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ACTIVITY OF SECONDARY AROMATIC AMINES AS CATALYSTS IN THE REACTION OF STERICALLY HINDERED AROXYL RADICALS WITH HYDROPEROXIDES

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UDC 541.128.34:547.553.1:541.515

Secondary aromatic amines AmH catalyze the reaction of the 2,4,6-tri-t-butylphenoxyl radical $ArO^{\cdot}$ with cumyl hydroperoxide ROOH [1-4]. This effect is closely connected with the antioxidant action of the mixtures of sterically hindered phenols and AmH which have a synergistic effect [5] and which are used in practice. The present work is directed to a study of the dependence of the catalytic activity of AmH on the temperature and on the substituents on the aromatic rings.

EXPERIMENTAL

Experimental procedures were as in [1, 3]. Eleven aromatic amines were tested, mostly ring-substituted diphenylamines (Table 1). The tests were carried out in CCl_4 in a current of argon at temperatures of 298.2, 320.1, and 341.6 K. The reactor was a thermostatted cell of 8.0 ml capacity set up in a Specord UV-VIS spectrophotometer. Changes in the concentration of $ArO^{\cdot}$ were determined from the spectra: either $\nu = 25{,}000\ cm^{-1}$, $\varepsilon = 1980$ liters/mole × cm, or $\nu = 16{,}000\ cm^{-1}$, $\varepsilon = 410$ liters/mole·cm [3]. Solutions of all the components of the reaction with the exception of $ArO^{\cdot}$ were transferred to the cell and warmed for a few minutes; after this a solution of $ArO^{\cdot}$ in CCl_4 at room temperature (up to 1.2 ml) was added and the necessary temperature corrections made at the point of commencement of the measurements on the basis of model dummy experiments.

The kinetic curves obtained for the consumption of $ArO^{\cdot}$ were rectified on coordinates of the equation

$$\frac{1}{[AmH]_0}\left\{\frac{[ArO^{\cdot}]_0+2[ArOH]_0}{[ArO^{\cdot}]_0-2[ROOH]_0}\ln\frac{[ArO^{\cdot}]_t}{[ArO^{\cdot}]_0}-2\frac{[ArO^{\cdot}]_0+[ROOH]_0}{[ArO^{\cdot}]_0-2[ROOH]_0}\times\ln\left(1-\frac{[ArO^{\cdot}]_0-[ArO^{\cdot}]_t}{2[ROOH]_0}\right)\right\}=kt \qquad \text{(I)}$$

(or $B = k_s t$) [1] where k_s is an empirical constant. We find the combined rate constant for $ArO^{\cdot}$ consumption from the equation $v_\Sigma = k\,[AmH]_0[ROOH]_0[ArO^{\cdot}]_0/[AmH]_0$, after which the initial rate of the catalytic reaction v is determined by subtracting from v_Σ the initial rate of the consumption of $ArO^{\cdot}$ by the noncatalyzed reaction (v'): $v = v_\Sigma - v'$. The value of v thus obtained is used for calculation of the rate constant for the catalytic process in each experiment, for each AmH, and at each temperature.

All the AmH samples were purified by preparative liquid chromatography [3] and corrections were applied for the ArOH content of the initial $ArO^{\cdot}$ solution.

RESULTS AND DISCUSSION

The reaction of $ArO^{\cdot}$ with ROOH, taking place as follows:

$$ArO^{\cdot} + ROOH \rightarrow ArOH + RO_2^{\cdot} \qquad (1)$$

$$RO_2^{\cdot} + ArO^{\cdot} \rightarrow X\ \text{(Quinolide peroxides)}\quad (QP) \qquad (2)$$

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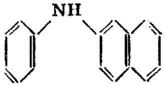
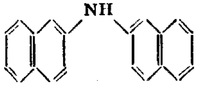
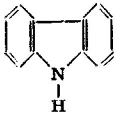
TABLE 1. Experimental Results* from a Study of the Catalytic Activity of AmH on the Reaction of ArO$^\bullet$ with ROOH

