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Photochemical elimination of singlet oxygen from oxodiperoxo-molybdenum(VI) complexes with aromatic N,N′-chelate ligands

I. Hatzopoulos [a], W.R. Thiel [b], H.-D. Brauer [a,*]

[a] *Institut für Physikalische und Theoretische Chemie, Universität Frankfurt, Marie-Curie-Str. 11, 60439 Frankfurt/Main, Germany*
[b] *Anorganisch–Chemisches Institut der Technischen Universität München, Lichtenbergstraße 4, 85747 Garching, Germany*

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Abstract

The photolysis of the oxodiperoxo-molybdenum(VI) complexes of the type (L-L)$MoO(O_2)_2$ (L-L = 2-(1-alkyl-3-pyrazolyl)pyridine, alkyl = n-butyl **1a**, n-octyl **1b**, n-octadecyl **1c**; L-L = 2-(3-n-octyl-2-imidazolyl)pyridine **2**) leads to the elimination of molecular oxygen both in its singlet and in its triplet state. Solutions of the diperoxo complexes contain two isomers A and B. The overall quantum yields of the photolysis, $\bar{Q}_{Ph}$, and of the generation of singlet oxygen ($^1O_2$), $\bar{Q}_\Delta$, were measured in methylene chloride as a function of the irradiation wavelength. From the wavelength dependence of $\bar{Q}_{Ph}$ it can be deduced that the quantum yields of the photolysis of the two isomers are not equal. Furthermore, for all diperoxo complexes fluorescence is observed, and for the complexes **1a**, **1b**, and **1c**, additionally, phosphorescence is observed. The overall quantum yields of the fluorescence, $\bar{Q}_F$, are in the range $(1.5–8.0) \times 10^{-3}$.

Keywords: Mo(VI)-peroxocomplexes; Phosphorescence; Photolysis; Singlet oxygen

1. Introduction

Photochemical reactions of diperoxo-molybdenum complexes, such as $MoO(O_2)_2[HMPA]\cdot H_2O$ (oxodiperoxo-molybdenum(VI)–(hexamethyl phosphoric triamide, water)), were reported to eliminate singlet oxygen by irradiation exclusively from one upper excited singlet state [1]. This rare type of adiabatic photoreaction has been observed for the first time for the photocycloreversion of endoperoxide of aromatic compounds. It was found that the reaction occurs in some cases from more than one upper excited singlet state [2]. Furthermore, it was observed that the elimination of molecular oxygen also occurs from upper excited triplet states [3]. In addition to that, investigations on Vaska's complex $Ir(CO)Cl(PPh_3)_2(O_2)$ have shown generation of $^1O_2$ by irradiation with an efficiency of 0.03 parallel to the generation of $^3O_2$ with an efficiency of 0.41 [4].

Increasing solubility in non-polar solvents is an important feature for the efficiency of an epoxidation catalyst. Recently it was shown that diperoxo complexes of the types 1 and 2 (Fig. 1) are highly efficient catalysts for the synthesis of sensitive epoxides in non-polar solvents [5]. Furthermore, this increased solubility allows investigations of the luminescence properties in degassed solvent matrices at 77 K, which consist of non-polar solvents. In this paper we report on investigations of the photochemical and photophysical properties of the diperoxo complexes shown in Fig. 1.

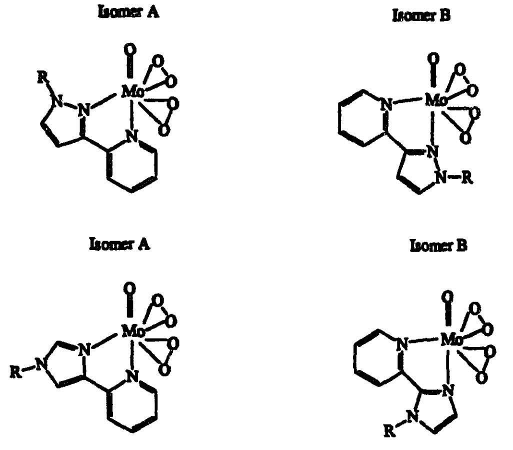

Fig. 1. Diperoxo complexes **1a**, **1b**, **1c** and **2**. R = n-butyl (**1a**); R = n-octyl (**1b**); R = n-octadecyl (**1c**); R = n-octyl (**2**).

