2426 *Russian Chemical Bulletin, International Edition, Vol. 58, No. 12, pp. 2426—2431, December, 2009*

Electrosynthesis of glutaric acid and regularities of electrocatalytic oxidation of cycloalkanones at a NiOOH anode in aqueous NaOH

B. V. Lyalin and V. A. Petrosyan*

N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences,
47 Leninsky prosp., 119991 Moscow, Russian Federaion.
Fax: (499) 135 5328. E-mail: petros@ioc.ac.ru

Methods of preparation of glutaric acid by oxidation of cyclopentanone or pentane-1,5-diol in an undivided cell at a NiOOH electrode in aqueous alkali are developed. Yields of the products were 51% or 90%, respectively. The mechanism of electrooxidation of cycloalkanones at a NiOOH electrode is discussed on the basis of literature data and the regularities of the oxidative transformations of cyclohexanone investigated earlier and those of cyclopentanone.

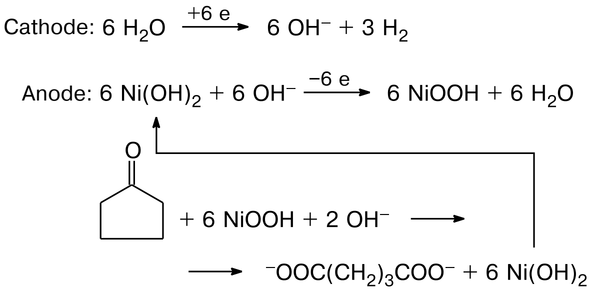
Key words: electrosynthesis, undivided cell, nickel hydroxide electrode, cyclohexanone, cyclohexane- and cyclopentanediones, adipic, glutaric and succinic acids, pentane-1,5-diol.

We have been investigating the electrosynthesis in aqueous alkaline media using a Ni anode and the undivided cell for several years.[1—3] On the surface of a Ni anode, NiOOH is formed during electrolysis under these conditions. This is an efficient oxidant, which is self-generated in the electrolysis. In particular, this approach was used for the development of two methods for the preparation of adipic acid[1]: by direct oxidation of cyclohexanol (the yield was 47%) and by the oxidation of cyclohexanol to cyclohexanone (the yield was 75%) followed by oxidation of the latter to adipic acid (the yield was 52%). The same approach was used in the present work for the preparation of glutaric acid, which is used in the synthesis of herbicides[4] and pharmaceuticals.[5] It is of note that cyclopentanol is not promising as the starting material because it is prepared by hydrogenation of cyclopentanone, which is more accessible.[6,7] Therefore, it was cyclopentanone that we chose as an initial subject of investigations. Besides, the oxidation of an available acyclic 1,5-diol was studied as an alternative for the synthesis of glutaric acid. The results of the investigations were used for generalization of the existing literature data on the regularities of the electrooxidation of cycloalkanones.

Results and Discussion

The first experiments on the electrooxidation (EO) of cyclopentanone (CP) at a NiOOH anode in aqueous NaOH (Table 1, entry *1*) resulted in the formation of glutaric acid in the yield of ~44% (based on the consumed ketone). The oxidation process is presented in Scheme 1, it is similar to the scheme of EO of cyclohexanone under the same conditions.[1]

Scheme 1



In the studies of EO of CP it was found that a decrease in the temperature of electrolysis from 25 to 10 °C led to the increase in the yield of glutaric acid by 13% (*cf.* entries *1* and *2*, Table 1). With increased concentration of alkali from 0.8 to 1.0 mol L^{-1}, the yield of the acid also increased, however it decreased again at higher (1.2 mol L^{-1}) concentration of the alkali (*cf.* entries *2*, *3*, and *4*, Table 1).

The increase in the concentration of CP from 0.1 to 0.2 mol L^{-1} is favorable for the increase in the yield of glutaric acid (by 20% based on loaded CP) and the conversion of CP (by 35%) (*cf.* entries *2*, *5*, and *6*, Table 1). The dependence of the yield of glutaric acid on current density passes through a maximum (*cf.* entries *2*, *7*, and *8*, Table 1) reaching 58% (based on consumed cyclopentanone) at $J = 6$ mA cm^{-2}. Thus, the conditions were found ($T = 10$ °C, $C_{CP} = 0.15$—0.2 mol L^{-1}, $C_{NaOH} = 1.0$ mol L^{-1},

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Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 12, pp. 2348—2353, December, 2009.

