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Kinetics and Mechanism of Liquid Phase Oxidation of Organic Compounds in the Presence of Cyclohexanone Transformation Products Acting as Antioxidants

K.S. Sharaeva*, I.V. Safarova, Sh.M. Okhirov, E.R. Latypova and A.Ya. Gerchikov
Ufa State University of Science and Technology, 32 Z. Validi St., Ufa, Republic of Bashkortostan, Russia, 450076

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The antioxidant properties of cyclohexanone transformation products were investigated using model radical-chain oxidations of 1,4-dioxane and isopropyl alcohol by molecular oxygen (air) at a temperature of 348 K. Oxygen-uptake kinetic curves during the oxidation process were obtained using a universal differential manometric setup under various initial conditions. It was shown that the compounds studied exhibit antioxidant activity, the mechanism of which varies depending on the nature of the oxidized substrate. For the two substrates examined, a reaction mechanism was proposed that satisfactorily agrees with the experimental data. Rate constants were determined that quantitatively characterize the efficiency of the antioxidant action.

Keywords: Radical-chain oxidation, Antioxidant activity, Lactam, Oxime, Free radicals

INTRODUCTION

One of the most important tasks of chemistry and chemical engineering is to preserve the performance properties of products of organic synthesis during storage and transportation. This concerns the protection of these products, as well as foods and pharmaceuticals, from destructive degradation caused by oxidation by molecular oxygen (air). This process, which has been well studied for a wide class of organic compounds [1-3], is a complex reaction proceeding by a chain mechanism with participation of radicals of various types and labile molecular products. The method of inhibiting this process is also known and consists of the use of a broad class of substances called antioxidants [4,5]. Adding them in small amounts to an organic substrate practically does not affect the target properties of the substrate but provides protection against undesirable thermo-oxidative decomposition.

Despite the wide range of antioxidants available from the fine-chemicals sector, interest in the synthesis of new highly efficient compounds remains topical. In particular, products of cyclohexanone transformation attract attention: the oxime 2,4-dioxaspiro[5,5]undecan-7-one and the lactam 2,4-dioxa-7-azaspiro[5,6]dodecan-8-one. These compounds contain N–H and O–H bonds that can serve as active centers conferring antioxidant properties. It is also important to note that the newly synthesized substances are transformation products of cyclohexanone, several derivatives of which exhibit biological activity, including use as drugs for the treatment of bacterial and fungal infections [6,7], as well as psychiatric disorders [8]. Therefore, the named compounds are promising as biologically active agents whose antioxidant properties add to the suite of characteristics desirable in pharmaceuticals. Additionally, the feedstock for such compounds is cyclohexanone, which is relatively inexpensive and readily available.

In view of the above, the aim of the present work was to investigate the effectiveness of the antioxidant action of the newly synthesized compounds in the oxidation of model organic substrates.

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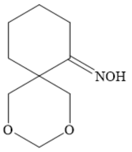
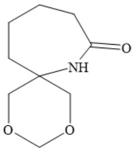
*Corresponding author. E-mail: sharaevs221000@mail.ru

REAGENTS AND EXPERIMENTAL METHODS

The substances investigated were the compounds whose structures are presented below:



Oxime 2,4-dioxaspiro[5,5]undecan-7-one (Oxime)



2,4-dioxa-7-azospiro[5,6]dodecan-8-one (Lactam)

These compounds were synthesized in the Laboratory of Organic and Bioorganic Chemistry of the Institute of Chemistry and Protection in Emergency Situations, using the Prins reaction [9] as the key step. Identification spectra are given in the supplementary materials to this article.

