2. Conditions were found ensuring a 98-99% selectivity of the conversion of C_2H_4 to acetaldehyde, and C_3H_6 to acetone.

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COTELOMERIZATION OF ETHYLENE AND ISOBUTYLENE WITH METHYL ISOBUTYRATE

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A study of telomerization and cotelomerization of various monomers with telogens, which react by rupture of the C−Cl bond, shows that the polar properties of the growing radicals and reacting molecules exert a considerable influence on the distribution of the reaction products. In this connection the most significant results are obtained when monomers containing substituents with different polarities, e.g., propylene and vinyl chloride, are used in cotelomerization [1]. Reactions with methyl isobutyrate in which a C−H bond is ruptured are considerably less sensitive to the polar properties of the reactants. In this case the course of the reaction is determined mainly by the structure of the growing radicals (primary, secondary) from the point of view of its effect on their ability to join onto a monomer and participate in a chain-transfer reaction with a telogen. The results of telomerization of ethylene and propylene with methyl isobutyrate showed that the yield of both homo- and cotelomers having ethylene as the last monomer unit is greater by a factor of 2.5-3 than compounds containing a propylene monomer unit at the end. This is due to the greater difficulties in the growth and chain-transfer stages in the latter [2]. It seemed interesting to compare, in cotelomerization with methyl isobutyrate, the monomers ethylene and isobutylene, which differ in structure still more. In the last, the growing radicals are tertiary and sterically hindered, which should cause a reduction in their capability for chain transfer

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TABLE 1. Data for Cotelomerization of Isobutylene (M_1) and Ethylene (M_2) with Methyl Isobutyrate (S) [150°, 0.15 mole % of tert-butyl peroxide (TBP), 40 min]

Expt. No.	S	M_1	M_2	$\frac{M_1}{S}$	$\frac{M_2}{S}$	$\frac{M_1}{M_2}$	Conversion, %		P*, %	Content of fractions T_1^e-T_3^i, %	Content of components in fraction $T_1^e-T_3^i$, mole %†																	
	mmoles						M_1	M_2			T_1^e	T_1^i	T_2^e	P_0	T_2^{ei}	T_2^{ie}	T_3^e	T_2^i	T_3^{eie}	T_3^{iee}	T_3^{eei}	P_1	T_4^e	T_3^{eii}	Φ_1‡	Φ_2‡	$T_5^e+T_3^i$
1	51,1	12,9	3,2	0,25	0,063	4,0	17,7	4,2	3,3	15,1	33,4	11,1	6,0	6,8	10,0	9,3	0,9	3,2	4,1	1,5	1,2	1,8	0,3	2,7	3,8	3,3	0,6
2	50,9	13,2	6,8	0,26	0,134	2,0	16,5	7,2	5,1	15,5	35,4	6,8	9,9	5,1	8,4	9,1	2,3	1,8	5,5	2,1	1,7	1,3	0,7	1,8	3,6	4,2	0,3
3	51,1	13,2	13,5	0,26	0,26	1,0	18,2	16,8	6,7	16,7	35,1	3,4	15,3	3,4	5,2	7,7	5,9	0,7	5,8	2,8	1,7	0,7	2,3	0,8	3,4	5,0	0,8
4	50,7	12,9	25,7	0,25	0,51	0,5	16,6	16,3	8,2	16,4	29,2	1,7	19,1	2,3	2,8	6,5	10,7	0,3	4,7	3,4	1,2	0,3	5,7	0,3	3,3	5,6	2,9
5	51,3	26,4	13,5	0,51	0,26	2,0	13,0	4,9	7,8	9,0	23,3	7,1	8,9	3,7	6,5	14,0	3,2	3,5	6,1	4,1	1,5	1,5	1,3	1,4	4,5	8,5	0,9
6	51,0	26,4	26,4	0,52	0,52	1,0	20,8	15,9	10,6	9,0	22,7	4,9	11,7	2,1	3,6	14,0	5,9	1,6	5,5	6,6	1,5	1,0	3,0	0,6	4,7	9,3	1,3
7	98,4	52,5	52,5	0,53	0,53	1,0	38,4	35,8	16,7	–	14,7	3,0	8,9	5,2	5,8	9,9	6,6	0,6	8,5	4,9	2,7	2,7	3,8	1,7	6,8	12,5	1,7

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* Content of products in the reaction mixture.

† T_1^e, T_1^i, T_2^{ei}, etc., are homo- and cotelomers with n monomer units; the index denotes the sequence of monomer units in the molecule.