Table 1. Dependence of the yield (%) of glutaric (GA) and succinic (SA) acids in the electrooxidation of cyclopentanone (CP)[a]

Entry	C_{CP} /mol L^{-1}	J_a /mA cm^{-2}	Q/Q_{theor}	Conversion of CP (%)	Yield[b]		
					GA		SA,
					I	II	I
1[c]	0.15	6	1.0	80	35	44	8
2	0.15	6	1.0	49	28	58	4
3[d]	0.15	6	1.0	34	20	58	6
4[e]	0.15	6	1.0	51	28	54	7
5	0.20	6	1.0	68	38	55	7
6	0.10	6	1.0	33	17	50	3
7	0.15	16	1.0	25	12	47	2
8	0.15	3	1.0	54	18	34	4
9	0.15	6	1.5	66	41	62	8
10	0.15	6	3.0	86	51	59	14

[a] Undivided-cell electrolysis in 1.0 *M* aqueous NaOH, Ni anode, Ti cathode, $T = 10$ °C, $Q_{theor} = 6\,F$ (mol CP)$^{-1}$; J_a is current density, Q is the amount of electricity.

[b] Calculated from ^{1}H NMR data for isolated mixture of electrolysis products: I based on the starting CP; II, based on the consumed CP.

[c] $T = 25$ °C.

[d] $C_{NaOH} = 0.8$ mol L^{-1}.

[e] $C_{NaOH} = 1.2$ mol L^{-1}.

$J = 6$ mA cm^{-2}, $Q = Q_{theor}$) providing the moderate yield (55—58% based on the consumed CP) of glutaric acid.

At the same time, the conversion of CP is relatively low under these conditions (49—68%) (*cf.* entries *2* and *5*, Table 1) and the yield of the target product (based on loaded CP) varies in the range of 28—37%. This shortcoming was partially eliminated by increasing the amount of passed electricity (*cf.* entries *2*, *9*, and *10*, Table 1). Thus the threefold increase (compared to theoretical) in electricity passed (entry *10*, Table 1) increased the conversion of the ketone to 86%, the yield of glutaric acid reaching 51% based on the loaded ketone (or 59% based on the consumed ketone).

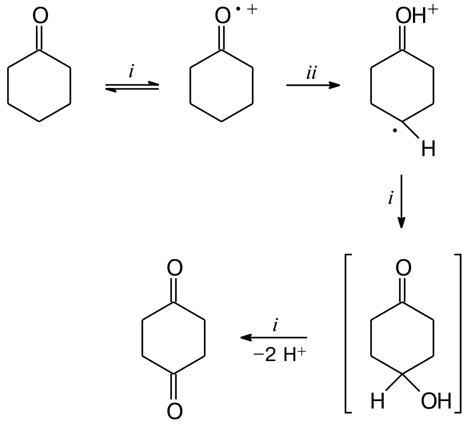
From the comparison of the results obtained with the data of the dependence of the yield of adipic acid on conditions of EO of cyclohexanone[1] it is obvious that the regularities of EO of cyclohexanone and CP to the corresponding acids are similar. At the same time, EO of CP to glutaric acid (the yield of acid is 51% based on the loaded ketone and its conversion of 86%) is more efficient than EO of cyclohexanone to adipic acid (the yield of the acid is 37% based on the loaded ketone and its conversion of 70%). These results can probably be rationalized by the fact that it is enol forms of ketones that are subject to EO,[8] the molar fraction of the enol form being 3 times higher in CP than in cyclohexanone.[9]

In EO of cyclohexanone under the conditions analogous to those used in the present work, not only adipic acid[1] but also small amounts of two other dicarboxylic acids formed: glutaric (up to 10%) and succinic (up to 4%), whose molecules contain one and two carbon atoms less than the molecule of adipic acid, respectively. The same was observed in EO of CP, where the moderate amount of succinic acid (8—14%) was present along with glutaric acid in the electrolysis products.

Let us note that adipic, glutaric, and succinic acids also are formed in chemical oxidation of cyclohexanone (with oxygen in the presence of cobalt naphthenate[10] or upon γ-irradiation,[11] with H_2O_2 and V_2O_5 as a catalyst,[12] with peracetic acid in the presence of Ru/C as a catalyst[13]). This prompted us to analyse and summarize separate data on the oxidation of cyclic ketones.