Compound	4,4'-substituent of diphenylamine	T, K	$k = K_3k_4$, liter/mole·sec	E, kJ/mole	lg A	E_{-4}	$\Delta H^{\ddagger}_{298}$	$\Delta F^{\ddagger}_{298}$	$-\Delta S^{\ddagger}_{298}$, J/mole·K
						kJ/mole			
(I)	CH_3O, CH_3O	298,2 320,1 341,6	36,8±7,4 84,4±6,4 186,0±14,7	31,5±2,1	7,08±0,35	2,2±2,1	29,0	64,1	118
(II)	CH_3O, H	298,2 320,1 341,6	7,88±0,17 19,6±6,1 43,6±7,7	32,1±7,9	6,49±1,32	2,8±7,9	29,6	68,0	129
(III)	CH_3, CH_3	298,2 320,1 341,6	5,93±0,45 14,8±1,1 32,8±1,7	33,3±2,0	6,61±0,34	4,1±2,0	30,8	68,6	127
(IV)	$(CH_3)_3C$, $(CH_3)_3C$	298,2 320,1 341,6	5,53±0,29 13,0±1,2 29,5±3,9	30,9±3,8	6,16±0,66	1,6±3,8	28,5	68,8	135
(V)	$(CH_3)_3C$, H	298,2 320,1 341,6	2,86±0,27 7,5±0,88 19,5±2,1	37,1±2,4	6,94±0,40	7,8±2,4	34,6	70,4	120
(VI)	H, H	290 [1] 298,2 320,1 341,6	1,05±0,1 1,62±0,06 4,75±0,6 11,5±1,5	38,2±1,1	6,90±0,19	8,9±1,1	35,7	71,8	121

TABLE 1 (Continued)

Compound	4,4'-substituent of diphenylamine	T, K	$k = K_3k_4$, liter/mole·sec	E, kJ/mole	lg A	E_{-4}	$\Delta H^{\ddagger}_{298}$	$\Delta F^{\ddagger}_{298}$	$-\Delta S^{\ddagger}_{298}$, J/mole·K
						kJ/mole			
(VII)	Br, Br	298,2 320,1 341,6	2,82±0,15 6,84±0,47 14,2±1,5	31,4±1,4	5,96±0,24	2,1±1,4	29,0	70,4	139
(VIII)	NO_2, H	298,2 320,1 341,6	0,62±0,11 1,92±0,38 5,24±1,43	41,6±2,2	7,07±0,36	12,3±2,2	39,1	74,2	118
(IX)		290 [2] 298,2 320,1 341,6	2,45±0,15 3,42±0,32 9,85±0,44 20,3±0,78	34,2±1,2	6,54±0,20	4,9±1,2	31,7	69,9	128
(X)		298,2 341,6	6,4±0,7 38,0±2,5	34,8±2,0	6,90±0,34	5,5±2,0	32,3	68,4	121
(XI)		298,2 320,1 341,6	0,16±0,07 0,49±0,25 1,24±0,23	39,6±5,8	6,19±0,96	10,6±5,8	37,4	77,6	135

$ArO^{\bullet} + ROOH \rightarrow ArOH + RO_2^{\bullet}$

		290 [1] 298,2 320,1 341,6	0,157+0,008 0,249+0,014 0,773+0,036 2,031+0,083	40,64±1,06	6,52±0,18		38,2	76,4	128

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*Statistical treatment was carried out at P = 0.95; $\Delta H^{\neq}_{298} = E - 2.48$ (kJ/mole), $\Delta F^{\neq}_{298} = 2.48\,(29.46 - \ln k)$ (kJ/mole), $\Delta S^{\neq}_{298} = (\Delta H^{\neq}_{298} - \Delta F^{\neq}_{298})/298$. A correction was introduced for the thermal expansion of the solvent.

