Recent Developments in Rare-Earth Mössbauer Studies. I

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This review describes recent nuclear resonance fluorescence experiments with low-energy γ rays of rare-earth nuclides using the Mössbauer effect.[1] These studies allow one to determine in favorable cases the hyperfine interaction of nuclear states with a rare-earth ion in a solid. The observed magnetic dipole interaction is mainly produced by the magnetic field from the $4f$ electrons polarized by exchange interaction in a magnetically ordered solid. An aligned charge distribution of $4f$ electrons also produces an electric field gradient, which leads, together with field gradients from the crystal, to a quadrupole interaction with the quadrupole moments of the nuclear states. In a typical experiment one can determine the magnetic dipole and the electric quadrupole coupling constants of the nuclear ground state and an excited state with an accuracy of a few percent. If both nuclear states possess nuclear moments, their ratio can be deduced readily from the data. Magnetic moments of the ground states of rare-earth nuclei have been measured very precisely recently by the atomic beam NMR technique. In these cases accurate values of the excited state moments can be determined from Mössbauer data. Also, the effective magnetic field at the nucleus can be derived. In experiments with even–even nuclei which have $I = 0$ ground states and low-lying $I = 2^+$ rotational states, one has to determine the magnetic field separately in order to deduce the moment of the rotational state from a measurement of the coupling constant of the interaction of an odd mass isotope (with known magnetic moment) in the same material. For the determination of quadrupole moments from Mössbauer experiments, the situation is different. In appropriate cases one can determine the ratio of quadrupole moments precisely but not their absolute values, because one has to calculate the field gradients based on electron wave functions and crystal field effects.

The situation in the field will be illustrated by some resonance absorption experiments which we did recently on the following transitions: 8.42 keV in Tm^{169}, 80.0 keV in Er^{166}, and 21.7 keV in Eu^{151}. These transitions are populated by the β^- decays of $Er^{169}(T_{\frac{1}{2}} = 9.4d)$, $Ho^{166}(T_{\frac{1}{2}} = 27.2h)$, and Sm^{151} $(T_{\frac{1}{2}} = 90y)$, respectively. In order to observe directly the hyperfine pattern in an absorber, we tried to avoid any splitting of the emission line in the source material, which is mainly a problem of achieving small electric field gradients at the nuclei. The resonance absorbers could be cooled by cold He gas to any desired temperature between 5 and 300°K. The sources were moved by double loud speakers with feedback regulation. They could be cooled by mounting the loud-speaker drive in the cryostat. The absorption spectra were recorded by a multichannel analyzer operated in multiscaler mode.

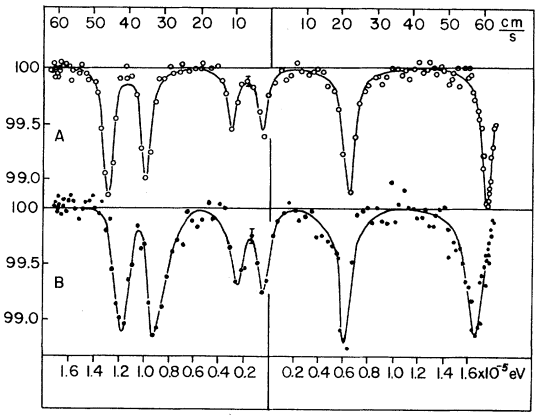

FIG. 1. Hyperfine splitting of 8.42-keV transition of Tm^{169} in a Tm-metal absorber at 5°K(a) and 25°K(b). The Er_2O_3 source heated to 800°K emitted a single line. The relative transmission in % is plotted as a function of the Doppler velocity v.

Recently, the hyperfine splitting of the 8.42-keV γ rays of Tm^{169} has been determined very accurately in Tm metal[2] and the Laves phase compound Fe_2Tm.[3] The experiments showed a well-resolved six-line absorption pattern for this $\frac{1}{2} - \frac{3}{2}$ magnetic dipole transition. The absorption pattern of Tm metal is shown at 5 and 25°K in Fig. 1. The source was Er_2O_3 heated to 800°K. At this temperature the alignment of the $4f$ electrons in the crystal field is small leading

[1] R. L. Mössbauer, Z. Physik **151**, 124 (1958).

[2] M. Kalvius, P. Kienle, H. Eicher, W. Wiedemann, and C. Schüler, Z. Physik **172**, 231 (1963).