Due to the symmetry of the ligands the peroxo complexes **1a**, **1b**, **1c** and **2** appear in two isomeric forms, with the terminal oxoligand in the trans position either to pyridine (isomer A) or to pyrazole (isomer **1B**) and imidazole (isomer **2B**), respectively. The isomers are distinguishable by NMR and IR spectroscopy. They cannot be isolated in pure

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* Corresponding author.

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forms as they are in equilibrium in solution, which could be shown by means of EXCY NMR experiments [5]c. For all peroxo complexes studied here the ratio of the isomers A:B was found to be about 2:1.

2. Materials and methods

2.1. Materials

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The complexes **1a**, **1b**, **1c** and **2** were prepared according to published procedures [5a]. Methylene chloride was of spectroscopic grade (Aldrich) and purified by column chromatography with basic Al_2O_3 (Woelm). Toluene and ethanol were both of spectroscopic grade and used as received.

2.2. Methods

Irradiations were performed in 1 cm cuvettes at room temperature using an Osram HBO 100/2 high pressure mercury lamp. Variation of the wavelength was achieved using a Schoeffel GM 252 monochromator in the range 248 nm–366 nm. The light intensities were measured by a SDC SD 444 silicon photodiode. Photon flux measurements were performed by chemical actinometry [6]. The progress of the photolysis was monitored photometrically with a Lambda 5 spectrophotometer (Perkin-Elmer). Complex concentrations were in the range $(0.8–1.7) \times 10^{-4}$ M.

For laser flash photolysis experiments, the following lasers were used: Lambda Physics EMG 100 and 200 E excimer lasers with emission wavelengths at $\lambda = 248$ nm (KrF), $\lambda = 308$ nm (XeCl) and $\lambda = 337$ nm (nitrogen). The pulse energy was controlled by a monitor photodiode. The exciting light was focused into the fluorescence cell containing the sample solution. The $^1O_2$ phosphorescence was detected after passing a 1274 nm interference filter (Schott) by a North Coast EO 817 P germanium diode positioned perpendicular to the laser beam. The data were recorded with a Gould 4072 transient recorder. $\bar{Q}_\Delta$ was determined using a method described in Ref. [7] by comparison of the initial intensity of $^1O_2$ phosphorescence after exposure of the complexes to a laser pulse with that from an optically matched standard (perinaphthenone in air-saturated CH_2Cl_2 with $Q^{st}_\Delta = 0.95 \pm 0.05$ [8]). The optical density of the solutions was chosen to be approximately 1 at the wavelengths of excitation.

All fluorescence spectra were recorded with a Perkin–Elmer fluorescence spectrometer 650-40. Quinine bisulfate (Aldrich p.a.) in 1 N H_2SO_4 was used as standard for measurements of the fluorescence quantum yields Q_F [9a]. Phosphorescence spectra were taken at 77 K in degassed glassy toluene/ethanol (2:1) solutions with a luminescence apparatus described in Ref. [9b]. Excitation of the samples was performed at $\lambda = 366$ nm, the half band of emission monochromator was $\lambda_{1/2} = 40$ nm. All spectra were corrected using the spectral sensitivity curves of the instruments [10].

3. Results and discussion

3.1. Photolysis of the diperoxo complexes

Irradiation of the diperoxo complexes at 313 nm in methylene chloride resulted in spectral changes such as those shown in Fig. 2 for the complex **2**.

The absorption spectra of **1a**, **1b**, **1c** and **2** exhibit a shoulder at about 360 nm (see Figs. 2 and 5), an intensive absorption band at about 300 nm and a second absorption band at $\lambda < 270$ nm (see Fig. 2). The wavelengths and the absorption coefficients of the shoulders and the first absorption bands are summarized in Table 1. As discussed for $MoO(O_2)_2[HMPA] \cdot H_2O$ in detail [1], the absorption spectra of diperoxo complexes must originate from ligand-to-metal charge transfer (LMCT) transitions. The absorption band at about 300 nm is characteristic for η^2-coordinated diperoxo-Mo-complexes in less polar solvents like methylene chloride [11]. The reaction spectrum in Fig. 2 demonstrates a decrease of this absorption band which is interpreted to correspond with the photochemical elimination of molecular oxygen. During the first 7 min of irradiation two isobestic points are observed (see inset of Fig. 2) at 293 nm and 325 nm, respectively, indicating that during this time the reaction is uniform. This is confirmed by the absorbance difference (AD) diagram [12] depicted in Fig. 3 which is obtained from the spectral changes given in the inset of Fig. 2.

