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Reaction of Tri- and Tetrasubstituted Alkenes with the Low-Temperature Oxidizing System Aluminum *tert*-Butylate–*tert*-Butyl Hydroperoxide

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Abstract—The system consisting of aluminum *tert*-butylate and *tert*-butyl hydroperoxide under mild conditions (20°C) oxidizes tri- and tetrasubstituted ethylenes containing at least one α-methyl group. The reaction proceeds via formation of tertiary allylic hydroperoxides and their subsequent transformations into unsaturated alcohols and epoxy alcohols, and also into carbonyl compounds. The presence of the latter products suggests degradation of the carbon skeleton of alkenes.

The system consisting of aluminum *tert*-butylate **I** and *tert*-butyl hydroperoxide **II** is an oxidant of alkanes, alkylarenes [1], as well as of their oxygen-containing derivatives: alcohols, aldehydes, ketones [2], ethers [3], and esters [4]. Reaction of the components **I** and **II** of the system leads to the evolution of oxygen [1–4] through the intermediate formation of di(*tert*-butoxy)-*tert*-butylperoxyaluminum and aluminum-containing trioxide [scheme (1)].

$$(t\text{-BuO})_3\mathrm{Al} + t\text{-BuOOH} \xrightarrow[-t\text{-BuOH}]{} (t\text{-BuO})_2\mathrm{AlOOBu}\text{-}t$$

$$\xrightarrow{t\text{-BuOOH}} \left[\begin{array}{c}(t\text{-BuO})_2\mathrm{Al{-}O{-}O{-}Bu}\text{-}t\\ t\text{-BuO{-}O{-}H}\end{array}\right] \rightarrow (t\text{-BuO})_2\mathrm{AlOOOBu}\text{-}t$$

$$\rightarrow (t\text{-BuO})_3\mathrm{Al} + \mathrm{O}_2 \qquad (1)$$

Radical oxidation of methyl and methylene bonds with this system depending on the structure of aliphatic and alkylaromatic hydrocarbons leads in the first stage to introduction of oxo, hydroxy [1, 2, 4], and hydroperoxy groups [1, 3]. The resulting functional derivatives are involved in subsequent reactions with di(*tert*-butoxy)-*tert*-butylperoxyaluminum as well as with the system **I**–**II**. In most cases their oxidation is accompanied by the cleavage of carbon–carbon bonds in the starting substrate. Note that, in spite of the fairly wide range of the studied compounds, direct oxidation of methyl group was observed only in toluene. It was noted previously that the carbon–hydrogen bonds of the benzene ring do not react with the above system [1]. The oxidation of substrates proceeds with a high conversion when a stable radical can form (benzhydryl and especially benzyl). It is known that the stability and the energy of formation of the benzyl radical [$E(\mathrm{PhCH}_2^{\bullet})$ 85 kcal mol^{-1}] are close to those of the allyl radical [$E(\mathrm{CH_2{=}CH{=}CH}_2^{\bullet})$ 89 kcal mol^{-1}] [5]. The latter can be formed in the course of oxidation of α-C–H bonds in alkenes.

In this work we studied the reactions of tetra- and trisubstituted alkenes (2,3-dimethyl-2-butene **III**, 2-methyl-2-butene **IV**, 1,1-diphenylpropene **V**, 1,1-diphenyl-2-methylpropene **VI**) with the oxidizing system **I**–**II**. We expected oxidation of the methyl group with the intermediate formation of allylic radicals and epoxidation of the double bond. According to published data [6], peroxy compounds of boron, silicon, and gallium oxidize alkenes to the corresponding epoxides. Note that hydroperoxide **II** reacts with the double bond in alkenes only in the presence of transition metal (vanadium, molybdenum, chromium, etc.) derivatives [7].

