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# Thermal and Oxidative Transformations of Polycyclohexadiene in the Solid Phase and Solution

V. E. AGABEKOV, ZH. D. CHAPLANOVA and YU I. GUDIMENKO

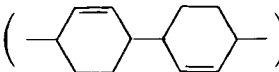
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The paper deals with the investigation of polycyclohexadiene oxidation mechanism in the solid phase and solution so as its inhibition. The schemes of these processes are proposed and kinetic parameters have been determined. Polymer degradation in solution is associated with transformation of peroxide radicals which lead to the formation of low-molecular products in the initial oxidation stage.

**KEYWORDS:** Polycyclohexadiene, thermodegradation, oxidation, solid state, solution, kinetics, mechanism

The possibility of using polycyclohexadiene (PCGD), the cyclohexadiene-1.3 oligomer, in the processes of microlithography<sup>1</sup> suggests a number of requirements concerning its thermal and oxidative stability upon storage and application. The features of the PCGD structure, namely: the presence of a tertiary carbon atom at a double bond in every monomer unit, specifies easiness of its interaction with oxygen which substantially affects physico-chemical properties of the oligomer.

The PCGD was prepared by the anionic polymerization of cyclohexadiene-1.3 in the presence of a catalytic complex of butyllithium with tetrahydrofuran.<sup>2</sup> The PCGD samples of 3500–4000 Mm and 0.92 g/cm<sup>3</sup> density were oxidized in the form of finely dispersed powders of a specific surface of 0.2 m<sup>2</sup>/g and solutions in chlorobenzene and toluene.

The analysis of PMR-spectra for the synthesized PCGD showed that the monomer units are added mainly in position 1.4 (  ), while the data of X-ray and electronographic studies confirmed its amorphous structure.<sup>3</sup>

## THERMAL AND OXIDATIVE TRANSFORMATIONS OF PCGD IN SOLUTION

In the first reaction stage, the autoxidation of PCGD in a chlorobenzene solution is described by the parabolic law  $(\Delta[\text{O}_2])^{1/2} = Ft$ , where  $F = \frac{1}{2}(k_2/\sqrt{k_6})[\text{PH}]\sqrt{k_i}$  is

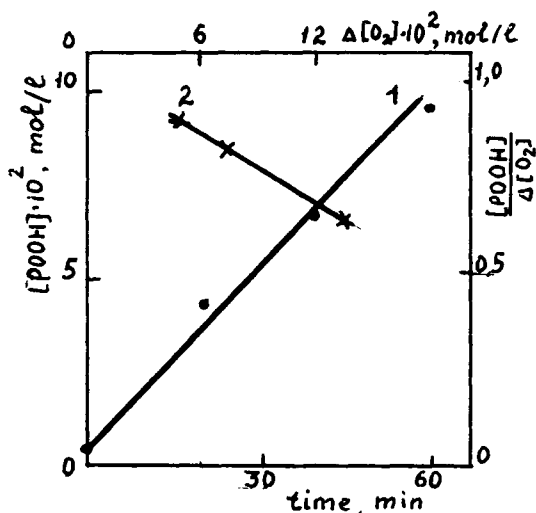


FIGURE 1 Dependence of the  $[\text{POOH}]/\Delta[\text{O}_2]$  relation on the oxidation depth of PCGD solution in chlorobenzene (1) and POOH (2) accumulation kinetics (2) at 363 K ( $[\text{PH}] = 0.62$  base-mol/l).

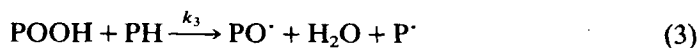
the selfacceleration factor of a radical reaction,  $k_2$  and  $k_6$  are the rate constants of propagation and termination of chains, correspondingly,  $k_i$  is the rate constant of initiation at the expense of hydroperoxide groups of the oxidized PCGD being practically always present in the starting oligomer. The reaction order to PCGD (PH) within initial concentration,  $[\text{PH}]_0 = 0.25\text{--}1.0$  base-mol/l, is equal to 1.33. The autoxidation rate of PCGD linearly depends on  $[\text{POOH}]_0^{1/2}$  ( $\text{ROOH}_0$  is the starting PCGD hydroperoxide concentration in the oligomer) which evidences the quadratic termination of kinetic chains.

