

Isomeric Forms of Arylnitroso Oxides: Electronic Spectra and Reactivity

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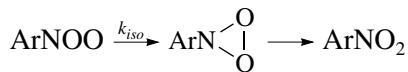
Received April 6, 2005

Abstract—The electronic spectra were measured and the kinetics of unimolecular decomposition of the isomeric forms (*cis* and *trans*) of phenylnitroso oxide, (4-methylphenyl)nitroso oxide, (4-nitropenyl)nitroso oxide, (4-bromophenyl)nitroso oxide, 4-(*N,N*-dimethylamino)phenylnitroso oxide, and (4-methoxyphenyl)nitroso oxide in acetonitrile, benzene, and *n*-hexane was studied using flash photolysis. In all of the nitroso oxides except for 4-(*N,N*-dimethylamino)phenylnitroso oxide, the *cis* form absorbed in a shorter wavelength region and was more labile than the *trans* form. The difference between the reactivity of the two species increased on going from *n*-hexane to acetonitrile. The temperature dependence of reaction rate constants was studied for both of the isomeric forms. Unlike the *trans* isomer, the *cis* isomer almost did not react with tetramethylethylene.

DOI: 10.1134/S0023158406040100

INTRODUCTION

Nitroso oxides are intermediates in the photooxidation of aromatic azides; they are formed by the interaction of triplet nitrenes with oxygen [1, 2]. In the absence of an oxidation substrate from the system, they undergo unimolecular conversion into cyclic isomers, dioxaziridines. In turn, dioxaziridines afford corresponding nitro compounds as stable reaction products (Scheme) [3, 4].



Scheme.

The molecular structure of nitroso oxides implies the occurrence of isomeric *cis* (**I**) and *trans* (**II**) conformations:



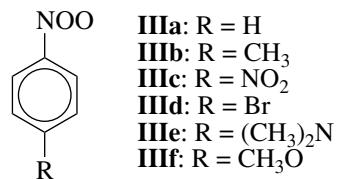
I

II

Gritsan and Pritchina [5] detected the formation of *cis*-(4-aminophenyl)nitroso oxide (**I**, R = 4-NH₂) and *trans*-(4-aminophenyl)nitroso oxide (**II**, R = 4-NH₂) with absorption maximums at 495 and 588 nm, respectively, in the photolysis of 4-aminophenyl azide in a low-temperature vitreous matrix of THF–toluene (1 : 1) in the presence of oxygen at 77 K. The assignment was made based on the quantum-chemical calculations of the electronic spectra of these species. The mutual conversion of the two species was observed under irradia-

tion with light at a wavelength of 436 or 578 nm. The *cis* form was found more stable, and it remained the only product as the temperature was increased to 100 K.

To our knowledge, quantitative experimental data on the reactivity of isomeric nitroso oxide species are absent from the literature. In this work, we managed to obtain the rate constants of decay of the *cis* and *trans* isomers of nitroso oxides in addition to the electronic spectra. Previously, we started with a study of the effect of the electronic properties of substituents and the nature of solvents on the reactivity of aromatic nitroso oxides [4, 6–8]. The kinetics of decay of arylnitroso oxides **IIIa**–**IIIe** was studied:



It was found that all of these arylnitroso oxides were consumed in unimolecular reactions. The rate constants of decay for **IIIa**–**IIIe** in acetonitrile at room temperature varied over the range 0.09–0.24 s⁻¹ [4]; in the case of **IIIa**, this rate constant changed from 0.10 s⁻¹ in acetonitrile to 0.36 s⁻¹ in *n*-hexane [7]. The rate constant of decay of **IIIe** essentially depended on the nature of the solvent; it increased from 1.16 s⁻¹ in *n*-hexane or 2.3 s⁻¹ in benzene to 214 s⁻¹ in acetonitrile [7].

