## Chemiluminescence in the reaction of the Ru<sup>II</sup> trisbipyridyl complex with dimethyldioxirane

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The reaction of dimethyldioxirane (1) with the Ru<sup>II</sup> trisbipyridyl complex accompanied by chemiluminescence (CL) was studied. It is established that the intensity of CL and the rate of its decay increase proportionally with the concentration of Ru<sup>II</sup>. The bimolecular rate constant ( $k_2$ ) of the reaction of 1 with Ru<sup>II</sup> was determined. The activation parameters ( $E_a$ and logA) for this reaction were calculated from the temperature dependence of  $k_2$ . The excitation yield of Ru<sup>II\*</sup> ( $\eta^*_{Ru}$ ) was estimated. The quenching of Ru<sup>II\*</sup> by dioxirane was studied, and the bimolecular quenching constant and the coefficient of excitation regeneration were determined. It was suggested that the catalysis of the decomposition of 1 and the excitation of Ru<sup>II</sup> occur via a mechanism of chemically initiated electron exchange.

Key words: chemiluminescence,  $Ru^{11}$  tris-2,2'-bipyridyl complex, dimethyldioxirane; catalysis, electron transfer.

Dioxiranes are three-membered cyclic peroxides. Presently, they are widely used as strong oxidants in synthesis of oxygen-containing compounds.<sup>1,2</sup> In the absence of an oxidizable substrate, dioxiranes isomerize to esters. The spontaneous isomerization of, for example, dimethyldioxirane (1) to methyl acetate is a strongly exothermic process  $(-\Delta H_0 > 80 \text{ kcal mol}^{-1})$ , and the energy released in this process is assumed to be sufficient for the formation of the product in the electron-excited state.<sup>1</sup> We have shown<sup>3</sup> that the chemiluminescence (CL) that accompanies the isomerization of compound 1 in the presence of 9,10-dibromoanthracene is related to the radiative deactivation of an excited activator that appears due to the transfer of energy from the triplet methyl acetate.

At the same time, dioxiranes are likely capable of reacting with substrates not only by the transfer of O atoms but also via an electron transfer mechanism. These reactions should be expected for the transition metal complexes. In the case of luminescent metal complexes, the reactions can be accompanied by CL. In a previously published work,<sup>4</sup> we established that enhancement of CL of 1,2-dioxetanes in the presence of the Ru(bpy)<sub>3</sub>Cl<sub>2</sub> complex (bpy is 2,2'-bipyridyl) is related to the intermolecular chemically initiated electron exchange luminescence (CIEEL). The present work is devoted to studying the CL that we observed and the kinetics of the reaction of compound 1 with the Ru(bipy)<sub>3</sub>Cl<sub>2</sub> complex.

## Experimental

Dioxirane 1 was obtained as described in a previously published work.<sup>5</sup> Compound 1 was identified by  $^{13}$ C NMR on

a Bruker AM 300 spectrometer (a signal at 101.35 ppm from the C atom bound to the peroxide group is characteristic) and spectrophotometrically on a Specord M-40 spectrophotometer  $(\lambda = 335 \text{ nm}, \epsilon = 10 \text{ L mol}^{-1} \text{ cm}^{-1}$ , see Ref. 5). The kinetics of the decomposition of 1 in acetone in the absence of Ru(bpy)<sub>3</sub>Cl<sub>2</sub> was monitored spectrophotometrically by a decrease in its optical density at  $\lambda_{max} = 335$  nm. The products of the decomposition of 1 were analyzed by GLC. Ru(bpy)<sub>1</sub>Cl<sub>2</sub> was synthesized according to a procedure described previously.6 Chemiluminescence was recorded using a FEU-140 photomultiplier on a photometric setup calibrated in absolute units by a known method.<sup>7</sup> The spectral sensitivity of the photocathode was determined by an SIRSh-6-100 ribbon tungsten lamp and a series of interference light filters in the 365-670 nm region. The sensitivity of a photocathode in the region of luminescence of  $Ru^{II}$  ( $\lambda_{max} = 630$  nm) was 5% of the maximum value in the 410–420 nm region. Chemiluminescence spectra were recorded using boundary light filters, an MZD-2M aperture monochromator, and a FEU-119 photomultiplier. Photoluminescence spectra were obtained on a Hitachi MPF-4 spectrofluorimeter. The quantum yield of PL of RuII in saturated and degassed acetone was determined using the quinine standard.<sup>8</sup>

## **Results and Discussion**

Methyl acetate (2) is the main product of decomposition of dioxirane 1.

