# *NUCLEAR MAGNETIC RESONANCE IN METALLIC THALLIUM*

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Nuclear magnetic resonance has been investigated in metallic thallium in a sample of natural isotopic abundance and in samples enriched in $Tl^{205}$ and $Tl^{203}$. Measurements were carried out over a range of fields from 550 to 5500 oe at helium temperatures. In a sample with natural isotopic abundance, it was observed that in weak fields the indirect exchange interaction between the nuclei, due to the presence of conduction electrons, caused the lines due to the two isotopes to merge. The experimental results agree well with calculations based on the Kubo and Tomita theory of magnetic resonance absorption. A value A/h = 37.5 kc/sec has been obtained for the indirect exchange interaction constant.

## INTRODUCTION

SEVERAL years ago Ruderman and Kittel [1] noted that in metals, in addition to the usual dipole-dipole interaction between nuclear spins, there can also exist an indirect exchange interaction due to conduction electrons. The operator for the indirect exchange interaction between the nuclear spins $I_\alpha$ and $I_\beta$ has the form $\tilde{A}_{\alpha\beta} I_\alpha \cdot I_\beta$. The constant $\tilde{A}_{\alpha\beta}$ depends on quantities characterizing the behavior of electrons in the metal and an expression for it was obtained in the one electron approximation by Ruderman and Kittel. [1]

In the case of a metal containing several different isotopes possessing magnetic moments, this interaction must lead to additional broadening of the nuclear magnetic resonance (n.m.r.) line, and this was shown in a convincing manner by Bloembergen and Rowland [2] who measured widths of n.m.r. lines in samples of metallic thallium of different isotopic composition. However, in addition to line broadening such an interaction, if it is sufficiently large, must give rise to another curious effect: it must make the resonance lines of the individual isotopes approach one another, and it must make them merge completely in weak fields when the energy difference between the Zeeman levels of the individual isotopes becomes smaller than the energy of the exchange interaction. This is due to the fact that in weak fields the nuclei of the different isotopes coupled by the exchange interaction behave as a single system. In strong fields the coupling between them is broken and each isotope gives rise to its own absorption line. This effect, which is to a certain extent analogous to the Paschen-Back effect, has been observed in electron paramagnetic resonance, [3,4] and we have attempted to observe it in the case of nuclear spins in metallic thallium.

Thallium has two stable isotopes, $Tl^{203}$ and $Tl^{205}$, whose natural abundance is respectively 30 and 70%. Both isotopes have spin $\frac{1}{2}$, and their magnetic moments differ by only 1%. Such a small difference between the magnetic moments on the one hand, and the appreciable value of the indirect exchange interaction on the other hand, have caused us to observe a complete merging of the lines of the two isotopes into one relatively narrow symmetric line even in fields $\lesssim 1$ koe. To convince ourselves that the observed effects are associated specifically with the presence in the sample of two different thallium isotopes, and are not due to some other causes, we have carried out measurements on isotopically enriched samples, one with 97.6% $Tl^{205}$ and the other with 90% $Tl^{203}$. These measurements also enabled us to determine accurately the displacement of the lines from their initial position in a sample of natural isotopic abundance. Moreover, they have enabled us to obtain quantitative data characterizing the narrowing of the line as the magnetic field is decreased.

In order to describe the results obtained we have carried out calculations on the basis of the theory of Kubo and Tomita, [5] which have turned out to be in good agreement with experiment. The indirect exchange interaction constant enters into several quantities which can be measured experimentally and the values obtained from these different measurements agree with one another.

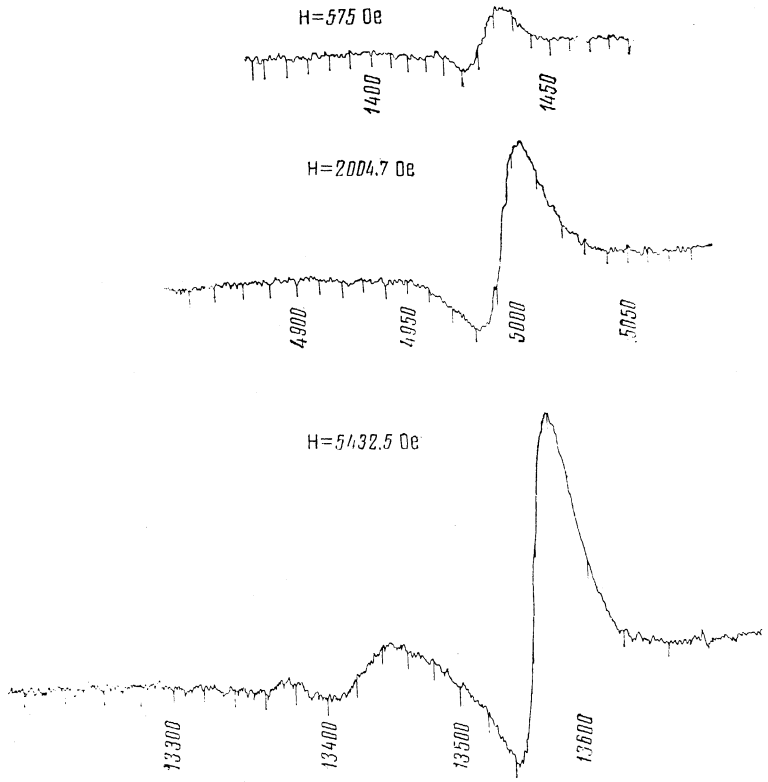


FIG. 1. Recorded derivatives of absorption lines in a sample of natural isotopic abundance.

