Reviews

The role of the Baeyer—Villiger reaction in the liquid-phase oxidation of organic compounds

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The data on the composition of ester products formed in the Baeyer—Villiger reaction in the liquid-phase oxidation of organic compounds with molecular oxygen, on production channel of peroxy acids, as well as on the influence of a carbonyl compound structure on its reactivity in reactions with peroxy acids have been classified and considered. The Baeyer—Villiger reaction was shown to be the main source of accessory primary alcohol esters and lactones in industrial processes of aerobic oxidation of cyclohexane and paraffin hydrocarbons.

Key words: Baeyer—Villiger reaction, ketones, peroxy acids, esters, liquid-phase oxidation.

The Baeyer—Villiger reaction, discovered[1] in 1899, forms the basis for high-demand synthetic approaches to obtain esters and lactones.[2—6] An increasing number of new oxidizing agents and catalysts are drawn into the orbit of the Baeyer—Villiger reaction.[6—9] However, the reviews[2—6] provide no data on the other aspect of the Baeyer—Villiger reaction, namely, its participation in the formation of esters and lactones in the liquid-phase oxidation of organic compounds with molecular oxygen.[10—17] Contrary to synthetic works, the object of studies directed on improving the oxidation processes, concerns a decrease, rather than an increase, of the ester yields. Aliphatic peroxy acids formed in these reactions are less reactive and find relatively rare applications in preparative synthesis. Moreover, reactions of alkane and cycloalkane oxidation proceed generally in apolar media.

The object of the present review is to organize data on the composition of ester products formed in the liquid-phase oxidation processes involving the Baeyer—Villiger reaction, production channels of peroxy acid, features of the reaction course in apolar media, as well as on the influence of a carbonyl compound structure on its reactivity.

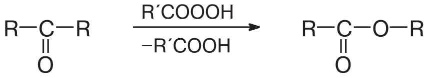
Classification of ester formation channels in the processes of the liquid-phase oxidation of organic compounds with molecular oxygen

Esters are the main byproducts in the liquid-phase oxidation of saturated hydrocarbons and their oxygen-containing derivatives.[18—23] In reviews,[18,19,22] all diversity of esters is divided into three groups depending on the main mechanisms of their formation.

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 5, pp. 0779—0786, May, 2018.

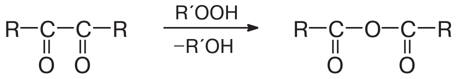
The first group comprises esters of primary (more rarely, secondary) alcohols and lactones having the same number of carbon atoms as the oxidized compound. It includes, *e.g.*, ethyl acetate formed upon oxidation of butane or methyl ethyl ketone,[21] isopropyl acetate formed upon oxidation of methyl isopropyl ketone,[24] ε-caprolactone formed upon oxidation of cyclohexane and its oxygen derivatives,[10,11] benzyl benzoate formed upon oxidation of benzyl phenyl ketone,[12,25] benzyl *p*-chlorobenzoate formed upon oxidation of benzyl *p*-chlorophenyl ketone,[26,27] benzyl phenylacetate formed upon oxidation of dibenzyl ketone,[12] and formate esters formed upon oxidation of aldehydes.[28—30] These compounds are formed predominantly *via* the Baeyer—Villiger reaction using peroxy acids (Scheme 1).[10—12,18,19,24—30]

Scheme 1



The existence of the second carbonyl group in a vicinal position to the first one results in the formation of a carboxylic acid anhydride.[18,31,32] Here, an oxidizing agent can be represented not only by peroxy acids but also by hydrogen peroxide and hydroperoxides (Scheme 2).[18,31,32]

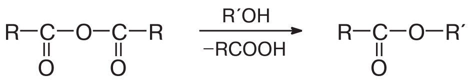
Scheme 2



R′ = H, Alk, acyl

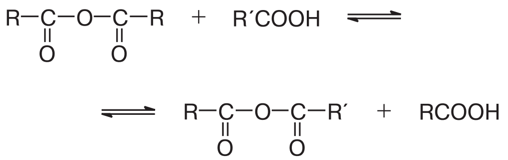
The second group comprises esters of secondary alcohols (having the same number of the carbon atoms as the parent compounds) and acids containing less carbon atoms then an alcohol. The esters include, *e.g.*, *s*-butyl acetate formed upon oxidation of butane and *s*-butanol, as well as mono- and dicarboxylic acid cyclohexyl esters formed upon oxidation of cyclohexane and cyclohexanol. These esters are formed with the participation of carboxylic acid anhydrides (Scheme 3).[18,19,22,33—36]