Reaction of alkenes with the system **I**–**II** was performed in benzene at 20°C. The ratio of the starting components (alkene : alcoholate **I** : hydroperoxide **II**) was 1 : 1 : 2. The highest conversion (up to 75%) was observed with the most substituted alkene, 2,3-dimethyl-2-butene, which agrees with the published data [7].

The primary oxidation products of alkenes **III**–**VI** are tertiary allylic peroxides $\mathrm{R_2C(OOH)C(R'){=}CH_2}$ [$\mathrm{R = R' = CH_3}$ (**VII**); $\mathrm{R = CH_3, R' = H}$ (**VIII**); $\mathrm{R = Ph, R' = CH_3}$ (**IX**); $\mathrm{R = Ph, R' = H}$ (**X**)]. Hydroperoxides **VII**–**X** undergo subsequent transformations to give unsaturated alcohols, epoxy alcohols, and carbonyl compounds (Table 1).

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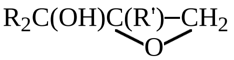
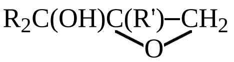
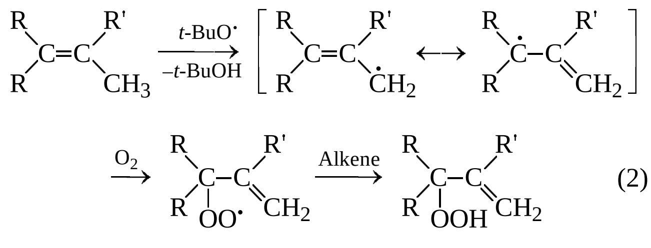
Table 1. Products of the reaction of alkenes $R_2C{=}CR'CH_3$ with the system $(t\text{-BuO})_3Al$–t-BuOOH (1 : 1 : 2 molar ratio) in benzene at 20°C, reaction time 24 h (mol mol^{-1} of alkene)[a]

Reaction products	R = R' = CH_3	R = CH_3, R' = H	R = Ph, R' = CH_3	R = Ph, R' = H
		Volatile products		
t-BuOH	2.48	2.81	2.76	2.47
t-BuOOH	Traces	0.12	0.30	0.27
$R_2C{=}CR'CH_3$ (initial)	0.26	0.51	–	–
$R_2C{=}O$	0.21	0.17	$Me_2C{=}O$ 0.12	$CH_3C(O)H$ (qualitatively)
$R_2C(OOH)C(R'){=}CH_2$	0.10	0.07	–	–
$R_2C(OH)C(R'){=}CH_2$	0.11	Traces	–	–
 $R_2C(OH)\underbrace{C(R')-CH_2}_{O}$	0.10	0.06	–	–
		Hydrolysis products of nonvolatile residue[b]		
t-BuOH	2.20	1.82	1.59	1.98
t-BuOOH	0.25	0.23	0.30	0.26
$R_2C{=}CR'CH_3$ (initial)	–	–	0.55	0.35
$R_2C{=}O$	0.04	$CH_3C(O)H$ (qualitatively)	0.20	0.30
$R_2C(OOH)C(R'){=}CH_2$	0.03	0.05	0.10[c]	0.11[c]
$R_2C(OH)C(R'){=}CH_2$	0.12	0.15	0.10	0.18
 $R_2C(OH)\underbrace{C(R')-CH_2}_{O}$	0.05	0.04	0.11	0.12
$CH_3C(O)OH$	–	0.13	–	0.16
PhOH	–	–	0.02	0.01
$PhC(O)CR'{=}CH_2$	–	–	0.03	0.02

[a] Averaged results. [b] Amount of aluminum in all the experiments is ~1 g-at. [c] Hydroperoxides were identified by the products of catalytic decomposition in the presence of benzenesulfonic acid, PhOH, and phenylalkenyl ketones.