[3] R. L. Cohen, Bull. Am. Phys. Soc. **8**, 43 (1963).

to a small quadrupole interaction and therefore to a single emission line. The observed splittings in Tm metal are produced by the fields from Tm^{3+} ions which are polarized by an exchange interaction at low temperature where Tm metal shows a simple ferrimagnetic antiphase structure.[4] At 5°K the Mössbauer spectrum is produced by a unique magnetic and electric field gradient at all nuclei. The magnetic coupling constants $gH \cdot \mu_K$ are 7.00×10^{-6} eV in the excited state and -9.04×10^{-6} eV for ground state. The quadrupole coupling $\frac{1}{4} eQV_{zz}$ of the 8.42 level is $+2.04 \times 10^{-6}$ eV. At about the same time Ritter[5] directly measured, with the atomic beam technique, the magnetic moment of the ground state of Tm^{169} [$\mu g = -(0.229 \pm 0.003)$ mm]. From these data a magnetic moment of $\mu_e = +(0.550 \pm 0.015)$ is derived for the 8.42-keV state. Based on these measurements a rather complete discussion of the magnetic properties of the $K = \frac{1}{2}$ rotational band of Tm^{169} in terms of the unified model is possible.[1,6]

The magnetic field at the Tm nuclei in Tm metal was found to be $(6.96 \pm 0.30) \times 10^6$ Oe at 5°K. We assume that due to the exchange interaction the lowest state of the $4f^{12}\,{}^3H_6$ configuration has $j_z = -j$ and that the contribution of the core polarization and conduction electron polarization to the magnetic field is small compared to the contribution of the 4*f* electrons. One can then account for the observed magnitude of H_{eff} in Tm metal by taking $\langle r^{-3}\rangle_{4f}$ effective, for magnetic dipole interaction, as about 75Å^{-3} which is about the value which is measured for the free ion.[5]

To derive a value for the quadrupole moment for the 8.42 keV state, we have to estimate the effective field gradient. If one only takes into account the contribution from the polarized 4*f* electrons and makes the same assumptions as in the calculation of H_{eff}, the value $(1 - R)Q = -1.13$ b is derived. R is the Sternheimer shielding factor which accounts for the shielding of the field gradient by the 4*f* electrons. In this estimate it is assumed that $\langle r^{-3}\rangle_{eff}$ for the electric field gradient is the same as $\langle r^{-3}\rangle_{eff}$ for the magnetic field, which is only a very crude approach. Furthermore, we neglected the direct contribution of the crystal field to the field gradient which follows from the small quadrupole splittings in the metal at higher temperatures. Another attempt was made to determine Q from the quadrupole splitting with ethyl sulfate. In this paramagnetic salt the field gradient is produced by the 4*f* electrons aligned by the crystal field and by the direct contribution of the crystal field modified by Sternheimer antishielding effects. Although the parameters describing the crystal field acting on the 4*f* electrons can be derived from optical spectra,[7] one even has difficulties estimating the field gradient of the 4*f* electrons because the eigenfunctions of the lowest crystal field term have to be known very accurately. Although we measured the temperature dependence of the field gradient,[8] it is difficult to estimate the temperature-independent direct contribution from the crystal field, because we could not make measurements with the decomposing salt to high enough temperatures. An estimate based on the presently accepted crystal field parameters gives $(1 - R)Q = -1.09$ b in close agreement with the result using the metal. Taking the shielding factor $R = 0.3$ from an early work of Sternheimer[9] with a proper value for $\langle 1/r3\rangle$, $Q(8.42\text{ keV}) = -1.5$ b. This leads to a positive intrinsic quadrupole moment (prolate shaped nucleus) of +7.5 b in close agreement with results from *E*-2 transition probabilities in the ground-state rotational band of Tm^{169} ($Q_0 = 7.50 \pm 0.15$ b).[10]

In the Tm metal we observed complex absorption spectra between 25 and 38°K, the origin of which is not understood.

In the following, the hyperfine splitting of the 80.0-keV γ rays of Er^{166} in Er metal is discussed to illustrate the problems involved in studying a $2^+ - 0^+$ transition in a rotational band of an even–even nucleus. The main interest is to determine the g factor of this rotational state accurately in order to make a comparison with recent theoretical calculations.[11] In order to do that, the effective magnetic field would have to be determined separately by measuring the magnetic coupling of Er^{167} or Er^{169}, the magnetic moments of which are now known from atomic beam measurements.[12,13] In the absence of precise data on H_{eff},[12] we may best calculate the effective fields taking the experimental value of H_{eff} in Tm metal as starting point.