Under the conditions of our experiment, the kinetic curves of consumption of 1 are exponential irrespective of

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Fig. 1. Kinetics of consumption of 1 (1) and semilogarithmic anamorphosis (2) at 308 K.

**Table 1.** Values of the first-order rate constant  $(k_1)$  of thermolysis of dioxirane 1 in acetone

<i>T/</i> K	$k_1 \cdot 10^5$ /s <sup>-1</sup>	$[1]_0 \cdot 10^2$ /mol L <sup>-1</sup>	
300	1.0	6.5	
303	2.0	6.5	
308	5.1	6.6	
313	22.5	3.7	
323	28.5	3.5	

Note. The error of determination of  $k_1$  is not greater than 15%.

the temperature  $(27-50^{\circ} \text{ C})$  and initial concentration of 1 ([1]<sub>0</sub> = 0.035÷0.066 mol L<sup>-1</sup>) (Fig. 1). The first-order rate constants ( $k_1$ ) calculated from the semilogarithmic anamorphoses of the kinetics curves of consumption of 1 are presented in Table 1. The Arrhenius parameters of reaction (1) were determined from the temperature dependence of  $k_1$ :  $E_a = 28.0\pm4.5$  kcal mol<sup>-1</sup> and logA = 16.0±2.0.

However, we failed to detect CL during the thermolysis of dioxirane 1 in the presence of  $O_2$ . It is known that the isomerization of 1 results in the formation of triplet-excited methyl acetate  $(2_T^*)$ .<sup>3</sup> It is likely that the radiative deactivation of  $2_T^*$  is almost absent due to the efficient quenching processes (quenching by oxygen, nonradiative transfer of energy to the solvent molecules, and others). This agrees with the fact that for the thermolysis of 1,2-dioxetanes, although the yield of triplet-excited carbonyl products is 10-100-fold higher than that of singlet-excited products), the observed CL is caused by the fluorescence of carbonyl compounds.



Fig. 2. Dependence of the maximum intensity of CL on the concentration of  $Ru^{II}$  ([1] =  $6 \cdot 10^{-3} mol L^{-1}$ ) (1) and dioxirane 1 ([ $Ru^{II}$ ] =  $1.25 \cdot 10^{-4} mol L^{-1}$ ) (2) at 313 K. Insertion: kinetics of CL at [ $Ru^{II}$ ] =  $1.25 \cdot 10^{-4}$  and [1] =  $6 \cdot 10^{-3} mol L^{-1}$  (moment of mixing the reagents is indicated by arrow).

When  $Ru(bpy)_3Cl_2$  is present in a solution containing dioxirane 1, CL is observed. The maximum intensity of CL is proportional to the concentration of the complex when [1] = const and to the concentration of 1 when  $[Ru^{II}] = const$  (Fig. 2). The observed dependences of the intensity of CL on the concentrations of the reagents testify that CL appears when  $Ru^{II}$  reacts with compound 1. The kinetics of the CL decay is exponential, and its rate considerably exceeds the rate of consumption of 1 in the absence of the complex (see Fig. 2).

The spectral region of the luminescence with a maximum at 590-620 nm corresponds to the photoluminescence (PL) of  $\text{Ru(bpy)}_3^{2+}$  (Fig. 3). Therefore,  $\text{Ru}^{II}$  is an emitter of the luminescence. The analysis of the reaction mixture after the CL is completed shows that compound 2 is the main product of the conversion of dioxirane 1. Thus, it can be assumed that the CL is caused by the deactivation of the excited  $\text{Ru}^{II}$  complex, which is formed in the reaction with 1:

$$\frac{Me}{Me} \subset \binom{0}{1} + Ru(bpy)_{3}^{2+} \xrightarrow{k_{3}} 2 + Ru^{*}(bpy)_{3}^{2+} \xrightarrow{} 2 + Ru(bpy)_{3}^{2+} + hv \qquad (2)$$

The observed first-order rate constant  $(k_{obs})$  of the CL decay is proportional to the concentration of Ru<sup>II</sup> (Fig. 4) and can be determined from the equation:

$$k_{\rm obs} = k_1 + k_2 [{\rm Ru}^{\rm II}]_0, \tag{3}$$

where  $k_2$  is the bimolecular rate constant.



