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Photoredox Reactions Occurring in Methanolic Solutions of Iron(III) Azido Complexes with *N,N′*-Ethylenebis (salicylaldiminato) *Schiff* Base Derivatives

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Summary. Photoredox reactions occurring in irradiated methanolic solutions of *trans*-[Fe(N_2O_2)(CH$_3$OH)N$_3$], where $N_2O_2{}^{2-}$ are tetradentate open-chain N_2O_2-*Schiff* base *N,N′*-ethylenebis(*R*-salicylaldiminato) or *N,N′*-1-methylethylenebis(*R*-salicylaldiminato) ligands denoted as *R*-salen and *R*-sal(*Me*)en, respectively ($R = $ H, 5-Cl, 5-Br, 4-OCH$_3$), have been investigated and their mechanism has been proposed. The complexes are redox stable in the dark. Ultraviolet and/or visible irradiation of methanolic solutions of the complexes induces photoreduction of Fe(III) to Fe(II). As an intermediate, $^{\bullet}$CH$_2$OH radicals were identified by EPR spin trapping technique. The final product of the photooxidation coupled with the photoreduction of Fe(III) is formaldehyde. The efficiency of the photoredox processes is strongly wavelength dependent and influenced by the peripheral groups *R* of the tetradentate ligands. Differences between the course of photochemical changes induced by 254 nm radiation and the other wavelengths of incident radiation is rationalized by involving azide anions photoreactivity in observed redox changes.

Keywords. Azide; Iron(III) complexes; Mechanism; Photolysis; Quantum yield.

Introduction

Comparing with the photoredox chemistry of structurally similar halogeno iron(III) complexes, two specific factors relate to azido complexes. The first one lies in very peculiar and rich photoreactivity of free and bonded azido groups, which can convert in the primary photochemical step to a six-electron triplet [1] or singlet [2] nitrene intermediate, azidyl radical [3], radical N$_3^{\bullet}$ [4], or a nitrido ligand coordinated to the central atom by a triple bond [5]. Of the mentioned primary products, nitrido complexes were isolated and characterized also in the solid state, radicals N$_3^{\bullet}$ were identified in solutions by spin trapping EPR, and the formation of

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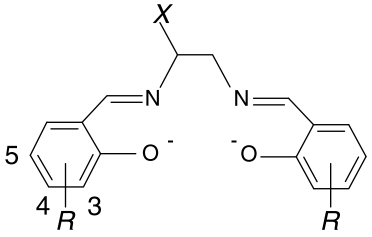

Fig. 1. Schematic representation of the N_2O_2-ligands *R*-salen^{2-} ($X = $H; $R = $H, 5-Cl, 5-Br, 4-$CH_3O$) and *R*-sal(*Me*)en^{2-} ($X = CH_3$; $R = $H, 5-Br)

nitrene and azidyl intermediates was postulated based on products of subsequent secondary reactions. In some cases only transformation of coordinated N_3^- to dinitrogen molecules [6] or a photoredox process not involving the azido ligand [7] was found.

Photochemistry of iron(III) azido complexes has been limited so far to compounds with macrocyclic porphyrin or cyclam ligands [5, 8]. Prolonged low-temperature irradiation of such complexes has led to the formation of iron(V) nitrido compounds.

Contrary to halogenides, azide anions can undergo intraligand decomposition which opens a possibility to investigate a "bell-like" dependence of the integral quantum yield of the central atom reduction on the wavelength of incident radiation [9].

In this contribution, the results on photochemical properties of the complexes *trans*-[Fe(N_2O_2)(CH_3OH)N_3], obtained within the attempts to examine the significance of the above mentioned factors, are presented. The structures of the $N_2O_2{}^{2-}$ ligands are shown in Fig. 1.

Results and Discussion

Given the known tendency of the high-spin Fe(III) to form hexacoordinated complexes both in the solid state and solutions, that of salen-type ligands to form their equatorial plane, and the previous potentiometric determination of a number of coordinated acido ligands [10], the composition of the complexes present in methanol at our conditions can be expressed as *trans*-[Fe(N_2O_2)(CH_3OH)N_3].

Typically, the electronic absorption spectra of the complexes [Fe(N_2O_2) (CH_3OH)N_3] consist of several distinct bands and shoulders. As an example, the spectra of [Fe(salen)(CH_3OH)N_3] and [Fe(salen)$(CH_3OH)_2$]$^+$ are shown in Fig. 2.

