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Effect of Solvents on the Rate of Epoxidation of α-Pinene and Δ^3-Carene with Peroxydecanoic Acid

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Abstract—Reaction of epoxidation of α-pinene and Δ^3-carene with peroxydecanoic acid in various organic solvents was studied. Effective activation energies of oxidation of α-pinene and Δ^3-carene with peroxydecanoic acid in various media are evaluated. It is shown that reaction medium significantly affects the rate of the process. Correlation dependences connecting the rate of epoxidation with main parameters of solvents are found.

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Reactions of monoterpene hydrocarbons with peroxyacids are typical electrophilic processes [1, 2]. Nowadays it is reliably established that epoxidation of pinenes and carenes proceeds stereoselectively [3]. The effect of solvent in these reactions is poorly studied though it may be expected that the reaction medium would influence the rate of the process as well as the yield of reaction products. Therefore the investtigation of solvent effect on the rate of epoxidation of monoterpene hydrocarbons should give useful information for optimal performance of the process.

We report here on the results of investigation of effect of the reaction medium on the rate of α-pinene and Δ^3-carene epoxidation with peroxydecanoic acid and the correlation equations connecting the rate constant and the basic properties of solvents.

By the preliminary experiments it was established that the reaction has first order with respect to each component, hence, overall second order. In the first stage of the reaction the intermediate complex between the terpenoid and peroxydecanoic acid is quickly formed, and this reaction can be regarded as the equilibrium one. In the second, rate-determining stage the intermediate complex decomposes to give the corresponding epoxide and decanoic acid. Proceeding from the above reaction mechanism it may be expected that kinetic curves will be linear in the coordinates ln (c_0/c_t) vs time (t). The slope these dependences permits calculation the rate constants of the pseudounimolecular reaction (k). Electrophilic oxidation of pyridine and DMSO with peroxyacids proceeds according to analogous mechanism [4, 5]. The epoxidation of terpenoids under investigation is stereoselective. High stereoselectivity of the process is ascribed to the shielding effect of *gem*-dimethyl group which prevents the attack of peroxy acids from β-region [3]. Kinetic curves of epoxidation of Δ^3-carene with peroxydecanoic acid in some solvents are presented in the Fig. 1. Dependence of ln (c_0/c_t) on t was linear up to high degrees of conversion, though in [3] in the case of epoxidation of α-pinene with peroxyacids the linear dependence was observed only in the initial stages of the process.

Yet in our experiments we do not observe any decrease in the reaction rate with the increase in conversion (Fig. 2) which may indicate the absence of significant influence of the reaction products on the rate of the process (Figs. 1, 2). Besides, in our experi-

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† Deseased.

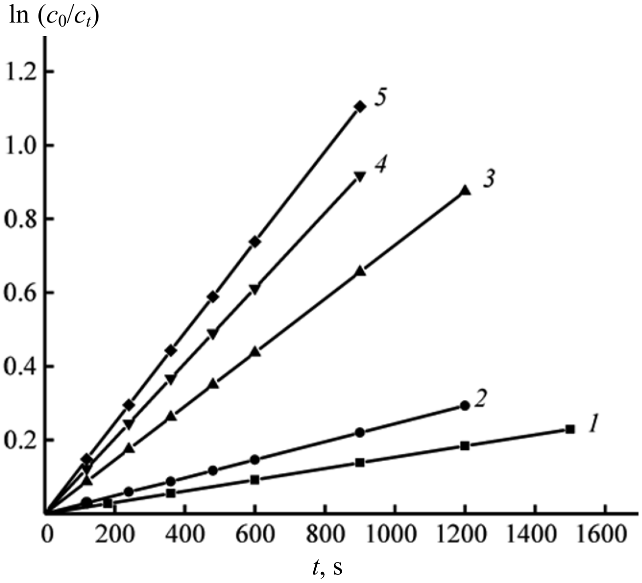

Fig. 1. Kinetic curves of epoxidation of Δ^3-carene with peroxydecanoic acid in organic solvents: (*1*) dioxane, (*2*) propanol-2, (*3*) toluene, (*4*) acetic acid, and (*5*) benzene. $T = 308$ K.