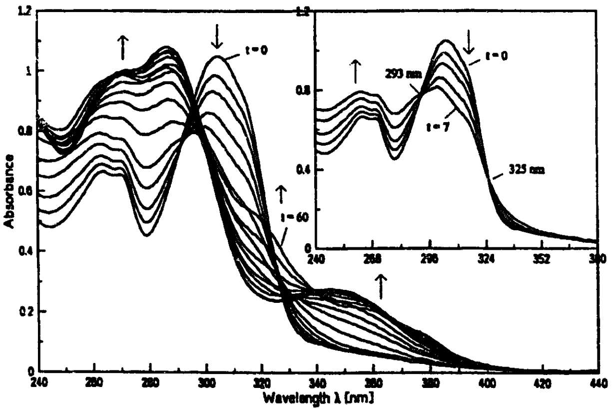

Fig. 2. Spectral changes during irradiation of 2 in methylenechloride; $\lambda_{exc} = 313$ nm; $[2] = 8.7 \times 10^{-5}$ M. Times, *t*, in min.

Table 1

Wavelengths λ_{max} and absorption coefficients ϵ_{max} of the shoulders (S) and the first absorption bands (AB) of the absorption spectra of the complexes **1a**, **1b**, **1c** and **2**

Complex	λ_{max} (S) (nm)	ϵ_{max} (S) [a] ($M^{-1} cm^{-1}$)	λ_{max} (AB) (nm)	ϵ_{max} (AB) [a] ($M^{-1} cm^{-1}$)
1a	363	940	300	9700
1b	363	1070	300	10400
1c	370	980	300	9100
2	350	1500	303	12000

[a] The error amounts to about ±7%.

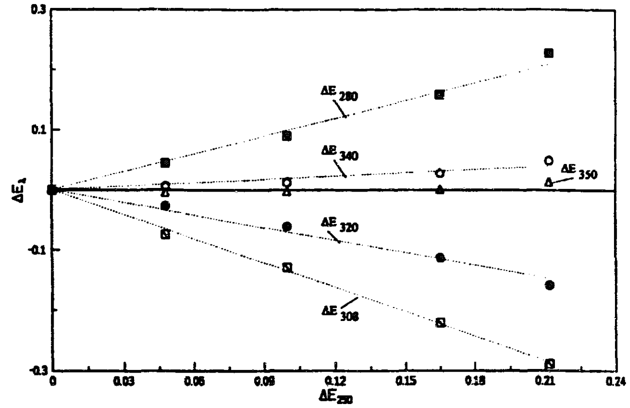

Fig. 3. Absorbance difference (AD) diagram for the spectral changes shown in the inset of Fig. 2.

In addition the reaction spectrum of **2** shows that further irradiation leads to formation of two new isobestic points at 285 nm and 304 nm, respectively, indicating that a consecutive photoreaction takes place. Irradiation of the complexes **1a**, **1b**, and **1c** shows similar results. Here we discuss only the first photochemical step.

3.2. Wavelength dependence of $\bar{Q}_{Ph}$

Generally, measurements of the irradiation wavelength dependence of the quantum yield of a photoreaction allow one to decide from which electronically excited state of the species the photoreaction originates [1–4]. We have performed such measurements for the photolysis of the diperoxo complexes in methylene chloride in the wavelength region 248 nm–366 nm at six different wavelengths hoping: (i) that from both electronically excited isomers molecular oxygen is eliminated, and (ii) that the quantum yields of both isomers are equal. As Eq. (1) shows in this case the overall quantum yield of the photolysis at the excitation wavelength λ_{exc}, $\bar{Q}_{Ph}^{\lambda_{exc}}$, is identical with the quantum yields of the isomer A, $\bar{Q}_{Ph}^{\lambda_{exc}}(A)$, and of the isomer B, $\bar{Q}_{Ph}^{\lambda_{exc}}(B)$, respectively. $\bar{Q}_{Ph}^{\lambda_{exc}}$ is defined as:

$$\bar{Q}_{Ph}^{\lambda_{exc}}=\frac{A_A^{\lambda_{exc}}\cdot\bar{Q}_{Ph}^{\lambda_{exc}}(A)+A_B^{\lambda_{exc}}\cdot\bar{Q}_{Ph}^{\lambda_{exc}}(B)}{(A_A^{\lambda_{exc}}+A_B^{\lambda_{exc}})} \qquad (1)$$

where $A_A^{\lambda_{exc}}$ and $A_B^{\lambda_{exc}}$ denote the absorbance of the two isomers at λ_{exc}.