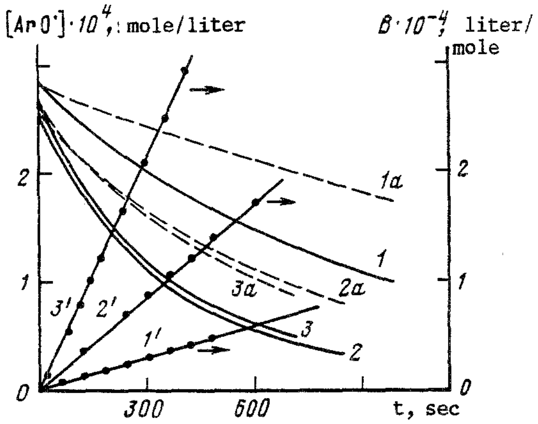

Fig. 1. Kinetic curves of ArO· consumption in the presence of ROOH, ArOH and 4,4-dibromodiphenylamine. Concentrations in moles/liter: 1) 2.81, 10.0, 10.6, 1.25. T = 298.2 K; 2) 2.54, 9.75, 11.6, 1.22, T = 320.1 K; 3) 2.64, 4.43, 25.1, 2.375, T = 341.6 K; 1a) and 3a) curves of ArO· consumption in absence of AmH. 1')-3') rectified curves 1) to 3) on coordinates of Eq. (I).

is accelerated in the presence of AmH (see Fig. 1) as a result of the rate limiting step in (1) being replaced by two, quite rapid, successive reactions (3) and (4) [1-3]:

$$\mathrm{ArO^{\cdot} + AmH \rightleftarrows ArOH + Am^{\cdot}} \tag{3, -3}$$

$$\mathrm{Am^{\cdot} + ROOH \rightarrow AmH + RO_2^{\cdot}}\ . \tag{4}$$

It is possible to set up experimental conditions such that side reactions do not occur in the system and the consumption of ArO· proceeds only according to reactions (1)-(3) with concurrent catalytic and noncatalytic decomposition of ArO· into ArOH and QP in accordance with the scheme given in [3]. Here, stages (3) and (-3) form a quasiequilibrium ($v_3 \approx v_{-3} >> v_4$) and the expressions $v' = 2k_1[\mathrm{ArO^{\cdot}}][\mathrm{ROOH}]$, $v = 2K_3k_4[\mathrm{ArO^{\cdot}}][\mathrm{ROOH}][\mathrm{AmH}]/[\mathrm{ArOH}]$ are valid for v and v', the factor 2 appearing in both expressions as a stoichiometric coefficient.

In order to understand the reasons for the exchange of the endothermic stage (1) for the longer route (reactions (3) and (4)) it is necessary to examine the effect of temperature on k_1 and the effective rate constant $K_3k_4 = k$ for each of the AmH studied. These results are given in Table 1. It should be noted that each value of k given in the Table is a statistical average of 6-12 experiments carried out with different initial concentrations of the reactants but restricted to the conditions which avoided side reactions in the system [3]. The results in Table 1 show that the values of K_3k_4 for all AmH increase with temperature but to different extents. It can be seen from Fig. 2 and Table 1 that for the majority of AmH $\log A \approx \log A_1$ and $E < E_1$. The value of E decreases with increase in the electron-donor properties of the substituent but the dependence of E on the σ^+ constant [6] is not precisely expressed and is more S-shaped than linear (Fig. 2); for (IV) and (VII) there is hardly any such dependence.

The energy profile of the catalyzed reaction is shown in Fig. 3. It can be seen from the figure that by changing the substituent on the rings it is possible to reduce the activation energy by not more than the value of E for the reaction