Several pathways of formation of hydroperoxides can be suggested. The first is the reaction of alkene with singlet oxygen partially generated by the studied system [8]. The process can also be initiated by *tert*-butoxy radicals formed by homolytic decomposition of aluminum-containing trioxide and identified with spin traps. *tert*-Butoxy radicals abstract hydrogen from the methyl groups of alkenes **III**–**VI**. The resulting primary radical undergoes rearrangement and reacts with the evolution of oxygen according to scheme (1) [scheme (2)].

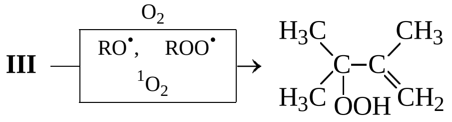


(2)

R = R' = CH_3 (**III**); R = CH_3, R' = H (**IV**); R = Ph, R' = CH_3 (**V**); R = Ph, R' = H (**VI**).

The possibility of this pathway was confirmed by the reaction of alkene **III** with oxygen, initiated by the system **I**–**II** taken in amount of 10% relative to alkene. Under these conditions the conversion of alkene was 80%. All the products listed in Table 1 were isolated. The major product was hydroperoxide **VII** (0.50 mol). We also performed oxidation of **III** with oxygen using *tert*-butyl peroxalate as initiator and obtained hydroperoxide **VII** and unsaturated alcohol.

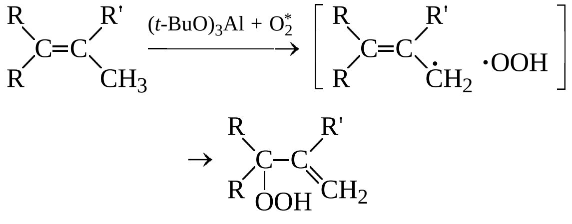
Our results do not contradict the published data on formation of tertiary hydroperoxides in oxidation of alkenes, because it is known that alkene **III** transforms into hydroperoxide **VII** either by homolytic oxidation in the presence of alkoxy and alkylperoxy radicals [9] or by the reaction with singlet oxygen [10].



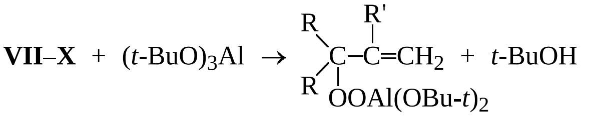
(3)

Along with the above-mentioned reactions, the third pathway is possible, namely, oxidation of alkene with available oxygen as suggested in [1, 2].

At present, we cannot choose between these three pathways. More definite conclusion can be made about further transformations of the oxidation products. Hydroperoxides **VII**–**X** react with alcoholate **I** to form new peroxy compounds of aluminum **A**.



VII–**X** (4)



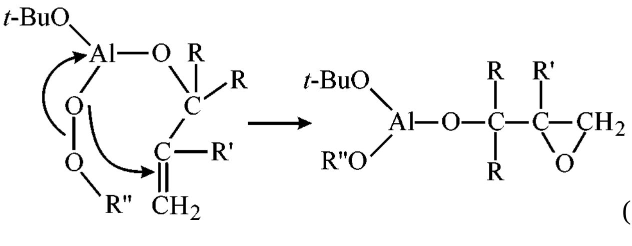
$$\textbf{VII–X} + (t\text{-BuO})_3Al \rightarrow R_2\underset{OOAl(OBu\text{-}t)_2}{C}\text{–}\overset{R'}{C}{=}CH_2 + t\text{-BuOH} \quad (5)$$

A

All the other compounds listed in Table 1 are formed by transformations of peroxides **A**. It was confirmed by the comparison of the products formed by oxidation of 2,3-dimethyl-2-butene with the system **I**–**II** and by transformation of specially prepared hydroperoxide, 3-hydroperoxy-2,3-dimethyl-1-butene **VII**, in the presence of aluminum *tert*-butylate (Table 2).

Aluminum-containing peroxides **A** decompose by several pathways. One of them is the formation of alcohols, probably by the reaction of peroxide **A** with tertiary hydroperoxide similarly to the release of *tert*-butanol [scheme (1)] [1–4].