‡ Content of T_3^{iei} = 2.5 (Φ_1) and T_3^{iie} = 3.4 (Φ_2), calculated from PMR spectra of the pertinent fractions isolated from the reaction mixtures of preparative experiment 7, conducted in a stainless-steel autoclave with a volume of about 28 ml in the presence of 2.5 mole % TBP on loaded telogen; reaction time 2 h.

and changes in the distribution of homo- and cotelomers. The reactions take place according to the general scheme

$$(CH_3)_2CHCO_2CH_3 + nCH_2{=}CH_2 + m(CH_3)_2C{=}CH_2 \xrightarrow{R\dot{O}}$$

$$\begin{array}{l} \longrightarrow H(CH_2CH_2)_nC(CH_3)_2CO_2CH_3(T_n{}^{e}) + H[(CH_3)_2CCH_2]mC(CH_3)_2CO_2CH_3(T_m{}^{i}) \\ \qquad\qquad\qquad\qquad \text{(homotelomers)} \\ \longrightarrow H\{(CH_2CH_2)_n[(CH_3)_2CCH_2]_m\}C(CH_3)_2CO_2CH_3(T_{n+m}) \\ \qquad\qquad\qquad\qquad \text{(cotelomers)} \end{array}$$

The experimental data obtained during a study of cotelomerization of ethylene and isobutylene with methyl isobutyrate (Table 1) show that in this system the basic mechanisms in the distribution of homo- and cotelomers observed in the reaction of methyl isobutyrate with ethylene and propylene are retained [2]. The particular difficulties in interpreting the results obtained in the present work are associated with the lack of a precise link between the variation in the ratios M_1/M_2 and M/S and the kind of changes in the distribution of the reaction products. Thus, with $M_1/M_1 = 1.0$ the change in M/S (0.26 in experiment 3 and 0.52 in experiment 6 in Table 1) had an effect not only on the ratio of homotelomers but also affected the ratio of cotelomers containing an identical number of monomer units (for example, $T_2{}^{ei}/T_2{}^{ie}$, $T_3{}^{eie}/T_3{}^{iee}$) which was not seen in the cotelomerization of ethylene and propylene with methyl isobutyrate. The course of the reaction will be examined for the example of experiment 6, where the ratio of filling the autoclave with the reactants is about 97%, which eliminates the possible effect of a heterophase system on the type of distribution of the cotelomerization products. The total yield of homotelomers of isobutylene is lower by a factor of 6.7 than the ethylene homotelomers (against 3 in favor of ethylene homotelomers in the ethylene–propylene system). The content of homo- and cotelomers which have a last ethylene unit (T – E) is higher by a factor of 3.5–4 than for cotelomers with the same number of monomer units but terminating in isobutylene (T – I). Thus, $T_2{}^{e}/T_2{}^{ei} \approx 3.3$; $T_3{}^{e}/T_3{}^{eei} \approx 3.9$; $T_2{}^{ie}/T_2{}^{ei} \approx 3.9$. On the other hand, the yields of products with the same number of monomer units which have ethylene as the last unit are approximately the same: $T_2{}^{e}/T_2{}^{ie} \approx 0.8$; $T_3{}^{e}/T_3{}^{eie} \approx 1.1$; $T_3{}^{eie}/T_3^{iee} \approx 0.8$. Infringement of these relations occurs in a case in which a cotelomer includes in succession two isobutylene units, which reflects the difficulties at the growth stage of the chain. A similar pattern is also observed for the ethylene–propylene system. The fact that the ratio of the product yields T – E/T – I is even somewhat higher than T – E/T – P (2.5–3 times) for the ethylene–propylene system confirms the hypothesis which we stated that the reactivity of the radicals at the chain-transfer stage, caused by their structure, plays a considerable part in this. The more stable, sterically hindered tertiary radicals with a terminal isobutylene unit transfer the chain worse in a reaction with methyl isobutyrate than primary radicals with a terminal ethylene monomer unit and secondary radicals with a terminal propylene monomer unit (in the ethylene–propylene system) [2]. This agrees with findings on the copolymerization of ethylene ($r_1 = 3.1$) and propylene ($r_2 = 0.77$), ethylene ($r = 2.6$) and isobutylene ($r_2 = 0.56$) [3]. Among the T_3 cotelomers, compounds are preferentially formed containing two ethylene and one isobutylene monomer unit; the fraction of the alternated cotelomer $T_3{}^{eie}$ amounts to about 40% of the total cotelomers $T_3{}^{eie}$, $T_3{}^{iee}$, and $T_3{}^{eei}$. In the range of reactant ratios studied their variation, like the methyl isobutyrate–ethylene–propylene system also, has a substantial effect only for homotelomers, while the change in the distribution and content of cotelomers is less pronounced. It may be pointed out, however, that the ratios $M_1/M_2 = 2.0$; $M_1/S = 0.51$; $M_2/S = 0.26$ (see Table 1, Expt. 5) are optimum for preparing cotelomers with n = 2 and 3. Among the reaction products the unsaturated compounds P_0 and P_1 have also been identified with the general formula