Based on previously published results [11–14] the absorption of a photon in the visible region can be attributed to ligand-to-metal charge transfer (LMCT) transitions O2p or N2p → Fe3d, the ultraviolet bands centered at $\lambda = 230$–260 and 300–350 nm to the intraligand transitions IL($\pi \rightarrow \pi^*$) localized predominantly on the phenyl rings and on the azomethine C=N fragment of the N_2O_2-ligands, respectively. Photons with $\lambda \leq 270$ nm are absorbed also by N_3^- anions, $\varepsilon_{254\,\mathrm{nm}}(N_3^-) \cong 100\,\mathrm{mol^{-1}\,dm^3\,cm^{-1}}$ for free anions in methanol [15]. Owing to their spin-forbidden nature, bands of ligand field (LF) states were not observed in solution spectra.

Control experiments confirmed that the complexes [Fe(N_2O_2)(CH_3OH)N_3] do not undergo spontaneous redox changes in methanolic solutions in the dark.

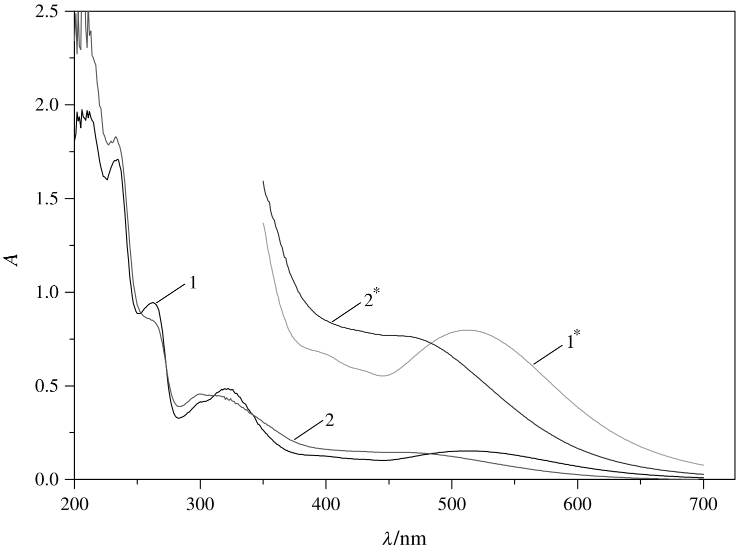

Fig. 2. Electronic absorption spectra of 2.0×10^{-4} *M* Fe(salen)$(CH_3OH)_2]^+$ (1, 1*) and [Fe(salen)$(CH_3OH)N_3$] (2, 2*) in methanol measured in 0.2 cm (1, 2) and 1.0 cm (1*, 2*) cells

Irradiation of methanolic solutions of the investigated complexes by ultraviolet or visible radiation leads to the photoreduction of Fe(III) to Fe(II). Calculation of quantum yields of net Fe(II) and CH_2O formation was performed by a standard procedure [16, 17].

The quantum yield values of Fe(II) formation, $\Phi_{Fe(II)}$, depend on the wavelength of incident radiation and on the peripheral groups *R* of the N_2O_2-ligands (Table 1).

In accord to our previous experience with the photoredox reactions of Fe(III) complexes in methanolic solutions [14, 16–18] we proposed that using *DMPO* and *ND* spin traps we could obtain evidence on the formation of radicals in the irradiated systems. Similarly as in case of iodo complexes [17], using *ND* in saturated ($c \sim 10^{-2}$ mol dm^{-3}) solutions of the [Fe(salen)$(CH_3OH)N_3$] complex, only poor-quality low-intensity EPR spectra of the $^{\bullet}CH_2OH$-*ND* adduct were obtained.

Upon irradiation of 5×10^{-3} mol dm^{-3} [Fe(salen)$(CH_3OH)N_3$] in the presence of *DMPO* spin trap, an EPR signal of $^{\bullet}CH_2OH$-*DMPO* was observed (Fig. 3).