Epoxy alcohols are formed by intramolecular oxidation of the double bond [Eq. (6)]. The possibility of epoxidation of allylic alcohols by the system **I**–**II** at 25°C was shown in [11, 12].



(6)

R'' = *t*-Bu, $C(CH_3)_2\text{–}C(CH_3){=}CH_2$.

However, intermolecular epoxidation cannot be ruled out, as it was shown for decomposition of allylic hydroperoxides in the presence of titanium alcoholates [13]. Direct epoxidation of the double bond by the reaction of hydroperoxy and carbon-centered radicals [Eq. (4)] is also possinle.

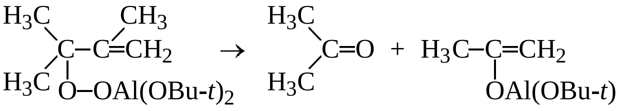
Table 2. Products of the reaction of $(CH_3)_2C{=}C(CH_3)_2$ with the system $(t\text{-BuO})_3Al\text{–}t\text{-BuOOH}$ (1 : 1 : 2) (a) and of transformation of $(CH_3)_2C(OOH)C(CH_3){=}CH_2$ in the presence of $(t\text{-BuO})_3Al$ (b) in benzene [20°C, 24 h; mol mol^{-1} $(t\text{-BuO})_3Al]^a$

Product	Reaction (a)	Reaction (b)
Volatile products		
t-BuOH	2.48	0.89
$(CH_3)_2C{=}C(CH_3)_2$	0.26	–
t-BuOOH	Traces	–
$(CH_3)_2C(OOH)C(CH_3){=}CH_2$	0.10	–
$(CH_3)_2C{=}O$	0.21	0.03
$(CH_3)_2C(OH)\underbrace{C(CH_3){-}CH_2}_{O}$	0.10	0.06
$(CH_3)_2C(OH)C(CH_3){=}CH_2$	0.11	0.13
Hydrolysis products of nonvolatile residue[b]		
t-BuOH	2.20	2.03
t-BuOOH	0.25	–
$(CH_3)_2C(OOH)C(CH_3){=}CH_2$	0.03	0.16
$(CH_3)_2C{=}O$	0.04	0.13
$(CH_3)_2C(OH)C(CH_3){=}CH_2$	0.12	0.20
MeOH	0.04	Not determined
$(CH_3)_2C(OH)\underbrace{C(CH_3){-}CH_2}_{O}$	0.06	0.20

[a] Averaged results. [b] Aluminum content in all the experiments 1 g-at.

The presence of carbonyl compounds among the volatile products (acetone, acetaldehyde) and among the hydrolysis products of the nonvolatile residue (benzophenone) suggests degradation of the carbon skeleton of alkene. In particular, Table 2 shows that acetone is formed in the reaction of 2,3-dimethyl-2-butene with the system **I**–**II** as well as in the reaction of the corresponding hydroperoxide with alcoholate **I**, which suggests that acetone is the product of decomposition of peroxide **VII**.

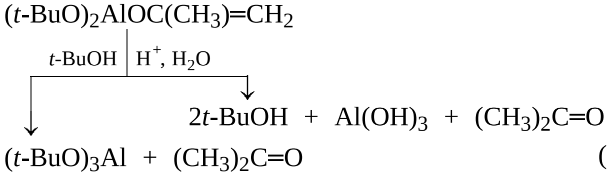
For example, in the case of 2,3-dimethyl-2-butene the reaction proceeds as follows.



$$(H_3C)_2\underset{O\text{–}OAl(OBu\text{-}t)_2}{C}\text{–}\overset{CH_3}{C}{=}CH_2 \rightarrow (H_3C)_2C{=}O + H_3C\text{–}\underset{OAl(OBu\text{-}t)_2}{C}{=}CH_2 \quad (7)$$