In this work, we studied the kinetics of decay of nitroso oxide **IIIf** in acetonitrile, benzene, and *n*-hex-

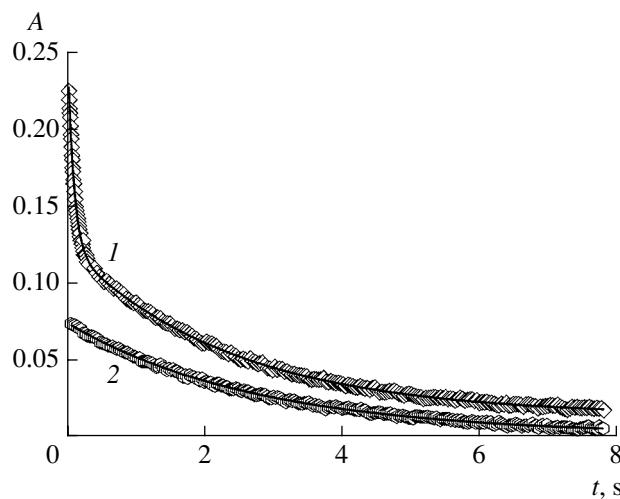
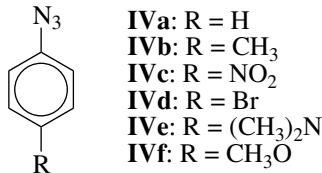


Fig. 1. Kinetic curves of decay of the absorbance of **III**f in an acetonitrile solution measured at wavelengths of (1) 440 and (2) 500 nm ($T = 297$ K). Solid lines refer to theoretical kinetic curves.

ane by flash photolysis with time-resolved spectrophotometric detection (for a preliminary communication, see [9]). The results of this study were unexpected, and they allowed us to newly consider previously obtained data on the kinetics of consumption of nitroso oxides **IIIa–IIIe**.

EXPERIMENTAL

Acetonitrile, benzene, and *n*-hexane were purified in accordance with a well-known procedure [10]. Azide **IVa** was synthesized according to Lindsay and Allen [11]; compounds **IVb–IVf** were synthesized according to Smith and Boyer [12]. Compounds **IVa** and **IVb** were distilled in a vacuum; **IVc** and **IVd** were recrystallized from ethanol, and **IVf** was recrystallized from *n*-hexane; **IVe** was purified on a column with Silica Gel 60 from Merck (eluent: *n*-hexane–ethyl acetate (2–3 vol %)).



Kinetic experiments were performed using a system for flash photolysis with time-resolved spectrophotometric detection of a well-known design [13]. The photolytic source was an IFP 5000-2 lamp; a maximum pulse energy was 400 J at $U = 5$ kV; $C = 32$ μ F; ~90% light energy was emitted in 50 μ s. Diffraction grating no. 1 (operating wavelength region of 200–500 nm) or diffraction grating no. 2 (400–700 nm) was used in a monochromator. The reactor was a quartz cell with the optical path length $l = 10$ cm and an inner diameter of

~1 cm. The flash photolysis of the aryl azide–solvent–air (O_2 in the case of **IVc**) system was performed with filtered light (UFS-2 light filter; transmittance range $\lambda = 270$ –380 nm). The initial concentration of aryl azides was $(1\text{--}25) \times 10^{-5}$ mol/l.

RESULTS AND DISCUSSION

*Kinetics of Decay and the Electronic Spectra of 4-Methoxyphenylnitroso Oxide **III**f*

A signal in the wavelength region 390–500 nm was observed in the flash photolysis of **IVf** in solutions saturated with atmospheric oxygen. This signal decreased as the concentration of oxygen in solution decreased; this allowed us to attribute this signal to the absorption of nitroso oxide **III**f. In acetonitrile and benzene, the kinetic curves of decay of the absorbance of **III**f (A) measured at $\lambda \leq 480$ nm consisted of two segments (fast and slow) and contained residual absorbance due to the absorption of reaction products (Fig. 1, curve 1). At $\lambda \geq 490$ nm, the kinetic curves were uniform; they consisted of only a slow segment (Fig. 1, curve 2) and were adequately described by first-order rate Eq. (1), which takes into account the formation of reaction products that absorb at the given wavelength and transforms into an ordinary first-order equation in the absence of these products ($A_\infty = 0$):

$$A - A_\infty = (A_0 - A_\infty) e^{-kt}. \quad (1)$$

The kinetic curves that consist of two segments ($\lambda \leq 480$ nm; Fig. 1, curve 1) correspond to the consumption of two intermediates different in reactivity. Therefore, the following five-parameter biexponential equation was used for the treatment of these curves:

$$A = A_\infty + A_0^I e^{-k^I t} + A_0^{II} e^{-k^{II} t}, \quad (2)$$

where A_0^I , A_0^{II} , k^I , and k^{II} are the initial absorbances and rate constants of unimolecular decay of the former and latter species, respectively; A_∞ is the final absorbance of due to reaction products. The kinetic curves of consumption of **III**f in acetonitrile and benzene measured at $\lambda \leq 480$ nm were adequately described by Eq. (2) (Fig. 1, curve 1). The rate constants k^{II} thus calculated were equal to the constants obtained by the treatment of kinetic curves that consisted of only a slow segment ($\lambda \geq 490$ nm) using Eq. (1).