Table 1. Quantum yields of Fe(II) formation, $\Phi_{Fe(II)}$, in irradiated methanolic solutions of 2.0×10^{-4} mol dm^{-3} [Fe(N_2O_2)$(CH_3OH)N_3$]

N_2O_2-ligand//λ_{irr}/nm	$\Phi_{Fe(II)} \times 10^3$			
	254	313	366	436
salen	2.64	4.14	0.522	0.062
5-Cl-salen	1.46	4.57	0.603	0.205
5-Br-salen	2.86	9.08	1.13	0.616
4-CH_3O-salen	0.702	26.5	1.18	0.184
sal(*Me*)en	0.006	5.33	1.02	0.009
5-Br-sal(*Me*)en	0.667	11.2	0.918	0.516

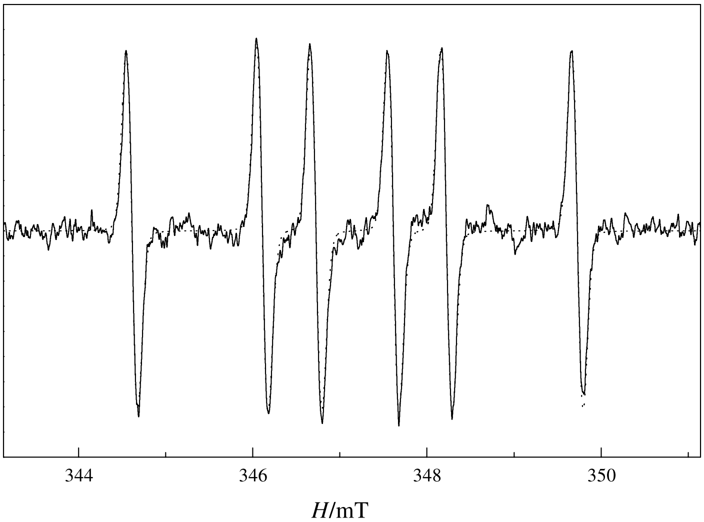

Fig. 3. Experimental (—) and simulated (···) room temperature EPR spectrum measured at irradiation of methanolic solution of $5 \times 10^{-3}\,M$ [Fe(salen)(CH_3OH)N_3] in the presence of $1.0 \times 10^{-2}\,M$ *DMPO* by radiation with $\lambda_{irr} \geq 300\,nm$; the EPR spectrum was simulated using the following parameters: $a_N = 1.50\,mT$, $a_H = 2.115\,mT$; $g = 2.0057$

As the final products of photoredox processes, formaldehyde and Fe(II) were formed. When irradiating the solutions at $\lambda = 313$, 366, or 436 nm, CH_2O and Fe(II) were formed in a 1:2 molar ratio. At irradiation by 254 nm, the ratio was 1.95:1. Within irradiation of $2.0 \times 10^{-3}\,mol\,dm^{-3}$ NaN_3 in methanol in the absence of any Fe(III) complex, formaldehyde was formed at the rate $1.5 \times 10^{-5}\,mol\,min^{-1}$.

Introducing oxygen into the systems after switching off the irradiation led to a slow reappearance of the parent iron(III) complex spectra.

Based on the above spectra interpretation, radicals identification, determined molar ratio of the final products, and literature data [19, 20], a mechanism of the processes occurring in irradiated systems of azido complexes may be proposed (in it, GS, IL and LMCT mean the corresponding ground state, intraligand and ligand-to-metal excited states of Fe(III) complexes, respectively, left superscripts denote the multiplicity of a given state; composition of the complexes is tentatively proposed to satisfy the stoichiometry requirements; for the sake of simplicity, non-redox reactions are not presented).

At irradiation of investigated systems by ultraviolet or visible radiation, spin-allowed excited states of complexes (Eqs. (1), (2)) or azide anions/ligands (Eq. (3)) are populated.

$$_{GS}{}^{6}[Fe^{III}(N_2O_2)(CH_3OH)N_3] \xrightarrow{h\nu(UV)} {}_{IL}{}^{6}[Fe^{III}(N_2O_2)(CH_3OH)N_3] \quad (1)$$

$$_{GS}{}^{6}[Fe^{III}(N_2O_2)(CH_3OH)N_3] \xrightarrow{h\nu(VIS)} {}_{LMCT}{}^{6}[Fe^{III}(N_2O_2)(CH_3OH)N_3] \quad (2)$$

$$N_3{}^{-} \xrightarrow{h\nu(UV,\lambda=254\,nm)} {}_{IL}{}^{1}N_3{}^{-} \quad (3)$$

Excited molecules can undergo physical deactivations from their higher energy to lower energy excited states or even to the ground state by intersystem crossing (isc) or internal conversion (ic) processes (Eqs. (4)–(7)).