Scheme 3



The latter are obtained either through oxidative destruction of a carbon chain or through transacylation of anhydrides with carboxylic acids (Scheme 4).[18,19,22,32—36]

Scheme 4



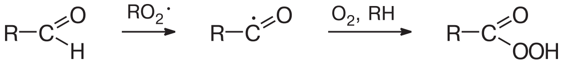
In an industrial cyclohexane oxidation process, the contribution from esterification of carboxylic acids with cyclohexanol to the formation of cyclohexyl mono- and dicarboxylates does not exceed 0.2%.[35]

The third group comprises γ- and δ-lactones containing the same number or often less carbon atoms than the substrate, the lactones being formed as a result of intramolecular chain-transfer reactions.[18,19]

Formation of peroxy acids in the liquid-phase oxidation of organic compounds with molecular oxygen

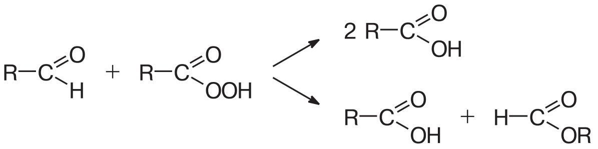
Formation of esters of the first group involves the participation of organic peroxy acids. It is known[18,21,28—30,37] that aldehyde groups are oxidized to carboxylic ones through the step of the peroxy acid formation (Scheme 5).

Scheme 5



Oxidation of aldehydes with peroxy acids affords two molecules of a carboxylic acid or a carboxylic acid and formate (Scheme 6).[18,19,28—30]

Scheme 6



When oxidizing other organic compounds, peroxy acids are presented in far more low concentrations. Development of sensitive gas chromatography techniques for peroxy acid determination based on reactions similar to those shown in Schemes 7 and 8 (see Refs 38, 39) enabled determination of these acids among the products of oxidation of not only aldehydes, but also cyclohexanone, ethyl methyl ketone, benzyl phenyl ketone, benzyl *p*-chlorophenyl ketone, dibenzyl ketone, benzyl benzoate, *tert*-butyl phenylacetate, benzyl phenylacetate, 2-hydroxycyclohexanone, and butyric acid.

Apart from oxidation of aldehydes, peroxy acids can be also obtained *via* homolysis of peroxy compounds af-

Scheme 7

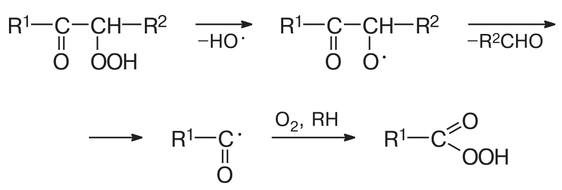
$$\mathrm{RCOOOH + Ph_2S \longrightarrow RCOOH + Ph_2SO}$$

Scheme 8

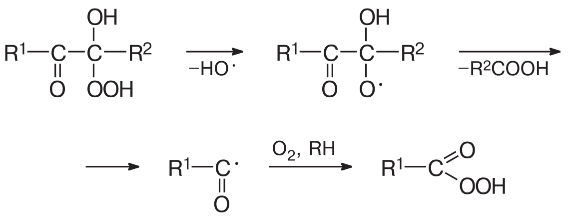
$$\mathrm{RCOOOH + Ph_2SO \longrightarrow RCOOH + Ph_2SO_2}$$

fording acyl radical, *e.g.*, α-ketohydroperoxide being the primary product of a ketone oxidation (Scheme 9),[18] or a product of α-ketoalcohol oxidation, namely, α-hydroxy-α-hydroperoxy ketone (Scheme 10).[18]

