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Mechanism of Photoinduced Processes in Solutions of Iodo Iron(III) Complexes Containing *Schiff* Base Ligands

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Summary. The mechanisms of photoinduced processes occurring in methanolic solutions of *trans*-[Fe(4-*R*-*benacen*)(CH_3OH)I] (4-*R*-*benacen*$^{2-}$: N,N′-ethylene-*bis*-(4-*R*-benzoylacetoneiminato) tetradentate open-chain *Schiff* bases with $R=$ H, Cl, Br, CH_3, OCH_3, or NO_2) were investigated by electronic absorption spectroscopy and EPR spin trapping. The complexes are redox-stable in the dark both in the solid state and in methanolic solutions. Ultraviolet and/or visible irradiation in methanol induces photoreduction of Fe(III) to Fe(II). No formation of $I^{\bullet}$ or I_3^- was observed. $^{\bullet}CH_2OH$ radicals and/or solvated electrons were identified in irradiated systems using nitrosodurene or 5,5-dimethyl-1-pyrroline-N-oxide as spin traps. The final product of the photooxidation coupled with the photoreduction of Fe(III) is formaldehyde, the molar ratio of Fe(II) and CH_2O being close to 2:1. The efficiency of the photoredox process is strongly wavelength dependent and influenced by the peripheral groups *R* of the tetradentate ligands. It is suggested that the primary photoredox step starts from thermally nonequilibrated ligand-to-metal charge transfer excited states.

Keywords. Mechanism; Photochemistry; Iron(III) complexes; Iodo ligand; *Schiff* bases; EPR spin trapping.

Introduction

Due to the reducibility of Fe^{3+} and the reducing properties of I^- it had long been believed that ferric iodide FeI_3 and iodo iron(III) complexes cannot exist [1]. These 'unusual' compounds are not mentioned even in fundamental inorganic compendia [2, 3], or only few data are given [4]. The existence and redox stability of iodo iron(III) complexes are conditioned predominantly by three factors: *i*) the solvent used influencing the electrode potentials $E^0(Fe^{III}/Fe^{II})$ and $E^0(I_2/I^-)$, *ii*) the impact of external energy (heat, photons), and *iii*) the coordination of ligands stabilizing the Fe(III) oxidation state. As an example of such ligands, tetradentate open-chain *benacen*-type compounds (in the following abbreviated as N_2O_2) may be given.

Apart from the ground state redox stability of iodo iron(III) complexes, their photoredox reactivity may be of interest due to two counter-acting factors being specific for iodo complexes when compared to structurally analogous complexes

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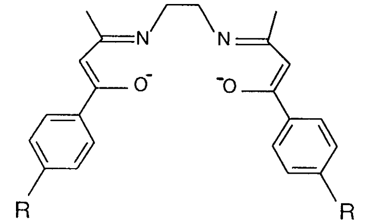

Fig. 1. Schematic representation of the ligands 4-*R-benacen*$^{2-}$

with other halo ligands. The first one lies in the easy thermodynamic oxidizability of I^-, opening a chance to reach the highest photoredox quantum yield; the second is the so-called 'heavy-atom effect' causing in some cases significantly lower photoreactivity of iodo complexes [5].

In this contribution, the results of the investigation of the mechanism photochemical reactions of *trans*-[Fe(N_2O_2)(CH_3OH)I] are presented. The structure of the $N_2O_2^{2-}$ ligands is shown in Fig. 1.

Results and Discussion

The electronic absorption spectra of all investigated complexes are very similar and consist of several broad bands and shoulders. As a typical example, the spectrum of *trans*-[Fe(*benacen*)(CH_3OH)I] is shown in Fig. 2.