Antioxidant activity of the synthesized compounds was studied using model-initiated oxidations of 1,4-dioxane and isopropyl alcohol (T = 348 K; initiator 2,2′-azobisisobutyronitrile, AIBN; initiation rate $V_i = 2.4 \times 10^{-7}$ M s^{-1}). The oxidation substrates were preliminarily purified by known procedures described in the literature [10,11]. 1,4-Dioxane was subjected to multiple fractional distillations over potassium hydroxide, followed by removal of acetaldehyde by treatment with hydrochloric acid and purging with a stream of nitrogen. Isopropyl alcohol was kept over iodine for 24 h, then zinc dust was added, and the mixture was refluxed until the iodine color disappeared. Solutions of both substrates were further fractionally distilled. Substrate purity was assessed by their oxidation parameters, which were comparable with literature data [12].

Manometric Installation

The manometric method is based on studying the mechanism and kinetics of liquid-phase oxidation of organic compounds. Kinetic experiments were carried out using a universal manometric setup (UMS), the design of which is described in detail elsewhere [13,14], as well as in the supplementary information. The UMS comprises two thermostatted glass reactors of identical volume; one functions as the working reactor and the other is used for pressure balancing. To maintain equal and constant temperatures in the reactors, each is equipped with a jacket connected to a circulating thermostat. The reactors are connected via capillary tubing to a differential pressure transducer and to a system of gas valves used both for pressure equalization between the reactors and for filling the reactors with the desired gas atmosphere. The transducer and valve system are mounted in the main unit of the apparatus. The reactors are charged with reaction mixtures that may differ from one another in concentration or in the presence of a catalyst or inhibitor. The valves are then closed, and the pressure in the working reactor changes in accordance with the volume of gas consumed or produced during the chemical reaction. The pressure difference is measured in volts (V) by a high-sensitivity differential pressure sensor based on a silicon membrane element and recorded as a function of time. By differentiating the resulting pressure–time curve, the gas uptake rate can be determined, which corresponds to the reaction rate in the reactors.

During the experiments, a solution of AIBN in the oxidation substrate was loaded into the working reactor, and the inhibitor dissolved in the same substrate was added to the desired concentrations using a microsyringe. During the development of the manometric setup, a calibration was performed, which showed that, for the given reactor volume and with 4 mL of solution introduced into a preheated reactor, isothermal conditions are reached within 5 min. Information on the time required to maintain the reaction mixture at constant temperature under analogous conditions is also given in [15]. Consequently, all measurements were started only after the reaction mixture had reached a steady temperature.

RESULTS AND DISCUSSION

Kinetics and Mechanism of 1,4-Dioxane Oxidation in the Presence of Additives: Oxime 2,4-Dioxaspiro[5.5]undecan-7-one (Oxime) and 2,4-Dioxa-7-azaspiro[5.6]dodecan-8-one (Lactam)

The reactivity of the examined compounds as antioxidants was studied in the model AIBN-initiated oxidation of 1,4-dioxane by measuring the rate of oxygen uptake at 348 K (initiator AIBN, initiation rate $V_i = 2.4 \times 10^{-7}$ M s^{-1}).

Figures 1 and 2 show the oxygen-uptake kinetic curves in the presence of the oxime and lactam additives (denoted InH).

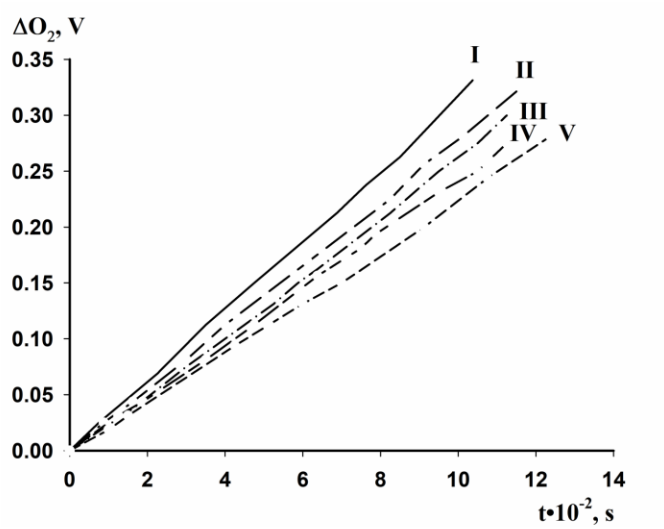


Fig. 1. Kinetic curves of oxygen uptake during the oxidation of 1,4-dioxane in the absence and in the presence of the oxime additive: [InH] × 10^4 M = 0.0 (I), 1.0 (II); 2.6 (III); 3.9 (IV); 5.1 (V). T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

