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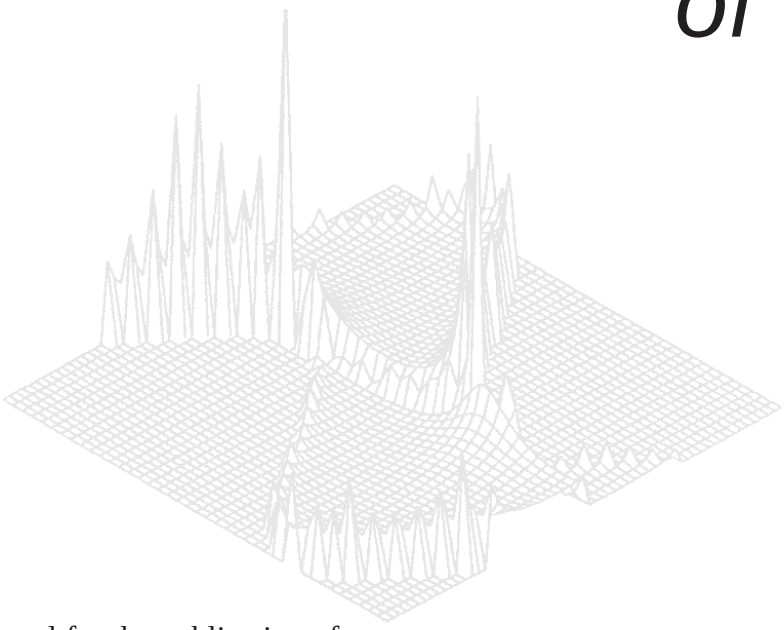

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Spin-lattice Relaxation Rates of Os and Other 5d Elements in Fe*

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Abstract

We give final results for the nuclear spin-lattice relaxation (SLR) rates of 5d series elements as dilute impurities in iron using the method of thermal cycling on oriented nuclei. In this report, we emphasise some aspects of the analysis of the relaxation curves, especially for Os and Au impurities. A comparison of the results with theory is given.

1. Introduction

The nuclear spin-lattice relaxation (SLR) rate of impurity nuclei in solids provides an interesting test of theory, in that it is particularly sensitive to the local density of states at the Fermi energy. In the past, a number of determinations of the SLR of dilute impurities in Fe host has been carried out using several methods (see Klein 1986 for a review), but its systematics as a function of impurity atomic number, even for such a well-studied host as iron, are by no means completely determined at present. A number of earlier measurements are unreliable and exhibit considerable scatter in the reported rates, for a variety of reasons. Here, we present some new results for the heavier members of the *5d* impurity series, and quote complete results for this series as obtained by our group. We note that the results for ^{182}Ta*Fe* and ^{186}Re*Fe* have already been reported (Bobek *et al.* 1993), as have those for ^{191}Pt*Fe* (Beck *et al.* 1996). A preliminary survey of the whole series was given in an oral presentation at the 10th International Conference on Hyperfine Interactions (Leuven, 1995). A more complete description of the experiments and results for the *5d* impurities in Fe will be published elsewhere (Beck *et al.* 1998*a*). In a companion paper, we give new results for 3d and 4d impurities in Fe (Beck *et al.* 1998*b*, present issue p. 267).

We mention briefly the applied-field dependence of the relaxation rates, which has caused some confusion and inaccuracy of reporting relaxation constants in the past (see Klein 1983, 1986). A good fit to the observed field dependence in all cases so far studied is offered by the 'enhancement factor model' (Kopp *et al.* 1981; Klein 1983), which assumes a proportionality of the additional rate at low

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applied fields to the square of the ferromagnetic enhancement factor known from NMR in ferromagnets. The precise origin of this proportionality is not certain, but it may be due to coupling of the impurity nuclear moments to magnons in the host material (Seewald *et al.* 1997). We will not discuss the source of the field dependence here, but will use the enhancement factor model where appropriate to ensure that our quoted relaxation constants represent the high-field saturation or intrinsic SLR rates.

Sources of uncertainties in past reported values of the SLR rate are incorrect analysis of data and lack of knowledge of the initial conditions for the relaxation. The method used in the present work avoids these difficulties and is conceptually simple: thermal cycling of oriented nuclei (TCON) (see Klein 1977, 1986). A detailed discussion of this method with comparisons with other techniques has been given elsewhere (Klein 1986; Bobek *et al.* 1993). We point out here that its principal drawback lies in its being an integral method, so that the nature of the observed anisotropy of nuclear radiations must be established by careful sample preparation and determination of the temperature dependence of the anisotropy. However, the presence of a non-orienting background has only a negligible effect on the results of the SLR measurement.