In *n*-hexane, the kinetic curves did not clearly exhibit two segments; however, they were inadequately described by Eq. (1) in the wavelength region of $\lambda \leq 440$ nm (Fig. 2a), and the rate constant somewhat increased on shifting to the short-wavelength region. It is likely that we were concerned with two species in this case; however, the contribution of a more reactive species to the overall kinetics of consumption of **III**f was small. The theoretical kinetic curves calculated from

Eq. (2) very adequately described the experimental kinetic curves obtained in *n*-hexane at $\lambda \leq 440$ nm (Fig. 2b).

The absorption spectra of the two species were constructed from the calculated initial absorbances (Fig. 3). The spectrum of a more active species was shifted toward the short-wavelength region. The positions of the absorption maximums of both of the intermediates in acetonitrile and benzene were shifted toward longer wavelengths, as compared with that in *n*-hexane. In this case, this shift was more significant for a longer lived species (Table 1). It can also be seen in Table 1 that the difference $\Delta\lambda_{\max}$ between the positions of the absorption maximums of the two species increased from 15 nm in *n*-hexane to 30 nm in acetonitrile.

By analogy with published data [5], it is believed that the intermediates with the absorption spectra in the short-wavelength and long-wavelength regions are the *cis* and *trans* forms of nitroso oxide **III**f, respectively. Note that, unlike published data [5], the *cis* isomer is more labile in the case under discussion. Table 2 summarizes the rate constants of decay of **III**f; in essence, this reaction is isomerization to a corresponding dioxaziridine (Scheme). The nature of the solvent had an opposite effect on the stability of the isomeric forms of **III**f: the reactivity of the *cis* form of the nitroso oxide increased, whereas that of the *trans* form decreased on going from *n*-hexane to acetonitrile. As a result, two segments can be clearly seen in the kinetic curves of consumption of **III**f in acetonitrile and benzene measured at $\lambda \leq 480$ nm.

Kinetics of Decay and the Electronic Spectra of Arylnitroso Oxides **III**a–**III**e

Previously, we studied the kinetics of decay of nitroso oxides **III**a, **III**b, and **III**d in acetonitrile and benzene. We found that the kinetic curves measured at wavelengths shorter than a certain value, which depends on nitroso oxide structure, were inadequately described by Eq. (1) and the rate constant increased on shifting to the short-wavelength region. That is, a behavior analogous to that found for the consumption of **III**f in *n*-hexane (Fig. 2) was observed. Previously, we failed to explain the effect observed. In this case, the kinetics of nitroso oxide consumption was studied over the spectral range where the rate constant was independent of wavelength. Thus, we monitored the decay of the *trans* isomers of corresponding nitroso oxides. The kinetic curves of decay of **III**a, **III**b, and **III**d in *n*-hexane were adequately described by first-order Eq. (1) over the entire wavelength range, and the resulting rate constants were equal to within the experimental error. Note that, in the study of the absorption spectra of nitroso oxides, the test compounds were generated in low concentrations in order to minimize the consumption of parent azides. More recently, we found that two segments could be observed in the kinetic curves of

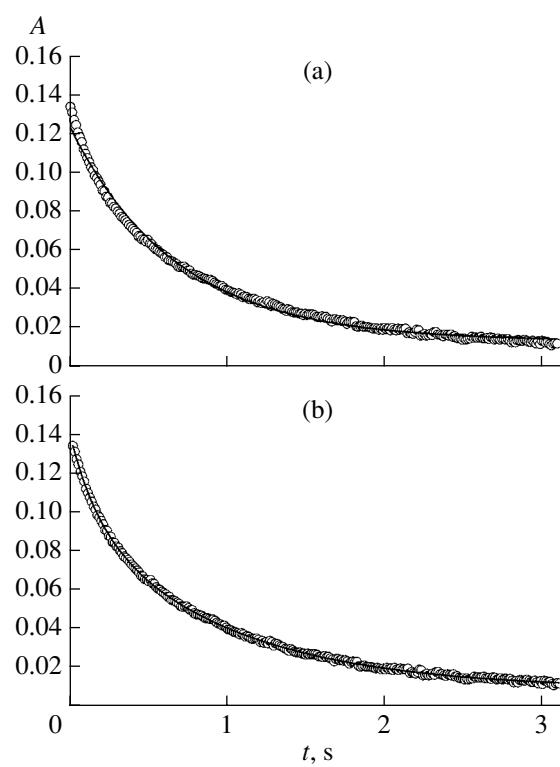


Fig. 2. Kinetic curve of decay of the absorbance of **III**f in an *n*-hexane solution measured at a wavelength of 390 nm and its theoretical descriptions by (a) Eq. (1) and (b) Eq. (2). $T = 297$ K.