$$_{\mathrm{IL}}{}^{6}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \xrightarrow{\mathrm{ic/isc}} {}_{\mathrm{LMCT}}{}^{6\ \mathrm{or}\ 4}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \tag{4}$$

$$_{\mathrm{LMCT}}{}^{6}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \xrightarrow{\mathrm{isc}} {}_{\mathrm{LMCT}}{}^{4}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \tag{5}$$

$$_{\mathrm{LMCT}}{}^{6\ \mathrm{or}\ 4}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \xrightarrow{\mathrm{ic/isc}} {}_{\mathrm{LF\ or\ GS}}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \tag{6}$$

$$_{\mathrm{IL}}{}^{1}\mathrm{N_3}^{-} \rightarrow {}_{\mathrm{GS}}\mathrm{N_3}^{-} \tag{7}$$

Of all accessible excited states of the complexes, only the spin allowed sextet and spin forbidden quartet LMCT states have the electron distribution suitable for an inner-sphere electron transfer leading to the observed photoreduction of Fe(III) to Fe(II) [19]. One of the LMCT states deactivations is a redox decomposition of the complexes leading to the formation of Fe(II) and $^{\bullet}\mathrm{CH_2OH}$ or $\mathrm{N_3}^{\bullet}$ (Eqs. (8), (9)).

$$_{\mathrm{LMCT}}{}^{6\ \mathrm{or}\ 4}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \rightarrow [\mathrm{Fe^{II}}(N_2O_2)\mathrm{N_3}]^{-} + \mathrm{H^+} + {}^{\bullet}\mathrm{CH_2OH} \tag{8}$$

$$_{\mathrm{LMCT}}{}^{6\ \mathrm{or}\ 4}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \rightarrow [\mathrm{Fe^{II}}(N_2O_2)(\mathrm{CH_3OH})] + \mathrm{N_3}^{\bullet} \tag{9}$$

In spite of the fact that only $^{\bullet}\mathrm{CH_2OH}$ was detected in irradiated systems, the primary formation of $\mathrm{N_3}^{\bullet}$ radical (9) cannot be definitely excluded since it can react with methanol molecules forming observed $^{\bullet}\mathrm{CH_2OH}$ radical (Eq. (10)).

$$\mathrm{N_3}^{\bullet} + \mathrm{CH_3OH} \rightarrow \mathrm{HN_3} + {}^{\bullet}\mathrm{CH_2OH} \tag{10}$$

The course of reaction (10) was investigated [21] and is supported also by the electrode potentials $E^o({}^{\bullet}\mathrm{CH_2OH/CH_3OH}) = 1.29\,\mathrm{V}$ and $E^o({}^{\bullet}\mathrm{N_3/N_3}^{-}) = 1.32\,\mathrm{V}$ [22].

In case of redox reversible radicals, back reactions of the products formed in the primary processes can occur forming the parent iron(III) complexes (Eqs. (11), (12)).

$$[\mathrm{Fe^{II}}(N_2O_2)\mathrm{N_3}]^{-} + \mathrm{H^+} + {}^{\bullet}\mathrm{CH_2OH} \rightarrow {}_{\mathrm{GS}}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \tag{11}$$

$$[\mathrm{Fe^{II}}(N_2O_2)(\mathrm{CH_3OH})] + \mathrm{N_3}^{\bullet} \rightarrow {}_{\mathrm{GS}}[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] \tag{12}$$

As documented by the value $E^o({}^{\bullet}\mathrm{CH_2OH/CH_2O}) = -1.180\,\mathrm{V}$ [23], the radical $^{\bullet}\mathrm{CH_2OH}$ is a strong reducing agent able to reduce further Fe(III) in subsequent secondary thermal redox steps (13) forming thus the final products Fe(II) and $\mathrm{CH_2O}$ (Eq. (13)).

$$[\mathrm{Fe^{III}}(N_2O_2)(\mathrm{CH_3OH})\mathrm{N_3}] + {}^{\bullet}\mathrm{CH_2OH} \rightarrow [\mathrm{Fe^{II}}(N_2O_2)\mathrm{N_3}^{-} + \mathrm{H^+} + \mathrm{CH_2O} \tag{13}$$

The ratio of $c(\mathrm{Fe^{II}})\!:\!c(\mathrm{CH_2O}) = 2\!:\!1$ determined in the systems irradiated by 313, 365, or 436 nm in this work shows that, along with the back reaction (8), the reduction of Fe(III) by $^{\bullet}\mathrm{CH_2OH}$ (13) is the main (if not the only) reactivity mode of $^{\bullet}\mathrm{CH_2OH}$. Its transformation to $\mathrm{HOCH_2–CH_2OH}$ or other products would increase the mentioned ratio. Moreover, no such products have been found so far in irradiated methanolic solutions of Fe(III) complexes [19].