According to previously published results [6–9], the absorption of a photon in the visible region can be attributed to ligand-to-metal charge transfer (LMCT) transitions O2p or I5p → Fe3d (Eq. (2)), whereas the ultraviolet bands centered at

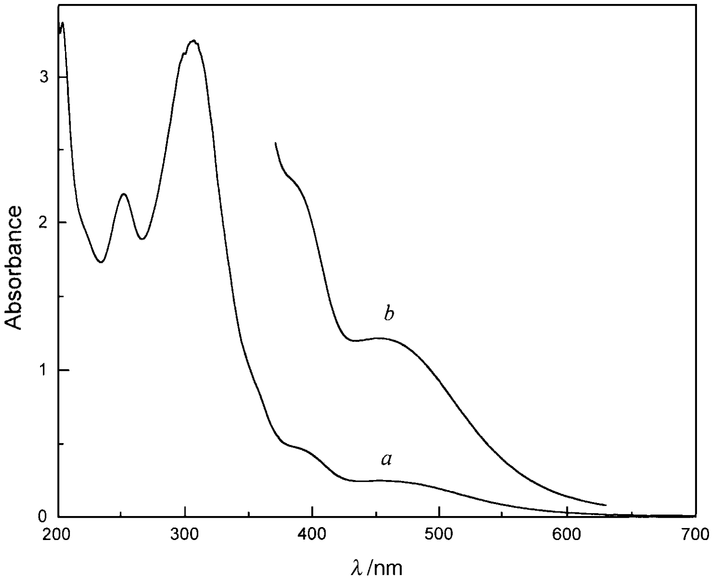

Fig. 2. Electronic absorption spectra of $5.046 \times 10^{-4}\,\mathrm{mol \cdot dm^{-3}}$ *trans*-[Fe(*benacen*)(CH_3OH)I] in methanolic $5.00 \times 10^{-3}\,\mathrm{mol \cdot dm^{-3}}$ [$N(CH_3)_4$]I measured in 1.0 cm (a) and 0.2 cm (b) cell

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

λ_{irr}/nm	254	313	366	546
benacen	6.55	3.90	3.00	1.75
4-Cl-*benacen*	7.15	5.50	4.30	0.90
4-Br-*benacen*	2.50	1.95	1.25	0.50
4-NO_2-*benacen*	3.90	3.30	2.50	1.75
4-Me-*benacen*	9.90	8.20	5.00	3.10
4-CH_3O-*benacen*	7.85	6.00	3.85	1.85

230–260 nm and 270–380 nm result from the $\pi \rightarrow \pi^*$ transitions localized predominantly on the phenyl rings and on the azomethine C=N fragment of the N_2O_2 ligands (Eq. (1)). Photons with $\lambda \leq 255$ nm are also absorbed by I^- anions. The bands of spin-forbidden LF transitions are not observed in solution spectra of high-spin iron(III) complexes.

The complexes $[Fe(N_2O_2)(CH_3OH)I]$ do not undergo spontaneous redox changes in methanolic solutions in the dark. Irradiation of methanolic solutions of the investigated complexes with UV and/or visible radiation leads to the photoreduction of Fe(III) to Fe(II). The integral quantum yields of Fe(II) formation, $\Phi_{Fe(II)}$, depend on the incident radiation wavelength and on the peripheral groups *R* of the N_2O_2 ligands (Table 1).

Complying with our previous experience with the photoredox reactions of Fe(III) complexes in methanolic solutions [7, 10, 11] we proposed that the use of nitrosodurene (*ND*) or 5,5-dimethyl-1-pyrroline-N-oxide (*DMPO*) spin traps could provide evidence on the radicals present in the irradiated systems. Using *DMPO* in solution of $10^{-3}\,\mathrm{mol \cdot dm^{-3}}$ [Fe(*benacen*)(CH_3OH)I], a reduced quality EPR spectrum clearly exhibiting features characteristic for a $^{\bullet}$*DMPO*-CH_2OH adduct ($a_N = 1.52$ mT, $a_H = 2.14$ mT, $g = 2.0059$ [12–16]) was obtained upon continuous irradiation ($\lambda > 300$ nm).