The present results complete the experimental systematics for the *5d*-series in Fe host and provide reliable values for comparison with theory. The implications for the *ab initio* calculations by the KKR–Green function method (Akai 1988; Akai *et al.* 1990), and for a possible spin-wave contribution to the intrinsic SLR rate (Weger mechanism, see Weger 1962), are discussed in detail by Bobek *et al.* (1993) and will be considered only briefly here. The theory of Akai (1988) for dilute impurities in iron represents the only *ab initio* calculations published to date; they reproduce the qualitative trends of the experimental data reasonably well, but show quantitative deviations. Nearly all of the calculated rates are *smaller* than the experimental values. The quantitative agreement is, however, relatively good for the 3d series (roughly 50%), becoming less satisfactory (80–100% deviations) in the 4d series (cf. Brewer *et al.* 1994*a*) and quite poor in the *5d*-series (factors of 2 to 5; see below). No calculations have yet been performed for the 4f-impurities, and very large deviations are seen for the light element impurities, in the few cases which have been measured (Brewer *et al.* 1994*b*). Various explanations for these deviations have been suggested. In order to provide reliable experimental values against which to test these hypotheses, we have performed the present set of experiments on the third transition series of impurities.

2. Experiments

The experiments were carried out by the TCON method using a metallic weak thermal link between the sample and the mixing chamber of a dilution refrigerator (base temperature 4·5 mK). The link was made of a thin plate of pure silver whose lower end served to mount the sample, together with a fast nuclear thermometer (^{54}MnAg, ^{54}MnAu, or ^{60}CoPd). The middle section of the silver plate was machined to produce a serpentine of typical width 1 mm and length ca. 50 mm; the thermal resistance of this link was then determined by the thickness of the silver (0·1–0·5 mm), and could be found by measuring its electrical resistance at liquid He temperature and applying the Wiedemann–Franz law (see Klein and Augustin 1988). The upper end of the link was attached to

a Cu mount which could be top-loaded into the dilution cryostat. The thermal behaviour of such a link is potentially quite complex, since both its thermal resistance and its heat capacity are temperature dependent. The heat capacities of the silver plate and of the samples and thermometers used in this work are small and only weakly temperature dependent (electronic heat capacities), but other materials used—especially soft solders—may have large nuclear Schottky heat capacities which depend strongly on the temperature (and the applied magnetic field). Therefore it is essential in practice to measure the momentary temperature of the sample using a fast nuclear thermometer. This temperature can then be taken into account in fitting the relaxation curves, by performing a numerical integration of the coupled differential equations describing the time evolution of the γ-ray anisotropy from the sample following a heat pulse. More details have been given by Klein (1986), Klein and Augustin (1988), and Beck (1997); some aspects of the fit procedure will be described below.

The samples used in this work were all made by melting a pure (4N) iron ingot with a small quantity of the desired impurity metal, giving an impurity concentration of the order of 0·1 at.%. The *5d*-metals are soluble in the Fe host lattice at this level, and homogeneity was ensured by remelting the ingots several times (either in Ar atmosphere with an electric arc furnace, or in high vacuum with an induction furnace). After pressing and rolling to give foils of ca. 50 μm thickness, we annealed the sample material for 8 hours at 850°C in high vacuum or in H_2. Pieces were then irradiated with thermal neutrons to give the desired radioactive isotope of the impurity element. Due to its low neutron capture cross sections, the Fe host lattice was not strongly activated in this process. Care was taken both to avoid fast neutrons and to anneal the samples after irradiation to remove radiation damage; when this was not done, deviations from the expected γ-ray anisotropy at low temperatures (computed from the known nuclear spins and moments and the static hyperfine fields B_{hf}) were seen, and no SLR data were collected in such cases. In general, agreement between the measured and calculated static anisotropies was good, although in some cases small corrections were applied in the fitting procedure for thermal gradients between sample and thermometer. In no case was evidence of a significant degree of oxidation or clustering of the impurities seen (i.e. as a reduced γ-ray anisotropy). More details of the sample preparation procedure are given by Bobek *et al.* (1993) for the cases of ^{182}Ta*Fe* and ^{186}Re*Fe* and will be given by Beck *et al.* (1998*a*) for the other samples. Exceptions to the above procedure were the ^{193}Os*Fe* and ^{191}Pt*Fe* alloys: here, due to the unfavourable natural abundances and neutron capture cross sections of the parent isotopes, it proved necessary to use isotope-enriched starting material in order to obtain sufficient γ-ray intensity from the desired radioisotopes. Initial experiments using natural starting materials gave very poor counting statistics and high parasitic heating due to unwanted isotopes in the samples, and were not used for further data evaluation. The alloys employed were made with 0·20 at.% Os in Fe, enriched to 99·4 at.% in ^{192}Os (natural abundance 41 at.%), and 0·07 at.% Pt in Fe, enriched to 0·79 at.% in ^{190}Pt (natural abundance 0·013 at.%). They were produced in a manner similar to the other samples and irradiated with a sufficient integrated flux of thermal neutrons to give about 10^5 Bq starting activity of the desired isotopes. Here, we concentrate on the experiments with ^{193}Os*Fe* and ^{198}Au*Fe*. Fig. 1 shows

