Fig. 1. ^1H NMR spectrum of the thiaselanes from reaction (b). The labels on the signals indicate the satellites due to ^{77}Se , ^1H coupling. Additionally, the number of bonds is given through which the nuclei are coupled; this number is repeated in the structural formulae; frequency 300.13 MHz; temperature 295 K.

tant relationships between the structure and NMR parameters are summarized here.

1. The number of Se atoms in a molecule can be derived from the number of ^{77}Se satellite pairs^[7] in the ^1H NMR spectrum (Fig. 1). Within a molecule, the value of the coupling constant decreases with the number of bonds separating the coupling nuclei.

2. The $\delta(\text{H})$ values^[8] increase with increasing chain length (1 → 2 → 3 → 4, 5 → 6, Table 1; 7 → 8 → 9, Table 2). A downfield shift also results, to a lesser extent, when one selenium atom is exchanged for one sulfur atom while the chain length remains constant (3 → 5, 4 → 6, Table 1).

3. The environment of the ^{77}Se nuclei has, as expected, a very strong influence on their magnetic shielding. According to the equation in Scheme 1, $\delta(\text{Se})$ of a given Se atom is dependant on the number of Se and S atoms in the α -, β -, and γ -positions. As is found for alkanes^[9] and phosphanes,^[10] the neighboring atoms in the α - and β -positions cause a downfield shift and those in γ -positions an upfield shift. Consistent with the greater electronegativity, the constants for sulfur are always more positive (stronger downfield shifts).

The constitution of the thiaselanes 1–9 follows unequivocally from the structural dependence of the NMR parameters (points

$$\delta(\text{Se}) = -227 + i\alpha_X + j\beta_X + k\gamma_X \pm 5; \text{X} = \text{S}, \text{Se}$$

i, j, k = number of S or Se atoms in α , β , and γ positions, respectively

$$\alpha_{\text{S}} = 377, \beta_{\text{S}} = 264, \gamma_{\text{S}} = -34, \alpha_{\text{Se}} = 311, \beta_{\text{Se}} = 254, \gamma_{\text{Se}} = -36$$

Scheme 1. Calculation of $\delta(\text{Se})$ with chemical shift increments.

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Magnetic Spin Effects on Photooxidation Quantum Yields of Ru^{II} -tris(bipyridine) Type Complexes in Magnetic Fields up to 17.5 Tesla**

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Today a large number of magnetic field and magnetic isotope effects on the rates and yields of chemical reactions are known.^[1] Their mechanisms are closely related to electronic- and nuclear-spin polarization effects arising from chemical reac-

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tions. The acronyms CIDEP and CIDNP have become established for these effects. The systematic study of all these effects is the object of *spin chemistry*.^[12] For magnetic field effects to influence chemical reaction rates, processes of electron spin must be kinetically coupled to processes of chemical change. In a magnetic field, the Larmor precession of the unpaired electron spins will modify the spin processes and thereby possibly alter the overall reaction kinetics. As a general rule, the magnetic field dependence of the observed kinetic effect reflects the ratio between the rates of inherent processes (spin processes and connected chemical processes) and the frequency of Larmor precession. Typically, the magnetic field effect has its strongest dependence on the relative change of the field at the point where both spin precession and internal processes occur at about the same rate (note that at a field of 1 Gauss (0.1 mT) the Larmor precession period is 0.36 μ s). The effect approaches a saturation value at field strengths several times higher. Most spin chemical experiments reported so far have been conducted in fairly weak magnetic fields (several 100 Gauss) for the obvious reason that this is sufficient if the internal spin processes are governed by hyperfine coupling. The field strength has, at least, been limited by the usual laboratory equipment for generating variable magnetic fields, that is, electromagnets which can only provide several Tesla of magnetic induction. Few investigations have utilized the fields of superconducting solenoids^[13] or have applied pulsed magnetic fields.^[4, 5] In one paper^[6] magnetic field effects at one high-field value of 14.5 T, produced by a Bitter type magnet, have been compared to effects at low fields; however, rather small effects were seen. In this communication we report the first investigations^[7] where the field is gradually varied from zero field to the highest field of 17.5 Tesla. Magnetic field dependent reaction kinetics could be measured for a system exhibiting the highest saturation fields observed so far and allowing determination of the fastest chemical and spin processes investigated so far by spin chemical methods.

The reaction we studied is the photooxidation of Ru^{II}-tris(bipyridine) type complexes **1a–1d** by methylviologen (MV²⁺).