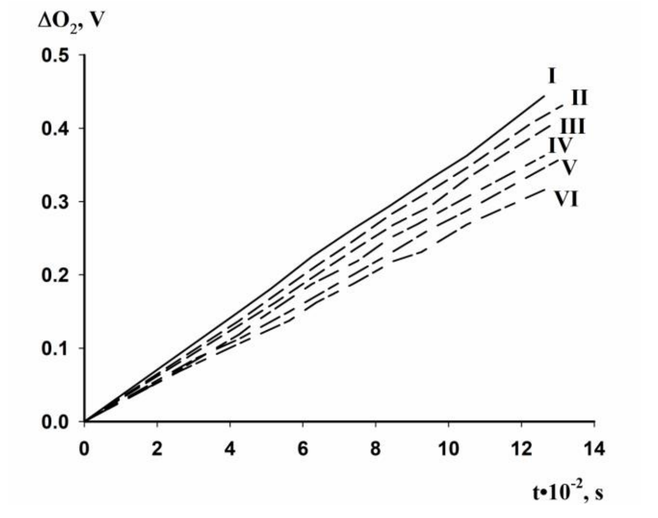
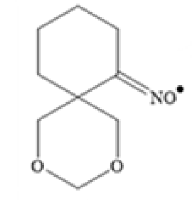


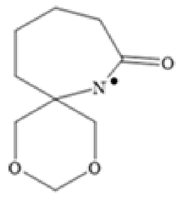
Fig. 2. Kinetic curves of oxygen uptake during the oxidation of 1,4-dioxane in the absence and in the presence of the lactam additive: [InH] × 10^4 M = 0.0 (I), 1.4 (II), 2.8 (III), 4.2 (IV), 5.5 (V), 8.3 (VI). T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

The obtained results indicate that the studied substances possess antioxidant properties, as evidenced by a decrease in the oxygen absorption rate in their presence, which systematically declines with an increase in the initial concentration of the additives.

Note that cyclohexanone is not known as an antioxidant, and these properties manifested in the discussed products of its modification as a result of introducing substituents containing N-H bonds (in the lactam) and N-O-H bonds (in the oxime), respectively. Evidently, these bonds can act as active inhibition centers, as is the case with derivatives of aromatic compounds. Aromatic compounds themselves may not exhibit antioxidant properties; however, their derivatives sterically hindered phenols (SHP) [16] and aromatic amines [17] possess antioxidant characteristics, effectively inhibiting the reactions of radical-chain oxidation of organic compounds. The mechanism of action of these compounds has been studied in great detail, and it has been established that the O-H and N-H bonds act as the active inhibition centers for SHP and aromatic amines, respectively. It should be noted that the newly synthesized transformation products of cyclohexanone contain substituents with these very bonds, which act as the active inhibition centers, analogous to the case of SHP and aromatic amines. It is known that for these inhibitors, the elementary chain termination event occurs as a result of the abstraction of a hydrogen atom from the active center of the inhibitor by the chain-carrying radical. Hereafter, we will introduce the notation InH, according to which the In• radical has the following structure:



Oxime radical



Lactam radical

Let us use the well-established mechanism for the oxidation of 1,4-dioxane [15,18,19], supplemented with elementary stages accounting for the antioxidant effect. This approach allows for the formal application of the well-known mechanism of inhibited oxidation of organic compounds:

r—N=N—r → $2r^{\bullet} + N_2$	(k_1)
$r^{\bullet}$ + RH → rH + $R^{\bullet}$	(k_2)
$R^{\bullet} + O_2 \rightarrow RO_2^{\bullet}$	(k_3)
$RO_2^{\bullet}$+ RH → ROOH + $R^{\bullet}$	(k_4)
$RO_2^{\bullet} + RO_2^{\bullet}$→ molecular products	(k_5)
$RO_2^{\bullet}$ + InH → $In^{\bullet}$+ ROOH	(k_6)
$RO_2^{\bullet} + In^{\bullet}$ → ROOIn	(k_7)