$$\mathrm{RO_2^{\cdot} + ArOH \rightarrow ROOH + ArO^{\cdot}} \tag{-1}$$

i.e., $E \geq D_{\mathrm{ROO-H}} - D_{\mathrm{ArO-H}} = \Delta H_1 = E_1 - E_{-1}$. The most exact value of ΔH_1 obtained in [7] was $\Delta H_1 = 29.3 \pm 7.1$ kJ/mole. Thus, theoretically, $E \geq 29.3 \pm 7.1$ kJ/mole which is in complete agreement with the results in Table 1. Further, it follows from the results which we obtained that E for diphenylamines with strongly electron-donor substituents (see, for example, for (I)) the theoretically possible limit is almost reached. Hence one must expect considerable further reduction of E for diphenylamines with even stronger electron-donor substituents on the rings. Apparently, the change in the nature of the dependence of E on σ^+ for (I) (Fig. 2) is also connected with this. Knowing ΔH_1 and E_1 it is possible to calculate $E_{-1} = E_1 - \Delta H_1 = (40.6 \pm 1.1) - (29.3 \pm 7.1) = 11.3 \pm 8.2$ kJ/mole which within the limits of experimental accuracy coincides with the value of $E_{-1} = 18.8 \pm 2.5$ kJ/mole for cumene [8]. The quite large difference in the statistical average values of E_{-1} (7.5 kJ/mole) can be ascribed to the

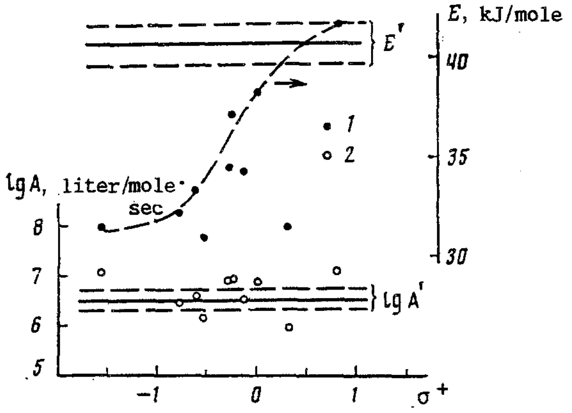

Fig. 2. Dependence of activation energy (1) and the logarithm of the pre-exponential factor A (liter/mole·sec) (2) on σ^+ (CCl_4, Ar).

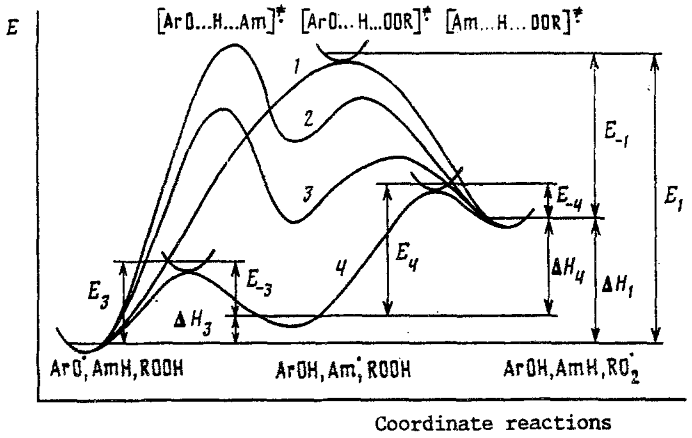

Fig. 3. Diagram of change in potential energy for noncatalyzed (1) and catalyzed (2-4) reactions for N–H bond strengths in AmH greater than (2), equal to (3) and less than (4) the strength of the O–H bond in ROOH.

difference in solvation effect in CCl_4 and aromatic solvents. It should be noted that the expression obtained in $k_1 = 10^{6.52\pm0.18}\exp(-40{,}600\pm 1100)/RT$ (CCl_4) differs somewhat in the value of E from the expression $k_1 = 10^{7.1\pm0.4}\exp(-45{,}600 \pm 2500)/RT$ obtained in a study of reaction (1) in benzene [9] which, apparently, is due to greater solvation of ArO˙ in benzene than in CCl_4.

Since $E = E_3 - E_{-3} + E_4 = \Delta H_3 + \Delta H_4 + E_{-4} = \Delta H_1 + E_{-4}$, then from the data of Table 1 it is possible to find the value of E_{-4} for the reaction

$$RO_2^{\cdot} + AmH \rightarrow ROOH + Am^{\cdot} \qquad (-4)$$

$E_{-4} = E - \Delta H_1 = E - (29.3 \pm 7.1$ kJ/mole). The results obtained are shown in Table 1. The value of E_{-4} increases monotonically with increase in σ^+ and only for (VII) is a deviation again observed. The inaccuracy in the absolute value of ΔH_1 introduces a systematic error into the values found for E_{-4} given in Table 1 are more reliable than those obtained on the basis of a study of the antioxidant activity of AmH.