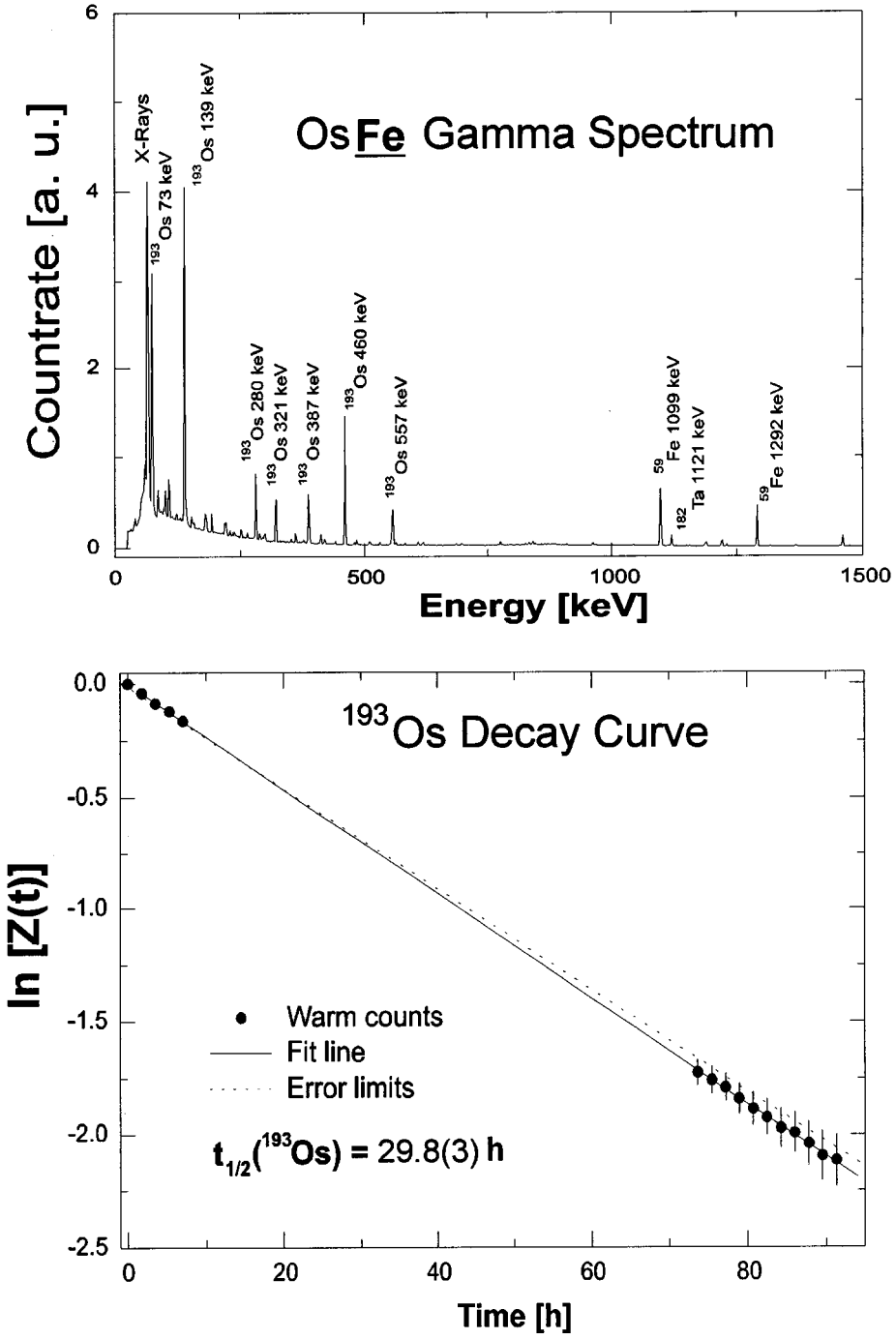

Fig. 1. Gamma-ray spectrum of a ^{193}OsFe sample (upper part), showing the strong 139 keV line used for TCON data analysis. The lower part shows our redetermination of the decay half-life of ^{193}Os using the 'warm counts' from the sample.