For the determination of $\bar{Q}_{Ph}^{\lambda_{exc}}$ the absorbance changes were monitored at $\lambda = 280$ nm. The total amount of the photolysis was limited in all cases to 10%. Under this condition $\bar{Q}_{Ph}^{\lambda_{exc}}$ was found to be independent of the initial concentration of the different diperoxo complexes.

The wavelength dependence of $\bar{Q}_{Ph}^{\lambda_{exc}}$ for all investigated diperoxo complexes is illustrated in Fig. 4. The values of $\bar{Q}_{Ph}^{\lambda_{exc}}$ of the complexes **1a**, **1b**, and **1c** show a similar course with two maxima at 334 nm and 302 nm. The values of $\bar{Q}_{Ph}^{\lambda_{exc}}$ of **2** vary from 0.05 at 336 nm to 0.65 at 284 nm passing through a maximum of 0.27 at 334 nm and a minimum of 0.15 at about 302 nm. The course of the wavelength dependence of $\bar{Q}_{Ph}^{\lambda_{exc}}$ observed for the diperoxo complexes is complex compared to that found for $MoO(O_2)_2[HMPA]\cdot H_2O$ [1], indicating that the assumption $\bar{Q}_{Ph}^{\lambda_{exc}}(A)=\bar{Q}_{Ph}^{\lambda_{exc}}(B)$ is not fulfilled.

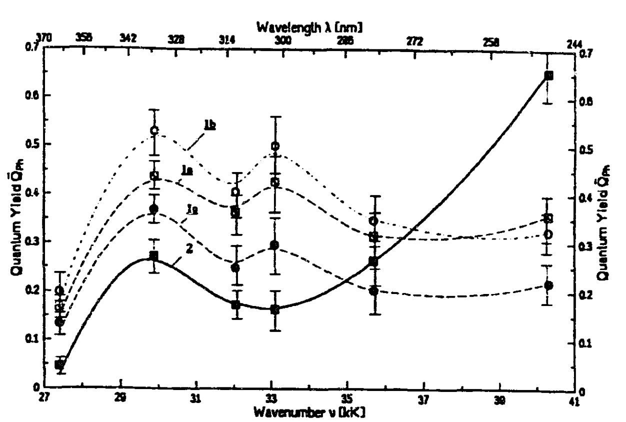

Fig. 4. Plots of $\bar{Q}_{Ph}$ of the diperoxo complexes versus the wavenumber $\tilde{\nu}$ ($kK = 1000\ cm^{-1}$) of excitation.

Since the absorption coefficients of the different isomers, ϵ, are unknown, quantum yields $\bar{Q}_{Ph}^{\lambda_{exc}}(A)$ and $\bar{Q}_{Ph}^{\lambda_{exc}}(B)$, respectively, cannot be calculated and consequently statements about the contributions of the isomers to $\bar{Q}_{Ph}^{\lambda_{exc}}$ cannot be made.

However, from the sharp decrease of $\bar{Q}_{Ph}^{\lambda_{exc}}$ at $\lambda < 334$ nm, which is observed for all diperoxo complexes, it can be suggested that the lowest excited state cannot be the initial state of the photolysis. The wavelength dependence of $\bar{Q}_{Ph}^{\lambda_{exc}}$ of the diperoxo complexes is consistent with the assumption that the photochemical elimination of molecular oxygen originates in all cases from upper excited electronic states. Because of the possibility that $\bar{Q}_{Ph}^{\lambda_{exc}}(A)\neq\bar{Q}_{Ph}^{\lambda_{exc}}(B)$, interpretation in more detail with respect to the upper excited electronic states is not possible.

For the values of $\bar{Q}_{Ph}^{\lambda_{exc}}$ of the complexes **1a**, **1b**, and **1c**, the graduation $\bar{Q}_{Ph}^{\lambda_{exc}}(\mathbf{1b})>\bar{Q}_{Ph}^{\lambda_{exc}}(\mathbf{1a})>\bar{Q}_{Ph}^{\lambda_{exc}}(\mathbf{1c})$ is found in the wavelength region from 366 nm to 280 nm. This graduation cannot be explained by steric effects of the different alkyl groups. Since it can be assumed that $\bar{Q}_{Ph}^{\lambda_{exc}}(A)$ and $\bar{Q}_{Ph}^{\lambda_{exc}}(B)$ are not equal it seems likely that the observed sequence in $\bar{Q}_{Ph}^{\lambda_{exc}}$ results from small differences in the ratios of the isomers A and B of **1a**, **1b**, and **1c**.