For the experiment, an $HoAl_2$ source, which has a cubic structure, was used. The absorber was Er metal 500 mg/cm^2 thick. The magnetic structure of the

[4] C. W. Koehler, J. W. Cable, E. O. Wollan, and M. K. Wilkinson, Phys. Rev. **126**, 1672 (1962).
[5] G. J. Ritter, Phys. Rev. **128**, 2238 (1963).
[6] See F. Boehm, in Proceedings of the International Conference on Perturbed Angular Correlation, Upsala, 1963.
[7] E. Wong and I. Richman, J. Chem. Phys. **36**, 1889 (1962).
[8] S. Hüfner, M. Kalvius, P. Kienle, W. Wiedemann, and H. Eicher, Z. Physik **175**, 416 (1963).
[9] F. W. Sternheimer, Phys. Rev. **80**, 104 (1950).
[10] M. C. Oleson and B. Elbek, Nucl. Phys. **15**, 139 (1960).
[11] S. G. Nilsson and O. Prior, Kgl. Danske Videnskab. Selskab Mat. Fys. Medd. **32**, No. 16 (1961).
[12] See B. Bleany, J. Appl. Phys. **34**, 1024 (1963).
[13] W. M. Doyle and R. Marrus, Phys. Rev. **131**, 1586 (1963).