Irradiation of $10^{-3}\,\mathrm{mol \cdot dm^{-3}}$ [Fe(*benacen*)(CH_3OH)I] in the presence of *ND* resulted in the EPR spectrum shown in Fig. 4, which is in excellent agreement with hyperfine splittings of $^{\bullet}$*ND*-CH_2OH ($a_N = 1.444$ mT, $a_H(2H) = 1.188$, $g = 2.0054$ [14–17]). EPR spectra monitored during *in situ* photochemical experiments for solutions with different concentration of free I^- anions showed that higher values of $c(I^-)$ corresponded to a lower intensity of $^{\bullet}$*DMPO*-CH_2OH and $^{\bullet}$*ND*-CH_2OH signals.

In agreement with the analysis of the photoredox mechanism we propose that the excess of iodide in the irradiated systems is reflected in the fast consumption of $^{\bullet}CH_2OH$ radicals (Eq. (9)) which resulted in a lower yield of $^{\bullet}$*DMPO*-CH_2OH and $^{\bullet}$*ND*-CH_2OH adducts.

An evidence for the formation of solvated electrons was obtained by the addition of large amounts of benzyl chloride to the experimental systems. The reaction of benzyl chloride with electrons lead to the formation of $^{\bullet}CH_2C_6H_5$ radicals which were trapped by *ND* and easily identified ($a_N = 1.44$ mT, $a_H(2H) = 0.89$ mT [14–17]). The experimental EPR spectrum obtained upon continuous irradiation

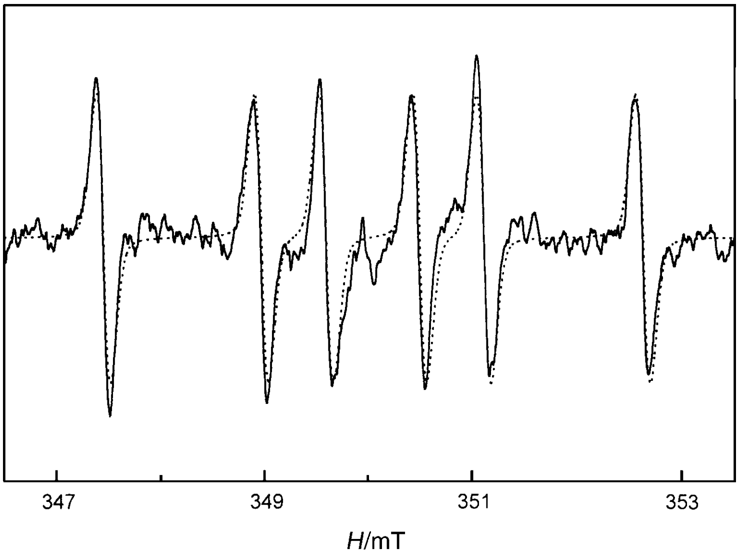

Fig. 3. Experimental EPR spectrum (solid line) measured upon continuous irradiation ($\lambda > 300$ nm) of a [Fe(*benacen*)(CH_3OH)I] methanol solution ($c = 10^{-3}\,mol \cdot dm^{-3}$) in the presence of *DMPO* ($c(DMPO) = 0.01\,mol \cdot dm^{-3}$); the simulated EPR spectrum (dotted line) was calculated using the *Hamiltonian* parameters $a_N = 1.52$ mT, $a_H = 2.14$ mT, and $g = 2.0059$)