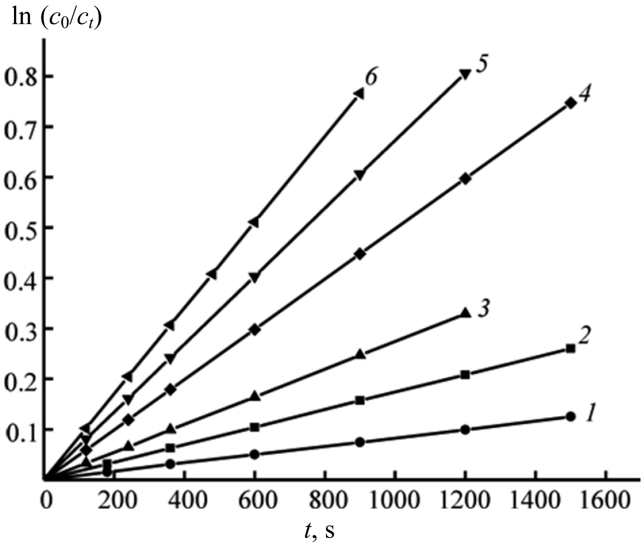

Fig. 2. Kinetic curves of epoxidation of α-pinene with peroxydecanoic acid in organic solvents: (*1*) ethyl acetate, (*2*) acetone, (*3*) dioxane, (*4*) toluene, (*5*) dichloroethane, and (*6*) acetic acid. $T = 308$ K.

ments epoxidation with peroxydecanoic acid was not complicated with the reactions of ring opening and isomerization of obtained products as it was expected in [3] in the case of α-pinene. In Tables 1, 2 the pseudounimolecular rate constants of epoxidation of α-pinene and Δ^3-carene with peroxydecanoic acid calculated for the initial stages of the process in the solvents under investigation are presented.

It follows from these data that the nature of the solvent influences the rate of the process. For example, at the performing of oxidation of Δ^3-carene with peroxydecanoic acid in dioxane instead of dichloroethane almost a 10-fold decrease in pseudounimolecular rate constant is observed.The oxidation of α-pinene with peroxydecanoic acid has the highest rate in nitrobenzene, while in acetone this rate is significantly

Table 1. Effective pseudounimolecular reaction rate constants of epoxidation of α-pinene with peroxydecanoic acid in the solvents under study (starting concentrations of reagents 0.05 mol/L)

Solvent	$k\times10^5$, s^{-1}, Δk ±0.04 k					E_a, kJ/mol
	303 K	308 K	313 K	318 K	323 K	
Dichloroethane	43.2	49.8	58.3	63.0	79.3	24.0
Benzene	68.6	82.3	100	121	143	29.7
Chlorobenzene	59.4	73.5	94.8	118	149	39.9
Tetrachloromethane	74.6	87.6	106	129	168	31.0
Nitrobenzene	84.8	106	135	171	213	38.6
Acetic acid	58.9	85.2	125	162	257	57.9
Toluene	52.8	67.3	79.8	100	116	32.6
m-Xylene	48.3	59.5	77.2	94.0	112	34.9
Acetone	6.82	8.33	11.7	13.6	16.9	39.2
2-Propanol	14.7	22.4	35.0	56.0	87.5	64.2
Ethyl acetate	14.8	17.4	22.8	28.7	33.5	36.4
Dioxane	24.3	27.4	33.1	40.0	47.6	27.3

Table 2. Effective pseudounimolecular reaction rate constants of epoxidation of Δ^3-carene with peroxydecanoic acid in the solvents under study (starting concentrations of reagents 0.05 mol/L)