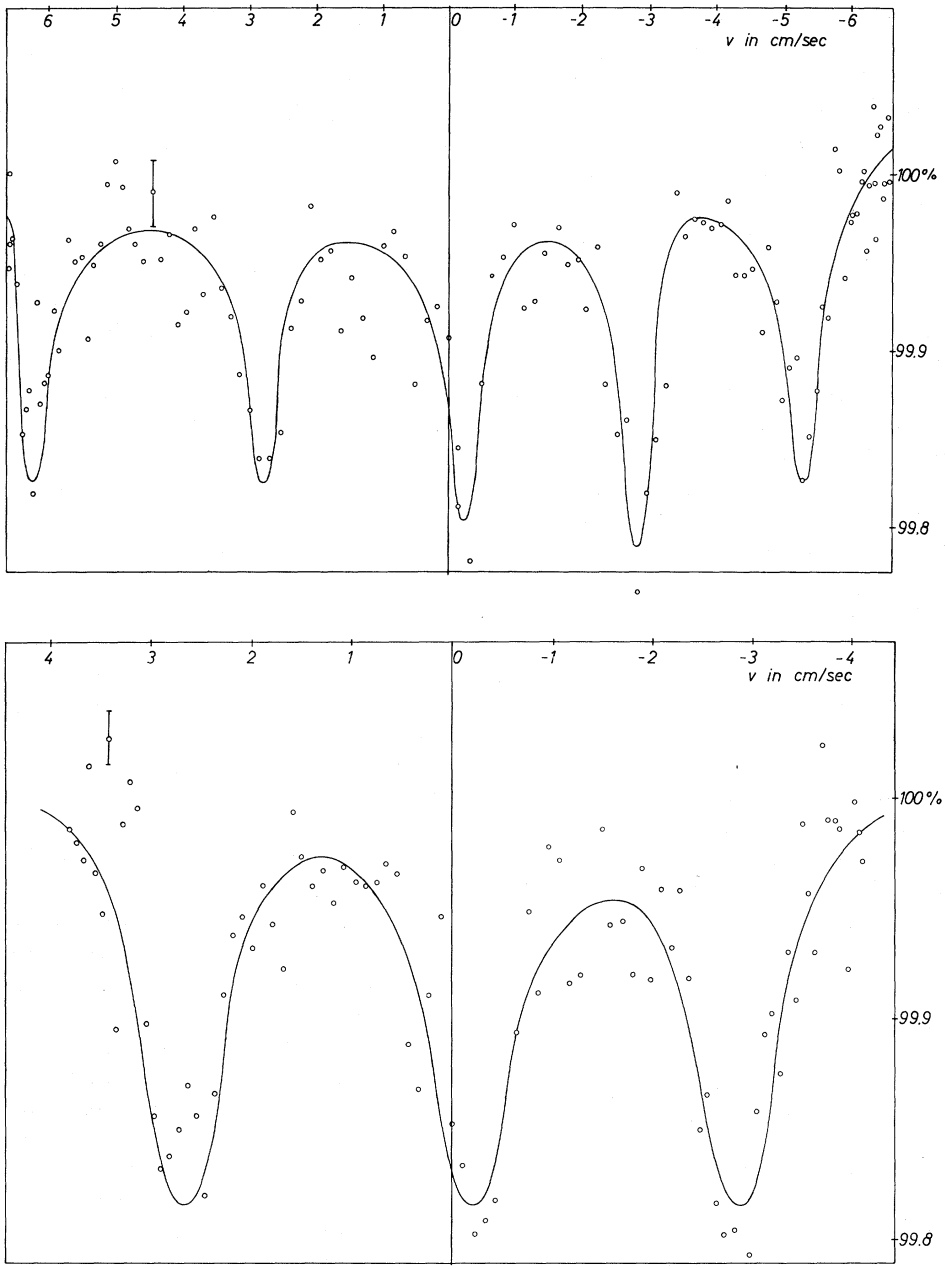

FIG. 2. Hyperfine splitting of the 80.0-keV transition of Er^{166} in an Er-metal absorber at 10°K(a) and 14°K(b). The spectrum (b) contains only the three inner lines. The source was $HoAl_2$ which emits an unsplit line (1 cm/sec Doppler velocity corresponds to 3.67×10^{-6} eV).

metal is known from neutron diffraction work.[14] Below 20°K, erbium has a ferromagnetic structure with moments ordered in a spiral along the c axis of the hexagonal crystal. Figure 2 shows the absorption patterns at 10 and 14°K. There are 5 absorption lines of about equal intensity revealing a unique magnetic field at all nuclei. The resonance absorption is very small, presumably because of the low Debye temperatures in the source and absorber. The temperature of the sources were slightly higher than the absorbers. They emitted single lines which were about 2.5 times the natural width. In order to achieve this the alloys had to be annealed after the neutron irradiation for several hours at 800°C. The following coupling constants were derived:

$$g_R \cdot H_{eff}\mu_k = (7.60 \pm 0.10) \times 10^{-6}\,\text{eV}\,,$$

$$\tfrac{1}{4}\, e \cdot Q \cdot V_{zz} = +(0.93 \pm 0.10) \times 10^{-6}\,\text{eV}\,.$$

In order to derive g_R and Q we had to estimate the

[14] J. W. Cable, E. O. Wollan, W. C. Koehler, and M. K. Wilkinson, J. Appl. Phys. **32**, 495 (1962).

magnetic field and the electric field gradient as in Tm metal.[2] It was assumed that the lowest state of the ion is $j_z = -j$. The contribution of core and conduction-electron polarization to H_{eff} was neglected; also the direct contribution of the crystal field to the electric field gradient. For $\langle 1/r^3 \rangle$ we assumed 72Å^{-3} for both interactions.[12] This value is deduced for a free ion from the atomic beam measurements. Thus, we obtain the following results:

$$g_R(2^+) = 0.31 \pm 0.03$$

and

$$(1 - R)Q(2^+) = -(1.4 \pm 0.2)\ \text{b}\ .$$

The quoted errors are estimates of the uncertainties which arise in calculating the fields. The value for $g_R = 0.31 \pm 0.03$ b is in good agreement with that derived by Gerdau *et al.*[15] from disturbed γ–γ angular correlation data. Nilsson and Prior[11] calculated $g_R = 0.30$ for the ground-state rotational band of Er^{166}. Cohen[16] reported the same values for g_R and $(1 - R)Q$ from Mössbauer experiments with an $ErFe_2$ absorber. Again assuming the Sternheimer factor $R = 0.3$ one finally deduces $Q(2^+) = -(2.5 \pm 0.3)$ b which indicates an intrinsic quadrupole moment $Q_0 = +(8.2 \pm 0.1)$ b again in agreement with $Q_0 = 7.50$ b derived from the E-2 transition probability.[17]

Finally, we will discuss the hyperfine coupling of Eu^{151} in europium compounds. They represent special cases because Eu^{3+} has a $4f^6\ {}^7F_0$ and Eu^{2+} a $4f^7\ {}^8S_{\frac{7}{2}}$ electron configuration. Measurements of the coupling constants in europium iron garnet (EuIG) and Eu metal as a function of the temperature will be reported. The Mössbauer effect of the 21.7-keV γ rays of Eu^{151} was first investigated by Shirley *et al.*[18] Quite recently, Barrett and Shirley also found the hyperfine splitting of this transition in Eu metal.[19] The spin of the 21.7-keV level is $\frac{7}{2}$ and the ground state has $I = \frac{5}{2}$.[19–21] The magnetic moment of the ground state is known from atomic beam results,[21] so that in principle accurate values for the excited state moment and H_{eff} may be derived. Barrett's measurements,[19] Ofer's experiments,[22] and our results showed that μ_a/μ_g is about 0.5, which means that many of the 18 expected lines coincide in the absence of a large quadrupole interaction so that eventually a simple 6-line hyperfine pattern is observed. This is discussed in detail by Barrett and Shirley.[19]