At the initial reaction stage (within 20 min) practically all the absorbed oxygen is consumed to form ROOH (Figure 1) that are first molecular intermediate products of PCGD oxydation.

The kinetics of POOH decomposition in chlorobenzene is described by the second-order reaction equation. A general expression for the rate constant of POOH decomposition ( $k_p$ ) has the form:

$$k_p = 7.64 \cdot 10^{17} \exp(-153.8 \pm 12.2/RT) \text{ l/mol} \cdot \text{s}$$

The initial stages of PCGD autoxidation in solution can be described by the following scheme:



A value of the reaction order according to PH, that is close to 1.5, indirectly confirms bimolecular decomposition of POOH according to reaction with PCGD.

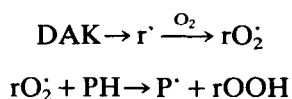
In accordance with the above scheme, the starting oxidation rate obeys Expression,<sup>4</sup>

$$W_0 = \frac{k_2}{\sqrt{k_6}} [\text{PH}] \sqrt{W_i}$$

where the initiation rate  $W_i = k_i[\text{POOH}][\text{PH}]$ , the initiation rate constant  $k_i = 2k_p e$  ( $e$  is the POOH initiation efficiency) which was observed tentatively. A general expression for  $k_i$  has the form:

$$k_i = 1.75 \cdot 10^7 \exp(-84.1 \pm 5.1/RT) \text{ l/mol} \cdot \text{s}$$

In the presence of a low-molecular initiator, azo-izo-butyric acid dinitrilo (DAK), the chain initiation runs according to the reactions:



The kinetic regularities for the PCGD initiated oxidation and autoxidation rates are similar (Figure 2).<sup>5</sup>

A general expression for  $k_i$  DAK in the PCGD solution has the form:

$$k_i = 1.7 \cdot 10^{14} \exp(-93.6 \pm 4.1/RT) \text{ s}^{-1}.$$

The specific rate of oxygen dissolution in the system was calculated from the dependence of  $W_0$  on  $p\text{O}_2$  and correspondingly, the conditions of oxidation ( $x$ ) were estimated. The values of  $x$  amount to  $2.7 \cdot 10^{-2}$  and  $6.2 \cdot 10^{-3} \text{ s}^{-1}$  at 333 and 353 K. The PCGD oxidation at 353 K proceeds under diffusion conditions ( $W_0/x \approx yp\text{O}_2$ ) upon about 40% oxygen content in the gas mixture, i.e. it is

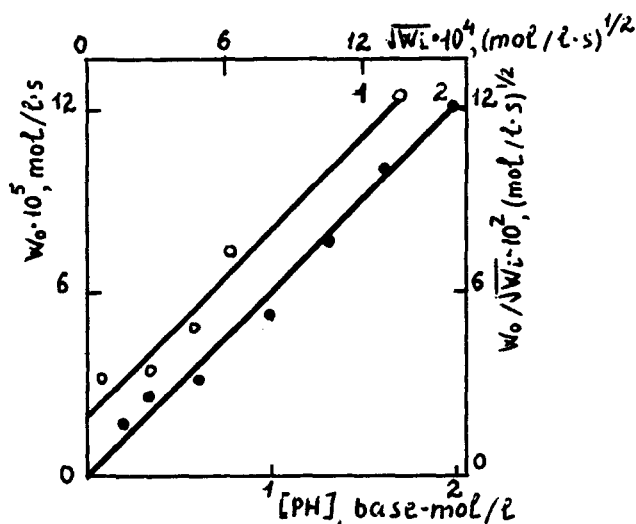


FIGURE 2 Dependence of the oxidation rate on  $\sqrt{W_i}$  (1) at  $[\text{PH}] = 1.94 \text{ base-mol/l}$  (343 K) and relation  $W_0/\sqrt{W_i}$  on the PCGD concentration (2) in chlorobenzene solution at 353 K.