Scheme 1

where r—N=N—r is the initiator, RH is the oxidized substrate, and $R^{\bullet}$ and $RO_2^{\bullet}$ are the alkyl and peroxyl radicals of 1,4-dioxane, respectively.

From the kinetic curves, the oxygen-uptake rates were calculated by the method of least squares (MLS); the values for the studied antioxidants depend on the initial concentration of InH (Fig. 3).

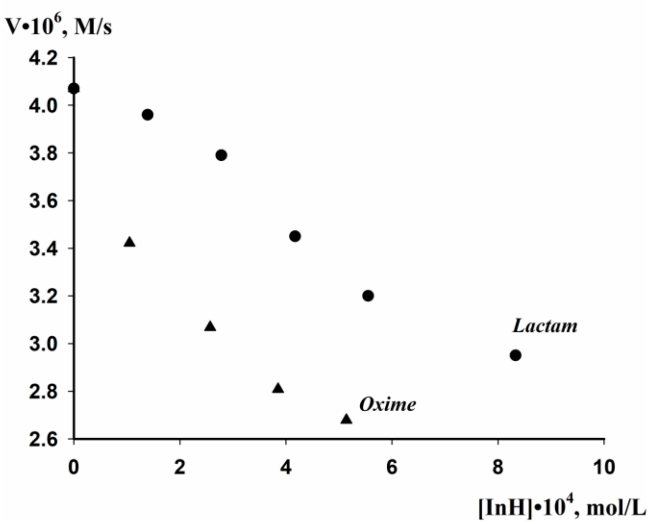


Fig. 3. Dependence of the oxidation rate of 1,4-dioxane on the concentration of the oxime and the lactam. T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

If the oxidation proceeds as a chain reaction in the presence of inhibitory additives, the following relationship holds between the oxidation rate and the concentration of the added inhibitor [12]:

$$F = \frac{V_0}{V} - \frac{V}{V_0} = \frac{f k_{ImH}\,[InH]}{\sqrt{2k_5 V_i}} \qquad (1)$$

where V_o, V and V_i are the oxygen-uptake rates in the absence and in the presence of the inhibitor, and the initiation rate, respectively.

F is the inhibitor efficiency parameter;

f – is the inhibition capacity;

k_{InH} – is the rate constant for chain termination on the inhibitor;

[InH] – is the inhibitor concentration;

$2k_5 = 6.67 \times 10^7$ M^{-1} s^{-1} is the rate constant for quadratic chain termination [12],

The experimental data are satisfactorily linearized in the coordinates of equation (Eq. (1)) with a correlation coefficient of R > 0.98

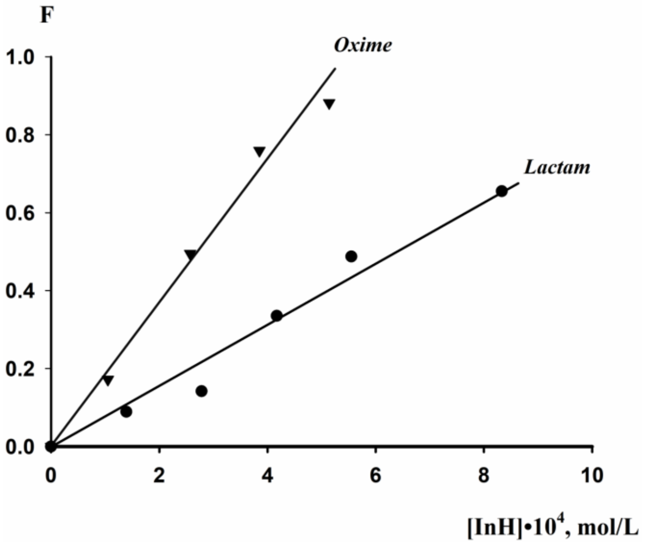


Fig. 4. Dependence of the inhibitor efficiency parameter on the concentration of the oxime and the lactam, plotted according to Eq. (1). T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