The situation is illustrated in Fig. 3 which shows

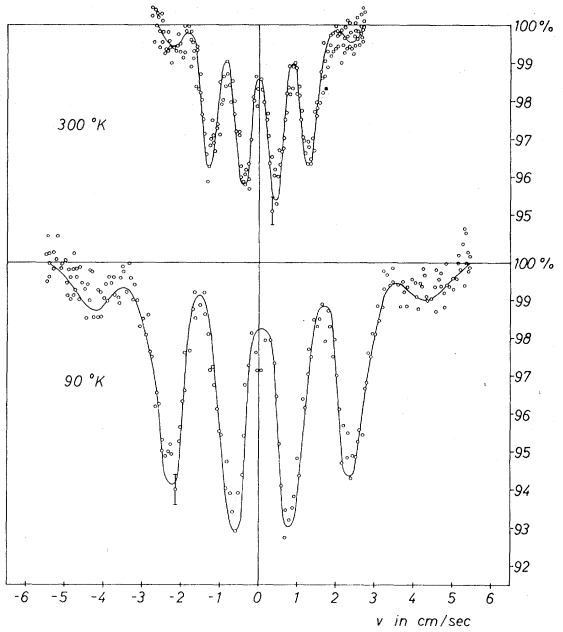

FIG. 3. Hyperfine splitting of the 21.7-keV γ line of Eu^{151} in europium iron garnet at 300 and 90°K. The source was SmF_3, which emits a single line. (1 cm/sec Doppler velocity corresponds to 7.24×10^{-7} eV.)

hyperfine spectra of the 21.7-keV γ ray in EuIG. The spectra in EuIG were taken at temperatures between 14 and 600°K in order to follow the variation of the hyperfine spectra in the temperature range of magnetic ordering. The measured points were fitted with Lorentz curves, determined by the magnetic splitting of the ground state, the ratio of the magnetic moments, the quadrupole-coupling constants of both states, and an isomeric shift. We assumed a single value for H_{eff} but determined the effective linewidth for each spectrum. For the ratio of the g factors of the excited state to that of the ground state, the average value from 15 measurements at different temperatures is $g_e/g_g = +0.530 \pm 0.001$. This is in excellent agreement with the result of Barrett and Shirley[19]:

$$g_e/g_g = +0.528 \pm 0.005\ .$$

Because the magnetic moment of the ground state is known (+3.419 nm),[21] we could derive the effective magnetic field from the observed magnetic ground-state splitting. In Fig. 4 this field is plotted as a func-

[15] E. Gerdau, W. Krull, L. Mayer, J. Braunsfurth, J. Heisenberg, P. Steiner, and E. Bodenstedt, Z. Physik **174**, 389 (1963).
[16] R. L. Cohen, Rev. Mod. Phys. **36**, 393 (1964) (this issue).
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[19] P. H. Barrett and D. A. Shirley, Phys. Rev. **131**,123 (1963).
[20] See E. Steichlee and P. Kienle, Z. Physik **175**, 407 (1963).
[21] F. M. Pichanik, P. G. H. Sandars, and G. K. Woodgate, Proc. Roy. Soc. (London) **A257**, 277 (1960).
[22] S. Ofer and I. Nowik, Phys. Rev. **132**, 241 (1963).

tion of the temperature. The extrapolation to 0°K yields $H_{eff}(0°K) = (600 \pm 6)$ kOe. The effective fields follow within the limit of errors the saturation magnetization measured recently by Geller *et al.*[23] The average of the widths of all lines decreased from 24×10^{-8} eV at 14°K to 11×10^{-8} eV at 510°K, whereas with Eu metal of the same effective thickness as absorber at low temperatures $\Gamma_{exp} = 10.4 \times 10^{-8}$ eV. The natural linewidth is $\Gamma_{nat} = (4.3 \pm 0.2) \times 10^{-8}$ eV.[24] These results indicate that there is not a unique H_{eff} activity at all nuclides in the