The variation of log (K_3k_4) with σ^+ for a temperature of 298.2 K is shown in Fig. 4 from which it can be seen that for diphenylamines with electron-donor substituents there is a close dependence which is also preserved at other temperatures.

$$\lg (K_3k_4)_{298.2} = (0.240 \pm 0.061) + (-0.860 \pm 0.077)\sigma^+ \quad (\rho = 0.9976)$$
$$\lg (K_3k_4)_{320.1} = (0.678 \pm 0.025) + (-0.802 \pm 0.032)\sigma^+ \quad (\rho = 0.9995)$$
$$\lg (K_3k_4)_{341.6} = (1.065 \pm 0.040) + (-0.766 \pm 0.051)\sigma^+ \quad (\rho = 0.9987).$$

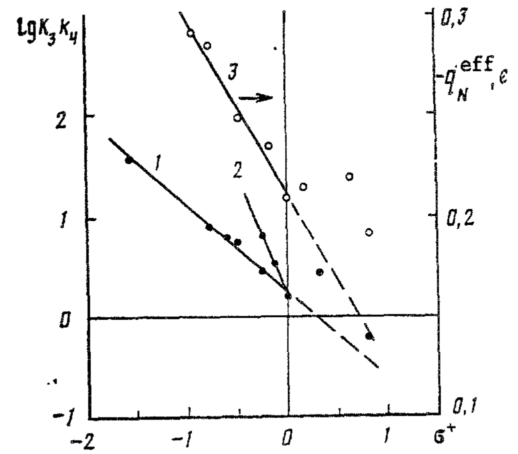

Fig. 4. Dependence of log (K_3k_4) on σ^+; 1) para-substituted diphenylamines; 2) the series diphenylamine—N-phenyl-2-naphthylamine—N,N-di-2-naphthylamine (CCl_4, 298.2 K); 3) dependence of the effective charge on the N atom of the amine radical on σ^+ (from the data of [10]).

The reduction in the absolute value of the σ^+ multiplier as the temperature increases will be noted; there is a tendency to a levelling out of the activity of these AmH as catalysts with increase in temperature.

The existence of a correlation between log (K_3k_4) and σ^+ makes it possible to suggest that there is a link between the values of (K_3k_4) and the charge characteristics of the reaction center (the N atom). For 4,4'-disubstituted diphenylamine radicals the following values are found for $-q_N^{ef}$ (e) [10]: (H, OCH_3) —0.2832; (H, H) —0.2086; (H, Br) —0.2142; (H, NO_2) —0.1920 (Ph, OCH_3) —0.2891; (Ph, CH_3) —0.2482; (Ph, H) —0.2343, and (Ph, NO_2) —0.2212. In fact there is good correlation between log (K_3k_4) and $-q_N^{ef}$ (Fig. 4), there being both electron-donor and electron-acceptor substituents, including even bromine derivatives, on the AmH.

The activation parameters for both catalyzed and noncatalyzed reactions are given in Table 1. One point that stands out is the high entropy loss in each route. For the majority of amines $\Delta S^{\neq} \approx \Delta S^{\neq}_1$ and only for (I) and (VIII) is $\Delta S^{\neq} > \Delta S^{\neq}_1$. In the case of AmH with bulky substituents the inequality is reversed. Since $\Delta S^{\neq} = \Delta S^{\neq}_5 - \Delta S^{\neq}_3 + \Delta S^{\neq}_4$ it is impossible to arrive at a reliable evaluation of the contribution of each separate component to the overall $\Delta S^{\neq}$. Some elaboration can, however, be introduced into the basic Hammond postulate if the strengths of the N—H bonds in the AmH molecule are known. In this way one can arrive at a qualitative explanation of the small sensitivity of $\Delta S^{\neq}$ to the nature of the para-substituent. The reason for the deviation in the behavior of (VII) and (IV) could be the interaction of the two bulky substituents which evidently distort the geometry of the AmH molecule and its radical Am$^\bullet$. In almost all cases $\Delta F^{\neq} < \Delta F^{\neq}_1$, i.e., the majority of AmH satisfy the classical definition of a catalyst. As one would expect (see Fig. 4), the values of $\Delta F^{\neq}$ for all diphenylamines and N-naphthylamines are found to bear a linear relationship to σ^+