A different situation was found when applying 254 nm radiation. In this case, part of the photons is absorbed by free or coordinated anion N_3^- (3) populating $(n \rightarrow \pi^*)$ excited state. One of its possible deactivation modes is its decomposition leading to nitrene anion N^- (Eq. (14)).

$$_{\mathrm{IL}}{}^1N_3^- \rightarrow N_2 + N^- \tag{14}$$

As rationalized previously in Ref. [24] this electronically unsaturated anion is a very strong two-electron oxidant able to react with surrounding molecules. In our systems it reacts with methanol producing further formaldehyde (Eq. (15)).

$$N^- + 2CH_3OH \rightarrow CH_2O + NH_3 + CH_3O^- \tag{15}$$

The sequence initiating by azide excitation (3) and terminating by formaldehyde formation (15) was proved independently by 254 nm irradiation of a methanolic solution of 2.0×10^{-3} mol dm^{-3} NaN_3 in the absence of iron(III) complex. Within such an irradiation formaldehyde was formed. The integral quantum yield of CH_2O formation was calculated based on the determined rate of CH_2O formation (1.11×10^{-7} mol min^{-1}), absorbance of solution at $\lambda = 254$ nm ($A = 0.1$ in 1 cm cell) and intensity of radiation emitted by the used low-pressure Hg-lamp determined by actinometry ($I_o = 5.5 \times 10^{-5}$ einstein min^{-1}) and reached the value of $\Phi \cong 1 \times 10^{-2}$. This "extra formed" formaldehyde causes the observed high molar ratio $n(CH_2O){:}n(Fe^{II}) = 1.95{:}1$ in the system of $[Fe(salen)(CH_3OH)N_3]$ irradiated by 254 nm incident radiation.

It should be pointed out that the molar absorption coefficient at $\lambda = 254$ nm of the complexes ($\varepsilon \cong 3500$ mol^{-1} dm^3 cm^{-1}) is significantly higher than that of azide anion ($\varepsilon \cong 100$ mol^{-1} dm^3 cm^{-1}). On the other hand, the concentration of N_3^- anions is ten times higher (2.0×10^{-3} mol dm^{-3}) than that of the complexes (2.0×10^{-4} mol dm^{-3}) in the irradiated solutions and, moreover, the quantum yield of N_3^- anions decomposition (*ca.* 10^{-2}) exceeded that of Fe(III) photoreduction (Table 1). Thus, though N_3^- anions absorb only a small part of the incident 254 nm photons (3), their participation (14, 15) in the photochemical processes occurring in irradiated systems cannot be fully excluded.

Tetradentate *Schiff* base *N2O2*-ligands behave as "redox innocent" ligands obviously not participating in the occurring redox processes. This conclusion is supported by dark reoxidation of Fe(II) expressed by stoichiometry given in Eq. (16).

$$4[Fe^{II}(N_2O_2)N_3]^- + O_2 + 4H^+ + 4CH_3OH \rightarrow 4[Fe^{III}(N_2O_2)(CH_3OH)N_3] + 2H_2O \tag{16}$$

A possible formation of Fe(V) nitrido complexes was checked by a prolonged 3 hour irradiation of $[Fe(salen)(CH_3OH)N_3]$ in methanol at $\lambda = 254$ or 313 nm, followed by vacuum room-temperature evaporation of methanol and measurement of IR spectra of the solid residue in Nujol mull. No characteristic band of triple Fe(V)≡N bond (centered at about $\bar{\nu} = 875$ cm^{-1}), however, appeared which is in agreement with observation that iron(V) nitrido complexes are stable predominantly at low temperature and only in case of complexes with macrocyclic ligands (cyclam, porphyrin) [8, 5].

Based on the rationalization published in Ref. [25], the observed decrease in the quantum yield, $\Phi_{Fe(II)}$, with increasing incident radiation wavelength may be understood and summarized in four points:

1) The communication between the populated IL and photoredox reactive LMCT states (4) is very effective.
2) The photoreduction (Eqs. (8), (9)) of Fe(III) to Fe(II) is the most significant (if not the only) mode of photoredox deactivation of the Fe(III) complexes.
3) The content of rovibrational energy of the photoreactive LMCT states is proportional to the energy of absorbed photon. The higher the difference between the rovibrational energy of a given LMCT state and the threshold energy of the primary photoproducts formation (Eqs. (8), (9)), the higher the probability of their separation, *i.e.* the lower the probability of their recombination (Eqs. (11, 12)) and, consequently, the higher $\Phi_{Fe(II)}$.
4) Since the investigated complexes provide no luminescence, continuous photolysis does not allow to distinguish between the photoredox reactivity of the quartet and sextet LMCT states.