$$CH_2{=}C(CH_3)CH_2[(CH_3)_2CCH_2]_nC(CH_3)_2CO_2CH_3$$

(n = 0, 1). They are formed as the result of the recombination of growing radicals with isobutenyl radicals [4]. The composition and structure of the homo- and cotelomers have been confirmed by ultimate analysis and the ^1H and ^{13}C NMR spectra (Table 2).

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra of the substances isolated (~ 30% solutions in CCl_4) were taken in Hitachi–Perkin–Elmer R-20 (60 MHz) and Bruker–Physik HX-90 (22.635 MHz) spectrometers. The chemical shifts of the signals were calculated relative to TMS. Analytical GLC: stainless-steel column, 3000 × 3 mm with 15% Carbowax 20M on Chromaton N-AW, 0.16-0.20 mm, temperature programming from 100°C with rate 4 deg/min, flow rate 55 ml/min, katharometer. The content by weight of unreacted telogen and the reaction products was calculated from the areas of the peaks corresponding to the components by the internal standards method. The telogen reference spot was methyl propionate. The reaction products reference spot was methyl α-butylcaproate. The conversion of ethylene and isobutylene was determined from the amount of unreacted gases and their

TABLE 2. Data on ^1H and ^{13}C NMR Spectra and Ultimate Analysis of Cotelomers (δ, ppm on TMS)

Compound	NMR spectrum	COO	CH_3O	C	$\underline{CH}CH_3$	$\underline{CH_3}C$	$\underline{CH_3}CH$	$\underline{CH_3}CH_2$	CH_2	Found/Calculated, %	
										C	H
$\overset{7}{(CH_3)_2}\overset{6}{C}H\overset{5}{C}H_2\overset{4}{C}H_2\overset{3}{C}H_2\overset{2}{C}\overset{3'}{(CH_3)_2}\overset{1}{C}O_2CH_3\ (T_2^{ei})$ (I)	^1H	–	3,60 s	–		1,13 s	0,88 d	–	1,44 m	71,08/70,92	12,00/11,91
	^{13}C	176,2	50,9	41,8	27,6	25,2	22,6	–	40,8 (3), 22,6 (4), 39,3 (5)		
$\overset{6}{C}H_3\overset{5}{C}H_2\overset{4}{C}\overset{5'}{(CH_3)_2}\overset{3}{C}H_2\overset{2}{C}\overset{3'}{(CH_3)_2}\overset{1}{C}O_2CH_3\ (T_2^{ie})$ (II)	^1H	–	3,60 s	–		1,17 s, 0,89 s	–	0,89 m	1,59 s 1,23 m	70,98/70,92	11,99/11,91
	^{13}C	177,0	51,0	41,6 (2), 33,9 (4)	–	27,0 (3′) 27,9 (5′)	–	8,3	51,0 (3), 36,4 (5)		
$\overset{8}{C}H_3\overset{7}{C}H_2\overset{6}{C}\overset{7'}{(CH_3)_2}\overset{5}{C}H_2\overset{4}{C}H_2\overset{3}{C}H_2\overset{2}{C}\overset{3'}{(CH_3)_2}\overset{1}{C}O_2CH_3\ (T_3^{eie})$ (III)	^1H	–	3,61 s	–		1,14 s, 0,85 s	–	0,85 m	1,25 m	73,23/72,84	12,16/12,23
	^{13}C	176,5	50,8	41,8 (2), 32,7 (6)	–	25,2 (3′), 26,6 (7′)	–	8,2	42,0 (3), 19,2 (4), 42,0 (5), 34,0 (7)		
$\overset{8}{C}H_3\overset{7}{C}H_2\overset{6}{C}H_2\overset{5}{C}H_2\overset{4}{C}\overset{5'}{(CH_3)_2}\overset{3}{C}H_2\overset{2}{C}\overset{3'}{(CH_3)_2}\overset{1}{C}O_2CH_3\ (T_3^{iee})$ (IV)	^1H	–	3,60 s	–		1,15 s, 0,86 s	–	0,86 m	1,59 s, 1,18 m	72,85/72,84	11,91/12,23
	^{13}C	177,0	50,9	41,6 (2) 33,8 (4)	–	27,6 (3′), 27,9 (5′)	–	14,1	51,5 (3), 44,1 (5) 26,1 (6), 23,5 (7)		
$\overset{9}{(CH_3)_2}\overset{8}{C}H\overset{7}{C}H_2\overset{6}{C}H_2\overset{5}{C}H_2\overset{4}{C}H_2\overset{3}{C}H_2\overset{2}{C}\overset{3'}{(CH_3)_2}\overset{1}{C}O_2CH_3\ (T_3^{eei})$ (V)	^1H	–	3,61 s	–		1,14 s	0,87 d	–	1,22 m	73,01/72,84	11,71/12,23
	^{13}C	177,0	51,0	41,8	27,9	25,2	22,6	–	40,8 (3), 24,8 (4), 30,2 (5), 27,1 (6), 38,9 (7)		
$\overset{9}{(CH_3)_2}\overset{8}{C}H\overset{7}{C}H_2\overset{6}{C}\overset{7'}{(CH_3)_2}\overset{5}{C}H_2\overset{4}{C}H_2\overset{3}{C}H_2\overset{2}{C}\overset{3'}{(CH_3)_2}\overset{1}{C}O_2CH_3\ (T_3^{eii})$ (VI)	^1H	–	3,64 s	–		1,14 s, 0,86 s	0,91 d	–	1,21 m	–	–
	^{13}C	176,2	50,8	41,9 (2), 33,5 (6)	27,8	25,2 (3′), 27,8 (7′)	23,9	–	41,5 (3), 19,3 (4), 42,8 (5), 50,8 (7)		