$$\Delta F^{\neq} = (70.67 \pm 0.70) + (3.97 \pm 1.04)\sigma^+ \text{ (kJ/mole)} \qquad (\rho = 0.9500).$$

It needs to be specified that up to the present the talk has not been about v and v' but about the rate constants (k_1 and K_3k_4) or about the standard changes in the functions on formation of activated complexes. Since [AmH] and [ArOH] enter into the equation for v, then any of the AmH of Table 1 is able to produce considerable (catalytic) acceleration of the reaction if one carries out experiments with sufficiently high $[AmH]_0$ and relatively low $[ArOH]_0$. It is also understood that v can exceed v' to a considerably greater extent than is possible as a result of the reduction of E by ~10 kJ/mole by the catalytic route (see Table 1). In fact, in contrast to catalysis with Arrhenius intermediates, in our case v depends not only on catalyst concentration but on the concentration of one of the reaction products: $v \propto [ArOH]^{-1}$. Since $[ArOH] \ll 1$ then $[ArOH]^{-1} \gg 1$. Hence, changing $[AmH]_0$ and/or $[ArOH]_0$ can have a considerable effect on v. Furthermore, if the experiments are carried out with small initial [ArOH] and the quantity of ArOH formed in the course of the experiment is comparable to, or even exceeds, $[ArOH]_0$, then v will fall continuously not only as a result of consumption of

TABLE 2. Estimated Parameters Based on Studies of the Catalytic Activity of Aromatic Amines (I)-(XI)

Parameters	(I)	(II)	(III)	(IV)	(V)	(VI)	(VII)	(VIII)	(IX)	(X)	(XI)
Q, kJ/mole	22,3	20,3	16,0	24,3	3,7	0	22,6	−4,9	13,3	11,3	−2,4
$D_{\mathrm{Am-H}}$, kJ/mole	345,9	347,9	352,2	343,9	364,5	368,2	345,6	373,1	354,9	356,9	370,6
E_4, kJ/mole	24,5	23,1	20,1	25,9	11,5	8,9	24,7	7,4	18,2	16,8	8,2
$k_{-4}^{348,5} \cdot 10^{-6}$, liter/mole·sec	3,4	2,8	1,8	4,2	0,5	0,34 *	3,6	0,11	1,4	1,1	0,19
$k_4^{318,5} \cdot 10^{-3}$, liter/mole·sec	0,5	0,8	2,3	0,3	45	110 *	0,4	180	4,4	7,2	140
$K_3^{348,5}$	0,46	$5,8 \cdot 10^{-2}$	$1,8 \cdot 10^{-2}$	0,11	$5,3 \cdot 10^{-4}$	$1,4 \cdot 10^{-4}$	$3,8 \cdot 10^{-2}$	$3,8 \cdot 10^{-5}$	$5,9 \cdot 10^{-3}$	$6,7 \cdot 10^{-3}$	$1,3 \cdot 10^{-5}$

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*Standard value.

the reagents ($ArO\cdot$ and ROOH) but also as a result of the accumulation of ArOH. Thus, the catalytic reaction which we are considering is auto-inhibiting, the final products taking the part of inhibitors.