Solvent	$k\times10^5$, s^{-1}, Δk ±0.04 k					E_a, kJ/mol
	303 K	308 K	313 K	318 K	323 K	
Dichloroethane	113	128	146	165	185	20.4
Benzene	99.5	123	157	205	253	40.4
Chlorobenzene	88.2	114	145	188	240	39.5
Tetrachloromethane	103	111	135	149	175	23.2
Nitrobenzene	85.6	108	132	169	206	35.5
Acetic acid	85.1	102	128	152	183	31.6
Toluene	54.1	72.9	104	136	168	49.6
m-Xylene	55.7	69.0	86.3	103	120	32.0
Acetone	20.3	25.2	33.4	41.6	55.2	39.5
2-Propanol	19.6	24.4	32.3	40.3	53.3	39.5
Ethyl acetate	12.5	16.7	25.6	34.5	52.7	57.1
Dioxane	11.9	15.3	20.0	25.7	33.6	40.9

lower (Table 1). Overall activation energies of both processes vary in the range 20.4–64.2 kJ/mol and agree with the values reported for epoxidation of ethylene hydrocarbons with peroxyacids [1–3].

Note that epoxidation of Δ^3-carene in the solvents under study proceeds faster than the reaction of α-pinene with peroxydecanoic acid save the cases of dioxane and ethyl acetate (Tables 1, 2). On the basis of obtained kinetic data the parameters of the transition state for both processes were calculated (Table 3).

An interconnection was found between $\Delta H^\#$ and $\Delta S^\#$ values of the transition state of the reactions under study. This means that under the conditions of our experiments in the oxidation of Δ^3-carene and α-pinene with peroxydecanoic acid a compensation effect is observed. $\Delta S^\#$ values are negative indicating the greater ordering of the transition state as compared to starting reagents which results in a high stereoselectivity of the process. $\Delta G^\#$ values of transition states for α-pinene are close. They vary within the limits 92.1–98.0 kJ/mol. For Δ^3-carene this parameter is lower and lies between 89.1–97.4 kJ/mol. The epoxidation of Δ^3-carene with peroxydecanoic acid proceeds faster than that of α-pinene, which is confirmed by the values of pseudounimolecular rate constants and $\Delta G^\#$ values of transition state.

For the establishing the dependence of pseudounimolecular rate constant of oxidation (k) on the main parameters of solvents the well known Eq. (1) [11–15] was used.

$$\log k = a_0 + a_1(n^2 - 1)/(n^2 + 2) + a_2(\varepsilon - 1)/(2\varepsilon + 1) + a_3B + a_4E_T + a_5\delta^2 + a_6V_M. \quad (1)$$

In Eq. (1) n and ε are the refraction index and the dielectric constant of solvent determining their polarizability and polarity, responsible for the nonspecific solvation. Parameters B (basicity according to Palm [16]) and E_T (electrophilicity according to Reichardt [17]) determine the ability of solvents to the acid-base interaction, that is, the specific solvation. Values of δ and V_M, the Hildebrandt solubility parameter whose square is proportional to the cohesion energy of the medium, and molar volume of solvent respectively reflect the influence of structural factors. Characteristics of solvents were taken from the reviews [13, 14], and the calculation algorithm, from the recommendations of the chemical correlation analysis group of IUPAC [15].

Generalization of data on oxidation of Δ^3-carene in 12 solvents at 308 K (Table 4) leads to the six-parametric equation with the coefficients of pair correlation r_i 0.657, –0.1117, 0.2286, 0.8808, –0.1054, and 0.1483. Starting multiple correlation coefficient is rather large, $R = 0.9929$, the mean-square deviation $S = 0.0430$.

$$\log k = 0.4697 + (4.3915\pm1.0712)f_1(n^2) + (-0.6956\pm0.2186)f_2(\varepsilon) + (-0.2\pm0.5)\times10^{-3}B + (-3.8\pm0.4)\times10^{-3}E_T + (0.0336\pm0.0061)\delta^2 + (-5.0\pm1.5)10^{-3}V_M. \quad (2)$$

Rate of oxidation of Δ^3-carene is satisfactorily described by two-parametric Eq. (3).