chromatography (stainless-steel column, 3000 × 3 mm with Chromosorb-102, 80-100 mesh, temperature programming from 100° with rate 25 deg/min, helium 55 ml/min, katharometer). Preparative GLC: stainless-steel column, 2600 × 9 mm with 15% Carbowax 20M on Chromaton N-AW, 0.16-0.20 mm, temperature programming from 125° at rate of 1 deg/min, helium rate 135 ml/min, katharometer. The experiments (the conditions and results of the GLC analysis for which are shown in Table 1) were run in stainless-steel autoclaves of about 10-ml capacity by the procedure described in [5]. To isolate the reaction products, 12 experiments were carried out under the conditions of Expt. 7 in Table 1. After distilling off unreacted telogen the residue, amounting to 20 g, was fractionated by vacuum distillation and the products were isolated from narrow fractions by preparative GLC. Identification of the ethylene homotelomers as well as those from isobutylene and the corresponding unsaturated compounds P_0 and P_1 was carried out by GLC according to the well-known examples produced in [4, 5].

The PMR spectra of compounds I, V, and VI (see Table 2) contain a doublet due to the CH_3 protons in the $(CH_3)_2CH$ group, which allowed them to be ascribed to cotelomers with a terminal isobutylene group. In the chromatogram of the cotelomerization reaction mixture, between the homotelomers T_2^e and T_3^e are located the components (I) and (II), which correspond, according to ultimate analysis, to T_2 cotelomers which include one ethylene and one isobutylene unit. The presence of a doublet at 0.88 ppm in the PMR spectrum of component (I) identifies it as T_2^{ei}. At the same time, in the ^{13}C NMR spectrum of component II there is a signal due to the methyl in the ethyl group with the quaternary carbon atom [6], which shows it to be T_2^{ie}. Between the homotelomers T_3^e and T_4^e are located compounds (III), (IV), and (V) corresponding in ultimate composition to T_3 cotelomers with an isobutylene and two ethylene units. Compound (V), as already stated, has a terminal isobutylene unit, i.e., it is T_3^{eei}. The choice between compounds (III) and (IV) was made on the basis of the ^{13}C NMR spectra. In the spectrum of (III) there is a signal due to the methyl in the ethyl group with the quaternary carbon atom, similarly to (II), which allowed this to be ascribed to T_3^{eie}. The spectrum of compound (IV) contains a signal due to the terminal methyl group in the long unbranched carbon chain and, thus, this compound is T_3^{iee}.