Scheme 9

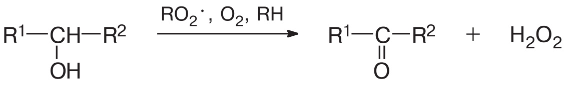


Scheme 10

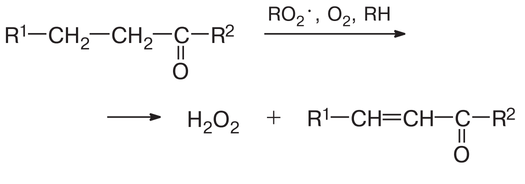


Formation of hydrogen peroxide during oxidation of secondary alcohols (Scheme 11)[18] and carbonyl compounds at β-CH-bonds (Scheme 12)[40,41] enables obtaining peroxy acids *via* acylation of H_2O_2 with anhydrides (Scheme 13) or carboxylic acids (Scheme 14).

Scheme 11

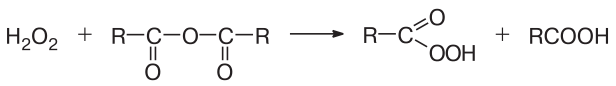


Scheme 12



R^2 = H, OH, OR, Alk

Scheme 13



Scheme 14

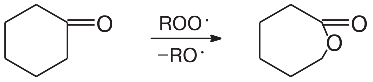
In contrast to the transformation illustrated in Scheme 13, reaction shown in Scheme 14 is reversible, and with consumption of the peroxy acid, a growing amount of hydrogen peroxide can be transformed into the peroxy acid. The reaction (see Scheme 14) proceeds with a well-marked rate even at 20 °C.[39]

The Baeyer—Villiger reaction as a main production channel for esters of primary alcohols and lactones in a process of the liquid-phase oxidation of alkanes, cycloalkanes, and their oxygen-containing derivatives

According to the Langenbeck—Pritzkov theory based on the data on the composition of the cyclohexanone oxidation products, esters are formed mainly *via* oxidation of ketones with peroxy acids.[18,19] Such conclusion seemed to be fully justified since ε-caprolactone identified among the cyclohexanone oxidation products is hard to obtain in an alternative way. Progress in the GLC techniques allowed passing to analysis of composition of individual ester products. It turned out that true (entering the transesterification reaction[18,19]) esters make just little contribution to the ester value, and their generation cannot only be due to the Baeyer—Villiger reaction.[19] In 50—70s of the previous century one attempted to account the ester formation for well-known reactions (such as esterification of carboxylic acids with alcohols, ester condensation of aldehydes) and hypothetical ones such as homolytic rearrangement of α-ketohydroperoxides, oxidation of ketones with peroxide raticals, *etc*.[20,21] These reaction could attribute the formation of any given compound in qualitative terms but they did not provide the observed rate of ester accumulation in autooxidation reactions.

Evidence for the Baeyer—Villiger reaction in formation of esters of the first group have been obtained in the studies.[9—11] During oxidation of cyclohexanone (chlorobenzene, 120 °C), a successive (in the order of enumeration) accumulation of 2-hydroperoxycyclohexanone, peroxy acid, ε-caprolactone, and adipic acid is observed (Fig. 1).[11] When carrying out the experiment under the same conditions but with adding diphenyl sulfide or diphenyl sulfone (reagents that reduce peroxy acids (see Schemes 7 and 8)), the formation of the lactone was inhibited almost completely (Fig. 2), though the radical-chain oxidation took place, and peroxide compounds with adipic acids were accumulated nearly with the same rate as in the experiment without such addition.[11] The same results have been obtained also for an open-chain compound such as benzyl phenyl ketone.[12] In this case, introduction of diphenyl sulfide also resulted in inhibition of benzyl benzoate formation. This unambiguously testify that lactones, on oxidation of cycloalkanones, and esters of primary alcohols, on oxidation of the open-chain compounds, are formed predominantly according to a reaction illustrated in Scheme 1, while an alternative mechanism involving peroxy radicals (Scheme 15)[23] does not take place in any noticeable rate.