3.3. Determination of $\bar{Q}\Delta$

To prove whether the photolysis of the diperoxo complexes generates $^1O_2$ we have performed time-resolved measurements of the phosphorescence of $^1O_2$ at 1276 nm in degassed methylene chloride. For the determination of the overall quantum yield of $^1O_2$ formation, $\bar{Q}_\Delta$, the method developed by Gorman et al. [7] was used. The intensity of the emission of $^1O_2$ at zero time, I_{po}, produced by the photolysis of the diperoxo complexes versus the energy of the exciting laser pulse at 248 nm, 308 nm and 337 nm was compared with the intensity of emission of $^1O_2$ obtained by excitation of perinaphthenone in air-saturated methylene chloride solution.

Table 2
Comparison of the values of $\bar{Q}_{\Delta}$ with the corresponding values of $\bar{Q}_{Ph}$ (shown in Fig. 4) for the complexes **1a**, **1b**, **1c** and **2**

Complex	$\bar{Q}_{\Delta}^{337}$	$\bar{Q}_{Ph}^{334}$	$\bar{Q}_{\Delta}^{308}$	$\bar{Q}_{Ph}^{308}$ [a]	$\bar{Q}_{\Delta}^{248253}$	$\bar{Q}_{Ph}^{248}$
1a	0.11 ± 0.02	0.44 ± 0.04	0.15 ± 0.02	0.40 ± 0.04	0.25 ± 0.03	0.36 ± 0.03
1b	0.11 ± 0.02	0.53 ± 0.05	0.13 ± 0.02	0.46 ± 0.04	0.25 ± 0.03	0.33 ± 0.02
1c	0.08 ± 0.01	0.37 ± 0.03	0.15 ± 0.02	0.28 ± 0.03	0.24 ± 0.03	0.22 ± 0.03
2	0.09 ± 0.01	0.27 ± 0.03	0.19 ± 0.02	0.17 ± 0.02	0.26 ± 0.03	0.65 ± 0.07

[253a] Mean values of the $\bar{Q}_{Ph}$ values at 313 nm and 302 nm.

Comparison of the values of $\bar{Q}_{\Delta}$, given in Table 2, with the values of $\bar{Q}_{Ph}$ at the corresponding wavelengths indicates that the released molecular oxygen is generated only partly as $^1O_2$. Only for **1c** at 248 nm and for **2** at 308 nm did we observe that $\bar{Q}_{\Delta} = \bar{Q}_{Ph}$ within the limits of error.

The fact that $^1O_2$ as well as $^3O_2$ is eliminated accounts for an intersystem crossing process which competes with the elimination of $^1O_2$ from the photochemically reactive singlet states of the isomers, since an increased intersystem crossing rate is expected due to strong spin–orbit coupling enhanced by Mo (internal heavy atom effect). However, this in contrast to the results obtained for $MoO(O_2)_2[HMPA] \cdot H_2O$ where the eliminated oxygen is generated completely as $^1O_2$. This discrepancy can be explained by the assumption that for the isomers A and B of the diperoxo complexes studied the energy difference between the reactive excited singlet state and the energetically next triplet state is smaller than in the case of $MoO(O_2)_2[HMPA] \cdot H_2O$ leading to a more effective intersystem crossing.

With respect to the wavelength dependence of $\bar{Q}_{\Delta}$ and the efficiencies of the formation of both $^1O_2$ and $^3O_2$ further ingenuous statements cannot be made, since two isomers are involved in the photochemical elimination of $^1O_2$ and $^3O_2$.

3.4. Fluorescence and phosphorescence spectra

Most organometallic complexes, which are luminescent, are diamagnetic in their ground state [13]. Molecular d^0 oxo complexes which have low energy LMCT states have not yet been observed to emit [14]. Some d^0-oxo-metallates are known to luminescence in the solid state [15]. The organometallic d^0-oxide CH_3ReO_3 emits from the lowest LMCT state in solvent matrices at 77 K ($\lambda_{max} = 640$ nm) [16].