The relevant reaction steps (Scheme 1) apply in general to electron transfer reactions with photoexcited triplet states. A triplet excited electron donor $^3\text{D}^m$ ($^3[\text{Ru}^{\text{II}}\text{L}_3]^{2+}$) undergoes elec-

Scheme 1.

tron *forward* transfer in a diffusion-controlled reaction (rate constant k_{set}) with a singlet ground state electron acceptor $\text{A}^n(\text{MV}^{2+})$ to generate a radical-ion pair $\text{D}^{m+1} \dots \text{A}^{n-1}$ ($[\text{Ru}^{\text{III}}\text{L}_3]^{3+} \dots \text{MV}^{+}$), which may dissociate (with rate constant k_{ce} of cage escape) to form the free radical ions, or else can undergo a *backward* electron transfer in the solvent cage to regenerate the ground-state reactants. The reaction scheme assumes conservation of spin multiplicity during the electron transfer steps. Thus the primary radical-ion pair (RIP) forms as a triplet spin state, and backward electron transfer (with rate constant k_{bet}) is only possible after a spin-flip transition to the corresponding singlet RIP state, occurring with a rate constant of k_{TS} and, for spin statistical reasons, of $3k_{\text{TS}}$ for the reverse process. An external magnetic field modifies the *rate* of spin equilibration in the RIP (for details see below) and thereby affects the overall efficiency of backward electron transfer and, indirectly, of cage escape (η_{ce}). The latter quantity is measured in our experiments.

The sources, preparation, and purification of the compounds employed in this investigation are described in Refs. [8, 9]. The experiments were carried out with a Bitter magnet at the High Magnetic Field Laboratory of MPI/CNRS at Grenoble. The magnetic field effect on the formation of free radicals was investigated by photostationary illumination in a continuous flow system.^[10, 11] The MV^{+} radicals produced in the photoreaction were protected against reoxidation through Ru^{III} complexes in the bulk by adding 2×10^{-2} M EDTA as a sacrificial donor to reduce Ru^{III} to Ru^{II}. The experiments were carried out in aqueous solutions with concentrations of 2×10^{-5} M for the complexes and 3×10^{-3} M for MV^{2+} . Further experimental details can be found in ref. [9].

The observed magnetic field dependence of the cage escape efficiency η_{ce} (which corresponds to the measured magnetic field effect on the quantum yield of photoinduced MV^{+} radical production) with the four complexes **1a–1d** is depicted in Figure 1.

Fig. 1. Relative magnetic field effect $R(B) = (\eta_{\text{ce}}(B) - \eta_{\text{ce}}(0)) / \eta_{\text{ce}}(0)$ on the quantum yield of MV^{+} radical production in the photoreaction with complexes **1a–1d**. The curves are results of theoretical simulations of the magnetic field dependence of η_{ce} using the parameters given in Table 1.

There is a pronounced ligand effect on the magnetic field sensitivity, which decreases regularly for every replacement of a bipyridine ligand by a phenanthroline ligand. In the low-field region the values measured with the Bitter magnet are in very good agreement with the results previously obtained up to 3.3 Tesla with the electromagnet in our laboratory.^[9] However, the new experiments with the fivefold extended field range clearly exhibit saturation behavior of the field effect above fields of about 5 Tesla.

The observed magnetic field dependence can be excellently reproduced by theoretical simulations based on our recently reported theoretical model,^[9, 12] which may be briefly summarized as follows: In the description of the spin state of the primary RIP ($[\text{Ru}^{\text{III}}\text{L}_3]^{3+} \cdots \text{MV}^{\cdot+}$) we make use of the concept of *effective spin* in order to account for the strongly spin-orbit-coupled nature of the Ru^{III} ground state in its low-spin d^5 electron configuration. The changes of the RIP spin state are governed by the fast relaxation of the effective spin at Ru^{III} described by a magnetic field independent relaxation time $\tau_s = T_1 = T_2$ (at zero field, k_{TS} in Scheme 1 is equal to $(4\tau_s)^{-1}$) and by the Zeeman interaction which, due to the strongly anisotropic g -tensor of Ru^{III} , enforces a fast loss of spin correlation between the effective spin at the complex and the spin at $\text{MV}^{\cdot+}$. Cage escape of the RIP and backward electron transfer are treated as first-order chemical rate processes with rate constants k_{ce} and $k_{\text{bet}, i}$, respectively; individual rate constants of backward electron transfer are employed for the four spin substates (denoted by index i) of the pair. These individual rate constants correspond to the products of the rate constant k_{bet} of the hypothetically spin-allowed process with the individual singlet characters $p_{s, i}$ of the respective spin states, one of them being essentially of singlet, three of them essentially of triplet nature. The efficiency of cage escape and its magnetic field dependence are calculated by numerically solving the pertinent stochastic Liouville equation and averaging the result over all orientations of the Ru^{III} complex.