Scheme 15



Quantitative results have been obtained on kinetic study of oxidation reactions of benzyl phenyl ketone[25] (Fig. 3) and benzyl *p*-chlorophenyl ketone.[27] A calculated curve (equation (1)) was found to correlate well with experimental values for benzyl benzoate concentrations

$$d[RC(O)OCH_2R]/dt = k_{eff}[RC(O)CH_2R][RCOOOH], \quad (1)$$

where k_{eff} is an effective reaction rate constant, [RC(O)-OCH_2R], [RC(O)CH_2R], and [RCOOOH] are concentrations of an ester, ketone, and peroxy acid respectively. When oxidizing benzyl *p*-chlorophenyl ketone, two peroxy acids were formed, however, in this case kinetics of an ester accumulation follows the equations of the type (1).[27]

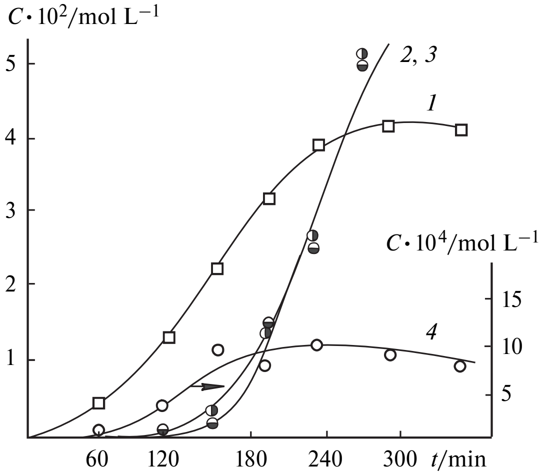


Fig. 1 Kinetic curves of accumulation of hydroperoxides (*1*), adipic acid (*2*), ε-caprolactone (*3*), and peroxy acids (*4*) upon oxidation of cyclohexanone (CH) (C_{CH} = 4.0 mol L^{-1}, chlorobenzene, 120 °C).

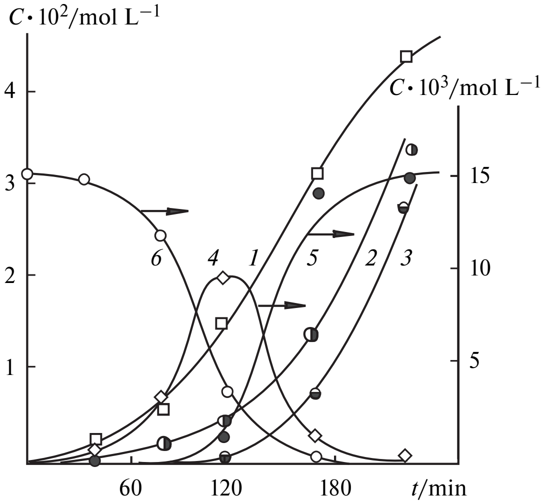


Fig. 2. Kinetic curves of accumulation of hydroperoxides (*1*), adipic acid (*2*), ε-caprolactone (*3*), diphenyl sulfoxide (*4*), and diphenyl sulfone (*5*), as well as consumption of diphenyl sulfide (*6*) upon oxidation of cyclohexanone and diphenyl sulfide (C_{CH} = 4.0 mol L^{-1}, chlorobenzene, 120 °C).

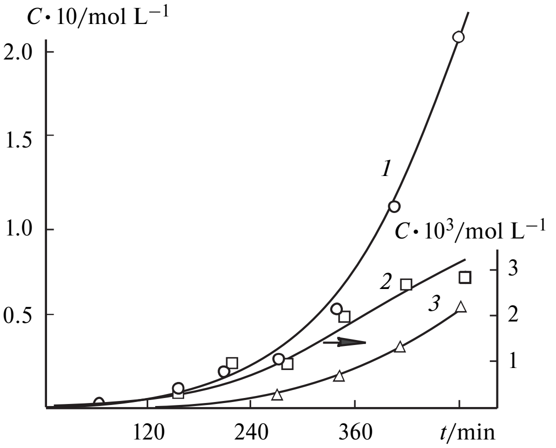


Fig. 3. Experimental values and calculated curves for accumulation of benzoic acid (*1*), peroxybenzoic acid (*2*), and benzyl benzoate (*3*) upon oxidation of benzyl phenyl ketone (BPK) (C_{BPK} = 1.0 mol L^{-1}, chlorobenzene, 110 °C).