Analysis of these dependencies allowed the determination of the effective rate constant for chain-breaking of the oxidation, which is given in Table 1:

Table 1. Values of the Effective Rate Constants for Inhibition of 1,4-Dioxane Oxidation by Oxime and Lactam Derivatives; T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}

Investigated compound	$fk_{InH} \times 10^{-3}$ M^{-1} s^{-1}
Oxime	7.5 ± 0.9
Lactam	3.1 ± 0.3

Kinetics and Mechanism of Oxidation of Isopropyl Alcohol in the Presence of the Additives Oxime 2,4-Dioxaspiro[5.5]undecan-7-one (Oxime) and 2,4-Dioxa-7-azaspiro[5.6]dodecan-8-one (Lactam)

The reactivity of the oxime and the lactam as antioxidants was studied in a model reaction: the initiated oxidation of isopropyl alcohol monitored by the rate of oxygen uptake at T = 348 K. Oxidation was initiated with 2,2'-azobisisobutyronitrile (AIBN) at an initiation rate $V_i = 2.4 \times 10^{-7}$ M s^{-1}. The addition of the oxime 2,4-dioxaspiro[5,5]undecan-7-one (oxime) and the 2,4-dioxa-7-azaspiro[5,6]dodecan-8-one (lactam) to the oxidizing isopropyl alcohol causes a concentration-dependent decrease in the oxygen-uptake rate. These results indicate that the tested compounds exhibit antioxidant activity.

Figures 5 and 6 show typical kinetic curves of oxygen uptake in the initiated oxidation of isopropyl alcohol: one curve for the reaction without additives and curves for the reaction in the presence of the oxime 2,4-dioxaspiro[5.5]undecan-7-one (oxime) and the 2,4-dioxa-7-azaspiro[5,6]dodecan-8-one (lactam).

Oxygen-uptake rates were calculated from the kinetic curves using a least-squares fitting routine. Figure 7 clearly shows that the addition of increasing concentrations of oxime 2,4-dioxaspiro[5.5]undecan-7-one (oxime) and 2,4-dioxa-7-azaspiro[5,6]dodecan-8-one (lactam) to the oxidizing isopropyl alcohol leads to a decrease in the oxygen-uptake rate.

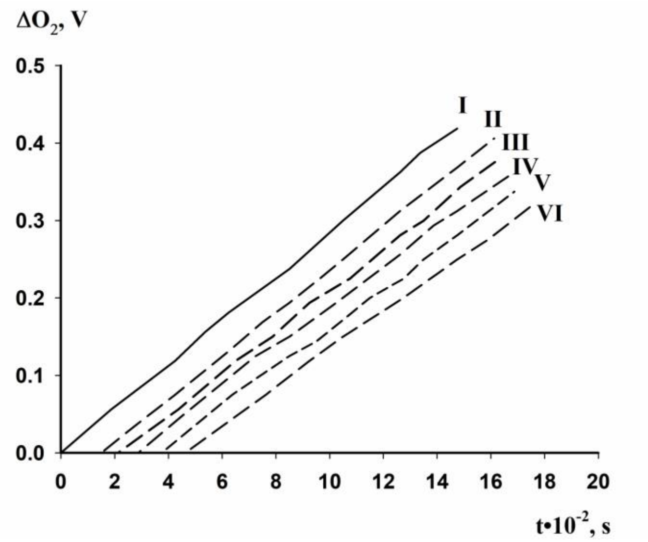


Fig. 5. Kinetic curves of oxygen uptake during oxidation of isopropyl alcohol in the absence (I) and in the presence of the additive 2,4-dioxaspiro[5.5]undecan-7-one (oxime): [InH] × 10^4 M = 0.0 (I) 1.3 (II); [III] = 1.8 (III); 2.5 (IV); 3.7 (V); 5.0 (VI). T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