It is known that alkenes can react with singlet oxygen to form 1,2-dioxetanes decomposing to carbonyl compounds [14]. But this reaction takes place only for alkenes with electron-withdrawing substituents. In our case formation of carbonyl compounds through decomposition of aluminum-containing peroxides is more probable. The suggested scheme [Eq. (7)] is experimentally confirmed by the reaction of 2,3-dimethyl-2-butene with the system **I**–**II** taken in an amount of 10% relative to alkene. From the suggested scheme (7) it follows that the amount of carbonyl compound must coincide with the amount of alcoholate **I** taken in the reaction. Indeed, in this experiment only 0.1 mol of acetone was isolated, which corresponds to the amount of aluminum *tert*-butylate.

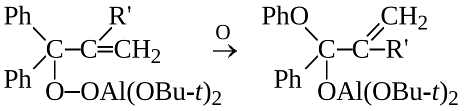
Hydrolysis of di-*tert*-butoxypropenyloxyaluminum yields acetone. However, Table 2 shows that the amount of acetone in the free state exceeds its amount in the bound state, which may be due to the reaction of excess *t*-BuOH with aluminum enolate [15].



$$(t\text{-BuO})_2\text{AlOC(CH}_3)\text{=CH}_2 \xrightarrow{t\text{-BuOH}} (t\text{-BuO})_3\text{Al} + (\text{CH}_3)_2\text{C=O}$$
$$(t\text{-BuO})_2\text{AlOC(CH}_3)\text{=CH}_2 \xrightarrow{\text{H}^+,\ \text{H}_2\text{O}} 2t\text{-BuOH} + \text{Al(OH)}_3 + (\text{CH}_3)_2\text{C=O} \quad (8)$$

Under these conditions aldehydes are oxidized to acyloxy derivatives of aluminum. The IR spectra of nonvolatile residues contain broad absorption bands in the range 1570–1600 cm^{-1}, characteristic of COO^- stretching vibrations, and in the hydrolysis products the carboxylic acids are found.

In the case of diphenyl-substituted alkenes the presence of phenylalkenyl ketones and phenol in the hydrolysis products of nonvolatile residues is due to partial rearrangement of peroxides **A** and decomposition of the resulting aluminum alkoxides [Eq. (9)].



$$\rightarrow \text{PhC(O)C(R')=CH}_2 + \text{PhOAl(OBu-}t)_2 \quad (9)$$

R' = H (**IX**), CH_3 (**X**).

Thus, the system consisting of aluminum tris-*tert*-butylate and *tert*-butyl hydroperoxide is a unique oxidant of tri- and tetrasubstituted alkenes under mild conditions. The process occurs by the radical pathway involving the stage of formation of tertiary hydroperoxides and their subsequent reaction with alcoholate **I** yielding new aluminum-containing peroxy compounds **A**. Heterolytic decomposition of these compounds yields the final products. The reaction occurs with partial cleavage of the starting alkene. Oxidation of carbon–hydrogen bonds of the benzene ring was not observed.

EXPERIMENTAL

The IR spectra were recorded on a Specord IR-75 spectrometer. Chromatographic analysis of the reaction products in the liquid phase was carried out on a Tsvet-2-65 chromatograph with a flame-ionization detector and argon carrier gas. Volatile components (*tert*-butanol, acetone, 2,3-dimethyl-2-butene, 2-methyl-2-butene, acetaldehyde, methyl acetate) were analyzed on a 2400 × 3-m column, stationary phase 10% polyethylene glycol on TZKM support, 55–70°C. For the analysis of benzophenone, 1-phenyl-2-propen-1-one, phenol, 1,1-diphenylpropene, 1,1-diphenyl-2-methylpropene, 1,1-diphenyl-2-propen-1-ol, and 1,1-diphenyl-2-methylpropen-1-ol a 3000 × 3-mm column was used; stationary phase 5% SE-30 on Inerton-AW support, 140–190°C. *tert*-Butyl hydroperoxide, 3-hydroperoxy-2,3-dimethyl-1-butene, 2,3-dimethyl-1-buten-3-ol, 3-methyl-1-buten-3-ol, and 1-methyl-1-(1-hydroxy-1-methylethyl)oxirane were analyzed on an LKhM-80 chromatograph; 1200 × 3-mm column, helium carrier gas, stationary phase 15% dinonyl phthalate on Chromaton N-AW-DMCS support, 40–80°C. Chromatograms were processed using external references. All the manipulations with aluminum compounds were done under argon.