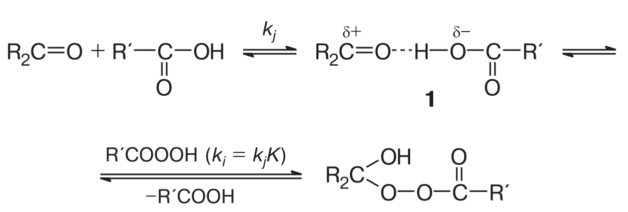
Mechanism of the Baeyer—Villiger reaction and reactivity of carbonyl compounds

Mechanisms of the Baeyer—Villiger reaction are still a subject of discussion. Questions remaining still unclear relate to the nature of intermediates, mechanisms of their formation and decompositions as well as catalysis of the corresponding steps.[1,2,6] In most studies, two-step mechanism is admitted comprising a reversible nucleophilic addition of a peroxy acid to a carbonyl compound affording a Criegee intermediate, and a heterolytic rearrangement of the latter to an ester or lactone. By an example of cyclohexanone oxidation with peroxylauric acid[13—17,42,43] and autooxidation reactions of aromatic ketones[25,27] the reaction was shown to be catalytic. Effective rate constants for all steps (k^{eff}_i) depend linearly on concentration of a carboxylic acid ([RCOOH]) including the one formed from a peroxy acid according Scheme 1:

$$k^{\text{eff}}_i = k_{i,0} + k_i[\text{RCOOH}], \qquad (2)$$

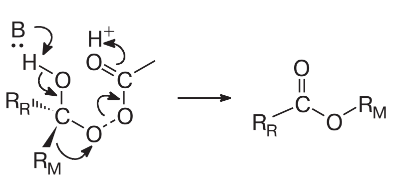
where $k_{i,0}$ is a rate constant of a step in the absence of a carboxylic acid, $k_i = k_jK$ (k_j is a rate constant for the complex formation involving carboxylic acid of a 1 : 1 ratio (*e.g.*, **1**), K is an equilibrium constant for the same reaction). In the case of an addition step, activation of a carbonyl group by the mechanism of general acid catalysis to generate complex **1** occurs, wherein the complex reacts with a peroxy acid to afford α-hydroxyperoxy ester (Scheme 16).

Scheme 16



The conversion of an α-hydroxyperoxy ester to an ester or lactone represents a nucleophilic rearrangement at an electron-deficient center.[44] Nowadays, the majority of studies concerning the Baeyer—Villiger reaction give priority to the synchronous mechanism (Scheme 17).[2,3,6]

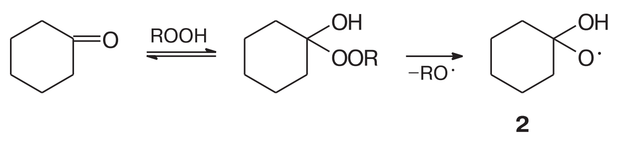
Scheme 17



This mechanism is in a good agreement with the fact that migrating groups retain their configuration, and is supported with quantum chemical calculations.[45—52]

Radical channel of transformation of a Criegee intermediate for oxidation of cyclohexanone with peroxylauric acid (Scheme 18, R is acyl) does not exceed 1—2% (see Ref. 14), and gives no lactone.[18,19]

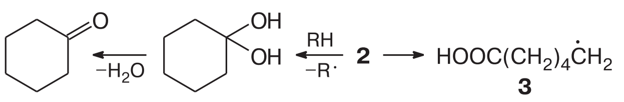
Scheme 18



R = H, Alk, acyl

The resulting α-hydroxycyclohexyloxy radical **2** either abstracts the hydrogen atom from the substrate, regenerating cyclohexanone, or undergoes destruction to form radical **3** (Scheme 19).

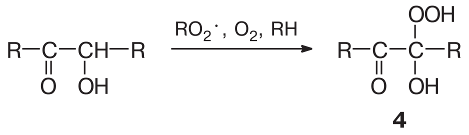
Scheme 19



Radical **3** can transform subsequently into caproic or 6-hydroxycaproic acid. Although possible, cyclization of the latter to ε-caprolactone proceeds in a moderate rate because of a low probability of a favorable approach of terminal groups to form a seven-membered ring. This fits well with the fact that in the experiment with the peroxy acid "subtraction" (see Fig. 2), no ε-caprolactone is formed with the participation of peroxide compounds. The observed contradiction with the available data on using hydrogen peroxide or hydroperoxides[5—9] in the Baeyer—Villiger reaction can be removed supposing that the reaction between these oxidizing agents and a carbonyl compound, *e.g.*, cyclohexanone, affords α-hydroperoxy- or α-hydroxy-α-alkylperoxycyclohexanes (see Scheme 18, R = H and Alk, respectively), which undergo a heterolytic rearrangement to form an ester or lactone[4,6,18,19] only in the presence of effective catalysts, particularly, Lewis acids.