Table 3. Kinetic and activation parameters of the reactions of oxidation of Δ^3-carene and α-pinene with peroxydecanoic acid[a]

Solvent	Δ^3-Carene					α-Pinene				
	$k\times10^5$,[b] s^{-1}	E_a, kJ/mol	$\Delta H^\#$, kJ/mol	$-\Delta S^\#$, J mol^{-1} K^{-1}	$\Delta G^\#$, kJ/mol	$k\times10^5$,[b] s^{-1}	E_a, kJ/mol	$\Delta H^\#$, kJ/mol	$-\Delta S^\#$, J mol^{-1} K^{-1}	$\Delta G^\#$, kJ/mol
Dichloroethane	165	20.4	17.9	236	89.4	63.0	24.0	21.4	228	93.9
Benzene	205	40.4	37.9	169	89.3	121	29.7	27.1	205	92.3
Chlorobenzene	188	39.5	36.9	173	89.4	118	39.9	37.3	173	92.3
Tetrachloromethane	149	23.2	20.8	225	89.1	129	31.0	28.4	200	92.1
Nitrobenzene	169	35.5	32.9	183	91.3	171	38.6	36.0	174	91.3
Acetic acid	152	31.6	29.1	199	89.2	162	57.9	55.4	135	96.2
Toluene	136	49.6	47.0	146	91.1	100	32.6	30.0	197	92.7
m-Xylene	103	32.0	29.4	199	92.6	94.0	34.9	32.3	191	93.0
Acetone	41.6	39.5	36.9	186	93.2	13.6	39.2	36.6	193	98.0
2-Propanol	40.3	39.5	36.9	184	92.7	56,0	64.2	61.6	103	94.2
Ethyl acetate	34.5	57.1	54.6	130	93.9	28.7	36.4	33.8	196	95.9
Dioxane	25.7	40.9	38.9	185	97.4	40.0	27.3	24.7	222	95.0

[a] $\Delta H^\#$, $\Delta S^\#$, $\Delta G^\#$ are calculated for 318 K. [b] Temperature 318 K. E_a is determined with the accuracy ±5.0 kJ/mol.

Table 4. Experimental and calculated [Eq. (2)] values of logarithms of rate constants of epoxidation of Δ^3-carene with peroxydecanoic acid

Solvent	308 K			318 K		
	5+log *k* (calculated)	Δlog *k*	5+log *k* (experiment)	5+log *k* (calculated)	Δlog *k*	5+log *k* (experiment)
Dichloroethane	2.1046	–0.0026	2.1074	2.2600	0.0425	2.2175
Benzene	2.0445	–0.0454	2.0899	2.2211	–0.0907	2.3118
Chlorobenzene	2.0747	0.0178	2.0569	2.2751	0.0009	2.2742
Tetrachloromethane	2.0675	0.0221	2.0453	2.2165	0.0433	2.1732
Nitrobenzene	2.0533	0.0199	2.0334	2.2521	0.0242	2.2279
Acetic acid	2.0212	0.0126	2.0086	2.1948	0.0129	2.1818
Toluene	1.9009	0.0381	1.8627	2.1119	–0.0216	2.1335
m-Xylene	1.1625	–0.0764	1.8388	2.0013	–0.0116	2.0128
Acetone	1.3346	–0.0668	1.4014	1.5768	–0.0423	1.6191
2-Propanol	1.3747	–0.0127	1.3874	1.5722	–0.0331	1.6053
Ethyl acetate	1.2868	0.0641	1.2227	1.5459	0.0081	1.5378
Dioxane	1.2138	0.0291	1.1847	1.4773	0.0673	1.4099

$$\log k = 1.2685 + (-4.8\pm0.4)\times10^{-3}E_\mathrm{T} + (0.0259\pm0.0058)\delta^2,\ N\ 12;\ R\ 0.9593;\ S\ 0.1021. \quad (3)$$

Values log *K* and their deviation from experimental data are listed in the Table 4.

Finally, the factor determining and intensifying the process is the electrophilicity of medium (E_T). For the achievement of value of multiple correlation coefficient $R > 0.9859$ the consideration of the cohesion energy density of the medium δ^2, increasing the rate of the process, of its polarizability *n* and of electrophilicity E_T decreasing the reaction rate is necessary. Basicity *B* and molar volume of solvent V_M in this case are insignificant.