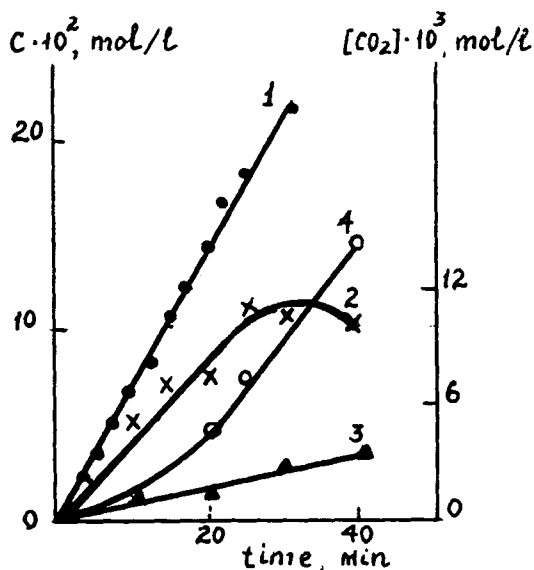


FIGURE 3 Kinetics of the O<sub>2</sub> absorption (1), POOH (2), PCOOH (3) and CO<sub>2</sub> accumulation (4) upon the PCGD solution oxydation in chlorobenzene in the presence of  $4.85 \cdot 10^{-3}$  mol/l DAK at 363 K.

limited by no chemical reaction but by a physical process of oxygen decomposition. A similar diffusion condition is observed at 333 K and 10% pO<sub>2</sub> while with 20% pO<sub>2</sub> it is kinetic, since  $W_0/r < ypO_2$ .

Along with hydroperoxides, acids and carbon dioxide are formed in PCGD but in considerably lower quantities. Among the reaction products, carbon oxide and ethylene were also identified. The kinetic regularities of POOH, PCOOH and CO<sub>2</sub> accumulation (Figure 3) evidence that at initial reaction stages (to oxidation depth of 0.2 mol/l of absorbed O<sub>2</sub>) their formation proceeds in parallel.

The oxidative transformations of PCGD in solution are accompanied by a change in its characteristic viscosity (Figure 4). In this case, the probability of occurring structurization and destruction processes substantially depends on the oxidation conditions and depth. In an argon atmosphere, the value of  $[\eta]$  PCGD does not practically change within 100 min at 363 K. At the same time, with DAK the structurization is observed (Figure 4, curve 3).

In PCGD, as well as in polyisoprene,<sup>6</sup> the cyanizopropylene radicals evidently are not able to abstract a hydrogen atom and attach a double bond of the oligomer which causes intermolecular linkages. In the oxygen, the hydrogen atom abstraction with radicals  $rO_2$  and  $PO_2$  completes with their addition to a double bond. So, with  $[DAK] = 1.45 \cdot 10^{-3}$  mol/l the value of  $[\eta]$  PGCD decreases (Figure 4, curve 4), while with  $[DAK] = 3.89 \cdot 10^{-2}$  mol/l an insoluble gel forms already in 20 min.