In the simulation of the observed magnetic field effect, the values of the g -tensor components of the Ru^{III} complexes were taken from ESR spectra measured at low temperature in a solid matrix of acetonitrile/sulfuric acid,^[13] whereas the rate constants τ_s^{-1} , k_{ce} , and k_{bet} were treated as empirical fit parameters. As it turned out, in order to reproduce the absolute values of $\eta_{\text{ce}}(0)$ (the cage escape efficiency at zero field) and the full magnetic field effect curves within the bounds of experimental error, all three parameters of the set could be fixed with certainty within a margin of $\pm 20\%$ for each of the complexes. Thus a fairly precise, *absolute* determination of τ_s^{-1} , k_{ce} , and k_{bet} is possible with our method. No supplementary information on these parameters is needed beforehand. The values determined for the four complexes are listed in Table 1.

Since the four spin substates of the primary RIP exhibit kinetic differences the overall kinetics of the RIP decay is not truly monoexponential. An effective overall decay time may be defined, however, as in Equation (a). As can be seen from Table 1,

$$\tau_{\text{eff}} = \frac{\eta_{\text{ce}}}{k_{\text{ce}}} \quad (\text{a})$$

τ_{eff} lies around 100 ps. Under conditions of diffusion-controlled formation of the RIPs in a homogeneous reaction, as in our

experiment, it would be impossible to observe the RIPs directly with ps time-resolution, since even with the highest acceptor concentration the time of formation of the RIP cannot be decreased below several ns. Direct observation of the RIP is, however, feasible and has been reported for a number of chemically linked systems of electron donor and acceptor (dyads) where the forward electron transfer step is not limited by diffusion (cf. Ref. [14]). In a ps time-resolved study of dyads composed of a Ru^{II} -tris(bipyridine) type electron donor and a methylviologen type electron acceptor connected by a covalent spacer of one or two CH_2 links, Yonemoto et al.^[15] observed RIP lifetimes between 8.5 and 70 ps, depending on the number n of links and the thermodynamic driving force of backward electron transfer ΔG_{bet}^0 . For $n = 2$ and $\Delta G_{\text{bet}}^0 = -1.65$ eV, a situation that can be considered as corresponding closely to the case of the RIPs formed in our free diffusion reaction, the observed value is 70 ps, which indeed comes rather close to the values determined by our spin chemical approach. Thus, while the ps time-resolved studies of the dyads corroborate the conclusions from our spin chemical investigations, the spin chemical approach yields additional, highly relevant information on the nature of the process that, in fact, determines the *observable* effective decay time of the RIP, not only in our freely diffusing systems but, of course, also in the dyads. The magnetic field effects described here give unequivocal evidence that the overall backward electron transfer is a two-step process involving spin conversion followed by spin-conserving electron transfer. Using relation (b) we can predict from our results the lifetimes $\tau_{\text{bet, eff}}$ (cf. Table 1) that would

$$\tau_{\text{bet, eff}} = \frac{\eta_{\text{ce}}}{(1 - \eta_{\text{ce}})k_{\text{ce}}} \quad (\text{b})$$

be observed for our RIPs if cage escape could be excluded. On comparison of these values with $\tau_{\text{bet}} = k_{\text{bet}}^{-1}$, the time constant of the spin-allowed backward transfer, it becomes clear that the $\tau_{\text{bet, eff}}$ values are significantly longer which implies that the observed lifetime must be strongly controlled by the spin relaxation process. This conclusion suggests that before discussing lifetimes of the RIP derived from the ps experiment in terms of electron transfer theory, the observed time constants must be decomposed into a spin conversion part and an electron transfer part. The spin chemical approach, as applied here, seems the only way to achieve such a kinetic separation. Hence, it should be a great experimental challenge to verify our conclusions by *directly* observing the magnetic field effect on the decay time of dyadic RIPs in ps time-resolved experiments.

For k_{ce} a value of $5 \times 10^9 \text{ s}^{-1}$ based on an estimation using the Eigen–Debye equation is usually referred to in the literature.^[16] The k_{ce} values resulting from our analysis are of the same order of magnitude. A deviation by a factor of 2–3 seems acceptable in view of the assumptions in the diffusional model. Further-

Table 1. Characteristic reaction parameters for the primary Ru^{III} complex/ $\text{MV}^{\cdot+}$ radical pair.