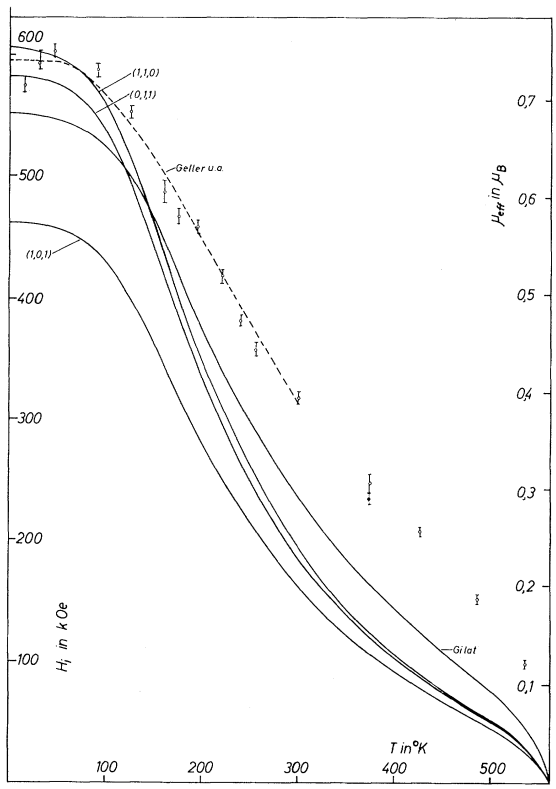

FIG. 4. Internal magnetic field H_i at Eu^{151} nuclei in europium iron garnet as a function of the temperature. The dotted line is the saturation magnetization from Geller *et al.* The full curves are calculated H_i by Gilat and Eicher. The calculation by Gilat does not take into account the crystal field whereas Eicher's calculation marked by (1,1,0; 0,1,1 and 1,0,1) does.

europium iron garnet. The effective magnetic fields in EuIG at the Eu nuclei are largely due to the fact that the exchange field and the crystal field mix the low-lying states of ^7F. Gilat and Nowik[25] calculated this taking into account only the exchange polarization. This gives rise to a unique field at all Eu nuclei. The temperature dependence of H_{eff} is also shown in Fig. 4. We scaled down the calculated curve given by Gilat *et al.*[25] using the values for H_{exch} given by Geller *et al.*[23] and $\langle r^{-3}\rangle_{eff} = 49\text{Å}^{-3}$ given by Bleany.[12] Eicher[26] calculated H_{eff} and its temperature dependence by including also the interaction of the crystal field. Assuming that the magnetization is along the (111) direction, the Eu ions at two different sites will produce different H_{eff} as was first found by Cohen in TmIG[27] where the two fields are very different. This is not the case in EuIG as Eicher's[26] curves marked (1,1,0) and (0,1,1) show. The temperature dependence of the line broadening observed in the experiment agrees well with Eicher's calculation. The agreement of the absolute value of H_{eff} at low temperatures is also excellent although a contribution to the field from spin polarization is neglected. One could argue that the agreement might be fortuitous, but this can be checked only if one could make an estimate of the spin polarization in this complicated case. Eicher's calculated values for H_{eff} are rather poor at higher temperatures because he included only the mixing of the first excited state in the calculations.

We fitted our transmission spectra including a quadrupole interaction in both the ground state and excited state. Taking the average of the results for these interactions below 90°K we find $eQ_gV_{zz}/h = -(17 \pm 15)$ Mc/sec and $eQ_eV_{zz}/h = -(16 \pm 16)$ Mc/sec. Although we did not find a quadrupole interaction within the limits of errors, the result indicates that the effective field gradients are rather small. In the present state it is very difficult to estimate the field gradient because one has to take into account different contributions which may partially cancel. We made a further attempt to measure the quadrupole coupling in Eu ethyl sulfate for which Judd *et al.*[28] reported a large interaction from a nuclear alignment measurement at very low temperatures. Figure 5 shows the spectrum of the Eu ethyl sulfate at 34°K. There is a single line having a linewidth of 13×10^{-8} eV, again indicating a rather small quadrupole interaction at 34°K.