Thus a mechanism of the oxidation of linear and some cyclic ketones with $Na_2S_2O_8$—$FeSO_4$ was discussed.[14,15] Presumably, in the first step of the oxidation of cyclohexanone as an example, an O-centered radical cation is formed, one of its reaction routes involves 1,5-shift of a hydrogen atom to generate C-centered radical cation, which is oxidized to cyclohexane-1,4-dione (the yield is ~2%).

Scheme 2



Reactants and conditions: *i.* $Na_2S_2O_8$—$FeSO_4$; *ii.* 1,5-Shift.

But if this were the case, the simultaneous 1,3- and 1,4-migration of a hydrogen atom would occur. However, no products of such transformations (cyclohexane-1,2-dione and -1,3-dione, respectively) were found in the reaction mixture containing much of resinous substances.[14,15] Most likely, this was caused by high reactivity of cyclohexane-1,2-dione and -1,3-dione under these conditions. It was confirmed by the data[11] on the γ-radiation-induced oxidation of cyclohexanone with oxygen, which proceeds *via* cyclohexane-1,2-dione, -1,3-dione, and -1,4-dione to form adipic, glutaric, and succinic acids, respectively. It was also noted[11] that due to the high reactivity, the mentioned cyclic diketones could only be found in solutions with pH < 11.5. This, in turn, explains the absence of cyclohexanediones in the resulting reaction mixture in the EO of cyclohexanone or CP at a NiOOH electrode in the alkaline medium.

In view of the aforesaid, the mechanism of EO of cyclohexanone in the alkaline medium at a NiOOH electrode corresponds to Scheme 3, where the starting structure subjected to EO is the enol form of cyclohexanone (see above). It has sufficiently low[9] pK = 11.3 and exists in 1.0 M NaOH solution in the form of the enolate anion. In the first step of the process, O-centered radical **1** is generated, whose further transformations include 1,3-, 1,4-, and 1,5-hydrogen shifts to afford cyclohexane-1,2-dione, -1,3-dione, and -1,4-dione *via* hydroxycyclohexanones **2a**, **2b**, **2c**. Further oxidation of cyclohexanediones **3a**, **3b**, and **3c**, occuring through several respective steps (Scheme 3) results in adipic (**4a**), glutaric (**6b**), and succinic (**7c**) acids. This conclusion is confirmed by the data on EO of cyclohexanediones.

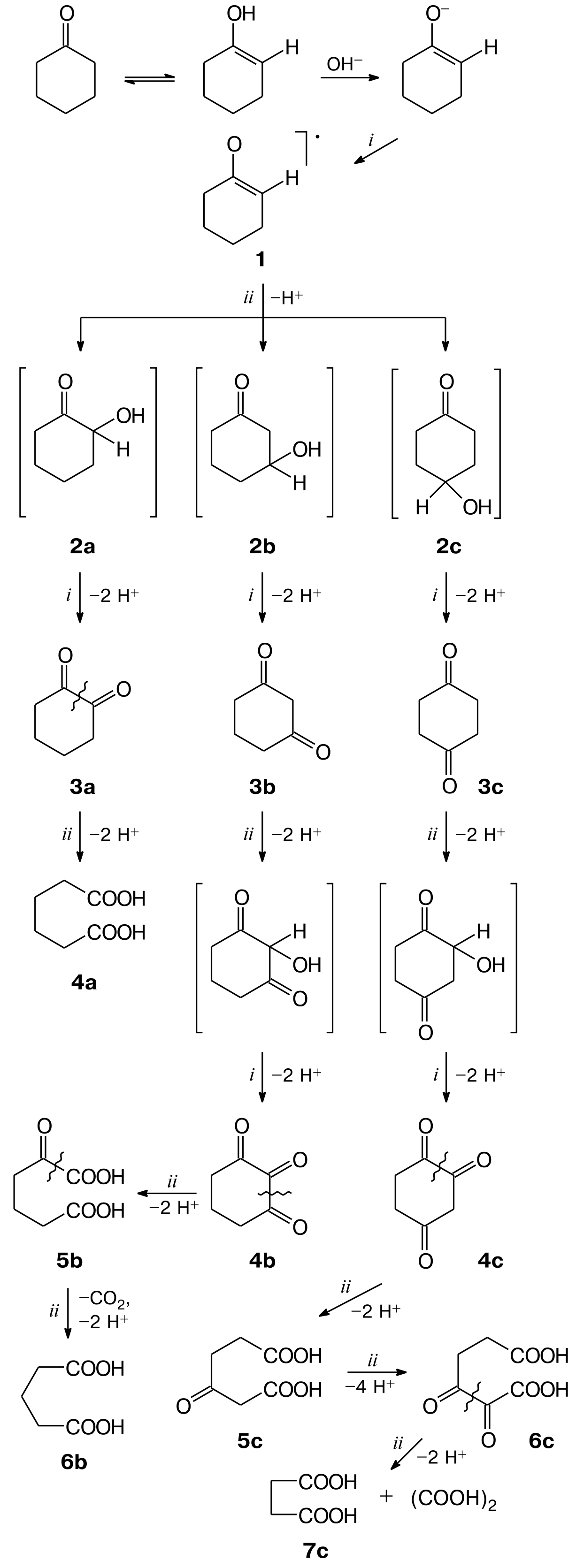
No sample of cyclohexane-1,2-dione (**3a**) labile under ordinary conditions was at our disposal, but it is known[8] that it is diones that are the primary electrooxidation products of the respective cyclic diols. Consequently, the electrosynthesis of adipic acid with a yield of 75% in EO of *cis*- and *trans*-cyclohexane-1,2-diols in aqueous K_2CO_3 occurs[16] *via* cyclohexane-1,2-dione, which in turn causes the appearance of the step **3a** → **4a** in Scheme 3. It is of note that in EO opening of the cyclohexane ring takes place between the carbon atoms of the C=O groups.[8,17]

