**4f**, 85 (74)	74

[a] Determined by gas chromatography and NMR spectra, isolated yields are given in parentheses.
[b] 16 mmol of **3a**.
[c] 60°C.
[d] Without addition of NaOH.



3a-f

4a-f

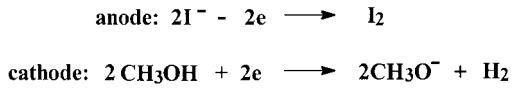
a Ar = Ph, R = CH_3
b Ar = Ph, R = $(CH_2)_2CH_3$
c Ar = Ph, R = $CH(CH_3)_2$
d Ar = Ph, R = $(CH_2)_4CH_3$
e Ar = 4-$CH_3C_6H_4$, R = CH_3
f Ar = 6-$MeOC_{10}H_6$, R = CH_3

Scheme 2.

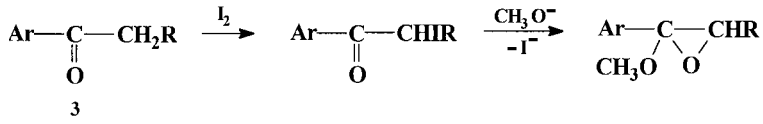
Results and Discussion

The reactions on electrodes, which take place during the process are usual for the mediatory system NaI–NaOH in methanol and lead to the formation of iodine at anode and methoxide anions at cathode (Scheme 3).

The formation of iodine at the anode is observed by the corresponding colour when electrolysis is conducted without stirring of the reaction mixture, as well as evolution of hydrogen at the cathode.



Scheme 3.



Scheme 4.

Then α-monoiodination of the enol form of ketone takes place in the solution in the same manner as it was previously established for the cases of cyclic[17] and aliphatic ketones.[21] α-Iodoketone thus formed undergoes CH_3O^- anion attack on carbonyl group with further cyclization (Scheme 4).

The subsequent interaction of cyclic intermediate with the second CH_3O^- anion results in the formation of the end product of the process—the corresponding α-hydroxyketal (Scheme 5).

The analogous mechanism of the indirect electrooxidation was previously demonstrated for the electrolytic transformation of cyclohexanone into 2,2-dimethoxycyclohexanol.[17] The same mechanism is also known for the reaction of α-halogenoketones with alkoxide anions in usual organic chemistry.[22]

The electrooxidation of **3a** when its concentration is 0.8 M (16 mmol of ketone in 20 ml of MeOH) and at the current density 100 mA/cm^2 gave rise to **4a** (52%) along with 3,4-diphenyl-3,4-hexanediol **5a** in 45% yield (exp. 1, Table 1). The formation of **5a** is a result of concurrent cathodic dimerization of **3a**[23] under the conditions studied (Scheme 6).

The two-fold decrease in **3a** concentration to 0.4 M (8 mmol of ketone in 20 ml of MeOH) and an increase in the current density up to 200 mA/cm^2 depress cathodic dimerization of **3a** to a sufficient degree and the main cathodic process becomes the formation of CH_3O^- anions. Under the last conditions, **4a** was formed in 82% yield and **5a** only in 13% yield (exp. 3, Table 1). Further increasing the current density gives rise to a decrease in the selectivity of **4a** formation because of the pathways of direct electrochemical oxidation[7–10] and electrochemically induced oligomerisation[24] of ketones, which take place under these conditions.

Under the optimal conditions of **3a** oxidation into **4a**, other aryl alkyl ketones **3b–e** were converted into **4b–e** in 75–86% substance yield (65–75% current yield).

Using NaI without addition of NaOH decreases the current yield of α-hydroxyketals from 70% to 45%. The addition of NaOH has two effects. First, it increases the concentration of CH_3O^- anions in solution and thus accelerates all processes with participation of CH_3O^- anions. Second, it increases the quantity of enol form in the starting ketone and thus facilitates the reaction of iodination of ketone. So the addition of NaOH increases the general current efficiency of the indirect electrooxidation of ketone in the undivided cell.