From the cotelomerization reaction mixture, component (VI) was isolated, and, according to the PMR spectral data, it contains a terminal isobutylene group (see Table 2). The presence in the ^{13}C NMR spectra of a signal due to the methylene group with δ 19.3 ppm, similar to that from the CH_2 group in (III) (δ 19.2 ppm), besides other typical signals (see Table 2), enabled us to identify it as T_3^{eii}. The investigation of fractions Φ_1 and Φ_2 was particularly interesting. Their GLC consist of groups of broadened, poorly separated peaks. On the chromatogram these fractions are located between T_4^e and T_5^e instead of in the most probable place for finding cotelomers T_3^{iei} and T_3^{iie}. The task of identifying these components is greatly complicated because, in this region, T_4 cotelomers isomeric with them may be located which contain one isobutylene and two ethylene units (T_4 cotelomers containing two isobutylene and two ethylene units from the type of distribution of the cotelomerization products should be situated beyond T_5^e). According to the PMR spectrum, fraction Φ_2 represents a mixture including methyl esters of branched carboxylic acids with the general formula $C_{13}H_{27}COOCH_3$, which corresponds to the cotelomers indicated above. The ratio of the integrated strengths of the protons in the CH_3O group to the protons in the acyl part of the molecule is 3 : 27. The presence of singlets due to the two CH_2 groups located between the quaternary carbon atoms (δ 1.59 and 1.68 ppm) and the signals due to the terminal methyl groups (δ 8.4 and 14.1 ppm) in the ^1H and ^{13}C NMR spectra respectively shows the presence in this fraction of T_3^{iie}, T_4^{ieee}, and T_4^{eeie}. Besides this, in the PMR spectrum there are weak signals due to the CH_2 group near the double bond (δ 4.70 ppm) and the methyl protons of the $CH_2 = C(CH_3)$ group (δ 1.94 ppm) [4] belonging evidently to $CH_2 = C(CH_3)CH_2C(CH_3)_2CH_2CH_2CH_2C(CH_3)_2CO_2CH_3$. Fraction Φ_1, according to the PMR spectrum, is also a mixture of methyl esters, but the ratio of the integrated strengths of the CH_3O group protons to the protons in the acyl part of the molecule is in this case 3 : 31, indicating an admixture of hydrocarbons, evidently cooligomers of ethylene and isobutylene. The singlet at δ 1.59 ppm in the PMR spectrum and the signals at 14.1 and 19.3 ppm in the ^{13}C NMR spectrum show that Φ_1 includes both T_3^{iei} and T_4^{eiee}.

CONCLUSIONS

1. In the cotelomerization of ethylene and isopropylene with methyl isobutyrate, homotelomers and every possible cotelomer with two and three monomer units in the molecule have been identified.

2. The preferential formation of cotelomers with terminal ethylene groups is due in the system under consideration to the greater reactivity of the pertinent radicals at the chain-transfer stage.

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DECOMPOSITION OF TRICYCLOHEXYLIDENE PEROXIDE

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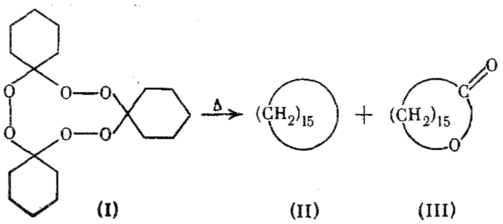
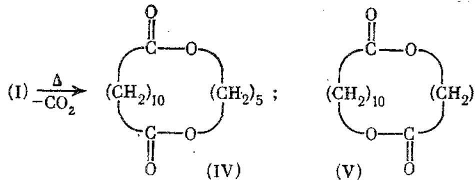
Cycloalkanes and lactones [2, 3] are formed in the thermal and photolytic decomposition of cyclic peroxides which are synthesized from cycloalkanones and H_2O_2 [1] Thus, the thermal decomposition of tricyclohexylidene peroxide (I) leads to cyclopentadecane (II) and the hexadecanolide (III) in amounts of 0.15 and 0.25 mole per mole of (I):



In the present work the thermolysis of (I) has been investigated with the aim of establishing the structure of the "dilactone" postulated in [4] and answering some questions related to the reaction mechanism. The composition and yield of the products obtained in the thermolysis of (I) in octanoic acid at 180°C are given in Table 1. These data show that the dilactone (IV) is formed together with the macrolactone (III) [(III) : (IV) ≈ 4:1]



The structure of the preparatively isolated dilactone (IV) was confirmed by ^1H and ^{13}C NMR and by IR and mass spectrometry. 1,10-Decanedicarboxylic acid is formed in the hydrolysis of (IV). However, the pres-

TABLE 1. Products of the Thermal Decomposition of Tricyclohexylidene Peroxide (I) in Octanoic Acid*

Products	Yield, moles/ mole peroxide	Products	Yield, moles/ mole peroxide
Cyclopentadecane	0,40	Cyclopentane	0,04
Hexadecanolide	0,12	Hexanoic acid	0,03
Dilactones (IV) and (V)	0,03	ε-Caprolactone	0,08
Cyclohexanone	0,19	CO_2	1,60

*Oxygen and pentadecene were identified qualitatively.

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