Analogously to the preceding case the multi-parametric equations for oxidation of Δ^3-carene at 318 K were obtained.

$$\log k = 0.4027 + (5.2529\pm1.0826)f_1(n^2) + (-0.5337\pm0.2209)f_2(\varepsilon) + (-0.8\pm0.5)\times10^{-3}B + (3.0\pm0.4)\times10^{-3}E_\mathrm{T} + (0.0351\pm0.0061)\delta^2 + (-4.2\pm1.5)\times10^{-3}V_\mathrm{M},\quad (4)$$
$$N\ 12;\ R\ 0.9914;\ S\ 0.0434.$$

The rate determining factors are the polarity of medium and the refractive index. Their exclusion leads to decrease in the correlation coefficient to 0.9550 giving a two-parametric equation. B and V_M factors are insignificant.

$$\log k = 1.5852 + (-4.4\pm0.4)\times10^{-3}E_T + (0.0219\pm0.0056)\delta^2,\ N\ 12;\ R\ 0.9550;\ S\ 0.0983. \qquad (5)$$

Analogous results were obtained by the generalizetion of data of Table 5 for epoxidation of pinene at 308 K. Six-parametric equation with the sufficiently high R value, 0.9739, was obtained. None of the considered parameters of solvents has the determining influence on log k value because the values of pair correlation coefficients (r_i) vary in the limits of 0.7587–0.7497. Correlation between log k values in 12 solvents and their properties permits to obtain the expression (6).

$$\begin{gathered}\log k = -1.2971 + (6.2414\pm1.9475)f_1(n^2) \\ + (-2.3744\pm0.3974)f_2(\varepsilon) \\ + (0.4\pm0.9)\times10^3B + (2.3\pm0.7)10^{-3}E_T \\ + (0.0536\pm0.0110)\delta^2 + (1.1\pm2.8)\times10^{-3}V_M, \\ N\ 12;\ R\ 0.9739;\ S\ 0.0781.\end{gathered} \qquad (6)$$

Exclusion of one of the parameters, for example, of themolar volume of solvent, from the six-parametric equation leads to Eq. (7) with the correlation coefficient $R = 0.9736$.

$$\begin{gathered}\log k = -1.2256 + (6.5979\pm1.7169)f_1(n^2) \\ + (-2.3177\pm0.3706)f_2(\varepsilon) + (0.3\pm0.9)\times10^{-3}B \\ + (2.2\pm0.7)\times10^{-3}E_T + (0.0518\pm0.0100)\delta^2, \\ N\ 12;\ R\ 0.9736;\ S\ 0.0786.\end{gathered} \qquad (7)$$

Characteristics determining the basicity and molar volume are insignificant. Their exclusion gives four-parametric equation with the insignificantly lower correlation (8).

$$\begin{gathered}\log k = -1.3765 + (7.1084\pm1.1227)f_1(n^2) \\ + (-2.2707\pm0.3530)f_2(\varepsilon) + (-2.0\pm0.4)\times10^{-3}E_T \\ + (54.6\pm7.0)\times10^{-3}\delta^2, \\ N\ 12;\ R\ 0.9732;\ S\ 0.0792.\end{gathered} \qquad (8)$$

Exclusion of the other parameters of correlation equation significantly decreases the correlation coefficient.

For the epoxidation of pinene at 318 K (Table 5) Eq. (9) with pair correlation coefficients

$$\begin{gathered}r_i = 0.6767, -0.2676, -0.0414, -0.6576, \\ -0.0697, 0.2333\end{gathered} \qquad (9)$$

was obtained. Multiple correlation coefficient R 0.9695, mean-square deviation s ±0.03. Data of the Table 5 in this case may be generalized by a six-parametric Eq. (10).

