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ON THE OVERHAUSER EFFECT IN NONMETALS. II.

G. R. KHUTSISHVILI

Tbilisi State University

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An expression has been derived for the degree of stationary polarization of nuclei in the case of partial saturation of one of the components of hyperfine structure of paramagnetic resonance in a paramagnetic salt or in a semiconductor with a donor or an acceptor impurity. The degree of polarization for the same partial saturation of all the paramagnetic resonance components has also been computed. In the case $I = \frac{1}{2}$ the role of noncontact terms in nuclear relaxation has been taken into account. Methods of measuring polarization are considered.

1. During the last several years several articles have appeared in which different variants of the Overhauser method for polarizing nuclei in paramagnetic salts and in semiconductors have been proposed.[1-6] The authors of several of these articles have succeeded in producing nuclear polarization. In most of these cases the articles deal with the problem of obtaining a nonstationary dynamic nuclear polarization.

In an earlier article[7] the author gave an expression for the degree of stationary nuclear polarization obtained in the case of complete saturation of one of the components of the hyperfine structure of paramagnetic resonance. In the case examined in that article the relaxation of nuclear spin is due only to the hyperfine (contact) interaction with the spin of the electron shell.

In this article we generalize those results. We examine the case of partial saturation of one or more components of hyperfine structure of paramagnetic resonance, and take into account the role of other mechanisms of nuclear relaxation.

We note that unlike Jeffries[4] we consider the saturation of an allowed transition in the paramagnetic resonance spectrum.

2. We consider a system consisting of an electron shell with an effective spin S equal to $\frac{1}{2}$ and of a nucleus with spin I placed into an external magnetic field H (this includes the cases of a trivalent acceptor or a pentavalent donor impurity in silicon or in germanium, and also the cases of many paramagnetic salts). The Hamiltonian of the system has the form* (we neglect quadrupole effects, and also the direct effect of an external field on the nuclear spin):

$$V = \beta\{g_{\parallel}H_zS_z + g_{\perp}(H_xS_x + H_yS_y)\} + \{AS_zI_z + B(S_xI_x + S_yI_y)\} \tag{1}$$

(cf. Bleaney's paper;[8] we follow his notation and consider the case of axial symmetry).

We restrict ourselves to the consideration of the case of a strong field: $\beta H \gg A$, $\beta H \gg B$. In this case the components of the electron and the nuclear spins along the direction of the external field (M and m respectively) are good quantum numbers. For the energy levels we have[8] (up to terms of order A and B):

$$E_{Mm} = Mg\beta H + KMm, \tag{2}$$

$$g = (g_{\parallel}^2\cos^2\vartheta + g_{\perp}^2\sin^2\vartheta)^{1/2}, \tag{3}$$

$$Kg = [(Ag_{\parallel})^2\cos^2\vartheta + (Bg_{\perp})^2\sin^2\vartheta]^{1/2}. \tag{4}$$

Alternating fields of suitable frequencies will give rise to resonance transitions between the levels (2). In particular, the transitions $\Delta M = \pm 1$, $\Delta m = 0$ give rise to paramagnetic resonance, while the transitions $\Delta M = 0$, $\Delta m = \pm 1$ give rise to nuclear magnetic resonance. The paramagnetic resonance spectrum has the form $\beta gH + Km$, i.e., we obtain $2I + 1$ equally spaced (up to terms of order A and B) hyperfine-structure components.

We neglect the spin-spin interaction in calculating the Boltzmann distribution among the energy levels. In this approximation we obtain $2I + 1$ pairs of levels coincident in energy with the energy difference between the components of each pair equal to $g\beta H$.

We have four types of relaxation transitions:

I) $\Delta M = \pm 1$, $\Delta m = 0$ — relaxation of electronic spin;

II) $\Delta M = -\Delta m = \pm 1$ — relaxation due to the hyperfine (contact) interaction;

III) $\Delta M = \Delta m = \pm 1$;

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*In the case of a pentavalent donor or a trivalent acceptor impurity in silicon or in germanium $g_{\parallel} = g_{\perp} = g \approx 2$, $K = A = B$.