It is known<sup>6</sup> that in some polymers the decomposition of hydroperoxide groups at a tertiary carbon atom according to the O—O bond results in the rupture of the neighbouring C—C bond. However, upon decomposition of PCGD hydro-

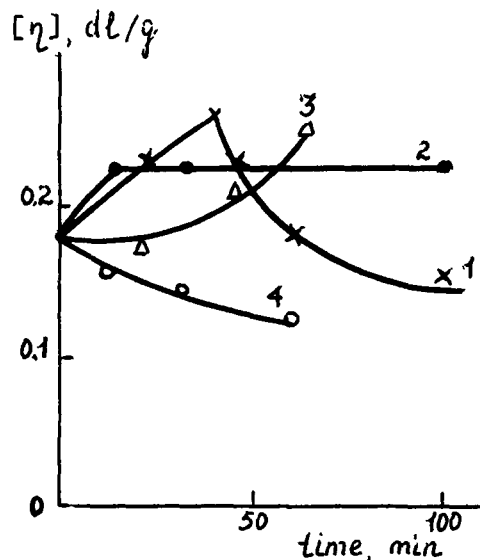


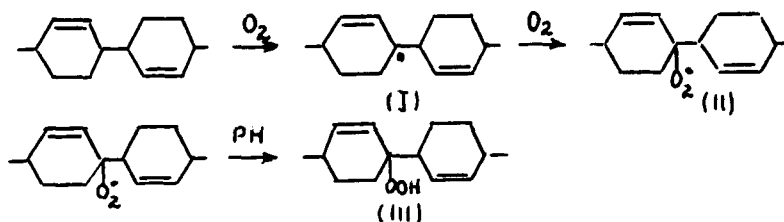
FIGURE 4 Kinetic curves of variation in characteristic viscosity upon thermal keeping of the PCGD solution in toluene;  $[PH] = 1.12$  base-mol/l; at 363 K: 1—oxygen; 2—argon;  $[DAK] = 1.45 \cdot 10^{-3}$  mol/l; 4—oxygen,  $[DAK] = 1.22 \cdot 10^{-3}$  mol/l.

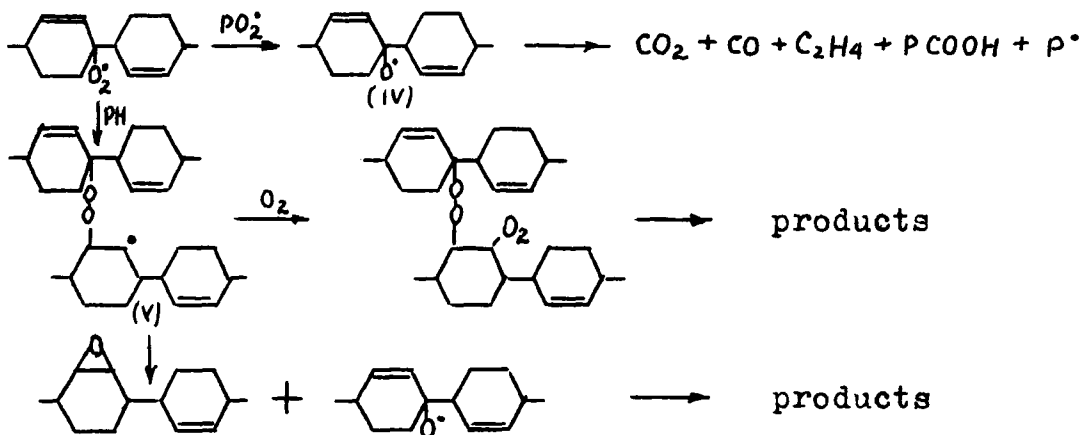
peroxides the destruction of its polymer chain does not occur. It is evidenced by the constant relative viscosity of the PCGD solution containing  $6.44 \cdot 10^{-2}$  mol/l POOH upon thermal decomposition of hydroperoxide to the concentration of  $3.0 \cdot 10^{-2}$  mol/l, as well as by the absence of  $CO_2$  in the gas phase.

Thus, the PCGD destruction in solution is associated with transformation of peroxide radicals which lead to the formation of low-molecular products in the initial oxidation stage. Two pathways of decomposition of peroxide radicals causing the polymer destruction are possible: monomolecular—by the C—C bond (then  $W_s \sim \sqrt{W_i}$ ) and bimolecular—upon interaction of peroxide radicals to form an unstable alkoxiradical.<sup>7</sup> In this case the following relation should be true  $W_s \sim W_i$ .