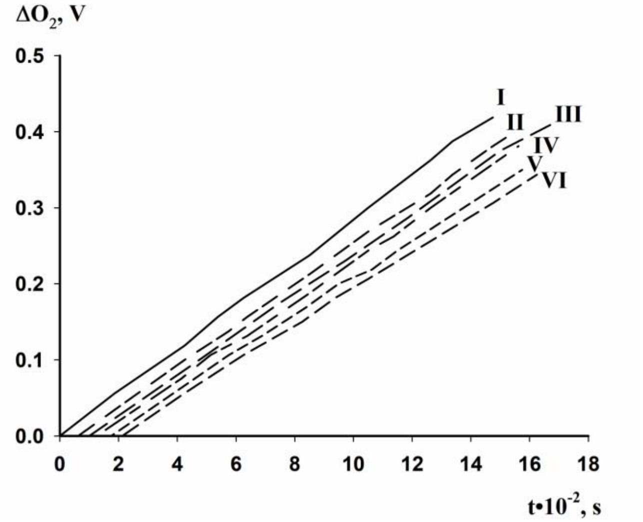


Fig. 6. Kinetic curves of oxygen uptake during oxidation of isopropyl alcohol in the absence (I) and in the presence of the additive 2,4-dioxa-7-azaspiro[5,6]dodecan-8-one (lactam): [InH] × 10^4 M = 0.0 (I) ; 5.0 (II); 7.5 (III); 1.3 (IV); 2.0 (V); 2.5 (VI). T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

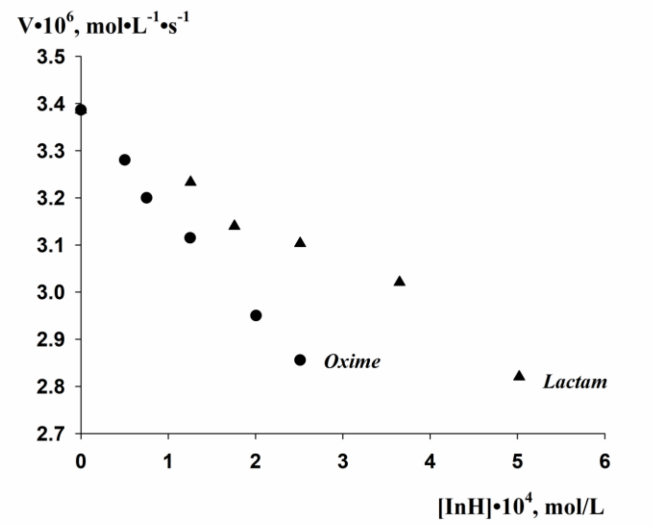


Fig. 7. Dependence of the oxidation rate of isopropyl alcohol on the concentration of oxime and lactam. T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

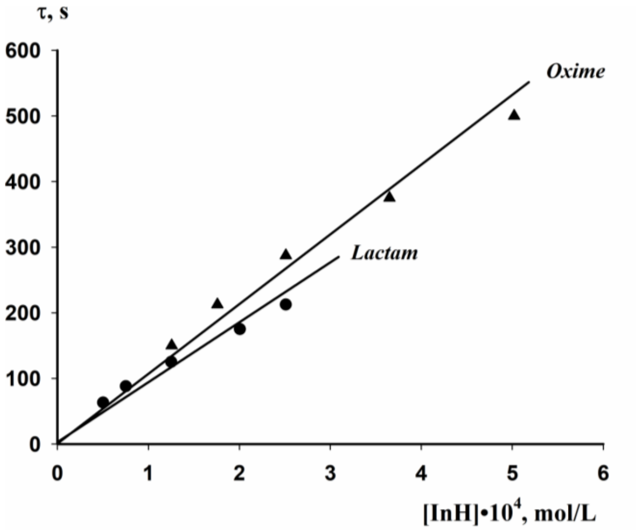


Fig. 8. Dependence of the induction period τ on the added concentration of oxime and lactam. T = 348 K, $V_i = 2,4 \times 10^{-7}$ M s^{-1}.