IV) $\Delta M = 0$, $\Delta m = \pm 1$.*

Relaxation processes of type III and IV are not important compared with relaxation of type II in the case when the wave function of the electron (or more accurately of the electron shell with $S = \frac{1}{2}$) taken at the position of the nucleus is large, in other words if the s-part of the wave function of the electron is not small.[9] In the case of a pentavalent donor or a trivalent acceptor impurity in silicon or in germanium, this condition is satisfied. One would expect this condition to be fulfilled also in the case of many paramagnetic salts (indeed, according to Abragam and Pryce[10] the large value for the hyperfine structure of paramagnetic resonance indicates that the hyperfine interaction in a paramagnetic salt is due to the admixture of an excited state into the paramagnetic-ion ground state with an unpaired s-electron).

3. To be specific, we shall first consider the case $I = 3/2$. We then have four pairs of levels (Fig. 1).

We denote by $W(a)$ the probability per unit time for the transition $a \longleftrightarrow a'$ brought about by an alternating field at right angles to the main field of frequency corresponding to this transition. It is well known that[11]

$$W(a) = \frac{\pi}{2}(\gamma_e H_1)^2 \varphi_a(\omega), \tag{5}$$

where γ_e is the gyromagnetic ratio for the spin of the electronic shell, $2H_1$ is the amplitude of the alternating field, ω is its (circular) frequency, while $\varphi_a(\omega)$ is the function which gives the line shape for the transition $a \longleftrightarrow a'$ (with $\int_0^\infty \varphi_a(\omega)\,d\omega = 1$).

We shall introduce the probabilities $W(b)$, $W(c)$ and $W(d)$ in an analogous manner. The frequencies of the corresponding four alternating fields differ from each other (they are equal to $(\beta g H + Km)/\hbar$, where $m = +3/2, +1/2, -1/2, -1/2, -3/2$).

We shall assume that only relaxation processes of types I and II occur.

We denote by $W(aa')$ the probability (per unit time) for the transition $a \longrightarrow a'$ brought about by interaction with the lattice. In a similar manner we introduce $W(a'a)$, $W(bb')$ etc. We have

$$\begin{gathered} W(aa') = W(bb') = W(cc') = W(dd') = We^{-\delta}, \\ W(a'a) = W(b'b) = W(c'c) = W(d'd) = We^{\delta}, \end{gathered} \tag{6}$$

where W is some function of the temperature,

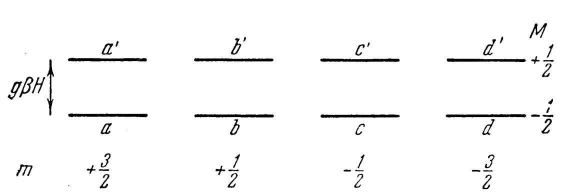

FIG. 1

while δ is given by the formula

$$2\delta = g\beta H / kT. \tag{7}$$

We further denote by $N(a), \ldots$ the populations of the corresponding levels and by $N^0(a), \ldots$ the equilibrium populations. We use N to denote the total number of systems under consideration. We then have

$$\begin{gathered} N(a) + N(a') + N(b) + N(b') + N(c) \\ + N(c') + N(d) + N(d') = N; \end{gathered} \tag{8}$$

$$\begin{gathered} N^0(a) - N^0(a') = N^0(b) - N^0(b') = N^0(c) - N^0(c') \\ = N^0(d) - N^0(d') = \frac{N}{4}\tanh\delta. \end{gathered} \tag{9}$$

In the stationary case we obtain

$$\begin{aligned} N(a)/N(a') &= (W(a) + We^{\delta})/(W(a) + We^{-\delta}), \\ N(b)/N(b') &= (W(b) + We^{\delta})/(W(b) + We^{-\delta}), \\ N(c)/N(c') &= (W(c) + We^{\delta})/(W(c) + We^{-\delta}), \\ N(d)/N(d') &= (W(d) + We^{\delta})/(W(d) + We^{-\delta}), \end{aligned} \tag{10}$$

$$N(a)/N(b') = N(b)/N(c') = N(c)/N(d') = e^{2\delta}. \tag{11}$$

Formula (11) means that the hyperfine interaction establishes equilibrium between the levels a and b′, b and c′, c and d′.