The PCGD destruction rate linearly depends on  $[DAX]$ . Relation  $W_s/W_i = 29 \pm 2$ , i.e. there are 29 events of alkoxiradical formation with their further decomposition per an event of chain termination.

The data obtained make it possible to describe the initial stage of the thermaloxidative transformation of PCGD in solution by the following scheme:<sup>8</sup>





**PCGD OXIDATION IN THE SOLID PHASE**

The autoxidation of PCGD in the solid phase, as well as oxidation in solution, represent a radical process with destructive branching and quadratic termination of kinetic chains:<sup>2</sup> the main branching agent is POOH PCGD, and the starting oxidation rate is described by the expression:

$$W_0 = \frac{k_2}{\sqrt{k_6}} [PH] \sqrt{W_i}$$

Upon autoxidation of PCGD in the solid phase at the initial stage, the relation of rates of the POOH formation and O<sub>2</sub> absorption is close to 1 and does not depend on pO<sub>2</sub> at pO<sub>2</sub> ≥ 20% (Figure 5). In PCGD mostly the intermolecular

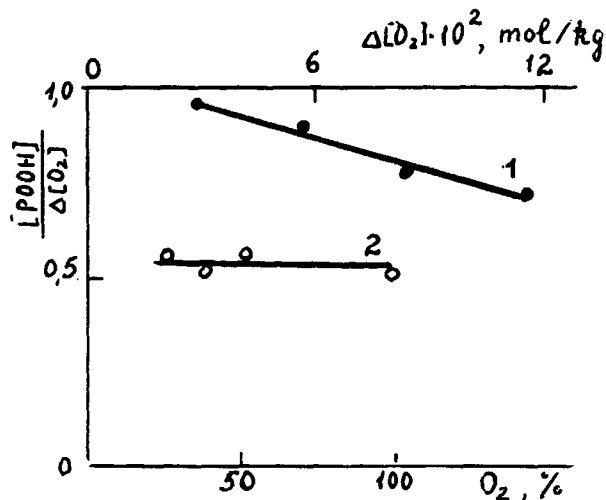


FIGURE 5 Dependence of relation [ROOH]/Δ[O<sub>2</sub>] on the oxidation depth (1) and pO<sub>2</sub> in the PCGD solid phase (2) at 353 K.

transfer of kinetic chains followed by the formation of single hydroperoxide groups evidently occurs.<sup>9</sup>

The POOH PCGD decomposition in the solid phase is described by the first-order kinetic equation with  $E = 129$  kJ/mol.<sup>10</sup> However, the POOH PCGD decomposition rate in the presence of 2,2'-methylene-bis(4-metil-6-tret-butylphenol) (InH), containing a mobile hydrogen atom and being a typical inhibitor of radical processes in polymers, markedly increases, the dependence  $W_p/[POOH] - [InH]$  being of a linear character, which permitted the calculation of a bimolecular constant for the reaction rate of POOH with InH ( $k_{InH} = 1.9 \cdot 10^{-2}$  kg/mol · s, 363 K). The POOH ( $k_p$ ) decomposition rate constants found from the first-order equation are evidently effective, and absolute constants of the bimolecular reaction rate of POOH with PH are found to be  $k'_p = k_p/[PH]$ .