The experimental data obtained from measurements of the oxygen-uptake rate in the presence of various concentrations of the tested sample allow the determination of the induction period τ of the oxidation process. The induction period for oxime 2,4-dioxaspiro[5.5]undecan-7-one (oxime) and 2,4-dioxa-7- azaspiro[5,6]dodecan-8-one (lactam) is linearly dependent on the inhibitor concentration (Fig. 8), which makes it possible, according to equation (Eq. (2)), to determine the parameter *f*, called the inhibition capacity:

$$\tau = \frac{f \cdot [InH]}{V_i} \qquad (2)$$

Based on the data processed in the coordinates of equation (Eq. (2)), the inhibition capacities *f* of the oxime and the lactam were determined; the values are listed in Table 2:

Table 2. Values of the Inhibition Capacities and Inhibition Rate Constants for Isopropyl Alcohol in the Presence of the Oxime and the Lactam; T = 348 K, $V_i = 2,4 \times 10^{-7}$ M s^{-1}

Investigated compound	f	$fk_{InH} \times 10^{-4}$ ($M^{-1} s^{-1}$)	$k_{InH} \times 10^{-4}$ ($M^{-1} s^{-1}$)
Oxime	0.24 ± 0.02	1.2 ± 0.1	5.0 ± 0.6
Lactam	0.21 ± 0.02	2.3 ± 0.2	10.9 ± 1.3
Amines			1÷9[11]

The table shows that the inhibition capacity *f* < 2, which means that alongside the classical stages (6) and (7) (Scheme 2), responsible for inhibition, there may be an additional stage of chain transfer to the inhibitor (9), leading to a decrease in the value of *f*. The emergence of this reaction results in an increased initiation rate and, consequently, a reduced effectiveness of inhibition.

The collected data allow for the explanation of experimental results using the well-known mechanism of the inhibited oxidation of isopropyl alcohol, which includes the regeneration reaction of the inhibitor from its radical [23]:

Using Eq. (1), the dependence of the oxidation-efficiency parameter F on the antioxidant concentration was obtained.

r—N=N—r $\rightarrow$ 2r$^\bullet$ + N_2	(k_1)
2r$^\bullet$ + $(CH_3)_2CHOH \rightarrow (CH_3)_2C^\bullet OH$ + rH	(k_2)
$(CH_3)_2C^\bullet OH + O_2 \rightarrow (CH_3)_2C(OH)OO^\bullet$	(k_3)
$(CH_3)_2C(OH)OO^\bullet \rightarrow HO_2^\bullet + (CH_3)_2C{=}O$	(k_4)
$HO_2^\bullet + (CH_3)_2CHOH \rightarrow HOOH + (CH_3)_2C^\bullet OH$	(k_5)
$2HO_2^\bullet \rightarrow HOOH + O_2$	(k_6)
$HO_2^\bullet + InH \rightarrow In^\bullet + H_2O_2$	(k_7)
$In^\bullet + HO_2^\bullet \rightarrow InH + O_2$	(k_8)
$In^\bullet + (CH_3)_2CHOH \rightarrow InH + (CH_3)_2C^\bullet OH$	(k_9)