In particular, let us assume that partial saturation of only the resonance $a \longleftrightarrow a'$ takes place. Then $W(b) = W(c) = W(d) = 0$, and the solution of (8), (10), and (11) yields

$$\begin{gathered} N(a) = N(b) = N(c) = N(d) \\ = N[W(a) + We^{\delta}]/[(5 + 3e^{-2\delta})W(a) + 4W(e^{\delta} + e^{-\delta})], \\ N(b') = N(c') = N(d') \\ = Ne^{-2\delta}[W(a) + We^{\delta}]/[(5 + 3e^{-2\delta})W(a) + 4W(e^{\delta} + e^{-\delta})], \\ N(a') = N[W(a) + We^{-\delta}]/[(5 + 3e^{-2\delta})W(a) \\ + 4W(e^{\delta} + e^{-\delta})]. \end{gathered} \tag{12}$$

We introduce the saturation parameter $s(a)$ for the resonance $a \longleftrightarrow a'$ by means of the following formula:

$$N(a) - N(a') = [1 - s(a)][N^0(a) - N^0(a')]. \tag{13}$$

We obtain

$$\begin{gathered} s(a) = (5 + 3e^{-2\delta})W(a)/[(5 + 3e^{-2\delta})W(a) \\ + 4W(e^{\delta} + e^{-\delta})]. \end{gathered} \tag{14}$$

For the degree of nuclear polarization we obtain

$$f = s(a)(1 - e^{-2\delta})/(5 + 3e^{-2\delta}). \tag{15}$$

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*A quadrupole nuclear relaxation $\Delta M = 0$, $\Delta m = \pm 2$, is also possible, but we neglect it. This relaxation will be weak, since it is not associated with the interaction between the nuclear and the electronic spins.

In a completely analogous manner we can discuss the case of partial saturation of the resonance $b \longleftrightarrow b'$. In this case we introduce the saturation parameter $s(b)$ for this resonance by means of

$$N(b) - N(b') = [1 - s(b)][N^0(b) - N^0(b')]$$

and obtain after a simple calculation

$$s(b) = (5 + e^{2\delta} + 2e^{-2\delta}) W(b) / [(5 + e^{2\delta} + 2e^{-2\delta}) W(b) + 4W(e^{\delta} + e^{-\delta})], \tag{16}$$

$$f = s(b)(1 + 3e^{2\delta} - 4e^{-2\delta}) / 3(5 + e^{2\delta} + 2e^{-2\delta}). \tag{17}$$

4. The results obtained above can be easily generalized to include arbitrary I. If partial saturation is produced for the transition between two states with nuclear spin components equal to m, the saturation parameter for this resonance and the degree of nuclear polarization become

$$\begin{gathered} s(m) = \frac{[2(I+1) + (I-m)e^{2\delta} + (I+m)e^{-2\delta}]\, W(m)}{[2(I+1) + (I-m)e^{2\delta} + (I+m)e^{-2\delta}]\, W(m) + (2I+1)\, W(e^{\delta} + e^{-\delta})}, \\ f(m) = s(m)\, \frac{2m + (I-m)(I+1+m)e^{2\delta} - (I+m)(I+1-m)e^{-2\delta}}{2I\,[2(I+1) + (I-m)e^{2\delta} + (I+m)e^{-2\delta}]}, \end{gathered} \tag{18}$$

where $W(m)$ is the probability (per unit time) of the transition between two states with nuclear spin components equal to m, produced by an alternating field of the corresponding frequency.

In particular, if $\delta \ll 1$, we obtain

$$s(m) = \frac{W(m)}{W(m) + W}, \quad f(m) = \frac{I(I+1) - m^2}{I(2I+1)}\, \delta s(m), \tag{18a}$$

while if $\delta \gg 1$:

$$s(m) = \frac{W(m)}{W(m) + (2I+1)\, We^{-\delta}}, \quad f(m) = \frac{I+1+m}{2I}\, s(m) \tag{18b}$$

when $m \neq I$, and

$$s(I) = \frac{2(I+1)\, W(I)}{2(I+1)\, W(I) + (2I+1)\, We^{\delta}}, \quad f(I) = \frac{s(I)}{2I+1} \tag{18c}$$

when $m = I$.