A general expression for  $k'_p$  and  $k_i$  of POOH PCGD in the solid phase has the form:

$$k'_p = 3.2 \cdot 10^{13} \exp(-129.8 \pm 8.1/RT) \text{ kg/mol} \cdot \text{s}$$

$$k_i = 3.6 \cdot 10^7 \exp(-84.4 \pm 5.1/RT) \text{ kg/mol} \cdot \text{s}$$

It is to be noted that  $k_{InH}$  is by 4 factors of magnitude higher than  $k'_p$  ( $6.6 \cdot 10^{-6}$  kg/mol · s at 363 K) despite close energy values of the O—H phenol bond (351.5 kJ/mol) and C—H bond in the PCGD molecule (395.0 kJ/mol). This is possibly associated with the diffusive phenol mobility in the PCGD solid phase. If the diffusion coefficient for phenol and in polypropylene is assumed the same<sup>11</sup> ( $D = 0.72 \cdot 10^{-8}$  cm<sup>2</sup>/s at 363 K), then the value of a diffusive constant  $k_{dInH} = 4\pi dDN \cdot 10^{-3}$  will amount to  $2.7 \cdot 10^6$  kg/mol · s (at  $d = 5$  Å). The diffusion coefficients for macromolecules in solid polymers are very low, as a rule,  $\ll 10^{-10}$  cm<sup>2</sup>/s. Assuming that  $D = 10^{-12}$  cm<sup>2</sup>/s,<sup>12</sup> we'll obtain the value of  $10^2$  kg/mol · s for  $k_{dPH}$ , which is by 4 orders of magnitude lower than  $k_{dInH}$ . Comparison of  $k_{dInH}$  and  $k_{InH}$  shows that the phenol diffusion in the PCGD solid phase proceeds much more rapidly than its reaction with hydroperoxide.

The initiation efficiency values,  $e$ , for POOH in the PCGD solid phase and chlorobenzene solution are very close (0.2 and 0.4, correspondingly). This indicates a general mechanism of radical generation for PVGD solid and liquid phases: the transfer of a free valency occurs by the chemical relay-race that is not enough sensitive to a change in the medium viscosity. The value of  $e$  decreases with temperature which can be explained by an increasing probability of radical recombination in a cell.

The kinetics of a change  $[\eta]$  in upon oxidation of solid PCGD resembles that of a liquid-phase process (Figure 6). A decrease in the quantity of a gel-fraction with temperature can evidence the decomposition of radical (V) (scheme) followed by formation of an epoxide and radical (IV).

The experimental results obtained convincingly evidence that there are no principal differences in kinetics of the PCGD autoxidation at initial stages in solution and solid phase.

The effect of the PCGD aggregate state on the kinetic regularities of its oxidation distinctly manifests itself upon using low-molecular initiators.<sup>13</sup> So, the



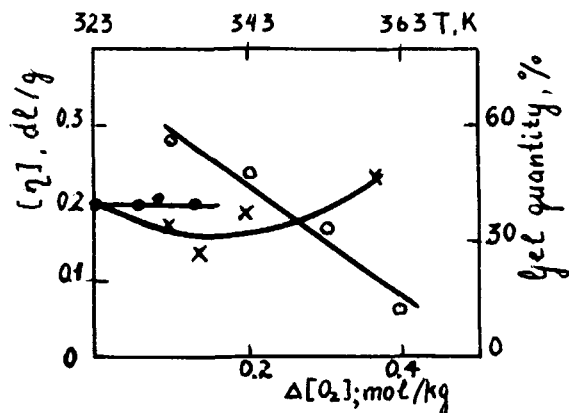


FIGURE 6 Dependence of the characteristic PCGD viscosity on oxidation depth (1,2) at 353 K and gel quantity on oxidation temperature (3) of the PCGD powder: 1—without initiator; 2—with  $1.16 \cdot 10^{-2}$  mol/kg DAK.

rate of  $O_2$  absorption in the presence of DAK and lauroyl peroxide (PL) drastically increases as compared with autoxidation and remains constant in a wide range of initiator concentrations (Figure 7) which can be associated with limited solubility of low-molecular additions in the PCGD solid phase. However, a linear dependence of  $W_i$  on  $[DAK]$  and  $[PL]$  indicates the absence of limitations in solubility of these compounds under the conditions studied.