We measured Mössbauer spectra in Eu metal between 12°K and room temperature. The results at 12°K are in good agreement with Barrett's and Shirley's[19] data at 4°K. The average value for $g_e/g_g = (0.534 \pm 0.002)$ derived from the spectra at

[23] S. Geller, H. J. Williams, R. C. Sherwood, J. P. Remeika, and G. P. Espinosa, Phys. Rev. **131**, 1080 (1963).
[24] D. J. Horen, H. H. Bolotin, and W. H. Kelly, Bull. Am. Phys. Soc. **8**, 127 (1963); A. C. Li, O. Kistner, and S. Monaro, *ibid.* **8**, 332 (1963).
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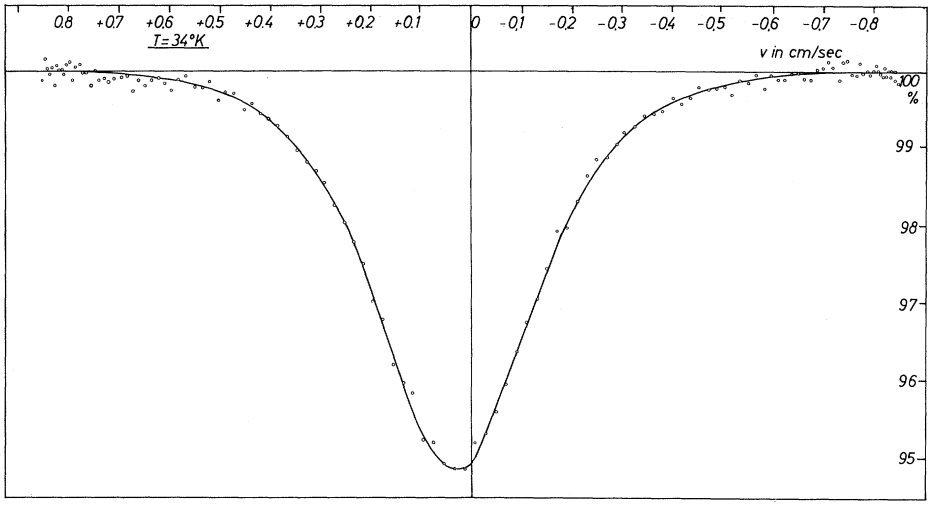

FIG. 5. Absorption line of the 21.7-keV line of Eu^{151} in Eu ethyl sulfate at 34°K.

different temperatures. Again we find good agreement with the previously cited data. Figure 6 shows the effective magnetic field at various kOe temperatures. The extrapolation to 0°K yields H_{eff}(°K) = (264 ± 2). In contrast to the spectra in EuIG, at all temperatures, the linewidths are the same, namely, 10.4×10^{-8} eV. This indicates that there is a unique magnetic field acting on all nuclei despite the complicated and not well understood magnetic structure of Eu metal.[29] The magnitude of $H_{eff}(0°K)$ = (164 ± 2) kOe in the metal may be compared with the field of Eu^{2+} in CaF which is −343 kOe.[30]

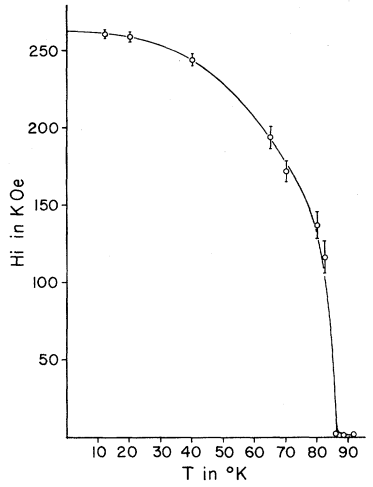

FIG. 6. Internal magnetic field H_i in Eu metal as a function of the temperature.

If one assumes the core polarization is the same in both cases, one would say that one has either +79 or +607 kOe additional contribution in the metal depending on the sign of the field, which is not yet known. It is probably more reasonable to assume that +80 kOe is produced by the polarized conduction electrons.

The temperature dependence of the internal field is what one expects for a ferromagnet with $S = \frac{7}{2}$. The fact that the saturation magnetization as function of temperature[31] is very different is not too surprising in the light of the complicated magnetic structure which is indicated by the neutron diffraction data.[29]

Finally, we would like to summarize some results on the isomeric shifts of transitions in the rare earths, although reliable results exist only for Eu^{151}. On the other hand, one might expect to receive valuable information from these measurements on the charge distribution of nuclear states within a rotational band, when the *s* electron density difference can be calculated.

In Eu^{151} Barrett and Shirley[19] reported a large isomeric shift of −0.81 cm/sec between Eu^{3+} and Eu metal. We found the same result for Eu metal and found a still larger shift in the divalent $Eu^{2+}(OH)_2$ of −(1.49 ± 0.03) cm/sec. The spectrum of $Eu(OH)_2$ is shown in Fig. 7. In Table I we have compiled all our present results on the isomeric shifts of Eu compounds in respect to EuF_3.