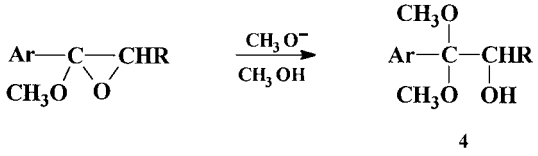
The system NaBr–NaOH is less effective for the indirect electrochemical oxidation of aryl alkyl ketones compared to the system NaI–NaOH. In the absence of NaOH and in the presence of only NaBr in the analogous process, **4a** was obtained in 78% substance and 20% current yield. When the mediatory system NaBr–NaOH was used, **4a** was formed in 83% substance and 37% current yield (exp. 9, Table 1). Nevertheless, it should be also mentioned that electrooxidation of **3a** under the conditions of exp. 1 (Table 1) using the NaBr–NaOH system after passing 4.5 F/mol of electricity gave rise to **4a** in 73% yield (32% current yield). This result might indicate that with the system NaBr–NaOH (even at the current density of 100 mA/cm^2) the potential of the anode is sufficient for the direct oxidation of **5a** into **3a** in accordance with the well-known reactions of the electrochemical cleavage of 1,2-diols.[25,26] To confirm this proposal the electrochemical oxidation of **5a** in the presence of NaBr–NaOH system was performed and **4a** was obtained in 62% yield (5 F/mol). On the other hand, **5a** is stable at the current density of 200 mA/cm^2 in the presence of NaI–NaOH system.

The temperature of the process has no sufficient influence on the electrooxidation **3a** into **4a** in the temperature interval of 30–60°C.

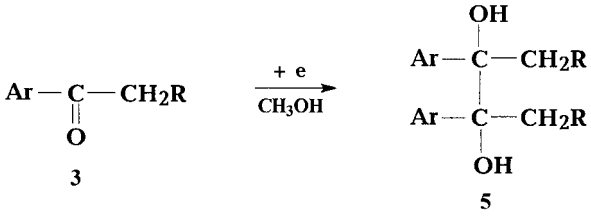
Benzyl ethyl ketone **6**, under the conditions studied, was transformed into methyl 2-methyl-3-phenylpropionoate **7** in 88% yield. The more acidic benzylic proton in **6** changes the reaction route and the Favorskii rearrangement becomes the main route for the process. In this case, the CH_3O^- anion attacks not the carbonyl group, but eliminates the benzylic proton in the intermediate α-iodoketone followed by cyclization into unstable cyclopropanone **8** (Scheme 7).

The analogous mechanism has already been proposed for the electrochemical oxidation of benzyl alkyl ketones in the presence of NaBr.[27]

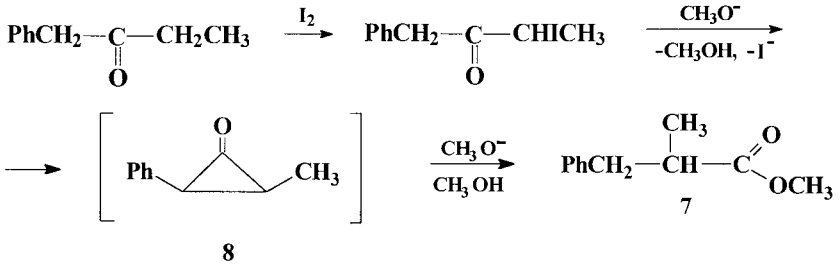
One more interesting fact is that dialkyl ketones **1** were transformed, under the same conditions, into unsaturated



Scheme 5.



Scheme 6.



Scheme 7.

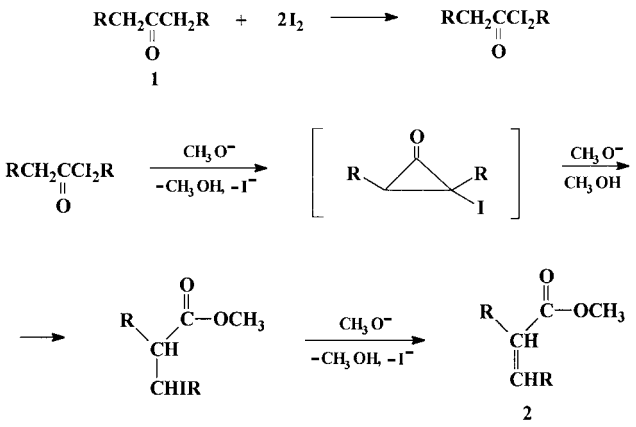
conjugated esters **2**.[21] In this case, the Favorskii rearrangement also takes place, but the Favorskii rearrangement of the double iodinated ketone (Scheme 8).

Thus, in the case of dialkyl ketones, the attack of CH_3O^- anion on the carbonyl group have sufficient sterical difficulties as compared with aryl alkyl ketones having one flat substituent.