General expressions for PL and DAK, respectively, have the form:

$$k_i = 2.5 \cdot 10^8 \exp(-88.7 \pm 2.0/RT) \text{ c}^{-1}$$

$$k_i = 6.9 \cdot 10^4 \exp(-70.4 \pm 1.2/RT) \text{ c}^{-1}$$

The total decomposition of PL in the PCGD solid-phase runs by the first-order

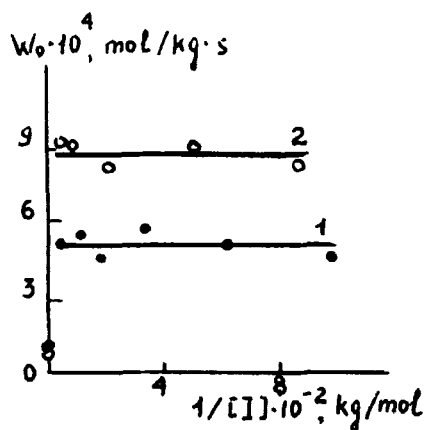


FIGURE 7 Dependence of the PCGD powder oxidation rate with DAK (1) and PL (2) on the initiator concentration at 353 K.

reaction with an effective rate constant

$$k_p = 6.9 \cdot 10^{10} \exp(-93.8 \pm 1.4/RT) \text{ c}^{-1}$$

In the PCGD solid phase, the values of  $k_p$  for POOH are by over two orders of magnitude lower than for PL; at the same time their initiation rates are close at comparable concentrations. This is evidently associated with a substantial difference in the initiation efficiency between PL ( $e = 0.01$  at 363 K) and POOH ( $e = 0.2$  at 363 K). The cyanizopropyl and undecyl radicals formed upon decomposition of DAK and PL are low-reactive and their removal from the cell is determined by diffusion whose rate, in contrast to chemical "relay race" competition, substantially depends on the medium viscosity.

If upon initiated oxidation of solid polymers the destruction of radicals proceeds mainly by the reaction



then the oxidation rate  $W = k_2[\text{PH}][\text{PO}_2]$  will depend on diffusive mobility of the initiator radicals

$$W = \frac{k_1 k_2 W_i [\text{PH}]^2}{8\pi d D k_i [\text{I}]}$$

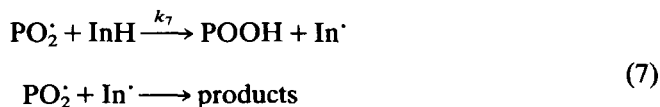
where  $8\pi d D (k_i [\text{I}]/k_1 [\text{PH}])$  is the effective rate constant of macroradical destruction in the presence of a low-molecular initiator (I).

$W_i = k_i [\text{I}] + k_3 [\text{POOH}][\text{PH}]$  is the total initiation rate.

The molecular volumes of cyanizopropyl and undecyl radicals calculated by use of the increments of corresponding groups<sup>14</sup> are equal to  $78 \cdot 10^{-30}$  and  $188 \cdot 10^{-30} \text{ m}^3$ , respectively. Consequently, upon substitution of DAK for PL, the PCGD oxidation rate has to increase which is confirmed by the experiment (Figure 7).

## INHIBITED PCGD OXIDATION IN THE LIQUID PHASE

The PCGD thermostability considerably increases in the presence of antioxidants (InH), substituted phenols and amines<sup>14,15</sup> which terminate kinetic chains by the reaction with peroxide radicals