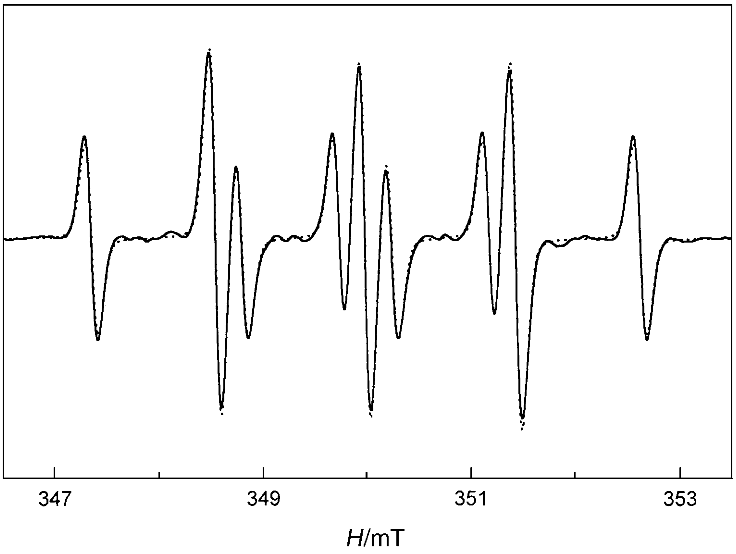

Fig. 4. Experimental EPR spectrum (solid line) measured upon continuous irradiation ($\lambda > 300$ nm) of a [Fe(*benacen*)(CH_3OH)I] methanol solution ($c = 10^{-3}\,mol \cdot dm^{-3}$) saturated with *ND* spin trap; the simulated EPR spectrum (dotted line) was calculated using *Hamiltonian* parameters $a_N =$ 1.444 mT, $a_H(2H) = 1.188$ mT, and $g = 2.0054$)

($\lambda > 300$ nm) of a solution of [Fe(*benacen*)(CH_3OH)I] in methanol : benzyl chloride = 1:1 ($c = 10^{-3}mol \cdot dm^{-3}$) containing excess of iodide ($c(I^-) = 10^{-2}\,mol \cdot dm^{-3}$) and saturated with nitrosodurene is depicted in Fig. 5. It was simulated as a superposition of three individual EPR spectra obtained from 64% of $^\bullet$*ND*-$CH_2C_6H_5$ adduct ($a_N = 1.444$ mT, $a_H(2H) = 0.894$ mT, $g = 2.0054$), 21% of $^\bullet$*ND*-CH_2OH

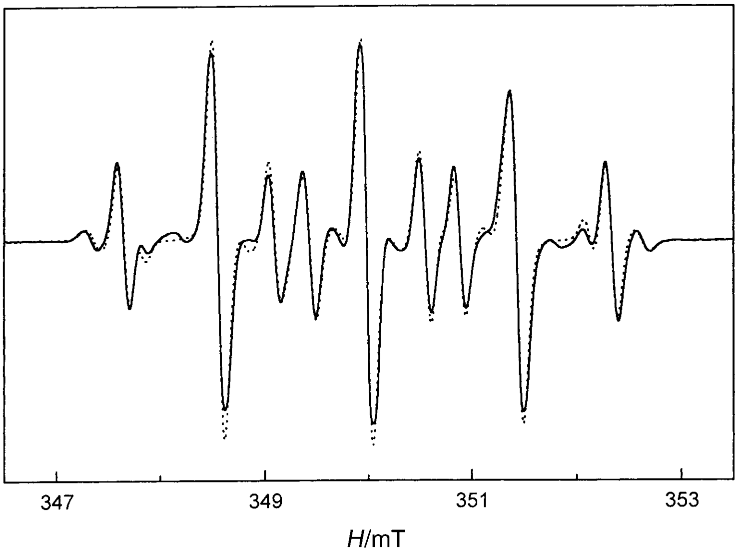

Fig. 5. Experimental EPR spectrum (solid line) measured upon continuous irradiation ($\lambda > 300\,\mathrm{nm}$) of a [Fe(*benacen*)($CH_3OH$)I] solution in methanol : benzyl chloride = 1 : 1 ($c = 10^{-3}\,\mathrm{mol \cdot dm^{-3}}$) in the presence of excess of iodide ($c(\mathrm{I}^-) = 0.01\,\mathrm{mol \cdot dm^{-3}}$) saturated with *ND* spin trap; the simulated EPR spectrum (dotted line) was calculated as a linear combination of three individual EPR spectra (see text)