Another explanation of the different reaction ability of dialkyl and aryl alkyl ketones, under the conditions studied, could be a higher reactivity of carbonyl group in aryl alkyl ketones owing to the conjugation with aryl-group.

In conclusion it should be mentioned that NaI–NaOH electrocatalytic system can produce under mild conditions direct 'one-pot' effective transformation of aryl alkyl ketones **3a**–**e** into corresponding α-hydroxyketals **4a**–**e** in high substance and current yields. This process is also a facile and economical method for the transformation of aryl alkyl ketones into α-hydroxyketals—convenient intermediates for the synthesis of the pharmacologically active 2-arylpropanoic acids.[4,5] The procedure utilises inexpensive reagents, simple equipment and undivided cell, it is easily carried out, and the work up is not complicated.

Three step or two step methods of α-hydroxyketals **4a,e,f** conversion into 2-arylpropanoic acids include rearrangement with 1,2-migration of the aryl group.[28–31] In this manner, for example, **4f** can be transformed into (2-methoxynaphth-2-yl)propanoic acid (Naproxen) in 80–85% yield.[28,29,31]



Scheme 8.

Experimental

GC analysis was carried out on Hewlett–Packard Model 5890 chromatograph with a flame-ionisation detector. Columns: (1) fused-silica capillary column HP-1 (5 m×0.53 mm×2.65 μm), 2) glass column 3 m×3 mm with 10% FFAP on Chromaton N-Super (0.13–0.16 mm). ^{1}H NMR spectra were run for solutions in $CDCl_3$ and recorded with Varian Unity 200 (200 MHz) spectrometer [^{1}H NMR spectrum of **7** on Varian Unity 500-PLUS (500 MHz) spectrometer]. Chemical shifts are presented in δ scale with tetramethylsilane (TMS) used as an internal standard. Mass-spectra (70 eV) were recorded with chromatographic injection using Hewlett–Packard Model 5988A spectrometer.

3,4-Diphenyl-3,4-hexanediol **5a** was obtained by the known method.[32]

General electrolysis procedure

A solution of ketone (8 mmol), NaI (10 mmol) and NaOH (1 mmol) in methanol (20 ml) was electrolysed in an undivided cell equipped with C-anode and Fe-cathode at 30°C under constant current density indicated in Table 1 until the quantity of the electricity indicated in Table 1 was passed. The reaction mixture was neutralised by dilute HCl, the solvent was then removed, and the reaction mixture was extracted with ether, washed with a solution of $Na_2S_2O_3$ in water, then with water, and dried with Na_2SO_4. After distillation, compounds **4a**–**f** were isolated.

1,1-Dimethoxy-1-phenyl-2-propanol (4a).[33,34] Bp 75–78°C (0.08 Torr), [lit.[34] bp 94–95° (0.5 Torr)]; ^{1}H NMR ($CDCl_3$): δ 0.93 (d, 3H, CH_3, J=6.5 Hz), 2.59 (d, 1H, OH, J=4.3 Hz), 3.18 (s, 3H, OCH_3) 3.33 (s, 3H, OCH_3), 4.10 (m, 1H, CH), 7.15–7.58 (m, 5H, C_6H_5).

1,1-Dimethoxy-1-phenyl-2-pentanol (4b).[35] Bp 93–95°C (0.08 Torr), [lit.[35] bp 80–82° (0.05 Torr)]; ^{1}H NMR ($CDCl_3$): δ 0.83 (t, 3H, CH_3, J=7.4 Hz), 1.20–1.58 (m, 4H, CH_2), 2.53 (d, 1H, OH, J=4.4 Hz), 3.21 (s, 3H, OCH_3) 3.33 (s, 3H, OCH_3), 3.94 (m, 1H, CH), 7.16–7.60 (m, 5H, C_6H_5).

1,1-Dimethoxy-3-methyl-1-phenyl-2-butanol (4c).[35] Bp 80–82°C (0.06 Torr), [lit.[35] bp 98–100° (0.3 Torr)]; ^{1}H NMR ($CDCl_3$): δ 0.69 (d, 3H, CH_3, J=6.9 Hz), 0.88 (d, 3H, CH_3, J=6.9 Hz), 1.45 (m, 1H, CH), 2.63 (d, 1H, OH, J=4.2 Hz), 3.22 (s, 3H, OCH_3) 3.28 (s, 3H, OCH_3), 3.72 (m, 1H, CH), 7.16–7.60 (m, 5H, C_6H_5).