Ligands	Parameters									
	ΔG_{bet}^0 [eV] [a]	η_{ce} [a]	$g_{\perp}$ [b]	$g_{\parallel}$ [b]	k_{ce} [ns ⁻¹] [c]	k_{bet} [ns ⁻¹] [c]	τ_s [ps] [c]	τ_{eff} [ps] [d]	τ_{bet} [ps] [e]	$\tau_{\text{bet, eff}}$ [ps] [f]
(bpy) ₃	-1.70	0.19	2.60	1.18	2.3 [g]	78 [g]	26.7 [g]	83	12.8	102
(bpy) ₂ (phen)	-1.70	0.19	2.60	1.18	2.2	66	23.5	86	15.2	107
(bpy) (phen) ₂	-1.71	0.18	2.54	0.95	1.9	46	21.3	95	21.7	116
(phen) ₃	-1.71	0.19	2.53	0.90	1.5	32 [g]	19.2 [g]	127	31.2	156

[a] Data from ref. [9]. [b] Data for ref. [13]. [c] From a simulation of the magnetic field dependence of η_{ce} . [d] Equation (a). [e] $\tau_{\text{bet}} = k_{\text{bet}}^{-1}$. [f] Equation (b). [g] Minor deviations from our preliminary values in [7] are due to more precise values of the g -tensor components employed in the present calculations. Here we use individual experimental values for each complex, whereas in [7] for both the (bpy)₃ and the (phen)₃ complex only a common, approximate set of values for the g -tensor components was used.

more, we note that the slight decrease of k_{ce} observed as bipyridine ligands are replaced by the somewhat larger phenanthroline ones is in agreement with the prediction of diffusion theory, according to which the rate constant of dissociation of a pair of reactants should decrease with increasing reaction distance. Taking into account that the backward electron transfer is a nonadiabatic process that depends on the value of the electronic coupling matrix element, the fact that k_{bet} too decreases by such a type of ligand substitution seems to support an assumption of increasing reaction distance because a concomitant decrease of the electronic coupling matrix element is expected for increasing separation. Since ΔG_{bet}^0 is constant, only a change in the coupling matrix element or in the internal reorganization energy associated with electron backward transfer or in both may account for a change of k_{bet} . Which of these two possibilities applies on substituting bipyridine by phenanthroline ligands cannot be decided at present.

The parameter τ_s is the paramagnetic relaxation time of the Ru^{III} complex in the RIP. It seems unlikely that the presence of the $\text{MV}^{+ \cdot}$ radical near the Ru^{III} complex has a significant effect on τ_s , and thus it may be conveniently compared to the corresponding value in the free Ru^{III} complex. Because of the short relaxation time and the large g -tensor anisotropy, an ESR signal of the present type of Ru^{III} complexes is not detectable in liquid solutions. However, indirect experimental access to τ_s is possible through the linewidth of ^1H resonances in NMR spectra.^[17, 18] Actually a value of $\tau_s \approx 20$ ps was evaluated by Doddrell et al.^[19] for $[\text{Ru}(\text{acac})_3]$ in a field range from 0.2 to 6 T. We have measured the ^1H - T_1 times for $[\text{Ru}(\text{bpy})_3]^{3+}$, from which the electronic relaxation times 25 ps and 9 ps, respectively, have been determined.^[20] These values are in rather good agreement with the spin chemical τ_s values. In particular these data confirm that our spin chemical inference of a decrease of τ_s on replacing bipyridine by phenanthroline ligands is correct.

In conclusion, we have shown that the spin chemical modeling of the magnetic field effects observed over a large range of magnetic fields allows an excellent simulation of the experimental data with a minimum of parameters. There is a substantial amount of evidence that the determined parameters represent reliable physical information that is not easily, if at all, available otherwise. This renders the method most valuable for a deep look into the details of fast backward electron transfer kinetics and paramagnetic relaxation occurring on a time scale of several tens of ps. with strongly spin-orbit coupled paramagnetic intermediates.

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Pronounced Electronic Coupling in Rigidly Connected N,C,N-Coordinated Diruthenium Complexes over a Distance of Up to 20 Å**

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Molecular systems able to ensure fast long-range electron transfer are an essential requirement for the application of thermal or photoinduced charge separation in artificial photosynthesis^[1] and in molecular electronics.^[2] Mixed-valence complexes are the prototypes of molecules able to undergo fast electron transfer usually between two metals and through a bridging ligand.^[3–11] We recently reported that a bridging ligand consisting of two cyclometalating bipyridylbenzenes connected back-to-back (**1**) affords diruthenium (5^3+)^[12, 13] and diosmium^[13] complexes displaying intense communication between both metal centers at a M–M distance of roughly 11 Å. (Strong intervalence coupling over this distance has also been recently observed in the “[Ru(pincer–pincer)Ru]” systems^[14]). In the present paper, we describe extended systems with two N,C,N coordination sites connected by various aromatic spacers, and show that strong communication can occur in diruthenium compounds with much longer bridging ligands. These ligands contain up to four phenyl rings and thus lead to a Ru–Ru distance of around 20 Å.

The synthesis of the ligand precursors **2-H₂** and **3-H₂**, as well as their diruthenium(II) complexes **6²⁺** and **7²⁺**, was described recently.^[15] In these complexes the metals are connected by a 4,4'-terphenyldiyl and 4,4'-quaterphenyldiyl ligand, respec-

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