For the diperoxo complexes studied both fluorescence and phosphorescence emission was observed, except for complex **2**, where no phosphorescence could be detected. To the best of our knowledge these are the first examples of luminescent diperoxo-Mo-complexes. As an example the emission spectra of complex **1a** are given in Fig. 5.

Generally only the phosphorescence emission is observed and only in rare cases can fluorescence emission be detected. This results from an enhanced intersystem crossing after excitation on account of the heavy atom effect which is particularly effective of metals of the fifth and sixth period. It is a little bit surprising that for complex **2** only the fluorescence emission is observed.

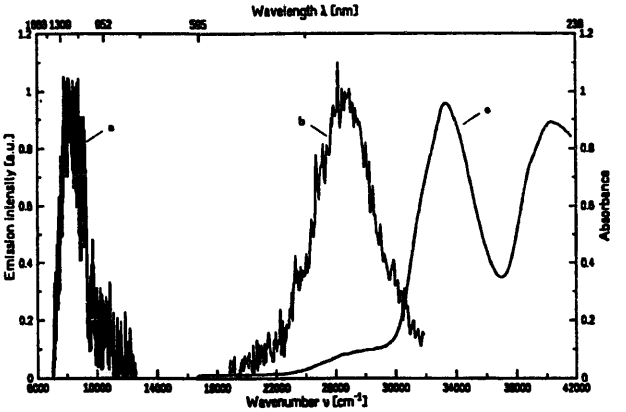

Fig. 5. (a) Corrected phosphorescence spectra of **1a** in toluene/ethanol (2:1) at 77 K, $\lambda_{1/2} = 40$ nm, $\lambda_{exc} = 366$ nm; (b) corrected fluorescence spectrum at room temperature in methylene chloride; (c) absorption spectrum in methylenechloride. Spectra (a) and (b) were recorded by different instruments (see Section 2.2 and Section 3.4).

The fluorescence spectrum of the different diperoxo complexes consists of a single band with a maximum which was independent of the excitation wavelength between 302 nm and 366 nm. Since this band overlaps with the lowest energy absorption (Fig. 5) of the single diperoxo complex we conclude that the fluorescence emission comes from the lowest LMCT excited states of the corresponding isomers. For all complexes a very small Stokes shift of at the most about 8 nm is observed. The overall quantum yields of the fluorescence, $\bar{Q}_F$, determined for the isomer mixtures are: **1a**, $\bar{Q}_F = 2 \times 10^{-3}$; **1b**, $\bar{Q}_F = 1.5 \times 10^{-3}$; **1c**, $\bar{Q}_F = 8.0 \times 10^{-3}$; and **2**, $\bar{Q}_F = 2.5 \times 10^{-3}$. The error limits amount to about 20%.

The fact that the diperoxo complexes are fluorescent supports the conclusion that the observed photoelimination of molecular oxygen does not originate from the lowest excited LMCT states of the isomers (vide supra). As has been found for endoperoxides of aromatic compounds the photoelimination of molecular oxygen reduces the lifetime of the reactive excited state to less than 3 ps [17]. Using the approximation (2) for the radiative lifetime τ^0_F of the lowest excited LMCT state [18]

$$\tau^0_F \sim \frac{1}{10^4 \epsilon_{max}} \qquad (2)$$

where ϵ_{max} denotes the absorption coefficient of the $S_0 \rightarrow S_1$ absorption (see Table 1) the fluorescence lifetime τ_F can be roughly estimated. According to Eq. (3)

$$\tau_F \sim \frac{\bar{Q}_F}{10^4\,\epsilon_{max}} \qquad (3)$$

For the diperoxo complexes values of τ_F are obtained in the range of 150–800 ps.

The phosphorescence spectrum of the single complexes **1a**, **1b**, and **1c** likewise consists of a single band with a maximum at 8000 cm^{-1}, i.e. the energy of lowest triplet states of the isomers of **1a**, **1b**, and **1c** should amount to 96 kJ mol^{-1}. Comparison with the energy of the lowest excited singlet states of the isomers of about 325 kJ mol^{-1} a high energy splitting $\Delta E(S_1 - T_1)$ is obtained. On the basis of this high value it is understandable that the fluorescence emission is observed, since the $S_1 \rightarrow T_1$ intersystem crossing should be rendered by the Franck–Condon prohibition. The reason for the fact that no phosphorescence emission of **2** is observed is not clear.

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