On the basis of the electron-configuration assignments given in Table I, one sees qualitatively that the *s* electron density should be higher in Eu metal compared with the *divalent* Eu compounds. The results show, furthermore, that the isomeric shift in all trivalent compounds is about the same and also that

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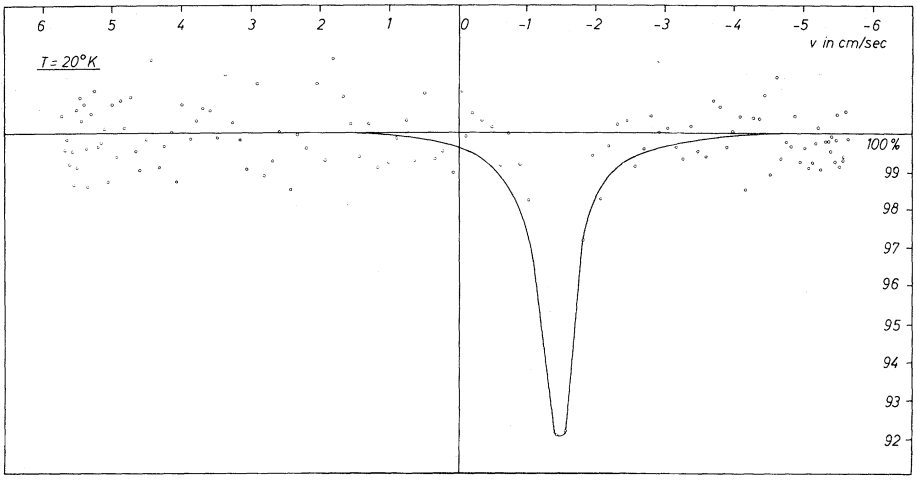

FIG. 7. Absorption line of 21.7-keV line of Eu^{151} in divalent $Eu(OH)_2$. There is an isomeric shift of -1.49 cm/sec in respect to the emission line in Eu^{3+}.

the s electron density is *smaller* compared with Eu metal and Eu^{2+}. This is also expected when one takes into account the pronounced shielding of the $5s$ electrons by adding electrons in the inner $4f$ shell. To calibrate the isomeric shift, it will probably be more accurate to use the difference of the s electron densities in the $4f^6$ and $4f^7$ configuration based on Hartree–Foch wave functions for Eu^{3+} and Eu^{2+} instead of estimating the conduction electron density in Eu metal. The experiments tell us also that it would be preferable to look for isomeric shifts in other rare-earth nuclides by comparing the lines in R^{2+} and R^{3+} compounds, in order to achieve reasonable estimates concerning the charge distributions in rotational states.

TABLE I. Isomeric shifts in Eu compounds.

Absorber	Δ, cm/sec	Δ, 10^{-8} eV	Electron configuration
Eu_2O_3	0.0	0.0	$4f^6$
$Eu_2(C_2H_5SO_4)_3 \cdot 9H_2O$	(+0.025 ±0.005)	(+2.0 ±0.4)	$4f^6$
EuIG	(+0.050 ±0.005)	(+4.0 ±0.4)	$4f^6$
Eu metal	(−0.78 ±0.01)	(−56.0 ±0.8)	$4f^7 + 6s^2$
$Eu(OH)_2$	(−1.49 ±0.03)	(−106.5 ±2.1)	$4f^7$

For Eu^{151} the given results show that the charge is *more* extended in the excited compared with the ground state. This may indicate either a $g_{7/2}$ and $d_{5/2}$ assignment for both states or a larger deformation for the excited state.

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Recent Developments in Rare-Earth Mössbauer Studies. II*

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1. INTRODUCTION

The recoil-free emission and absorption of nuclear γ rays (Mössbauer effect) has already been used for the measurement of hyperfine interactions of nuclear states. In some cases magnetic and quadrupole moments of the nuclear states involved have been deduced from these measurements. In most experiments of this kind carried out so far, ferrimagnetic (or ferromagnetic) materials containing the nuclei to be investigated were used as sources or absorbers and the recoil-free absorption spectra were measured. These spectra showed splittings produced by the magnetic Zeeman effect of the nuclear states in an effective magnetic field, H_{eff}, produced by the oriented hyperfine interaction within a magnetic do-

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