A decrease in $\Phi_{Fe(II)}$ with decreasing incident radiation wavelength is a consequence of opening a new chemical deactivation channel not involving the central atom. This channel is a decomposition of the azido group, which can occur only when applying 254 nm radiation populating directly an excited state of this group along with excitation of the complex present.

A comparison of the quantum yields, $\Phi_{Fe(II)}$, for $[Fe(N_2O_2)(CH_3OH)N_3]$ and those for their structural analogues with axial F^-, Cl^-, Br^-, or I^- ligands [14, 20] documented a different wavelength dependence of the integral quantum yield of Fe(III) photoreduction due to involvement of azido group decomposition initiated by absorption of photons with 254 nm.

Experimental

The *Schiff* bases H_2(salen), H_2(5-Cl-salen), H_2(5-Br-salen), $H_2(4\text{-}CH_3O\text{-salen})$, H_2(sal(*Me*)en), and H_2(5-Br-sal(*Me*)en) were synthesized using a general procedure [26] by condensation of the corresponding *R*-salicylaldehyde with 1,2-ethanediamine or 1-methyl-1,2-ethanediamine in 2:1 molar ratio in methanol. The purity of the ligands was checked by elemental analysis, fusion temperature, ^{13}C NMR and ^1H NMR spectra.

Methanol (Lachema, reagent grade) was dried before use by distillation from $Mg(OCH_3)_2$. 1,2-Ethanediamine and 1-methyl-1,2-ethanediamine (Lachema) were distilled at a reduced pressure prior to use. 5,5-Dimethyl-1-pyrrolidine-*N*-oxide, *DMPO* (Aldrich), was freshly distilled before use and stored under argon in a freezer. Potassium tris(oxalato)ferrate(III) (Oxford Organic Chemicals), nitrosodurene, *ND* (Sigma), NaN_3 (Sigma), and 1,10-phenanthroline (Aldrich) were used without further purification. The other chemicals were purchased from Lachema and used as received. All commercial chemicals were of analytical grade.

Solutions of $[Fe(N_2O_2)(CH_3OH)N_3]$ were prepared *in situ* from stock methanolic solutions of $Fe(NO_3)_3$, corresponding $H_2(N_2O_2)$ and NaN_3 so as to obtain solutions with the initial concentrations of $c(Fe^{III}) = 2.0 \times 10^{-4}\,mol\,dm^{-3}$, $c(H_2(N_2O_2)) = 2.05 \times 10^{-4}\,mol\,dm^{-3}$, and $c(N_3^-) = 2.0 \times 10^{-3}\,mol\,dm^{-3}$.

Steady state photolysis at $\lambda = 254$ nm was performed in a two-chambered quartz photoreactor equipped with a low pressure Germicidal Lamp G8T5, or at $\lambda = 313$, 366, or 436 nm in a three-chambered temperature-controlled (20 ± 1°C) quartz photoreactor (Applied Photophysics), radiation of the high-pressure 150 W Hg-lamp was monochromatized by solution filters [10]. The irradiated solutions were deoxygenated by purging with solvent-saturated argon 30 min prior and during irradiation. Intensity of the incident monochromatized radiation was determined by a ferrioxalate actinometer [27].

The course of photoredox changes was monitored by electronic absorption spectroscopy as time evolution of $c(\mathrm{Fe^{II}})$ and $c(\mathrm{CH_2O})$, Fe(II) being in the form of $[\mathrm{Fe(phen)_3}]^{2+}$ and formaldehyde CH_2O as 3,5-diacetyl-1,4-dihydrolutidine [16, 17]. The amount of photolyses was limited to less than 5% to avoid radiation absorption by photoproducts.

The EPR spectra were measured on a Bruker 200D spectrometer (Germany) interfaced to an Aspect 2000 computer (Germany) using freshly prepared solutions containing spin traps *DMPO* or *ND*. Measurement conditions and a mode of the EPR spectra simulations are given in our previous paper [17].

Electronic absorption spectra were recorded on a Specord 200 spectrophotometer, IR spectra were scanned on a FT spectrometer Nicolet, Magna 750 in Nujol mulls.

Details on the experiments performed and experimental data treatment are described elsewhere [16, 17].

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