From the results obtained above it follows, in particular, that for the complete saturation of the paramagnetic resonance that corresponds to the transition between levels with components of nuclear spin equal to I, it is necessary that the condition $W(I) \gg We^{\delta}$ be satisfied. In all the other cases ($m \neq I$) it is necessary only that the weaker condition $W(m) \gg We^{-\delta}$ be satisfied. It is easy to understand the physical significance of this difference. To be specific, we consider the case $I = 3/2$ (Fig. 1). When the transition $b \longleftrightarrow b'$ is saturated the alternating field gives rise to more transitions of type $b \rightarrow b'$ than of type $b' \rightarrow b$. But owing to the increase in the population of the b' level, the hyperfine interaction gives rise to transitions of type $b' \rightarrow a$. Thus the hyperfine interaction transfers the systems from states b and b′ into states a and a′, facilitating the saturation of the resonance $b \longleftrightarrow b'$. We obtain the same result when the resonances $c \longleftrightarrow c'$ and $d \longleftrightarrow d'$ are saturated. But in the case of saturation of the $a \longleftrightarrow a'$ resonance this effect will not occur, since the level a′ is not connected to other levels by the hyperfine interaction.

5. Let us now consider, for arbitrary I, the case of the same partial saturation of all the 2I + 1 components of hyperfine structure of paramagnetic resonance. We again assume that only relaxation processes of types I and II take place. We denote by $N(m)$ and $N(m')$ the populations of the states with nuclear spin components equal to m and spin components of the shell equal respectively to $-1/2$ and $+1/2$. In the stationary case we have

$$N(m) / N(m') = (W(m) + We^{\delta}) / (W(m) + We^{-\delta}).$$

From this we obtain

$$\begin{aligned} N(m) - N(m') &= \frac{N(m)}{W(m) + We^{\delta}}\, W(e^{\delta} - e^{-\delta}) \\ &= \frac{N(m')}{W(m) + We^{-\delta}}\, W(e^{\delta} - e^{-\delta}), \end{aligned} \tag{19}$$

or, since all the resonances are saturated to the same degree,

$$N(m) / (W(m) + We^{\delta}) = N(m') / (W(m) + We^{-\delta}) = \text{const},$$

where the constant does not depend on m.

The hyperfine interaction establishes equilibrium between the levels m + 1 and m′. Thus

$$\begin{gathered} N(m+1) / N(m') \\ = (W(m+1) + We^{\delta}) / (W(m) + We^{-\delta}) = e^{2\delta}. \end{gathered}$$

Therefore, for all the resonances to be saturated to the same degree it is necessary to have

$$W(m) = qe^{2m\delta}, \tag{20}$$

where q does not depend on m.

We introduce the saturation parameter s which, according to the assumed conditions, is the same for all the resonances. We then have

$$N(m) - N(m') = (1-s)\,[N^0(m) - N^0(m')] = (1-s)\,\frac{N}{2I+1}\tanh\delta. \tag{21}$$

Thus, in accordance with (19) we obtain

$$\frac{N(m)}{W(m) + We^{\delta}} = \frac{N(m')}{W(m) + We^{-\delta}} = \frac{N}{2I+1}\,\frac{1-s}{W(e^{\delta} + e^{-\delta})} \tag{22}$$

From this we obtain

$$N(m) + N(m') = (1-s)\,\frac{N}{2I+1}\left[1 + \frac{2W(m)}{W(e^{\delta} + e^{-\delta})}\right]$$

By making use of the normalization condition

$$\Sigma\,[N(m) + N(m')] = N,$$

we can easily establish the relation between q and s

$$2\,\frac{1-s}{s}\,q = (2I+1)\,W(e^{\delta} + e^{-\delta})\frac{\sinh\delta}{\sinh(2I+1)\,\delta}, \tag{23}$$

and thus obtain

$$N(m) + N(m') = \frac{N}{2I+1}(1-s) + Nse^{2m\delta}\frac{\sinh\delta}{\sinh(2I+1)\delta} \tag{24}$$

The total component of nuclear spin is

$$\sum_m m\,[N(m) + N(m')]$$

and taking into account the fact that

$$\sum_m me^{2m\delta} = I\,\frac{\sinh(2I+1)\delta}{\sinh\delta}\,B_I(2I\delta)$$