adduct ($a_N = 1.444\,\mathrm{mT}$, $a_H(2H) = 1.188\,\mathrm{mT}$, $g = 2.0054$), and 15% of $^{\bullet}$*ND*-CH_2R radical ($a_N = 1.385\,\mathrm{mT}$, $a_H(2H) = 0.772\,\mathrm{mT}$, $g = 2.0056$), which corresponds to the photoproduct of *ND* in methanol [14–17].

The identification of $^{\bullet}CH_2OH$ (and the absence of $CH_3O^{\bullet}$) radicals in the systems may be taken as evidence that instead of free (non-coordinated) methanol molecules or CH_3O^- anions, rather coordinated methanol molecules take part in the primary electron transfer reactions [12, 15, 18]. As the final products of the photoredox processes, formaldehyde and Fe(II) were formed in a 1:2 molar ratio. No indication of an accumulation of I_3^- anions [19] during irradiation was observed in electronic absorption spectra. Introducing oxygen into the systems after switching off the irradiation led to a reappearance of the parent iron(III) complex spectra (Eq. (11)).

From the above experimental results and literature data [20, 21], a mechanism of the processes occurring in irradiated systems of iodo complexes may be proposed. In this context, GS, IL, and LMCT will denote the ground state, intraligand, and ligand-to-metal excited states of Fe(III) complexes, respectively; left superscripts point at the multiplicity of a given state. The composition of the complexes is tentatively proposed to satisfy the stoichiometry requirements; for the sake of simplicity, non-redox reactions are not presented.

Excited states population:

$$ {}^{6}_{\mathrm{GS}}[\mathrm{Fe^{III}}(N_2O_2)(CH_3OH)I] \xrightarrow{h\nu\,(\mathrm{UV})} {}^{6}_{\mathrm{IL}}[\mathrm{Fe^{III}}(N_2O_2)(CH_3OH)I] \qquad (1) $$

$$ {}^{6}_{\mathrm{GS}}[\mathrm{Fe^{III}}(N_2O_2)(CH_3OH)I] \xrightarrow{h\nu(\mathrm{Vis})} {}^{6}_{\mathrm{LMCT}}[\mathrm{Fe^{III}}(N_2O_2)(CH_3OH)I] \qquad (2) $$

Physical deactivation by intersystem crossing (ISC) or internal conversion (IC):

$$ {}^{6}_{\mathrm{IL}}[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] \xrightarrow{\mathrm{IC/ISC}} {}^{6\text{ or }4}_{\mathrm{LMCT}}[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] \tag{3} $$

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

$$ {}^{6\text{ or }4}_{\mathrm{LMCT}}[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] \xrightarrow{\mathrm{IC/ISC}} {}_{\mathrm{LF\ or\ GS}}[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] \tag{5} $$

Primary photoredox step:

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

Back reactions of the products formed in the primary process(es):

$$ [\mathrm{Fe^{II}(N_2O_2)I}]^- + \mathrm{H}^+ + {}^{\bullet}\mathrm{CH_2OH} \rightarrow {}_{\mathrm{GS}}[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] \tag{7} $$

Subsequent secondary thermal redox steps:

$$ [\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] + {}^{\bullet}\mathrm{CH_2OH} \rightarrow [\mathrm{Fe^{II}(N_2O_2)I}]^- + \mathrm{H}^+ + \mathrm{CH_2O} \tag{8} $$

$$ {}^{\bullet}\mathrm{CH_2OH} + \mathrm{I}^- \rightarrow \mathrm{ICH_2OH} + \mathrm{e}^- \rightarrow \mathrm{HI} + \mathrm{CH_2O} + \mathrm{e}^- \tag{9} $$

$$ {}_{\mathrm{GS}}[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] + \mathrm{e}^- \rightarrow [\mathrm{Fe^{II}(N_2O_2)(CH_3OH)I}]^- \tag{10} $$