The same is not true for α-hydroxy-α-hydroperoxy ketones **4** and α-hydroxy-α-alkylperoxy ketones. The former are formed not only as intermediates in the reaction as illustrated in Scheme 2 (R′ = H), but also as a result of the carbon chain destruction at the step of the radical-chain oxidation of α-ketoalcohols (Scheme 20).[18,33,53,54]

Scheme 20



Conversion of compounds **4**, as well as α-hydroxy-α-alkylperoxy ketones to an anhydride occurs probably with a participation of the carbonyl group (Scheme 21).[18,53] The rearrangement takes place both in the absence of catalysts and under a general acid catalysis with carboxylic acids.[18,53]

Scheme 21

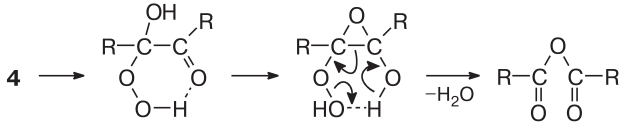


Table 1. Oxidation of aliphatic ketones (RH) with peroxylauric acid (RCOOOH) in the presence of lauric acid (RCOOH) (chlorobenzene, 80 °C, 240 min)

Ketone	[RH]	[RCOOH] $\cdot 10^2$	$[RCOOOH]_0 \cdot 10^2$	$[RCOOOH]^a \cdot 10^2$	Ester yield^b (mol.%)
	mol L^{-1}				
Nonan-5-one	1.05	4.71	4.1	0.3	26.3
Octan-2-one	1.28	3.76	3.7	1.2	48.0
Heptadecan-9-one	0.77	4.75	4.9	0.8	41.5

[a] At the end of the experiment.
[b] On the basis of the converted peroxy acid.

Esters or lactones yields obtained in the processes of the liquid-phase oxidation involving the Baeyer—Villiger reaction vary for various substrates. For example, if under uncatalyzed oxidation of cyclohexanone the yield of ε-caprolactone is close to that of adipic acid (see Fig. 1), then in oxidation of benzyl phenyl ketone the yield of benzyl benzoate is ~3 times less relative to benzoic acid (see Fig. 3). Apparently, it depends on reactivity of carbonyl compounds and peroxy acids, structure of migrating groups, and also on the availability of the channels of the peroxy acid formation and consumption. Reactivity of the carbonyl group is governed by electronic and steric factors.[44] Aromatic aldehydes and ketones are known to be less reactive than aliphatic ones. Enlargement of aliphatic radicals also decrease the reactivity of a carbonyl compound towards the nucleophilic attack. Either the step of nucleophilic addition to yield an Criegee intermediate or the rearrangement step can be a controlling step. For cyclohexanone and peroxylauric acid, the process is controlled by the rearrangement step. In this case, α-hydroxyperoxy ester is accumulated in considerable amounts, and the yield of ε-caprolactone relative to the converted peroxy acid at 20 °C is almost quantitative.[13,14] For long-chain aliphatic ketones, the rate of oxidation with peroxylauric acid is lower. Evidently, in these cases the controlling step represents the step of formation of α-hydroxyperoxy ester, rather than that of its decomposition. A decrease in ester yields when passing from long-chain aliphatic ketones (Table 1) is connected with the thermal decomposition of a peroxy acid.