Fig. 3. Absorption (1) and photoluminescence spectra (1') of Ru(bpy)<sub>3</sub><sup>2+</sup> at [Ru<sup>II</sup>] =  $5 \cdot 10^{-6}$  mol L<sup>-1</sup> and after the reaction with 1 ( $2 \cdot 10^{-2}$  mol L<sup>-1</sup>) (2, 2') in MeCN. Chemiluminescence spectrum in the reaction of Ru<sup>II</sup> ( $1.25 \cdot 10^{-4}$  mol L<sup>-1</sup>) with 1 ( $6 \cdot 10^{-3}$  mol L<sup>-1</sup>) (3).

The dependences of  $k_{obs}$  on the concentration of Ru<sup>II</sup> at different temperatures are presented in Fig. 4. The rate constants of the reaction of dioxirane 1 with Ru<sup>II</sup> ( $k_2/L$  mol<sup>-1</sup> s<sup>-1</sup>) were determined from the slope of these straight lines:

$$T/K$$
 298 303 313 318 323  
 $k_2 \cdot 10^{-2}$  1.39 $\pm 0.16$  1.69 $\pm 0.24$  3.10 $\pm 0.20$  3.54 $\pm 0.21$  4.46 $\pm 0.66$ 

The activation parameters were calculated from the temperature dependence of  $k_2$ :  $E_a = 9.6 \pm 1.6$  kcal mol<sup>-1</sup> and log  $A = 9.1 \pm 1.0$ . As can be seen from the data obtained, the reaction of Ru<sup>II</sup> with 1 occurs with an activation energy that is approximately 10 kcal mol<sup>-1</sup> lower, and the isomerization rate of dioxirane in the presence of Ru<sup>II</sup> ([Ru<sup>II</sup>] =  $1.25 \cdot 10^{-4}$  mol L<sup>-1</sup>) is  $k_2$ [Ru<sup>II</sup>]/ $k_1 = 170$  times (313 K) higher.

The comparison of the PL and absorption spectra of  $Ru(bpy)_3^{2+}$  before and after the cessation of the reaction with 1 (see Fig. 3) indicates that the starting complex undergoes some changes. The character of the change in the spectra corresponds to the formation of hydroxy derivatives of the Ru<sup>II</sup> trisbipyridyl complex in the reaction (see Ref. 9):



The intensity of the PL also decreases insignificantly due to reaction (4). However, reaction (4) has no effect on the kinetics of CL and does not affect the results, because noticeable changes in the absorption and PL spectra of  $Ru^{II}$  are observed only when 1 is present in a  $4 \cdot 10^3$ -fold excess (see Fig. 3).

It is known that  $Ru(bpy)_3^{2+}$  is efficiently excited in the reaction of one-electron reduction of  $Ru(bipy)_3^{3+}$ :10

$$Ru(bpy)_{3}^{3+} + e^{-} \rightarrow Ru^{*}(bpy)_{3}^{2+}.$$
 (5)

The excitation of  $Ru^{II}$  by the transfer of an energy from triplet-excited ketones is not efficient.<sup>11</sup> Therefore, when the formation of a radical anion of the product and  $Ru^{III}$  is initiated in the chemical reaction, the formation of  $Ru^{II*}$  can be expected.

The data obtained, as well as the results on the CL of  $Ru^{II}$  in the reaction with 1,2-dioxetanes,<sup>4</sup> suggest that  $Ru^{II}$  is excited in the reaction with 1 via the CIEEL mechanism:

$$Ru^{II} + 1 \implies [Ru^{II...} 1] \implies [Ru^{III...} 1^{--}] \rightarrow$$

$$\rightarrow [Ru^{II...} 2^{--}] \stackrel{\frown}{\longrightarrow} [Ru^{II*...} 2] \stackrel{\rightarrow}{\longrightarrow} Ru^{II} + 2 + hv$$

$$(6)$$

After an electron was transferred from  $Ru^{II}$  to dioxirane 1,  $Ru^{III}$  and the dioxirane radical anion are formed. As the theoretical calculations<sup>12</sup> showed, the transfer of an electron to the antibonding orbital of the peroxide bond leads to a sharp decrease in energy, and the irreversible cleavage of this bond occurs almost immediately after the insertion of an electron. Then radical anion 1<sup>--</sup> isomerizes to a radical anion of methyl acetate. The reverse electron transfer is a strongly exothermic process, which results in the excitation of  $Ru^{II*}$ .