In a special investigation, we showed that EO of cyclohexane-1,3-dione in aqueous K_2CO_3 at a NiOOH anode results in glutaric acid with the yield of 81%. Besides, it is known[17] that EO of cyclohexane-1,3-dione at a Pt anode in an acidic medium occurs *via* cyclohexane-1,2,3-trione (**4b**), which further oxidizes also to glutaric acid *via* α-ketodicarboxylic acid.

Thus, the mechanism of EO of 1,3-diketone (**3b**) represented in Scheme 3 involves 1,3-shift of a hydrogen atom and results in triketone (**4b**). The further oxidation of **4b** occurs with ring opening to α-ketodicarboxylic acid (**5b**), which in turn oxidizes to glutaric acid (**6b**) with elimination of CO_2.

Obviously, EO of cyclohexane-1,4-dione (**3c**) occurs by the mechanism analogous to that of EO of cyclohexane-1,3-dione (**3b**). This allows considering cyclohexane-1,2,4-trione (**4c**) and β-ketodicarboxylic acid (**5c**) as most likely intermediates of the oxidation process (Scheme 3). The oxidation of keto acid **5c**, apparently also occurs through 1,3-shift of a hydrogen atom, and compound **6c** that formed oxidizes with cleavage of the C—C bond between the carbon atoms of the carbonyl groups (see above) resulting in succinic and oxalic acids. This can indirectly proved by the data on EO of acetylacetone[17] to carboxylic acid and by the fact[10] that the oxidation of cyclohexane-1,4-dione with HNO_3 results in succinic (**7c**) and oxalic acids.

Scheme 3



Reactants and conditions: *i.* [NiOOH]; *ii.* [NiOOH], H_2O.

The resulting products (Scheme 3) of EO of cyclohexanone, *i.e.*, dicarboxylic acids, are present in the reaction mixture as dianions, because the electrolysis was carried out in the alkaline medium.

The analysis of the mechanism of the process suggested in Scheme 3 explains the ratio of adipic, glutaric, and succinic acids in the EO products of cyclohexanone. Thus the probability of hydrogen migration in the cyclohexanone-derived O-centered radical (**1a**) to the position 3 is much higher than that to the position 4, which is higher than that to the position 5. Therefore the concentration of intermediate cyclohexane-1,2-dione is much higher than those of cyclohexane-1,3-dione and -1,4-dione. As a result, the yield of adipic acid in the EO of cyclohexanone is noticeably higher[1] than those of glutaric or succinic acids.

These results allow describing the mechanism of the EO of CP at a nickel hydroxide electrode affording a mixture of glutaric and succinic acids by Scheme 4 analogous to Scheme 3 considered above.