The existence of the formation channels of peroxy acids and carbonyl compounds in the processes of liquid-phase oxidation of organic compounds with molecular oxygen results in the unavoidable Baeyer—Villiger reaction even in apolar media and with participation of relatively weak aliphatic peroxy acids. Contribution of this reaction in the ester formation can be decreased either due to decomposition of peroxy acids or due to promotion of the radical decomposition of α-hydroxyperoxy esters. Studying the influence of the mixed-valence metal compounds on the reaction of cyclohexanone oxidation with peroxylauric acid has shown that such property can be found in cobalt and manganese compounds.[15]

The above-mentioned ideas concerning the mechanism of formation of the group under consideration comprising esters, and data on formation of ε-caprolactone in model reactions correspond closely to experimental results obtained when studying the product composition of the

Table 2. Effect of cobalt(II) and chromium(III) naphtenates and ketone conversion (Δ[RH]) on the composition of cyclohexanone oxidation products ([RH] = 4.0 mol L^{-1}, chlorobenzene, 120 °C, [Co] = [Cr] = = $1.6 \cdot 10^{-3}$ mol L^{-1})[33,55]

Oxidation products	Composition of oxidation products (mol.%)					
	Δ[RH] = 0.1 mol L^{-1}			Δ[RH] = 0.25 mol L^{-1}		
	Without a catalyst	Co^{II}	Cr^{III}	Without a catalyst	Co^{II}	Cr^{III}
Peroxy compounds	37.6	7.1	14.0	18.7	3.7	9.3
2-Hydroxycyclohexanone	3.9	21.3	26.2	2.3	7.1	21.3
Cyclohexane-1,2-dione	1.3	3.4	0.7	1.0	0.8	0.7
Adipic anhydride	1.1	1.7	2.3	2.5	1.2	2.2
2-Hydroxycyclohexyladipate	0.2	1.0	0.1	0.4	1.2	0.4
ε-Caprolactone	21.5	6.6	24.3	29.0	4.5	26.1
Adipic acid	18.5	36.8	18.7	24.2	49.9	20.7
6-Oxocaproic acid	12.5	4.6	8.3	11.8	6.9	9.3
Glutaric acid	0.5	7.0	0.7	3.4	5.8	5.0
δ-Valerolactone	2.5	1.4	3.3	4.5	4.7	3.5
6-Hydroxycaproic acid	0.4	9.1	1.4	2.2	14.5	1.5

Table 3. Effect of cobalt(II) naphtenate and cyclohexane conversion (Δ[RH]) on the composition of cyclohexane oxidation products (without solvent, 150 °C, 1.47 MPa, [Co] = $8.5 \cdot 10^{-4}$ mol L^{-1})

Oxidation products	Composition of oxidation products (mol.%)			
	Δ[RH] = 0.6 mol L^{-1}		Δ[RH] = 1.2 mol L^{-1}	
	Without a catalyst	Co^{II}	Without a catalyst	Co^{II}
Hydroperoxides	9.3	2.7	4.2	0.2
Cyclohexanol	33.6	51.0	26.3	40.7
Cyclohexanone	46.5	39.3	61.7	49.0
2-Hydroxycyclohexanone	2.4	1.5	1.6	2.0
6-Oxocaproic acid	1.4	1.1	1.2	1.5
Adipic acid	2.9	1.8	2.2	3.6
Cyclohexyladipate	0.3	0.2	0.5	0.6
ε-Caprolactone	1.2	<0.1	0.7	<0.1
6-Hydroxycaproic acid	2.4	2.4	1.6	2.4

liquid-phase oxidation of cyclohexanone[33,55] (Table 2) and cyclohexane (Table 3). Referring to the data of Table 2, it can be seen that ε-caprolactone is actually the main side product of the uncatalyzed as well as chromium(III) naphtenate-catalyzed oxidation of cyclohexanone. A relative yield of the lactone decreases in the presence of cobalt(II) naphtenate. At the same time, the relative yield of 6-hydroxycaproic acid rises in the cobalt salt-catalyzed reaction (see Table 2). These results are in a good agreement with the concepts of the high activity of cobalt salts in the reactions of homolytic decomposition of peroxy compounds yielding, as noted above, no lactone (see Schemes 18 and 19). The same picture is observed also in cyclohexane oxidation (see Table 3). In this case, the lower yield of ε-caprolactone (compared to cyclohexanone oxidation) is associated with a lower ketone concentration. The main ester products of cyclohexane oxidation are cyclohexyl mono- and dicarboxylates.[34]

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Received September 1, 2017;
in revised form December 14, 2017;
accepted February 13, 2018