Since CL reflects the catalyzed route of isomerization of dioxirane 1, we can write:

 $I_{\rm CL} = \eta^*_{\rm Ru} \eta_{\rm PL} k_2 [{\rm Ru}]_0 [1]_0 \exp[-(k_1 + k_2 [{\rm Ru}^{\rm H}]_0)t], \quad (7)$ 

where  $I_{CL}$  is the intensity of CL,  $\eta^*_{Ru}$  is the excitation yield of Ru<sup>II\*</sup> in the reaction, and  $\eta_{PL}$  is the luminescence yield of the complex.



Fig. 4. Dependence of the observed rate constant of the CL decay  $(k_{obs})$  on the concentration of  $Ru^{II}$  ([1] =  $6 \cdot 10^{-3}$  mol  $L^{-1}$ ) at 298 (1), 303 (2), 313 (3), 318 (4), and 323 K (5).



Fig. 5. Dependences of the luminous sum of CL in the reaction on the concentrations of  $\operatorname{Ru}^{II}([1] = 6 \cdot 10^{-3} \text{ mol } L^{-1})$  (1) and 1 ([ $\operatorname{Ru}^{II}$ ] = 1.25  $\cdot 10^{-4}$  mol  $L^{-1}$ ) (2) at 313 K.

The maximum intensity of CL in the reaction of  $Ru^{II}$  with I can be calculated by the equation:

$$I_{\rm CL}^{\rm max} = \eta^*{}_{\rm Ru}\eta_{\rm PL}k_2[{\rm Ru}]_0[1]_0.$$
(8)

The luminous sum of the reaction is independent of  $[Ru^{II}]$  but increases linearly as the concentration of 1 increases (Fig. 5). Using Eq. (7) for the luminous sum of the reaction (S), we can write the following equation:

$$S = \int_{0}^{\infty} f dt = \eta^*_{Ru} \eta_{PL} k_2 [Ru]_0 [1]_0 \int_{0}^{\infty} \exp[-(k_1 + k_2 [Ru^{II}]_0)t] dt =$$
  
=  $\eta^*_{Ru} \eta_{PL} k_2 [Ru]_0 [1]_0 (k_1 + k_2 [Ru^{II}]_0)^{-1}.$  (9)

The yield of CL was estimated from Eq. (9) using the data in Fig. 5:  $\eta^*_{Ru}\eta_{PL} = (9.5\pm3) \cdot 10^{-5}$  Einstein mol<sup>-1</sup>. Taking into account the luminescence yield  $\eta_{PL} = 0.0095$ , the chemiexcitation yield of Ru<sup>II</sup> in the reaction with dioxirane was estimated as  $\eta^*_{Ru} = 0.01\pm0.005$ . This value is lower than the excitation yield of Ru<sup>II\*</sup> in the reaction with 1,2-dioxetane, which is equal to 0.2.<sup>4</sup> The difference in  $\eta^*_{Ru}$  is probably caused by the Ru<sup>II</sup>-induced isomerization of dioxirane 1 without formation of Ru<sup>II\*</sup>.

According to the statements of the electron transfer theory, the value of the bimolecular rate constant  $k_2$ depends on the oxidation potential of an activator ( $E_{\rm ox}$ ) and on the reduction potential of peroxide ( $E_{\rm red}$ ) according to the equation:<sup>12</sup>

$$k_2 = A \exp[-(E_{\rm ox} - E_{\rm red} - e^2/\varepsilon R_0)/(RT)], \qquad (10)$$

where e is the charge of an electron,  $\varepsilon$  is the dielectric permeability of a solvent, and  $R_0$  is the distance between ions in the transition state.



Fig. 6. Kinetics of quenching of photoiuminescence of  $Ru^{II}$  by dioxirane 1 in coordinates of the Stern-Volmer equation. ( $\lambda_{exc} = 450$  nm, [ $Ru^{II}$ ] = 2.5 · 10<sup>-5</sup> mol L<sup>-1</sup>, 289 K).

As known,<sup>13</sup> the oxidation potential of  $\text{Ru}^*(\text{bpy})_3^{2+}$ decreases by the value of the excitation energy, *i.e.*,  $E^*_{\text{Ru}II^*/\text{Ru}III} = E_{\text{Ru}II/\text{Ru}III} - E^* = 1.29 - 2.12 = -0.83 \text{ V}.$ 