1,1-Dimethoxy-1-phenyl-2-hexanol (4d). Bp 90–93°C (0.05 torr), ^{1}H NMR ($CDCl_3$): δ 0.81 (t, 3H, CH_3, J=6.6 Hz), 1.12–1.50 (m, 6H, CH_2), 2.28 (d, 1H, OH, J=4.3 Hz), 3.21 (s, 3H, OCH_3) 3.33 (s, 3H, OCH_3), 3.89 (m, 1H, CH), 7.15–7.58 (m, 5H, C_6H_5); ^{13}C NMR ($CDCl_3$): δ 13.98 (q), 22.60 (t), 28.50 (t), 30.49 (t), 49.26 (q), 49.86 (q), 74.52 (d), 103.39 (s), 127.68 (d), 127.76 (d), 127.88 (d), 137.56 (s); MS (70 eV): *m/z* (relative intensity): 207 ($M-OCH_3^+$, 2), 152 (8), 150 (100), 121 (14), 105 (33), 91 (12), 77 (21), 59 (4). Anal. Calcd for $C_{14}H_{22}O_3$: C, 70.56; H, 9.31. Found: C, 70.82; H, 9.39.

1,1-Dimethoxy-1-(4-methylphenyl)-2-propanol (4e). Bp 72–75°C (0.08 Torr), ^{1}H NMR ($CDCl_3$): δ 0.96 (d, 3H, CH_3, J=6.5 Hz), 2.34 (s, 3H, CH_3), 2.38 (d, 1H, OH, J=4.1 Hz), 3.21 (s, 3H, OCH_3) 3.34 (s, 3H, OCH_3), 4.07 (m, 1H, CH), 7.07 (d, 2H, Ar, J=7.5 Hz), 7.25 (d, 2H, Ar, J=7.5 Hz); ^{13}C NMR ($CDCl_3$): δ 16.46 (q), 21.05 (q), 49.26 (q), 49.93 (q), 70.73 (d), 103.59 (s), 127.81 (d), 128.40 (d), 134.26 (s), 137.65 (s); MS (70 eV): *m/z* (relative intensity): 179 ($M-OCH_3^+$, 8), 165 (100), 150 (24), 149 (78), 119 (79), 105 (15), 91 (75), 77 (14), 57 (84). Anal. Calcd for $C_{12}H_{18}O_3$: C, 68.55; H, 8.63. Found: C, 68.73; H, 8.87.

1,1-Dimethoxy-1-(6-methoxynaphtyl-2)-2-propanol (4f). Bp 102–105°C (0.05 Torr), viscous oil, ^{1}H NMR ($CDCl_3$): δ 1.11 (d, 3H, CH_3, J=6.5 Hz), 2.64 (d, 1H, OH, J=4.3 Hz), 3.37 (s, 3H, OCH_3) 3.53 (s, 3H, OCH_3), 4.01 (s, 3H, OCH_3), 4.30 (m, 1H, CH), 7.20–8.00 (m, 6H, Ar); ^{13}C NMR ($CDCl_3$): δ 16.53 (q), 49.35 (q), 50.01 (q), 55.20 (q), 70.81 (d), 103.60 (s), 105.28 (d), 118.71 (d), 125.97 (d), 126.03 (d), 127.40 (d), 128.56 (s), 129.85 (d), 132.62 (s), 134.12 (s), 157.87 (s); MS (70 eV): *m/z* (relative intensity): 276 (M^+, 4), 245 ($M-OCH_3^+$, 3), 231 (13), 216 (4), 185 (100), 157 (38), 142 (25), 114 (31), 89 (41), 45 (28). Anal. Calcd for $C_{16}H_{20}O_4$: C, 69.55; H, 7.30. Found: C, 69.73; H, 7.41.

Methyl 2-methyl-3-phenylpropionate (7).[36,37] Bp 64–66° (0.08 Torr), [lit.[37] bp 90–93° (1 Torr)]; ^{1}H NMR ($CDCl_3$): 1.15 (d, 3H, CH_3, J=7.0 Hz), 2.66 (d,d 1H, CH, J_1=8.0 Hz, J_2=13.5 Hz), 2.88 (m, 1H, CH) 3.25 (d,d 1H, CH, J_1=7.0 Hz, J_2=13.5 Hz), 3.63 (s, 3H, OCH_3), 7.15–7.28 (m, 5H, Ar).

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