consumption of **III**a ($\lambda \leq 420$ nm), **III**b ($\lambda \leq 430$ nm), and **III**d ($\lambda \leq 420$ nm) in acetonitrile and benzene if the pulse energy was increased and nitroso oxides were

Table 1. Absorption maximums of the isomeric forms of nitroso oxides (λ_{\max} , nm). $T = 295 \pm 2$ K

Nitroso oxide		Solvent		
		CH_3CN	C_6H_6	$n\text{-C}_6\text{H}_{14}$
III e	<i>cis</i> -	500	500	470
	<i>trans</i> -	600	550	520
III f	<i>cis</i> -	430	430	410
	<i>trans</i> -	460	450	425
III b	<i>cis</i> -	390	380	390
	<i>trans</i> -	425	410	
III a	<i>cis</i> -	380	380	360
	<i>trans</i> -	405	400	
III d	<i>cis</i> -	380	380	380
	<i>trans</i> -	415	410	
III c	<i>cis</i> -	380	–	–
	<i>trans</i> -	420	–	–

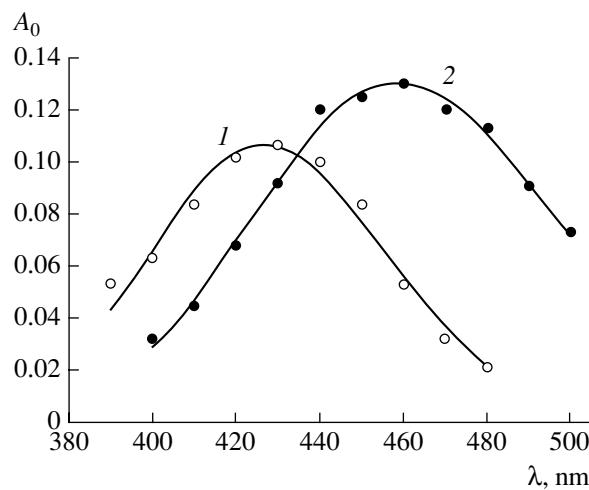


Fig. 3. Optical absorption spectra of the (1) *cis* and (2) *trans* forms of nitroso oxide **III**f in acetonitrile. $T = 297$ K.

generated in sufficiently high initial concentrations. The available experimental data were treated using biexponential Eq. (2), and the optical spectra (Table 1, Fig. 4) and the rate constants of decay (Table 2) of the isomeric forms of nitroso oxides **IIIa**, **IIIb**, and **IIIc** were obtained. The isomers of these three nitroso oxides in *n*-hexane were spectrally indistinguishable and consumed at equal rate constants. Unsubstituted

Table 2. Rate constants of decay of the isomeric forms of nitroso oxides (s^{-1}). $T = 295 \pm 2$ K

Nitroso oxide		Solvent		
		CH ₃ CN	C ₆ H ₆	<i>n</i> -C ₆ H ₁₄
IIIe	k_{iso}^I	150 ± 5	2.3 ± 0.2	1.86 ± 0.03
	k_{iso}^{II}	260 ± 10	8 ± 1	9.9 ± 0.5
III f	k_{iso}^I	11.3 ± 0.2	6.9 ± 0.1	4.7 ± 0.6
	k_{iso}^{II}	0.37 ± 0.02	0.56 ± 0.04	1.00 ± 0.05
III b	k_{iso}^I	2.25 ± 0.06	1.6 ± 0.2	0.26 ± 0.02
	k_{iso}^{II}	0.17 ± 0.01	0.23 ± 0.02	
III a	k_{iso}^I	1.19 ± 0.06	0.45 ± 0.04	0.30 ± 0.03
	k_{iso}^{II}	0.10 ± 0.01	0.12 ± 0.01	
III d	k_{iso}^I	1.45 ± 0.02	1.4 ± 0.1	0.28 ± 0.03
	k_{iso}^{II}	0.14 ± 0.01	0.19 ± 0.01	
III c	k_{iso}^I	0.09 ± 0.01	—	—
	k_{iso}^{II}			