(B_I is the Brillouin function) we obtain for the degree of nuclear polarization

$$f = sB_I(2I\delta).$$

6. We state without proof an expression for the degree of nuclear polarization in the case $I = 1/2$ (Fig. 2), when partial saturation (generally speaking with different degrees of saturation) of both components of paramagnetic resonance is produced (we here again assume that relaxation processes of only types I and II occur):

$$f = \frac{2W(a)\,W(b)\,(e^{\delta} - e^{-\delta}) + 2W\,[W(a) - W(b)] -}{2W(a)\,W(b)\,(e^{\delta} + e^{-\delta}) + 2W\,[W(a) + W(b)] +} \tag{25}$$

$$\rightarrow \frac{-\,W\,[e^{-\delta}\,W(a) - e^{\delta}\,W(b)]\,(e^{\delta} + e^{-\delta})}{+\,W\,[e^{-\delta}W(a) + e^{\delta}\,W(b)]\,(e^{\delta} + e^{-\delta}) + 2W^2\,(e^{\delta} + e^{-\delta})}$$

7. Finally we consider, for the case $I = 1/2$, the effects produced by relaxation processes of types III and IV. For the probabilities of transitions produced by interaction with the lattice we may write (Fig. 2):

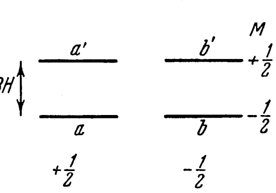

FIG. 2

$$\begin{aligned} &W(a'a) = W(b'b) = We^{\delta}, && W(aa') = W(bb') = We^{-\delta},\\ &W(b'a) = \lambda We^{\delta}, && W(ab') = \lambda We^{-\delta},\\ &W(a'b) = \lambda_1 We^{\delta}, && W(ba') = \lambda_1 We^{-\delta},\end{aligned}$$

$$W(ab) = W(ba) = W(a'b') = W(b'a') = \lambda_2 W, \tag{26}$$

where λ, λ_1, λ_2 and W are functions of the temperature.

In the stationary case we obtain

$$\begin{aligned} N(a')\,(\lambda_1 e^{\delta} + \lambda_2) + N(a)\,(\lambda e^{-\delta} + \lambda_2) \\ = N(b')\,(\lambda e^{\delta} + \lambda_2) + N(b)\,(\lambda_1 e^{-\delta} + \lambda_2), \\ N(a')\,[W(a) + We^{\delta}] + N(b')\,\lambda We^{\delta} + N(b)\,\lambda_2 W \\ = N(a)\,[W(a) + W(1+\lambda)\,e^{-\delta} + \lambda_2 W], \\ N(b)\,[W(b) + We^{-\delta}] + N(a)\,\lambda We^{-\delta} + N(a')\,\lambda_2 W \\ = N(b')\,[W(b) + W(1+\lambda)\,e^{\delta} + \lambda_2 W]. \end{aligned} \tag{27}$$

From the normalization condition we have

$$N(a) + N(a') + N(b) + N(b') = N. \tag{28}$$

We first consider the case of complete saturation of both components of paramagnetic resonance. The $N(a) = N(a')$, $N(b) = N(b')$ and we obtain

$$\frac{N(a)}{N(b)} = \frac{\lambda e^{\delta} + \lambda_1 e^{-\delta} + 2\lambda_2}{\lambda e^{-\delta} + \lambda_1 e^{\delta} + 2\lambda_2},\quad f = \frac{(\lambda - \lambda_1)\sinh\delta}{(\lambda + \lambda_1)\cosh\delta + 2\lambda_2}. \tag{29}$$

If, in particular, $\delta \ll 1$, we obtain

$$\frac{N(a)}{N(b)} = 1 + 2\delta\,\frac{\lambda - \lambda_1}{\lambda + \lambda_1 + 2\lambda_2},\quad f = \delta\,\frac{\lambda - \lambda_1}{\lambda + \lambda_1 + 2\lambda_2}. \tag{29a}$$

(the first of these formulas agrees with Abragam's result[2]). For $\delta \gg 1$ we obtain

$$\frac{N(a)}{N(b)} = \lambda/\lambda_1,\quad f = (\lambda - \lambda_1)/(\lambda + \lambda_1). \tag{29b}$$