the gamma-ray spectrum of an Os*Fe* sample, along with our redetermination of the half-life of the ^{193}Os $I = \frac{3}{2}$ ground state. In preliminary static nuclear orientation experiments, the gamma-ray anisotropy as a function of temperature was measured and the half-life of the orienting parent state was redetermined, since the literature values are often not very precise. At this stage, a check for thermal gradients between sample and (nuclear) thermometer was performed and any deviations of the observed anisotropy, which could indicate defects, clustering, or incomplete magnetic saturation of the sample, were determined.

For the TCON experiments, heating pulses (using nonresonant rf power from a coil around the sample holder) were applied during a fixed time period, controlled by the computer which acquires the data. Gamma-ray spectra (typically 1024 energy channels) were collected as a function of time (256 time channels, heating during the first half of each time sweep) to give a 1024×256 matrix of counting rates. This procedure was repeated, with addition of the resulting matrices, until sufficient counting statistics were obtained (usually, the sum matrix was stored after about each hour of counting, so that the data could be examined for temperature drifts and other instabilities before addition to give time-averaged data from a typical measuring time of 12 hours). The gamma rays of interest in the energy spectra, along with a pulser peak for deadtime correction, were then integrated and the resulting values stored in much smaller matrices which, corrected for radioactive decay and normalised by the 'warm' counting rates determined separately, contained the gamma-ray anisotropies $W(t)$ ('relaxation curves'). These served as the starting point for the fitting procedure to obtain the relaxation constants C_{K}.

Ideally, on the time scale of the observed relaxation curve, the sample temperature will be a step function, cycling between the extreme temperatures T_h and T_l, which are determined by the applied rf power and the thermal impedance of the thermal link to the mixing chamber, as well as the heat leaks to the sample. In practice, the sample holder has a finite thermal time constant τ_{L} (and in fact, its time dependence seldom has a simple exponential form). This time constant was kept as short as possible compared with the nuclear relaxation time being studied. The measured gamma-ray anisotropy of the fast nuclear thermometer (usually ^{54}Mn*Ag*) was used to construct curves of the lattice temperature $T_{\mathrm{L}}(t)$, which could also be used in the fitting procedure.

The multiexponential relaxation function which results from solving the $2I$ coupled rate equations describing the nuclear relaxation at low temperatures is well known (Bacon *et al.* 1972; Klein 1986). It can be obtained either by solving the resulting eigenvalue equation using matrix diagonalisation, or numerically using an integration method such as the Runge–Kutta procedure. The latter is preferable, since it allows the momentary lattice temperature $T_{\mathrm{L}}(t)$ to be inserted at each integration step as a function of time, thus taking the true temperature dependence of the sample during the relaxation process into account. This is extremely important when the ideal, step-function behaviour is not obtained, as is usually the case unless the relaxation time being measured is very long. For the fitting procedure, the (possible) input parameters are the temperatures T_h and T_l and the single relaxation constant C_{K} which determines the time dependence of $W(t)$, as well as anisotropy parameters A_{red} and W_{D} ('anisotropy reduction' and 'drift' parameters) which allow for incomplete static orientation

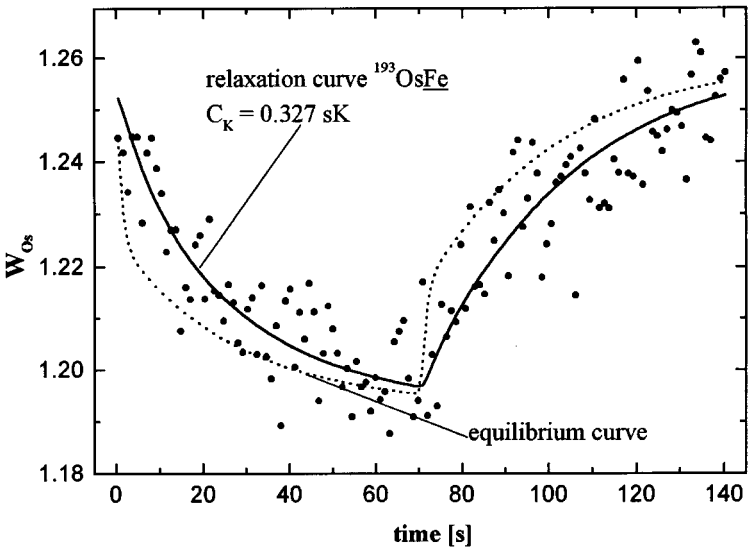

Fig. 2. Relaxation curve for ^{193}OsFe. The dotted curve shows the lattice temperature $T_{\mathrm{L}}(t)$ as determined by the fast nuclear thermometer. The solid curve is the fit to the multi-exponential relaxation function taking the momentary temperatures into account.