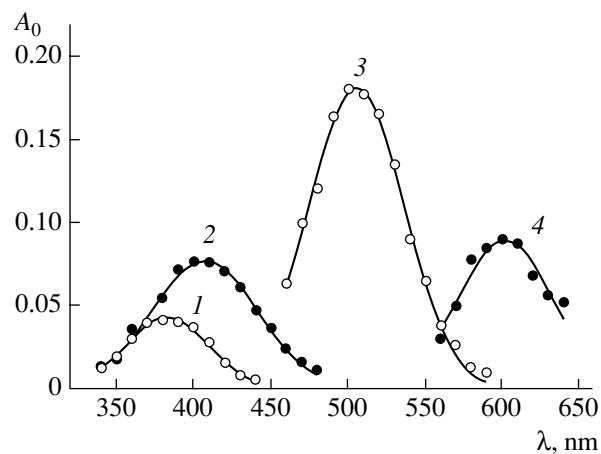


Fig. 4. Optical absorption spectra of the (1) *cis* and (2) *trans* forms of nitroso oxide **III**a and the (3) *cis* and (4) *trans* forms of nitroso oxide **III**e in acetonitrile. $T = 297$ K.

compound **III**a in benzene and acetonitrile exhibited shifts of the absorption maximums of both of the species to the long-wavelength region, as compared with that in *n*-hexane. In the case of **III**b and **III**d, the position of the absorption maximum of a *cis* isomer was independent of the nature of the solvent, whereas the spectrum of a *trans* isomer in benzene or acetonitrile was shifted to the red region (Table 1). Analogously to the case of **III**f, the reactivity of the *trans* forms of nitroso oxides **III**a, **III**b, and **III**d decreased and the reactivity of the *cis* forms increased on going from *n*-hexane to acetonitrile (Table 2).

Previously [7], monochromator diffraction grating no. 1 was used in a study of the kinetics of the decomposition of nitroso oxide **III**e (see Experimental). With diffraction grating no. 2, which allowed us to operate over a longer wavelength range, in place of the above diffraction grating, we found that, upon shifting to the red region of the spectrum, the absorbance curves of **III**e were inadequately described by Eq. (1) starting at 500 nm in *n*-hexane, 530 nm in benzene, and 550 nm in acetonitrile. In this case, the rate of nitroso oxide consumption increased analogously to that for **III**a, **III**b, **III**d, and **III**f upon shifting toward short wavelengths. Consequently, the *trans* isomer of nitroso oxide **III**e, which absorbs light in the long-wavelength region, was more reactive in this case. The resulting kinetic curves were adequately described by Eq. (2). In acetonitrile at $\lambda \geq 600$ nm, only the *trans* isomer absorbed light, and the kinetic curves of its decay corresponded to first-order Eq. (1). Table 1 summarizes the absorption maximums of the isomeric forms of **III**e, and Fig. 4 shows the optical spectra in acetonitrile. As can be seen in Table 1, the presence of a very strong electron-acceptor substituent in the arylnitroso oxide molecule resulted in, first, a considerable shift of the absorption spectra of both of the conformers to the long-wavelength region, as compared with those of other nitroso oxides, and,

Table 3. Activation parameters (E_a , kJ/mol) of the decay reaction of the isomeric forms of nitroso oxides in various solvents