However, if (complete) saturation is produced only for the resonance a ⟷ a′, we obtain for the degree of nuclear polarization

$$f = \frac{\lambda_2\,(\lambda - \lambda_1)\,(e^{\delta} - e^{-\delta}) + (\lambda + \lambda_1 + 2\lambda\lambda_1) - \lambda_1\,(1+\lambda)\,e^{2\delta} - \lambda\,(1+\lambda_1)\,e^{-2\delta}}{\lambda_2(1+\lambda)(3e^{\delta} + e^{-\delta}) + \lambda_2(1+\lambda_1)(e^{\delta} + 3e^{-\delta}) + (3\lambda + 3\lambda_1 + 2\lambda\lambda_1 + 4\lambda_2^2) + \lambda_1(1+\lambda)e^{2\delta} + \lambda(1+\lambda_1)e^{-2\delta}}, \tag{30}$$

which gives for $\delta \ll 1$

$$f = \delta\,(1 + \lambda_2)\,(\lambda - \lambda_1)\,/\,2\,[\lambda + \lambda_1 + 2\lambda_2 + \lambda_2^2 + \lambda\lambda_1 + \lambda\lambda_2 + \lambda_1\lambda_2]. \tag{30a}$$

For $\delta \gg 1$ (30) gives $f = -1$, which is easily understood since, if $\lambda_1 \neq 0$ in the case of complete saturation of the resonance $a \longleftrightarrow a'$, all the systems will accumulate in level b.

8. In conclusion, we consider the question of the possibility of a direct measurement of the degree of nuclear polarization in the case of the effects considered above. It is desirable to have methods by which f can be determined without applying the results obtained above (since these results are associated with certain assumptions).

We consider the case $I = 3/2$. We have

$$f = \{3[N(a) + N(a')] + [N(b) + N(b')] - [N(c) + N(c')] - 3[N(d) + N(d')]\}/3N,$$

or

$$f = \{3[N(a) - N(b)] + 3[N(a') - N(b')] + 4[N(b) - N(c)] + 4[N(b') - N(c')] + 3[N(c) - N(d)] + 3[N(c') - N(d')]\}/3N. \quad (31)$$

If we measure experimentally, for example, the signal due to the nuclear resonance $a \longleftrightarrow b$, we can find $N(a) - N(b)$. Thus, by measuring experimentally the nuclear resonances $a \longleftrightarrow b$, $a' \longleftrightarrow b'$, $b \longleftrightarrow c$ etc. we can determine f.

We also note that some of the differences $N(a) - N(b)$, $N(a') - N(b')$, $N(b) - N(c)$ must increase strongly when at least one of the components of the hyperfine structure of paramagnetic resonance is saturated, even if $\delta \ll 1$ [this can be seen, for example, from (12)]. Thus, nuclear magnetic-resonance measurements afford a check, at high temperatures, on the validity of the above theoretical results.

However, the actual situation is more complicated. According to (2) the energies of all the nuclear-resonance transitions are the same up to terms proportional to A and B, and are equal to K/2. In the next approximation the terms differ by an amount of order $B^2/g\beta H$. But to prevent the different components of nuclear magnetic resonance from overlapping the experiments have to be conducted at relatively weak external fields.

Moreover, according to Valiev,[12] nuclear magnetic resonance experiments on nuclei of paramagnetic atoms will be successful apparently only at liquid-helium temperatures. Such experiments are of considerable interest for their own sake.

It can be seen from (31) that when one of the components of paramagnetic resonance is saturated it is not possible to find f directly by measuring the paramagnetic resonance of the unsaturated components. However, such measurements may be carried out to check theoretical results.

For example, when the resonance $b \longleftrightarrow b'$ is completely saturated

$$N(a) - N(a') = N(e^{2\delta} - 1)/(e^{2\delta} + 5 + 2e^{-2\delta}),$$

while in the absence of saturation

$$N^0(a) - N^0(a') = \frac{N}{4} \tanh \delta.$$

Thus, if δ is of the order of or greater than unity, then in the case of complete saturation of the resonance $b \longleftrightarrow b'$ the signal corresponding to the resonance $a \longleftrightarrow a'$ must increase significantly (in particular, if $\delta \gg 1$, it will increase by a factor of four).

Finally, we note that in the case of polarization of radioactive nuclei the degree of polarization can be found directly from the angular anisotropy of γ radiation from these nuclei.

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