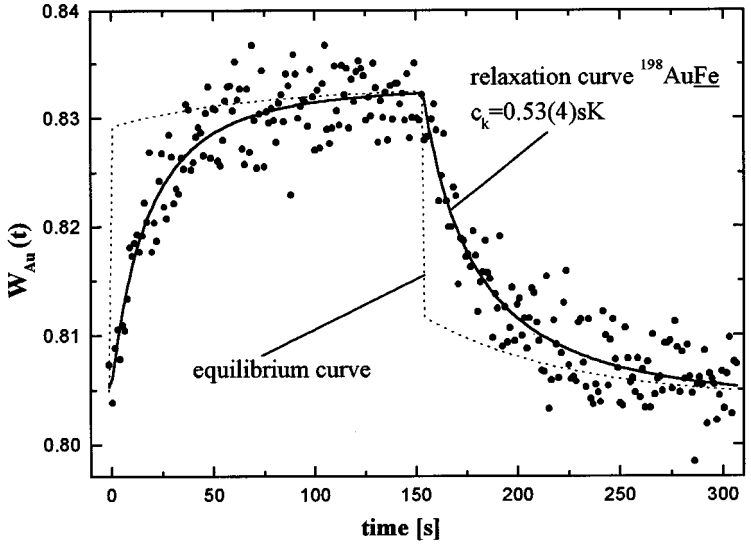

Fig. 3. Relaxation curve for ^{198}AuFe, similar to Fig. 2. Note that the lattice temperature follows nearly a step function, although a small 'tail' due to temperature-dependent heat capacities and warming of the surrounding materials by the heat pulse is still present.

of the sample nuclei (see above) and for count-rate drifts during the experiment; these latter parameters have ideal values of 1·00. In practice, it is not feasible to leave all five parameters free, in particular since their values are correlated in the fit. The temperatures T_h and T_l as well as the intermediate values $T_{\mathrm{L}}(t)$

are obtained from the thermometer data and fits are performed either with C_K as the only free parameter, or with the three parameters C_K, A_{red}, and W_D left free. In all cases reported here, the last two parameters were within a few per cent of 1·00, and their observed variations were used to estimate the uncertainties in the reported C_K values (along with the fit errors for C_K itself, which tend to underestimate the real experimental uncertainties). Great care was taken to verify that different fitting procedures gave the same result for C_K within the reported uncertainties. Figs 2 and 3 show relaxation curves for ^{193}Os*Fe* and ^{198}Au*Fe*, along with the corresponding lattice temperature curves $T_L(t)$ and various fits to the relaxation data. Relaxation curves for ^{182}Ta*Fe* and ^{186}Re*Fe* are given in Bobek *et al.* (1993), and for ^{191}Pt*Fe* in Beck *et al.* (1996). Curves for the other cases reported here can be found in Bobek (1994), Funk (1996) and Beck (1997).

3. Results and Discussion

We have determined relaxation constants C_K for dilute Ta, W, Re, Os, Ir, Pt and Au impurities in Fe. Aside from variations in dwell time δt and rf heating rate P_{rf} to optimise the experimental conditions, the measurement procedure was the same in all cases. The fitting procedure was varied as described above depending on the quality of the relaxation-curve data and the thermal response of the sample holder in the given case. For the favourable cases ^{182}Ta, ^{186}Re, ^{192}Ir and ^{198}Au, the applied-field dependence of C_K was determined (Bobek *et al.* 1993; Bobek 1994); see Fig. 4 for an example.

For the other, relatively short-lived isotopes ^{187}W, ^{193}Os and ^{191}Pt, only the high-field limiting value, $C_{K\infty}$, was determined. Table 1 summarises the values of $C_{K\infty}$ obtained, along with the other parameters relevant to each determination. The reduced relaxation rates r_∞, which are isotope-independent and are defined by $r_\infty = [\gamma^2 C_{K\infty}]^{-1}$ (Klein 1986), are also shown; they are the relevant quantities for comparison with theory. (Here γ is the nuclear gyromagnetic ratio of the impurity isotope used; convenient units are 10^{-15} T^2 s/K, as shown in the table).