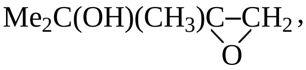
The amount of aliphatic acids in nonvolatile residues was determined by the procedure in [16], and the content of *tert*-butoxy groups was found by the Denigés method [17]. The amount of aluminum was determined by precipitation of aluminum hydroxide and its calcination to Al_2O_3. Carboxylic acids were identified in the form of methyl esters after treatment with diazomethane [4]. Carbonyl compounds were identified as 2,4-dinitrophenylhydrazones by the melting points and by TLC on Silufol UV-254 plates, elution with benzene. The amount of hydroperoxides **I**, **VII**, and **VIII** in the volatile fraction and hydrolysis products of the nonvolatile residue was determined chromatographically. Quantitative analysis of hydroperoxides **IX** and **X** formed by oxidation of diphenyl-substituted alkenes was performed by iodometric titration. Compounds **IX** and **X** were identified by products of their acid decomposition [18] (phenol and phenyl alkenyl ketones).

Aluminum tri-*tert*-butylate was prepared by the reaction of *tert*-butanol and aluminum [19] and purified by sublimation (160–170°C/1 mm Hg). Found, %: Al 11.12. $\text{C}_{12}\text{H}_{27}\text{AlO}_3$. Calculated, %: Al 10.96.

1,1-Diphenylpropene and 1,1-diphenyl-2-methylpropene were prepared by dehydration of the corresponding tertiary alcohols at 200°C without a solvent [20]. 2,3-Dimethyl-2-butene was synthesized by dehydration of 2,3-dimethyl-2-butanol with oxalic acid [21], and 2-methyl-2-butene, by treatment of 2-methyl-2-butanol with concentrated sulfuric acid [22].

2,3-Dimethyl-1-buten-3-ol [23] and 3-methyl-1-buten-3-ol [24] were prepared by treatment of methyl acrylate or methacrylate with excess methylmagnesium iodide. The refractive indices, boiling points, and melting points of the alkenes and alcohols agree with the published data. 1-Diphenyl-2-propen-1-ol was prepared by treatment of methyl acrylate with phenylmagnesium bromide, bp 130–132°C/2 mm Hg, n_D^{19} 1.5690. Found, %: OH 8.06; bromine number 75.02. $C_{15}H_{14}O$. Calculated, %: OH 8.10, bromine number 76.20. Phenylprop-2-en-1-one was prepared by decomposition of the corresponding Mannich salt [25], bp 57–59°C/2 mm Hg, n_D^{19} 1.5180, mp of 2,4-dinitrophenylhydrazone 264–266°C. 1-Methyl-1-(1-hydroxy-1-methylethyl)oxirane was prepared by treatment of 2,3-dimethylbut-1-en-3-ol with *tert*-butyl hydroperoxide in the presence of vanadyl acetylacetonate [7]. Colorless liquid, bp 34°C/24 mm Hg, n_D^{19} 1.4284. IR spectrum, ν, cm^{-1}: 860, 920, 1250, 3050 (epoxy ring), 3460 (OH). Found, %: OH 14.18. $C_6H_{12}O_2$. Calculated, %: OH 14.66. 3-Hydroperoxy-2,3-dimethyl-1-butene was prepared by treatment of 2,3-dimethyl-2-butene with 30% hydrogen peroxide and a solution of sodium hypochlorite in anhydrous methanol [26]; bp 46°C/8 mm Hg, n_D^{16} 1.4440. The concentration of *tert*-butyl hydroperoxide was no less than 99.5–99.8%.