Dark reoxidation of Fe(II):

$$ 4[\mathrm{Fe^{II}(N_2O_2)I}]^- + \mathrm{O_2} + 4\mathrm{H}^+ + 4\mathrm{CH_3OH} \rightarrow 4[\mathrm{Fe^{III}(N_2O_2)(CH_3OH)I}] + 2\mathrm{H_2O} \tag{11} $$

Of all accessible excited states, only the spin-allowed sextet and spin-forbidden quartet LMCT states have an electron distribution suitable for an inner-sphere electron transfer leading to the observed photoreduction of Fe(III) to Fe(II) [1, 20]. One possibility of LMCT state deactivation is a redox decomposition of the complexes leading to the formation of Fe(II) and ${}^{\bullet}\mathrm{CH_2OH}$ (Eq. (6)). As documented by the value $E^0({}^{\bullet}\mathrm{CH_2OH/CH_2O}) = -1.180\,\mathrm{V}$ [22], the radical ${}^{\bullet}\mathrm{CH_2OH}$ is a strong reducing agent able to reduce further Fe(III) (Eq. (8)). It should be pointed out, however, that even a formation of $\mathrm{I}^{\bullet}$ cannot be definitely excluded. Reactions of iodine atoms with I^- leading to I_3^- are characterized by nearly diffusion limited rate constants. Given its characteristic spectrum [19] ($\varepsilon(353\,\mathrm{nm}) = 26400\,\mathrm{mol^{-1}} \cdot \mathrm{dm^3} \cdot \mathrm{cm^{-1}}$), formation of I_3^- may be traced by absorption spectroscopy. The generation of $\mathrm{I}^{\bullet}$ or I_3^- was, however, not observed. A spectroscopic study of a methanolic solution of I_3^- in the absence of iron complexes in a control experiment documented that at the applied conditions the rate of anion scavenging by methanol was negligible when compared with the rate of photochemical reactions.

Solvated electrons could not be formed directly from excited I^- since in the EPR experiments only radiation with $\lambda > 300\,\mathrm{nm}$ was applied. On the other hand, their production was identified only in systems containing I^- anions (in systems of iron(III) complexes with the other halides, only ${}^{\bullet}\mathrm{CH_2OH}$ radicals were observed [1, 5, 18, 20]). A sequence of steps (Eq. (9)) is in accordance with the observation of increasing EPR signal intensity attributed to $ND^{\bullet -}$ with increasing concentration of I^- anions. The energy demand to extract an electron from I^- (approximately 3.2 eV) is partly compensated by an energy release in a C$-$I bond formation step (approximately 2.3 eV) and other energy contributions (changes in solvation energies *etc.*). In accordance with data known so far [20, 21], the 2:1 ratio of $c(\mathrm{Fe^{II}}) : c(\mathrm{CH_2O})$ manifests the effective reduction of Fe(III) by $^{\bullet}\mathrm{CH_2OH}$ (Eq. (8)).