Fig. 5 shows the reduced relaxation rates r_∞ for 5d impurities in Fe plotted across the series as a function of impurity atomic number. Also shown are some earlier data for the nuclear SLR rates, and the calculations of Akai (1988). We discuss briefly each case separately:

The isotope ^{182}**Ta** is readily produced from natural Ta and has a conveniently long half-life. The solubility of Ta in Fe is limited and, due to its high melting point, the alloys (final concentration 0·40 at.%) had to be prepared in an arc furnace. The nuclear parameters are reasonably well known, and the calculated interaction temperature T_{int} (= $B_{eff}\mu\mu_N/Ik$) is 23·9(5) mK, so that extremely low measuring temperatures are not required. The sensitivity obtained in the relaxation curves was adequate (see Bobek *et al.* 1993). The present results agree with an NMR/ON determination for ^{183}Ta*Fe* (Allsop *et al.* 1980) but not with the early spin-echo result on stable ^{181}Ta*Fe* (Masuda *et al.* 1974). This is probably due to an unresolved quadrupole splitting in the latter measurement.

The case of ^{187}**W** is much more difficult. Owing to its short half-life, modest anisotropy coefficient and low T_{int} of 10·8(4) mK, the statistical accuracy of the relaxation curves is relatively poor, and this is reflected in the large relative error of r_∞ (see Table 1). The present value agrees (within error limits) with the

relatively uncertain result of Ohya *et al.* (1987), obtained using a dilute WFe alloy (0·17 at.%; present work: 0·30 at.%) by NMR/ON at low applied fields. The spin-echo value of Masuda *et al.* (1974) is considerably higher, but their reported sample concentration of 3 at.% was well above the solubility limit and again, unresolved quadrupole splitting may have falsified the results.

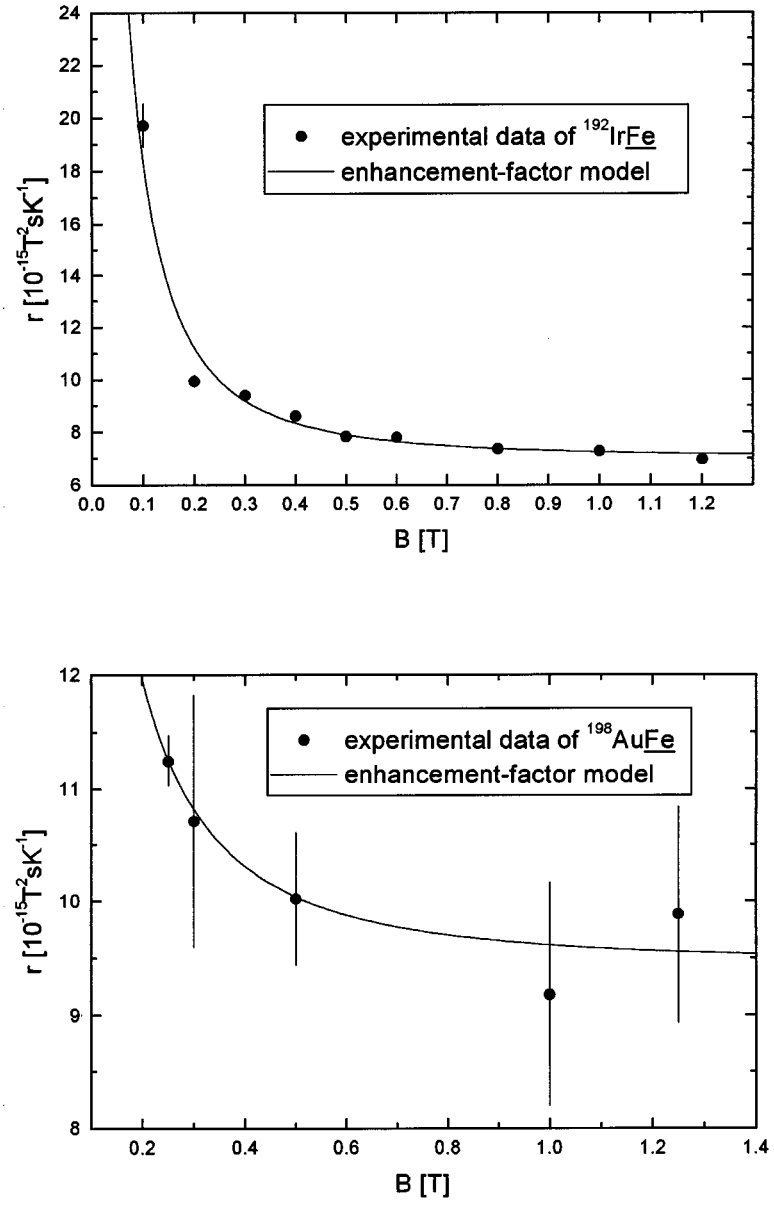

Fig. 4. Field dependence curves for the relaxation constants C_{K} of ^{192}IrFe and ^{198}AuFe, as determined by Bobek (1994). The fitted curves correspond to the enhancement-factor model (see Bobek *et al.* 1993).