Reaction of 2,3-dimethyl-2-butene with the system $(t\text{-}BuO)_3Al$–*t*-BuOOH in a 1 : 1 : 2 molar ratio. A mixture of 1.70 g of $(t\text{-}BuO)_3Al$, 1.24 g of *t*-BuOOH, and 0.58 g of 2,3-dimethyl-2-butene in 20 ml of benzene was kept for 24 h at room temperature. The solution acquired an intense yellow color. The solvent and the volatile products were distilled into a trap cooled with liquid nitrogen. The condensate contained 0.04 g of *t*-BuOOH, 1.16 g of *t*-BuOH, 0.06 g of $Me_2C{=}O$, 0.15 g of $Me_2C{=}CMe_2$, 0.05 g of $Me_2C(OOH)C(CH_3){=}CH_2$, and 0.07 g of $Me_2C(OH)C(CH_3){=}CH_2$. The residue, a viscous yellow mass, was dissolved in diethyl ether and treated with a 10% solution of sulfuric acid. The ether extract contained 0.05 g of *t*-BuOOH, 1.15 g of *t*-BuOH, 0.03 g of $Me_2C{=}O$, 0.04 g of $Me_2C(OOH)C(CH_3){=}CH_2$, 0.11 g of $Me_2C(OH)C(CH_3){=}CH_2$, and 0.06 g of  $Me_2C(OH)(CH_3)\underset{\diagdown\,O\,\diagup}{C{-}CH_2}$. Acetone was identified in the form of 2,4-dinitrophenylhydrazone, mp 127°C. The acidic aqueous phase of hydrolysis products contained 0.18 g of aluminum and 0.12 g of *t*-BuOH.

Reaction of 3-hydroperoxy-2,3-dimethyl-1-butene with alcoholate I. A mixture of 0.71 g of $(t\text{-}BuO)_3Al$ and 0.35 g of $Me_2C(OOH)C(CH_3){=}CH_2$ in 15 ml of benzene was kept for 24 h at room temperature and treated as described above. The volatile fraction contained 0.20 g of *t*-BuOH, 0.002 g of $Me_2C{=}O$, and 0.033 g of $Me_2C(OH)C(CH_3){=}CH_2$. The hydrolysis products contained 0.03 g of $Me_2C{=}O$, 0.45 g of *t*-BuOH, 0.07 g of $Me_2C(OOH)C(CH_3){=}CH_2$, 0.05 g of $Me_2C(OH)C(CH_3){=}CH_2$, 0.10 g of  $Me_2C(OH)(CH_3)\underset{\diagdown\,O\,\diagup}{C{-}CH_2}$, and 0.075 g of Al.

Reactions of alkenes **IV**–**VI** with the system **I**–**II** and the analysis of the reaction products were performed similarly. The IR spectra of nonvolatile residues after oxidation of diphenyl-substituted propenes contain an absorption band at 1660 cm^{-1} characteristic of the carbonyl stretching vibrations of benzophenone. This fact proves that benzophenone is formed before the hydrolysis. The melting point of authentic benzophenone 2,4-dinitrophenylhydrazone and the mixed sample is 236°C.

The presence of epoxy compounds in the products was proved by the qualitative reactions such as the coloration with pyridine on heating [27], formation of a precipitate of silver iodate [28], and the Deckert test [29]. Quantitative determination of epoxides was performed according to [30].

Hydroperoxides **IX** and **X** were identified by paper chromatography [1], and also by formation of phenol and phenylalkenyl ketones in the presence of *p*-toluenesulfonic acid [4].

The decomposition products were analyzed qualitatively and quantitatively.

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