The following expression is valid for the inhibited PCGD oxidation rate:

$$W = \frac{k_2 [\text{PH}] W_i}{f k_7 [\text{InH}]}$$

where  $f$  is the inhibitor capacity. This is confirmed by the linear dependence of  $W$  on  $1/[\text{InH}]$  and inhibition degree  $F = W/W_0 - W_0/W$  on  $[\text{InH}]$ . The efficiencies of

the majority of the space-inhibited phenols studied: 2,6-di-*tert*-butyl-4-methylphenol (ionol), 2,2'-methylene-bis-(4-methyl-6-*tert*-butylphenol) (anti-oxidant 22-46, and its analogue, bisalcofen BP), 2,2'-methylene-bis(4-methyl-6- $\alpha$ -methylcyclohexylphenol) (nonox BSP), bis-(2,2-methyl-5-*tert*-butyl-4-oxiphenyl) monosulphide (santonox) are the values of the same order and are equal to 250, 1050, 230, 320, 121, respectively at 363 K. For propionic acid pentaeritric ester (3,5-ditert-butyl-4-hydroxyphenyl) (igranox 1010), the value of  $k_7/k_2$  amounts to 5830. Comparison of the *N*-phenyl- $\beta$ -naphthylamine (neozone D) ( $k_7/k_2 = 72$ ) and *N*-isopropyl-*N'*-phenyl-*p*-phenylenediamine (diaphen FP) ( $k_7/k_2 = 226$ ) efficiencies shows that the reactivity of diamine relative to the oligomer peroxide radicals is approximately 3 times higher than that of amine. If we assume the value of  $k_2$  for the oxidizing PCGD being similar to its low-molecular analogue, 3,6-dimethylcyclohexene ( $k_2 = 22 \text{ l/mol} \cdot \text{s}$ ), then  $k_7$  for the phenols studied will amount to  $10^4\text{--}10^5 \text{ l/mol} \cdot \text{s}$ , which is close of  $k_7$  for space-inhibited phenols in hydrocarbons.

The general expressions of  $k_7/k_2$  and  $k_7/\sqrt{k_6}$  for bisalcofene BP solution have the form:

$$k_7/k_2 = 2.3 \cdot 10^{12} \exp(-68.8 \pm 3.1/RT)$$

$$k_7/\sqrt{k_6} = 5.9 \cdot 10^{10} \exp(-99.3 \pm 7.2/RT)(\text{l/mol} \cdot \text{s})^{1/2}$$

The space-inhibited phenols inhibit the PCGD destruction. So, e.g. in the presence of bisalcofene BP, the initial destruction rate ( $W_i$ ) at 363 K is  $W_i = 2.6 \cdot 10^{-6} \text{ mol/l} \cdot \text{s}$ ,  $[\text{InH}]_0 = 2.36 \cdot 10^{-3} \text{ mol/l}$  and  $[\text{PH}] = 1.12 \text{ base-mol/l}$  amounts to  $9.8 \cdot 10^{-7} \text{ mol/l} \cdot \text{s}$ , while under the same conditions, but without inhibitor,  $W_i = 3.2 \cdot 10^{-5} \text{ mol/l} \cdot \text{s}$ . With  $W_i = \text{const}$ ,  $W_i$  drops with increasing concentration of the inhibitor. It is to be noted that at  $[\text{InH}] = 2.36 \cdot 10^{-3} \text{ mol/l}$  and  $[\text{DAK}] = 7.9 \cdot 10^{-3} \text{ mol/l}$  the PCGD increases with the oxidation depth. In the presence of an acceptor of free radicals, the structurization evidently predominates due to the attack of the PCGD double-bond initiator with low-molecular radicals followed by formation of intermolecular linkages. Similar results were obtained in the absence of InH when keeping thermally the PCGD solution with DAK in an argon atmosphere (Figure 4, curve 3). At the same time, upon autoxidation of the PCGD solution with  $5.3 \cdot 10^{-4} \text{ mol/l}$  bisalcofene BP $[\eta]$  the PCGD remained unaffected in 20 h oxidation as compared to the initial one. These data confirm the suggestion reported by us earlier that the PCGD destruction in solution is associated with transformation of peroxide radicals.

The additions of bisalcofene BP at the CGD-1.3 polymerization stage permitted keeping (over 4 months) a 10% oligomer solution in the toluene/*n*-xylol mixture (polymeric base of the negative PCGD-FN-1 photoresist) without a change in characteristic viscosity and solubility.

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