Table 1. Summary of results

Isotope	Half-life[A] (days)	Nuclear spin I	Magnetic moment (μ_N)	T_l (mK)	T_h (mK)	Static hyperfine field B_{hf} (T)	$C_{K\infty}$(s K)	r_∞ (10^{-15} T^2 s K^{-1})
^{182}Ta	115·0	3	3·02(3)	28	33	−64·8(1)	0·46(1)	1·07(3)
^{187}W	0·99	$\frac{3}{2}$	0·621(15)	13	14	−71·4(1)	1·9(1)	1·3(1)
^{186}Re	3·78	1	1·739(3)	20	92	−75·97(13)	0·087(2)	1·65(4)
^{193}Os	1·24(1)	$\frac{3}{2}$	0·75(3)	17·2	20·5	−109·(5)	0·29(3)[B]	6·1(5)
^{192}Ir	74·2	4	1·924(10)	23	29	−137·3(7)	0·264(5)	7·12(14)
^{191}Pt	2·80(2)	$\frac{3}{2}$	0·500(2)	6·6	9·7	−128·0(3)	0·900(25)[C]	4·36(12)
^{198}Au	2·69	2	0·584(1)	14·6	16·4	−116·9(1)	0·53(4)	9·7(7)

[A] Values with error limits were redetermined in this work.
[B] Weighted averages of results from Funk (1996) and Beck (1997).
[C] The slightly different values (within quoted errors) from those given in Beck *et al.* (1996) are due to reevaluation including a second gamma-ray line.

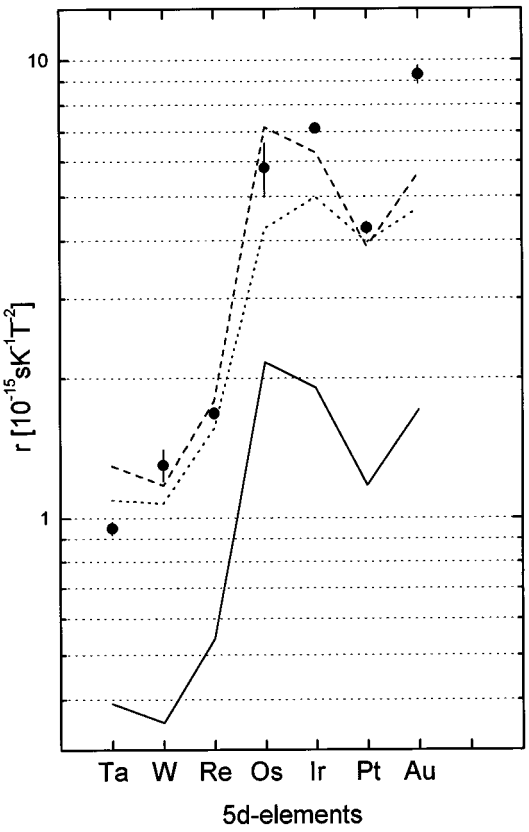

Fig. 5. Overall systematics (intrinsic SLR rates r_∞ versus impurity Z) for dilute 5d series impurities in Fe. The experimental points were obtained in this work or by Beck *et al.* (1996) and Bobek *et al.* (1993). The solid curve represents the calculations of Akai (1988), the dashed curve gives Akai's results scaled upwards by a factor of 3·3, and the dotted curve is the sum of Akai's values and an estimated virtual spin-wave contribution (cf. Masuda *et al.* 1974).

The ^{186}**Re** decay is also rather unfavourable, due to the small branching ratio and low anisotropy coefficient of the 137-keV gamma-ray transition. The interaction temperature is $T_{\rm int} = 48{\cdot}3(1)$ mK, so that experiments can be carried out at relatively high temperatures (see Table 1). The 0·4 at.% ReFe alloy used here nevertheless gave reasonable statistics for the relaxation curves within the measuring times permitted by the 3·8 day half-life, resulting in an overall accuracy of about 2·4%. Our datum for r_∞ is in agreement with the (less accurate) values previously reported [e.g. by Rüter *et al.* (1981); cf. the discussion in Bobek *et al.* (1993)].