Scheme 2

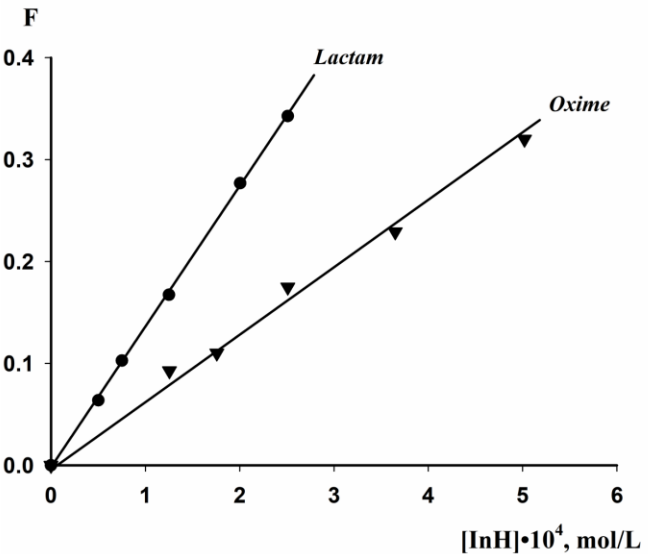


Fig. 9. Dependence of the oxidation-efficiency parameter F for the oxime and the lactam on inhibitor concentration, plotted according to equation (Eq. (1)). T = 348 K, $V_i = 2.4 \times 10^{-7}$ M s^{-1}.

Using the obtained dependencies and equation (eq.1), the values of the inhibition rate constants fk_{InH} were determined; they are presented in Table 2. Here $2k_6 = 1.2 \times 10^9$ M^{-1} s^{-1} is the rate constant for the bimolecular termination of hydroperoxyl radicals, used for IPA [22,23].

CONCLUSION

The kinetics and mechanism of the oxidation of 1,4-dioxane and isopropyl alcohol in the presence of cyclochexanone transformation products have been studied. It has been demonstrated for the first time that the transformation products of cyclochexanone a relatively inexpensive and readily available raw material for the synthesis of potential antioxidants possess the properties of oxidative process inhibitors. It has been established that the mechanism of action of these substances depends on the nature of the substrate and includes both well-known stages of chain termination on the antioxidant molecule (during the oxidation of 1,4-dioxane) and inhibitor regeneration (during the oxidation of IPA), as well as a chain transfer reaction to the inhibitor during the oxidation of IPA, which relatively reduces the inhibition efficiency due to the low value of the inhibitor capacity.

The difference in the values of the effective inhibition rate constants for the two studied antioxidants is noteworthy. A possible reason for this difference during the oxidation of 1,4-dioxane is likely the difference in the bond energies of the active centers and the O-H bond formed in reaction (6) of the hydroperoxide. Indeed, for the oxime, the strengths of the broken and formed bonds are similar (≈370 kJ mol^{-1}) [24], and the reaction is practically thermoneutral. In contrast, for the lactam, an N-H bond with a strength of ≈423 kJ mol^{-1} is broken while the same O-H bond in the hydroperoxide molecule is formed. As a result, reaction (6) in Scheme 1 is endothermic and requires a higher activation energy, which accounts for the lower value of fk_7 for the lactam.

However, when studying the reaction of oxidizing isopropyl alcohol in the presence of additives, the found values of the inhibition rate constants were similar for the two studied compounds but lower than in oxidizing 1,4-dioxane. This is apparently related to a change in the reaction mechanism (Scheme 2), according to which antioxidant radicals participate in the chain transfer reaction to the inhibitor (reaction 9). This leads to an increase in the initiation rate and, as a consequence, reduces the efficiency of the antioxidant action.

The established reaction mechanism and the determined rate constant values allow us to position these new antioxidants within the broad class of biologically active organic compounds possessing antioxidant properties, including those we studied earlier under the same conditions [25-28]. Although the effective inhibition rate constants for the newly obtained compounds are lower than those for the previously studied ones, for which the values vary within the ranges of 10^4 and 10^5÷10^6 for 1,4-dioxane and isopropyl alcohol, respectively, the appealing feature of these new compounds lies in the availability and relatively low cost of the starting raw materials. It is also important to note that close structural analogs of the newly synthesized compounds are used as pharmaceutical drugs [6-8], which allows the oxime and lactam to be considered promising biologically active compounds.

ACKNOWLEDGEMENTS

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