If the intermediate cyclopentanone-derived O-centered radical (**8**, Scheme 4) undergoes 1,3-hydrogen shift, then its oxidative transformation product occurring *via* diketone (**9a**) should be glutaric acid (**6b**). In the case of 1,4-shift, cyclopentane-1,3-dione (**9b**) and cyclopentane-1,2,3-trione (**10**) are transiently formed, which results in succinic acid (**7c**). Since the probability of 1,3-hydrogen shift in the EO of CP is higher than that of 1,4-shift (see above), the yield of glutaric acid more than fourfold exceeds that of succinic acid (see Table 1).

The EO mechanism in Scheme 4 is in accord with the data on the chemical ($Na_2S_2O_8$—$FeSO_4$) oxidation of CP to cyclopentane-1,3-dione[14] and on the oxidation of the latter to succinic acid at a Pt anode in an acidic medium.[17]

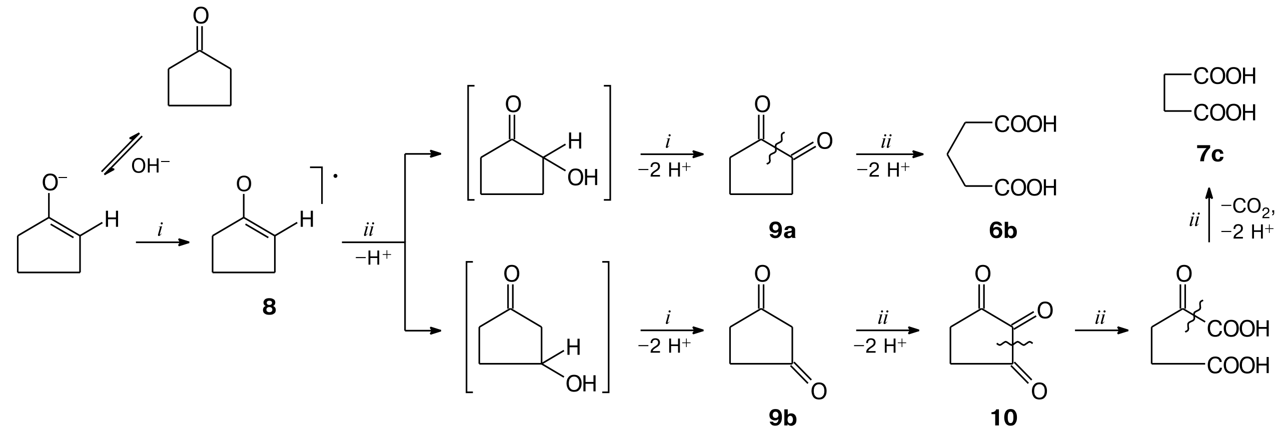
In the EO of CP, only 1,3- or 1,4-shifts of a hydrogen atom are possible, therefore only one by-product, *viz.*, succinic acid, is present in the reaction mixture together with glutaric acid as the major product (*cf.* with EO of cyclohexanone).

It was of interest to study the electrosynthesis of dicarboxylic acids in the EO (under the conditions analogous to those described above) of cyclic ketones with much larger number of carbon atoms in the ring. We chose cyclododecanone as an example the chemical (HNO_3) oxidation of which results in dodecanedioic acid.[10]

Cyclododecanone turned out to be poorly soluble in an aqueous alkaline solution. Therefore, its EO was carried out in an alkaline aqueous-alcoholic (50% Bu^tOH) solution. However, the yield of dodecanedioic acid in the electrolysis of cyclododecanone under these conditions was low (5—10%), and variation of different factors (current density, concentrations of ketone and alkali, temperature of the process, the amount of electricity passed) had not substantial effect on the yield of the target product. Thus, the extension of the ring size decreases the effectivity of the EO of cycloalkanones at a nickel hydroxide electrode.

We have also studied the preparation of glutaric acid by EO of available acyclic alcohol, pentane-1,5-diol, at a NiOOH anode in aqueous NaOH. It resulted in glutaric acid as the major product, but the formation of trace amounts (1—3%) of succinic acid was also observed. It was established that the yield of glutaric acid slightly (by ~5%) reduced (*cf.* entries *1* and *2* in Table 2) with the increase in temperature from 25 to 50 °C (which apparently was caused by the oxidation of glutaric acid itself), but still remained high (87—91%). The decrease in the yield of the target product by 4% (*cf.* entries *1* and *3*, Table 2) was caused by the increase in the alkali concentration in the range of 0.5—1.0 mol L^{-1} and by 13% (*cf.* entries *1* and *4*, Table 2) by the increase in pentane-1,5-diol concentration (from 0.14 to 0.22 mol L^{-1}). The dependence of the yield of glutaric acid on the current

Scheme 4



Reactants and conditions: *i.* [NiOOH]; *ii.* [NiOOH], H_2O.