Iron(III) complexes belong to non-emitting compounds, and it has been deduced that this phenomenon is a consequence of very fast deactivation processes competing to radiation deactivation. Femtosecond techniques confirmed this explanation [23, 24], showing that vibrational relaxation is a much slower process than intersystem crossing or internal conversion. This means that the vibrational energy content of a directly populated photoredox reactive LMCT state (Eq. (2)) may be lower than that of the LMCT states reached *via* intersystem crossing or internal conversion from electronically higher energy intraligand states (Eq. (3)). The excess of the vibrational energy 'accumulated' during the IL → LMCT transition is a main candidate for the observed wavelength dependence of the quantum yield [25]. The higher the difference between the vibration energy of a given LMCT state and the threshold energy of the primary photoproducts formation (Eq. (6)), the higher the probability of their separation, *i.e.* the lower the probability of their recombination (Eq. (7)). Consequently, a higher $\Phi_{\mathrm{Fe(II)}}$ results. 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 comparison of the quantum yields $\Phi_{\mathrm{Fe(II)}}$ for $[\mathrm{Fe(N_2O_2)(CH_3OH)I}]$ and those for their structural analogues with axial $\mathrm{F^-}$, $\mathrm{Cl^-}$, or $\mathrm{Br^-}$ ligands [21, 26] documented a substantially higher photoredox reactivity of the former. Based on this comparison it can be concluded that the effect of the high thermodynamic redox unstability of iodo iron(III) complexes overcomes the 'heavy atom effect' of iodide. In spite of the experimental data obtained, a direct participation of $\mathrm{I^-}$ ligands in the primary redox process(es) should not be definitely excluded; to understand their role, further experiments are to be performed.

Experimental

The *Schiff* bases H$_2$(4-*R*-*benacen*), where $R = \mathrm{H, Cl, Br, CH_3, OCH_3}$, and $\mathrm{NO_2}$, were synthesized by condensation of the corresponding 4-*R*-benzoylacetone with 1,2-ethanediamine in methanol [27]. The solid complexes $[\mathrm{Fe(N_2O_2)I}]$ were synthesized as described in Ref. [6] and characterized by elemental analysis and electronic absorption spectra.

Methanol (Lachema, reagent grade) was dried before use by distillation from $\mathrm{Mg(OCH_3)_2}$. 5,5-Dimethyl-1-pyrroline-N-oxide (*DMPO*, Aldrich) was freshly distilled before use and stored in a freezer under argon. The other chemicals were of analytical grade, purchased from Lachema (*ND* from Sigma) and used as received.

Solutions of $[\mathrm{Fe(N_2O_2)(CH_3OH)I}]$ were prepared either from solid $[\mathrm{Fe(N_2O_2)I}]$ and $[\mathrm{N(CH_3)_4}]\mathrm{I}$ or *in situ* from stock methanolic solutions of $\mathrm{Fe(ClO_4)_3}$, $\mathrm{H_2(N_2O_2)}$, and $[\mathrm{N(CH_3)_4}]\mathrm{I}$ to obtain solutions with initial concentrations of $c(\mathrm{Fe^{III}}) = 2.0 \times 10^{-4}\,\mathrm{mol \cdot dm^{-3}}$ and $c(\mathrm{I^-}) = 2.0 \times 10^{-3}\,\mathrm{mol \cdot dm^{-3}}$.

Formed Fe(II) was determined as $[\mathrm{Fe}(phen)_3]^{2+}$, formaldehyde as 3,5-diacetyl-2,6-dimethyl-dihydropyridine. Details on photochemical experiments, actinometry, and evaluation of experimental data are described in Ref. [7].

The EPR spectra were measured at 290 K using a Bruker 200D spectrometer (Germany) interfaced to an Aspect 2000 computer (Germany). The freshly prepared solutions containing spin traps ($c(DMPO) = 0.01\,\mathrm{mol \cdot dm^{-3}}$; saturation concentrations of *ND* under the given experimental conditions) were carefully purged with argon and then placed in a quartz flat cell optimized for the Bruker TM cavity. The samples were irradiated directly in the cavity by a 250 W medium pressure Hg lamp (Applied Photophysics, England), and the EPR spectra were monitored *in situ*. A pyrex filter was used to cutoff the radiation below 300 nm. The *g*-value was determined with an uncertainty of ± 0.0001 using a marker containing 1,1-diphenyl-2-picrylhydrazyl. The simulations of the EPR spectra were obtained using the program EPR SimFonia (Bruker, Germany). The complex experimental EPR spectra were fitted as the linear combinations of the individual simulations according to a least squares procedure using the Scientist program (MicroMath).

Electronic absorption spectra were recorded on a Specord 200 spectrophotometer.

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