Nitroso oxide		CH ₃ CN		C ₆ H ₆		<i>n</i> -C ₆ H ₁₄	
		log A	E_a	log A	E_a	log A	E_a
IIIe	<i>cis</i> -	$T = 288\text{--}338\text{ K}$		$T = 288\text{--}323\text{ K}$		$T = 285\text{--}323\text{ K}$	
	<i>trans</i> -	3.7 ± 0.1	7.8 ± 0.6	11.9 ± 0.2	65 ± 1	11.0 ± 0.4	61 ± 2
IIIff	<i>cis</i> -	$T = 287\text{--}323\text{ K}$		$T = 279\text{--}337\text{ K}$		$T = 285\text{--}323\text{ K}$	
	<i>trans</i> -	4.8 ± 0.1	13.7 ± 0.3	$T = 279\text{--}337\text{ K}$		11.8 ± 0.6	62 ± 3
IIIa	<i>cis</i> -	$T = 276\text{--}333\text{ K}$		$T = 279\text{--}337\text{ K}$		$T = 276\text{--}333\text{ K}$	
	<i>trans</i> -	11.7 ± 0.1	60.5 ± 0.3	12.4 ± 0.1	65.5 ± 0.5	11.5 ± 0.2	61 ± 1
IIIId	<i>cis</i> -	$T = 276\text{--}333\text{ K}$		$T = 279\text{--}337\text{ K}$		$T = 276\text{--}333\text{ K}$	
	<i>trans</i> -	11.4 ± 0.2	67.2 ± 0.9	12.2 ± 0.1	70.4 ± 0.6	11.8 ± 0.1	66.9 ± 0.7
IIIa	<i>cis</i> -	$T = 293\text{--}333\text{ K}$		$T = 293\text{--}333\text{ K}$		$T = 293\text{--}333\text{ K}$	
	<i>trans</i> -	12.6 ± 0.2	70 ± 1	$T = 293\text{--}333\text{ K}$		$T = 293\text{--}333\text{ K}$	
IIIId	<i>cis</i> -	$T = 282\text{--}323\text{ K}$		$T = 282\text{--}323\text{ K}$		$T = 282\text{--}323\text{ K}$	
	<i>trans</i> -	12.4 ± 0.1	68.9 ± 0.6	$T = 282\text{--}323\text{ K}$		$T = 282\text{--}323\text{ K}$	
		8.8 ± 0.2	54 ± 1				

second, an increase in $\Delta\lambda_{\max}$ to 50 nm in *n*-hexane and benzene or 100 nm in acetonitrile. Table 2 summarizes the rate constants of decay of **IIIe**. The values for the *cis* isomer were somewhat corrected as compared with previously published data [7]: 1.86 s⁻¹ in place of 1.16 s⁻¹ in *n*-hexane and 150 s⁻¹ in place of 214 s⁻¹ in acetonitrile. Note that, analogously to (4-aminophenyl)nitroso oxide [5], the *trans* isomer of **IIIe** is more labile than the *cis* isomer in all of the solvents used (Table 2); this distinguishes these two nitroso oxides from the others studied in this work. A polar solvent significantly increased the rate constants of decay of both of the isomeric forms of **IIIe**.

The absorption spectrum of nitroso oxide **IIIc** in acetonitrile exhibited two maximums at 380 and 420 nm [4]; however, the rate constant of consumption was independent of wavelength. That is, it is most likely that both of the isomers were consumed at equal rate constants in this case (Table 2).

Activation Parameters of the Isomerization of Arylnitroso Oxides

Table 3 summarizes the temperature dependence of the rate constants of decay of **IIIa** and **IIIId**–**IIIff**. In the case of **IIIff**, the activation energy of the *trans* form in all of the solvents was higher than that of the *cis* form by 5–7 kJ/mol, whereas the preexponential factors were equal to within the experimental error. In the case of **IIIe** in a nonpolar solvent, the activation energies of

decay of both of the species were equal, whereas the activation energies dramatically decreased in acetonitrile. Moreover, the activation energy of the *trans* conformer was almost twice as high as that of the *cis* conformer. The decrease in the activation energy was accompanied by a decrease in the preexponential factor. For unsubstituted phenylnitroso oxide **IIIa** and compound **IIIId** containing an electron-acceptor substituent in acetonitrile, the activation energies of decay of *trans* isomers were lower than those of *cis* isomers by 8 and 15 kJ/mol, respectively (Table 3). Figure 5 demonstrates the temperature dependence of the rate constants of decay of nitroso oxides **IIIa** and **IIIe**.

Comparative Reactivity of Arylnitroso Oxides toward Tetramethylethylene

Previously [8], we studied the reaction kinetics of arylnitroso oxides with olefins. We found that they are electrophilic species and electron-donor substituents at the olefin double bond, and electron-acceptor substituents at the aromatic ring of a nitroso oxide increased the rate constant of reaction. Tetramethylethylene is one of the most reactive olefins. In this work, we attempted to measure the reactivity of the isomeric forms of nitroso oxides toward this olefin using compounds **IIIId** and **IIIff** in acetonitrile. The kinetic curves of consumption of **IIIId** and **IIIff** in the presence of tetramethylethylene, which were measured at wavelengths at which both of the isomeric forms of nitroso oxides absorb, were treated with the use of Eq. (2). The dependence of the