Table 2. Effect of the experimental conditions on the yield of glutaric (GA) and succinic (SA) acids in the electrolysis of pentane-1,5-diol (PD) under the conditions of the undivided-cell electrolysis in aqueous NaOH solution*

Entry	C/mol L^{-1}		T/°C	J_a/mA cm^{-2}	Yield (%)	
	PD	NaOH			GA	SA
1	0.14	1.0	20	6	91	2
2	0.14	1.0	50	6	87	3
3	0.14	0.5	20	6	96	2
4	0.22	1.0	20	6	78	2
5	0.14	1.0	20	16	71	2
6	0.14	1.0	20	3	81	1

* Conditions: NiOOH anode, Ti cathode, $Q_{\text{theor}} = 8\ F(\text{mol PD})^{-1}$.

density passes through a maximum (*cf.* entries *1*, *5*, and *6*, Table 2), reaching 91% at $J_a = 6$ mA cm^{-2}.

It is known that the primary hydroxyl groups, unlike the secondary ones, are readily oxidized to the carboxyl groups,[8] easily overpassing the step of transient formation of an aldehyde. Therefore, the formation of glutaric acid as the major EO product of pentane-1,5-diol seems to be quite appropriate. The formation of trace amounts of succinic acid appears to be caused by the partial oxidation of glutaric acid itself.

This process probably occurs also through 1,3-migration of hydrogen to form a C=O group in α-position to the COOH group. The subsequent cleavage of the C—C bond with elimination of CO_2 results in succinic acid. The oxidation of aliphatic acids at a NiOOH electrode in an alkaline medium with the formation of lower homologs of acids has earlier been described in literature.[8]

Thus, we suggest two alternative and convenient methods for electrochemical preparation of glutaric acid: the oxidation of CP (the yield of the acid is 59%) and pentane-1,5-diol (the yield of the acid >90%). We discussed the regularities of EO of cyclic ketones (cyclohexanone and CP) that allow explaining the formation of the by-products, *i.e.*, dicarboxylic acids with one or two carbon atoms less in the chain, along with the target products (adipic and glutaric acids).

Experimental

The electrolysis was carried out in a galvanostatic mode using the direct current source B5-8 in an undivided jacketed cell with a Ni anode (S = 124 cm^2) and a Ti cathode (S = 40 cm^2). The coulometer designed at the SCB IOC was connected to the electric circuit. During the electrolysis, the reaction mixture was stirred with a magnetic stirrer and a thermostat U-1 was used to maintain constant temperature. Before the experiment, the Ni anode was activated according to the procedure described earlier:[18] preliminary electrolysis was carried out in the solution containing 0.1 M $NiSO_4$, 0.1 M NaOAc, and 0.005 M NaOH, at J_a = 1 mA cm^{-2} with periodical reverse of the electrodes polarization. This procedure is necessary for the formation of multi-layer coating containing NiOOH on the Ni anode surface.

Cyclopentanone, cyclohexane-1,3-dione, and pentane-1,5-diol (Acros) were used without additional purification. In order to determine the degree of conversion of cyclopentanone, it was converted to 2,4-dinitrophenylhydrazone according to the known procedure.[19] The obtained acids, glutaric and succinic, were identified by ^{1}H NMR spectroscopy (DMSO-d$_6$) on a Bruker AC-300 spectrometer by the comparison with the spectra of the authentic samples (Acros).