Preparation of dilute samples of ^{193}**Os**Fe requires the use of isotopically enriched Os starting material, as mentioned above. Our samples had an Os concentration of 0·20 at.%, within the solubility limit. The branching ratios of the observable gamma rays (Fig. 1) are very small and their anisotropy coefficients only moderate; the value of $T_{\rm int}$ [19·9(1·1) mK] is also moderate and favours temperatures in the 15 mK range for the measurement. As can be seen in Fig. 2, the thermal behaviour of the sample holder was far from ideal in this case, due

to strong radioactive heating and an overly large thermal impedance. As a result, the relative error of our reported r_∞ for OsFe is the largest in the 5d series (8·2%). It is about the same as the reported relative error of the spin-echo value (Masuda *et al.* 1974; nom. 2 at.% OsFe alloy), which is again nearly 40% larger than our result for r_∞. For the reasons discussed above, we are confident that our SLR rate for OsFe is closer to the true value.

The case of ^{192}**Ir**Fe is again relatively favourable. This comparatively long-lived activity can be readily prepared using natural iridium (here 0·23 at.% in Fe) and has three major gamma-ray transitions with favourable branching ratios and anisotropy coefficients. The interaction temperature is $T_{\rm int} = 24{\cdot}2(2)$ mK, permitting sensitive measurements over a range of 10–40 mK. This allows the most accurate evaluation of r_∞ for the 5d series (1·9% relative error). Our value is in good agreement with previous TCON results (Kopp *et al.* 1981; van Rijswijk 1986) for ^{191m}IrFe and ^{192}IrFe, respectively, but is again lower than the early spin-echo result obtained on a nominal 1 at.% sample (Masuda *et al.* 1974), as well as a very early NMR/ON value (Eska *et al.* 1971).

For the measurement of ^{191}**Pt**Fe, isotopically enriched Pt starting material was required (see above). Our results for 0·07 at.% alloys were given in Beck *et al.* (1996); the values of $C_{\rm K\infty}$ and r_∞ quoted here have been corrected slightly for an improved fitting procedure and inclusion of data from a second gamma-ray transition, and exhibit a moderate relative error of about 2·5%. The isotope studied has reasonably favourable nuclear properties and a simple gamma-ray spectrum. Its 2·8 day half-life and $T_{\rm int} = 15{\cdot}6(3)$ mK allow TCON measurements of good accuracy in the 5–15 mK range. A previous NMR/ON result (Ohya *et al.* 1985) is in good agreement after extrapolation to the high-field limiting value r_∞.

Finally, ^{198}**Au**Fe is an excellent case for TCON owing to its simple gamma-ray spectrum, with nearly 100% branching ratio, high anisotropy coefficient, and easy production of the 2·7 day ^{198}Au activity (our alloy contained 0·28 at.% Au). The interaction temperature $T_{\rm int} = 12{\cdot}5(1)$ mK allows sensitive measurements in the range 5–18 mK. A number of results have been reported in the literature, but they exhibit considerable scatter. The spin-echo value for ^{197}AuFe (Masuda *et al.* 1974) is, as in other cases, systematically high, while early NMR/ON values [including the adiabatic-passage measurement at 0·2 T by Callaghan *et al.* (1975), after extrapolation to the high-field limiting value] are much lower. More recent NMR/ON results (Romanski 1991) and previous TCON experiments in our own laboratory (Bobek 1994) agree with the present value. Its relative error of 7·2% could be improved upon by further optimisation of the measurement conditions (cf. Fig. 3).

In Fig. 5, our reported values for r_∞ are compared with the *ab initio* calculations of Akai (1988) and with some selected experimental data. The calculations as published give SLR rates r_∞ which are a factor of 3 to 5 smaller than the experimental rates. The agreement can be made nearly perfect by simply scaling the calculation by a factor of 3·3 (dashed line in the figure), except for the first and last members of the series studied here (Ta, Au). There is of course no justification for such a procedure, but it does illustrate that the theory predicts well the *shape* of the systematics curve. Correcting the as-published *ab initio* rates by adding the estimated rates due to the Weger mechanism (excitation of

virtual spin waves), as calculated phenomenologically by Masuda *et al.* (1974), was shown in Bobek *et al.* (1993) to improve the agreement considerably (dotted line in Fig. 5), although the present more detailed comparison indicates that the agreement obtained in this way is not as good as that given by the simple scaling procedure. This is not surprising in view of the approximate nature of the estimate of the spin-wave contributions. The recent results of Seewald *et al.* (1997) support the possible importance of such a dynamic contribution to the SLR rate. It is clear that new calculations, taking the effect of lattice relaxation into account in at least a few exemplary cases to test its importance, and including dynamic effects, are needed to resolve this discrepancy.

Acknowledgments

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