From the results in Table 1, it is possible to obtain evaluations of several quantities for which there are still no reliable experimental data. From the Polyani—Semenov formula $E_{-4} = \mathrm{const} - \alpha Q_{-4}$ one can calculate the heat effect Q and the strength of the N—H bond in AmH (D_{Am-H}). Assuming const = E_{-4} for diphenylamine (because here $D_{Am-H} \approx D_{ROO-H}$ [11, 12], i.e., $Q \approx 0$) and $\alpha = 0.3$ (as for a series of reactions of $RO_2^{\cdot}$ with phenols [13]), we obtain: $Q = (8.9 - E_4)\cdot 3.33$ and $D_{Am-H} = D_{ROO-H} - Q = 368.2 - Q$ (kJ/mole), if we take $D_{ROO-H} = 368.2$ kJ/mole [14]. From the relationship $E_4 = Q_{-4} + E_{-4}$ one can also find E_4. The results of these evaluations are given in Table 2. Using the formula

$$\ln k_i = \ln k_{i\,\mathrm{std}} - \frac{E_i - E_{i\,\mathrm{std}}}{RT}$$

one can arrive at the absolute values of k_{-4} and k_4 for all AmH. We take as standard diphenylamine, for which $k_{-4} = 3.4\cdot 10^5$ and $k_4 = 1.1\cdot 10^5$ liters/mole·sec at 348.5 K [11]. Knowing k_4 and calculating $k = K_3 k_4$ we can also estimate K_3. It is understood that in such a procedure the systematic errors in the values of E_i and $E_{i\,\mathrm{std}}$ compensate each other and that the reliability of the value for k_{-4} so obtained is higher than that for k_4 and consequently for K_3. It can be seen from Table 2 that whereas the values of k_{-4} are relatively insensitive to the nature of the para-substituent in AmH, the values of the other parameters change quite considerably. Although it is necessary in the end to consider the data in Table 2 as only tentative, they agree well with values and estimates found in the literature (see, for example, [12]). It is known that the diphenylamine radical does not split off H from toluene very rapidly even on heating [15], and such a reaction occurs with ethylbenzene only with difficulty (k = 0.7 liter/mole·sec at 348.5 K [16]). The 4,4'-dinitrodiphenylamine radical dehydrogenates toluene even at room temperature [17]. As we have seen, this does not contradict the results of Table 2 nor does it contradict the dependence, noted in [17], of the facile replacement of $Am_1^{\cdot}$ by $Am_2^{\cdot}$ on generating $Am_1^{\cdot}$ in the presence of Am_2H on the nature of the substituent on the radical and the amine. The values of k_{-4} characterize the potential ability of AmH to break the oxidation chain, and k_4 is a measure of the reduction in the antioxidant activity of AmH as a result of extending the oxidation chain through the amine radical. Aromatic amines can thus, depending on their k_{-4} values, considerably surpass antioxidants based on sterically hindered phenols, differing from the latter also in their small E_{-4} values.

Thus, the quantitative study of the dependence of the catalytic activity of AmH in the reaction of $ArO^{\cdot}$ with ROOH on the temperature and the nature of the substituents not only facilitates a deeper understanding of the mechanisms of and reasons for the catalysis but it also makes it possible to draw conclusions regarding the relative reactivity of $Am^{\cdot}$ and the effectiveness of AmH as an antioxidant.

The authors express their thanks to E. T. Denisov for advice and comments.

CONCLUSIONS

1. Secondary aromatic amines are catalysts in the reaction of sterically hindered aroxyl radicals with hydroperoxides. The rate of the catalytic reaction v is proportional to [$ArO^{\cdot}$], [ROOH], [AmH], and inversely proportional to [ArOH].

2. Values of k_{cat} have been found for eleven aromatic amines in reaction with 2,4,6-tri-t-butylphenoxyl radical with cumyl hydroperoxide in CCl_4 over the temperature range 298.2-341.6 K. The values of E and $\Delta F^{\neq}$ fall with increasing electron-donor properties of the substituents on the aromatic ring of the amine. On the basis of the values of k which were obtained, several parameters have been estimated including the strength of the N—H bond in the amine molecule.

3. For diphenylamines with electron-donor substituents there is a linear relationship between log k and the σ^+ function.

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