$$\begin{gathered}\log k = -1.5134 + (4.5395\pm2.0371)f_1(n^2) \\ + (-2.7659\pm0.4156)f_2(\varepsilon) + (1.3\pm0.9)\times10^{-3}B \\ + (-2.6\pm0.8)\times10^{-3}E_T + (0.0625\pm0.0115)\delta^2 \\ + (4.7\pm2.9)\times10^{-3}V_M, \\ N\ 12;\ R\ 0.9695;\ S\ 0.0817.\end{gathered} \qquad (10)$$

Analogously to the above-described cases excludeing of basicity B and molar volume of solvent V_M leads to expressions with R = 0.9637, S = 0.0890, and R = 0.9586, S = 0.0949 respectively. Signs of all the members of these equations are the same as in Eq. (6).

Table 5. Experimental and calculated [Eq. (2)] values of logarithms of rate constants of epoxidation of Δ^3-carene with peroxydecanoic acid

Solvent	308 K			318 K		
	5+log k (calculated)	Δlog k	5+log k (experiment)	5+log k (calculated)	Δlog k	5+log k (experiment)
Dichloroethane	1.7374	0.0402	1.6972	1.9209	0.1215	1.7993
Benzene	1.9503	0.0348	1.9154	2.0854	0.0026	2.0828
Chlorobenzene	1.9023	0.0306	1.8663	2.0639	–0.0080	2.0719
Tetrachloromethane	1.8613	–0.0812	1.9425	2.0141	–0.0965	2.1106
Nitrobenzene	1.9203	–0.1050	2.0253	2.1374	–0.0956	2.2330
Acetic acid	1.8853	–0.0451	1.9304	2.1578	–0.0517	2.2095
Toluene	1.8911	0.0630	1.8280	2.0635	0.0635	2.0000
m-Xylene	1.8517	0.0772	1.7745	2.0670	0.0939	1.9731
Acetone	1.0279	0.1073	0.9206	1.2142	0.0806	1.1335
2-Propanol	1.4043	0.0541	1.3502	1.7820	0.0339	1.7482
Ethyl acetate	1.1143	–0.1262	1.2405	1.3285	–0.1294	1.4579
Dioxane	1.3826	–0.0552	1.4378	1.5879	–0.0147	1.6021

Hence, insignificance of the effects of these factors on the reaction rate was established.

Different influence of the reaction medium on the epoxidation of α-pinene and Δ^3-carene is connected with different solvation of terpenoids leading to different dependences of ln k on physicochemical parameters of solvents. At the same time it is presumable that solvation of peroxydecanoic acid is the same in both processes.

EXPERIMENTAL

Synthesis of peroxydecanoic acid was carried out by the reaction of decanoic acid with hydrogen peroxide in the sulfuric acid medium [16]. Purification of peroxydecanoic acid was carried out by crystallization from hexane until the peroxyacid content reached 98.5–99.5%. Main admixture in all the samples of preparation was decanoic acid. Main advantage of peroxydecanoic acid is high stability to detonation and the possibility of obtaining samples of the high purity which is practically impossible for such well known epoxidating agent as peroxyacetic acid. Solvents used in our work were purified by known procedures [17, 18] and distilled under argon collecting the fractions with physicochemical constants corresponding to published data. α-Pinene (4,7,7-trimethylbicyclo[3.1,1]hept-3-ene) and Δ^3-carene (3,7,7-trimethylbicyclo[4.1.0]hept-2-ene) were isolated from turpentine and thoroughly purified by the procedure [19]. Content of α-pinene and Δ^3-carene in the samples under study according to GLC data was no less than 98%. The epoxidation of α-pinene and Δ^3-carene was carried out in glass reactor under argon. Solutions of terpenoid and peroxydecanoic acid were kept at definite temperature for 15–20 min and then quickly mixed marking the beginning of the reaction. After definite time intervals samples were taken, and peroxydecanoic acid content was evaluated in them iodometrically [20]. Error in the evaluation of reaction rate was no more than ±4%. In all kinetic experiments starting concentrations of α-pinene, Δ^3-carene and peroxydecanoic acid were the same, 0.05 mol/L. Rate of the process was found at five temperatures in the range 303–323 K.

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