Electrooxidation of CP to glutaric acid (entry *10*, Table 1). A 1 M NaOH solution (200 mL) and CP (2.6 mL, 0.03 mol) were placed in the cell. The electrolysis was carried out at 10 °C and a current of 0.744 A. After passage of 18 F (Q = 52110 C, threefold amount compared to theoretical) per mol of CP, the electrolysis was stopped. The reaction mixture was stirred for 0.5 h and neutralized with concentrated HCl to pH 6—7. An aliquot of this solution was withdrawn and the degree of conversion of CP was determined (see above). The degree of conversion was 86%. Concentrated HCl was added to the remaining reaction mixture (to pH 1—2), then water was distilled off under reduced pressure. The solid residue was treated with Me_2CO (4×25 mL) and the extract was concentrated to dryness to yield 2.88 g of a powder that was shown to be a mixture of glutaric and succinic acids, (^{1}H NMR spectroscopy). Their molar ratio equal to 3.75 : 1.0 was determined by the signal integrated intensities of glutaric (δ 1.70—1.88 (m, 2 H, CH_2)) and succinic (δ 2.40 (m, 4 H, 2 CH_2)) acids. The yields of these acids based on the loaded CP were 51 and 14%, respectively. In order to determine the melting point of the glutaric acid the powder (2.88 g, see above) was treated with hot C_6H_6 (5×10 mL) and after cooling of the extract, the precipitate of glutaric acid was filtered off. M.p. was 97—98 °C (m.p. 99 °C).[20]

Electrooxidation of cyclohexane-1,3-dione to glutaric acid. A 0.1 M K_2CO_3 solution (200 mL) and cyclohexane-1,3-dione (0.56 g, 0.005 mol) were placed in the cell. The electrolysis was carried out at 25 °C and a current of 0.744 A. After passage of 8 F (Q = 3860 C) per mol of cyclohexane-1,3-dione, the electrolysis was stopped. The reaction mixture was stirred for 0.5 h and neutralized with concentrated HCl (to pH 1—2), then water was distilled off under reduced pressure. After workup of the reaction mixture as described above, we isolated 0.71 g of a solid that was shown to be a mixture of glutaric and succinic acids (^{1}H NMR spectroscopy).The molar ratio of glutaric and succinic acids equal to 17.1 : 1.0 was determined by the signal integrated intensities. The yields of glutaric and succinic acids based on the loaded CP were 81 and 5%, respectively.

Electrooxidation of pentane-1,5-diol to glutaric acid (entry *1*, Table 2). A 1 M NaOH solution (200 mL) and pentane-1,5-diol (2.9 mL, 0.028 mol) were placed in the cell. The electrolysis was carried out at 20 °C and a current of 0.744 A. After passage of 8 F (Q = 21616 C) per mol of the diol, the electrolysis was stopped. Workup of the reaction mixture (as described above) afforded 3.36 g of the product, glutaric acid, with admixture (1.75%) of succinic acid (identified by ^{1}H NMR spectroscopy). The yield of glutaric acid was 91% (based on the loaded pentane-1,5-diol). In order to determine the melting point of glutaric acid, the powder (3.36 g, see above) was recrystallized from C_6H_6. M.p. 98—99 °C (m.p. 99 °C).[20]

Electrooxidation of cyclododecanone to dodecanedioic acid. A 0.2 M KOH solution (200 mL) in 50% aqueous Bu^{t}OH and cyclododecanone (3.64 g, 0.02 mol) were placed in the cell. The electrolysis was carried out at 20 °C and a current of 0.298 A (J_a = 2.4 mA cm^{-2}). After passage of 6 F (Q = 11580 C) per mole of cyclododecanone, the electrolysis was stopped. The reaction mixture was stirred for 0.5 h. Then Bu^{t}OH was salted out by the addition of solid NaCl. The aqueous and organic layers were separated. Bu^{t}OH was distilled off under reduced pressure. The residue, remained after the distillation (non-consumed ketone with the admixture of NaCl) was mixed with water (5 mL) and treated with Et_2O (2×10 mL). The non-consumed cyclododecanone was additionally extracted from the aqueous layer with Et_2O (3×60 mL). The organic solutions were combined and dried with Na_2SO_4. After evaporation of the solvent, cyclododecanone (3.46 g) was obtained (identified by ^{1}H NMR spectroscopy), the degree of conversion of the ketone was 5%. The aqueous layers (after isolation of the ketone) were combined, acidified with concentrated HCI (to pH 1—2), then water was distilled off under reduced pressure. The solid residue was extracted with Me_2CO (4×50 mL). After evaporation of the solvent, dodecanedioic acid (0.097 g) was obtained (identified by ^{1}H NMR spectroscopy). The yield of the target product was 2.1% (based on the loaded ketone).

This work was financially supported by the Division of Chemistry and Materials Science of the Russian Academy of Sciences (program DC-01).

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Received May 13, 2009;
in revised form July 2, 2009