According to Eq. (10), one can expect an increase in  $k_2$  by several orders of magnitude. These facts can serve as an additional argument in favor of the electronexchange mechanism of the interaction between Ru<sup>II</sup> and dioxirane. In fact, it is found from the measurements of the intensity of photoluminescence of Ru<sup>II</sup> in the presence of 1 that dioxirane quenches the excited complex (Fig. 6), and the negative deviation from the linear Stern-Volmer dependence is observed at the concentration  $[1] > 2.5 \cdot 10^{-4}$  mol L<sup>-1</sup>. The value  $K_{\rm S} = 1800\pm200$  L mol<sup>-1</sup> was obtained from the linear region of the quenching kinetics from the Stern-Volmer equation:

$$I_0/I = 1 + K_{\rm S}[1], \tag{11}$$

 $(I_0 \text{ and } I \text{ are the intensities of luminescence in the}$ absence and presence of a quencher,  $K_{\rm S} = k_{\rm b} \tau_0$  is the Stern-Volmer constant,  $k_b$  is the bimolecular rate constant of quenching, and  $\tau_0$  is the lifetime of Ru\*(bpy)<sub>3</sub><sup>2+</sup> 2+ in the absence of dioxirane). The lifetime of Ru\*(bpy)3 depends on the concentration of  $O_2$  in acetone at T =298 K: in the absence of oxygen, 600±30 ns; in the presence of dissolved air, 150±10 ns. Since the quenching of Ru<sup>II\*</sup> was studied in an atmosphere of air,  $k_{\rm b} =$  $(1.2\pm0.3)\cdot10^{10}$  L mol<sup>-1</sup> s<sup>-1</sup> was determined from Eq. (11). At the same time, the estimation of the rate constant of the reaction of Ru<sup>II\*</sup> with 1 by Eq. (10) gives a considerably greater value:  $k_b \approx 5 \cdot 10^{22} \text{ L} \text{ mol}^{-1} \text{ s}^{-1}$ . The seeming disagreement can be likely explained by the fact that  $k_{\rm b}$  is really limited by diffusion of reagents in acetone. In fact, the bimolecular quenching rate constant is close to the diffusion constant.

The bimolecular quenching rate constant is the sum of the rate constants of the "physical"  $k_{ph}$  and "chemi-

cal"  $k_{ch}$  routes of quenching of Ru<sup>II\*</sup>. Previously, for quenching of the excited ruthenium complex by dioxetane,<sup>14</sup> we showed that  $k_{ph} \approx 0$ . The quenching of Ru<sup>II\*</sup> by dioxirane probably also has a chemical nature.

The catalytic activity of  $Ru^{II} (k_b/k_2)$  increases more than  $8 \cdot 10^7$  times upon excitation. The mechanism of quenching of  $Ru^{II*}$  by dioxirane can be described by a series of reactions:

$$\operatorname{Ru}^{\parallel \ast} + 1 \longrightarrow [\operatorname{Ru}^{\parallel \ast} \cdots 1] \longrightarrow [\operatorname{Ru}^{\parallel \parallel} \cdots 1^{-1}] \longrightarrow$$

$$\longrightarrow [\operatorname{Ru}^{\parallel \parallel} \cdots 2^{-1}] \longrightarrow \operatorname{Ru}^{\parallel \ast} \cdots 2^{-1} \longrightarrow \operatorname{Ru}^{\parallel \ast} + 2 + hv$$

$$[\operatorname{Ru}^{\parallel \parallel} \cdots 2^{-1}] \longrightarrow \operatorname{Ru}^{\parallel \ast} + 2 . \qquad (12)$$

The negative deviation of the quenching kinetics from the linear dependence, which is determined by the Stern-Volmer equation (see Fig. 6), is likely related to an apparent decrease in efficiency of quenching due to the regeneration of the excited Ru<sup>II</sup> complex. The following equation has been previously obtained<sup>15</sup> for similar systems with regeneration of excitation:

$$I_0/I - 1 = \left(\frac{1 + k_b(1 - \alpha)\tau_0[\mathbf{1}]}{1 + k_b\tau_0[\mathbf{1}]}\right) k_b\tau_0[\mathbf{1}],$$
 (13)

where  $\alpha$  is the coefficient of excitation regeneration.

It follows from Eq. (13) that when the concentration of a quencher is small ([1]  $\rightarrow$  0), the effect of regeneration of Ru<sup>11\*</sup> does not manifest itself, and for the great concentration ([1]  $\rightarrow \infty$ ),  $k_{\rm b}' = k_{\rm b}(1 - \alpha)$ . The estimation based on the quenching kinetics gives the value  $\alpha = 0.3 \pm 0.1$ , which is much higher than  $\eta^*_{\rm Ru}$ . It is likely that the excitation of Ru<sup>11</sup> increases the channel of the catalyzed isomerization of dioxirane 1, occurring via a mechanism of reversible electron transfer.

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