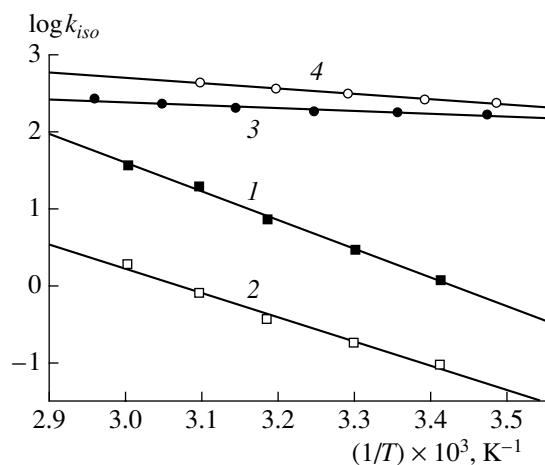


Fig. 5. Temperature dependence of the rate constants of decay of the following nitroso oxides: (1) *cis* form of **IIIa**, (2) *trans* form of **IIIa**, (3) *cis* form of **IIIe**, and (4) *trans* form of **IIIe**.

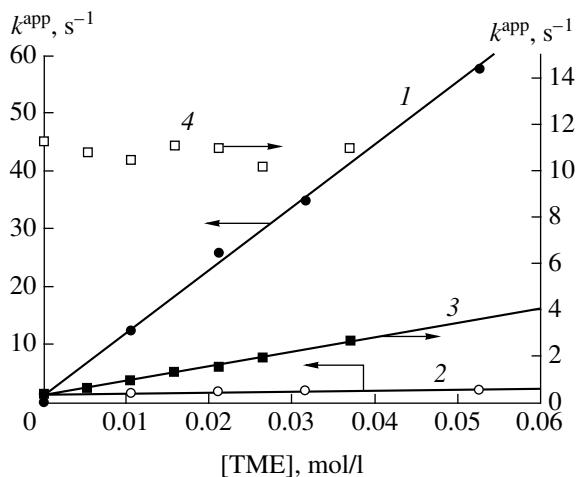


Fig. 6. Dependence of the apparent rate constants of consumption of the (1) *trans* and (2) *cis* isomers of nitroso oxide **IIId** and the (3) *trans* and (4) *cis* isomers of nitroso oxide **IIIf** on the concentration of tetramethylethylene in acetonitrile solutions. $T = 295$ K.

apparent rate constants of consumption of the *cis* and *trans* isomers of **IIId** and **IIIf** on the concentration of tetramethylethylene in solution was obtained (Fig. 6). The absolute rate constants of the reactions of the isomeric forms of nitroso oxides with tetramethylethylene (TME) were found from the slopes of these functions. It was found that the *cis* form of **IIIf** does not react with the olefin at all, whereas the rate constant of reaction of the *trans* form is $k_{\text{TME}}^{\text{II}} = 64 \pm 2$ l mol⁻¹ s⁻¹. In the case of **IIId**, the *cis* form reacted with tetramethylethylene with the very small rate constant $k_{\text{TME}}^{\text{I}} = 9 \pm 2$ l mol⁻¹ s⁻¹, and the rate constant of the *trans* form was $k_{\text{TME}}^{\text{II}} =$

$(1.09 \pm 0.03) \times 10^3$ l mol⁻¹ s⁻¹, that is, higher by two orders of magnitude. At a sufficiently high concentration of tetramethylethylene (0.21 mol/l), only unreactive *cis* isomer of nitroso oxide **IIId** remained in the system; we managed to obtain the absorption spectrum of this isomer separately from that of the *trans* isomer. This spectrum in the wavelength region 350–430 nm with a maximum at 390 nm was almost coincident with the absorption spectrum of the *cis* isomer of **IIId** obtained by the treatment of the kinetic curves of decay of the absorbance of **IIId** measured in the absence of TME in the wavelength range where both of the species absorb (Table 1).

ACKNOWLEDGMENTS

This work was supported by the Department of Chemistry and Materials Science of the Russian Academy of Sciences (program “Theoretical and Experimental Studies of the Nature of Chemical Bonds and Mechanisms of the Most Important Chemical Reactions and Processes”) and by the RF Ministry of Education and Science (analytical departmental purpose program “Development of the Scientific Potential of the Higher School,” project RNP.2.2.1.1.6332).

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