## EXPERIMENT

All the measurements were carried out at helium temperatures, since it is difficult to obtain a good signal-to-noise ratio in weak fields by any other method.

The sample of natural isotopic abundance was prepared from metal of 99.9% purity. The sample enriched in $Tl^{205}$ contained 97.6% $Tl^{205}$, 2.4% $Tl^{203}$ and less than 0.1% impurities, the principal one of which was Si. The sample enriched in $Tl^{203}$ contained 90% $Tl^{203}$, 10% $Tl^{205}$ and less than 0.2% impurities, the principal one of which was again Si. These samples consisted of finely dispersed metallic powder mixed with vacuum grease. The metallic powder was obtained by reducing the oxide $Tl_2O_3$ in an atmosphere of hydrogen at 265° C. The size of the particles in the samples did not exceed $1\mu$, which is much smaller than the skin depth in thallium at helium temperatures in the frequency range used. Measurements in the case of natural isotopic composition were also carried out on a sample obtained by filing thallium with a barette file. The size of the particles in this case was $10-30\mu$ and was, apparently, somewhat greater than the skin depth. The position and the shape of the lines obtained for all these different samples agreed within experimental error.

The n.m.r. spectrometer was briefly described previously.[6] The magnetic field was measured by means of proton resonance, and the accuracy of the measurement was limited by the instability of the magnetic field which did not exceed $10^{-2}$%. The modulation amplitude was different depending on the line width, and was so chosen that the cor-

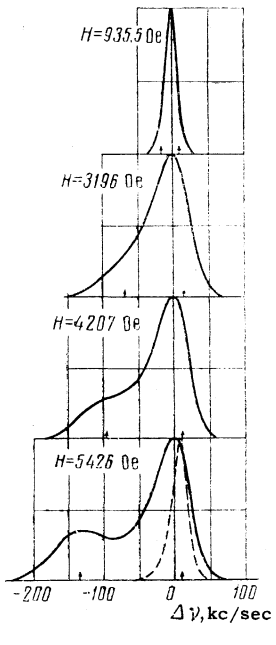


FIG. 2. Absorption lines in a sample of natural isotopic abundance for different intensities of the magnetic field. The dotted curve shows the form which the $Tl^{205}$ absorption line has in an enriched sample.

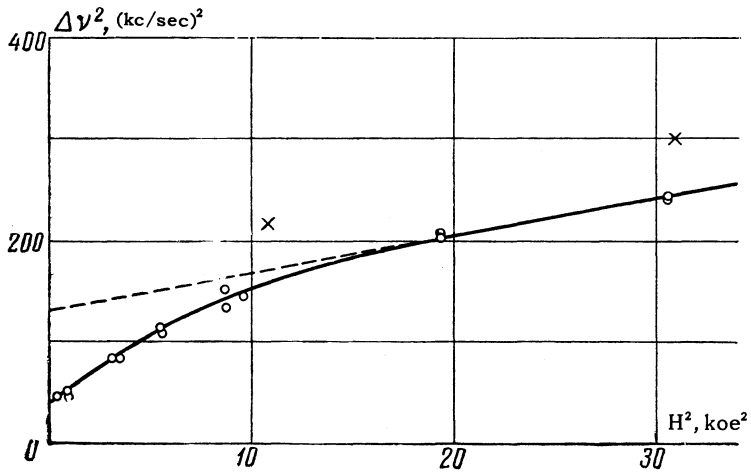


FIG. 3. Dependence on the square of the field of the second moment of the $Tl^{205}$ absorption line in an enriched sample.

rection to the second moment due to the finite modulation[7] did not exceed 1%.

## RESULTS

Figure 1 shows recorded derivatives of absorption lines in metallic thallium of natural isotopic abundance in three different magnetic fields. Figure 2 shows the integrated line shape in the different fields. It can be seen that in weak fields there exists only one relatively symmetric resonance line. As the field is increased the line broadens, becomes asymmetric, and in the highest fields two lines appear due to the two different isotopes of thallium, and these were the lines observed by Bloembergen and Rowland.[2]

The absorption lines of $Tl^{205}$ in the enriched sample behave differently. An increase in the magnetic field broadens the line only slightly. The absorption lines of $Tl^{203}$ in the enriched sample behave similarly, but the broadening is much more pronounced in this case since this sample contains a greater amount of the "foreign" isotope. From these measurements we have obtained for metallic $Tl^{205}$ the ratio $\nu_{res}/H_{res} = 2.4975 \pm 0.0003$ kc/sec · oe, and for metallic $Tl^{203}$ the value $\nu_{res}/H_{res} = 2.4723 \pm 0.0003$ kc/sec · oe. These values were used to indicate in Fig. 2 by arrows those frequencies at which the $Tl^{205}$ and $Tl^{203}$ lines occur in the corresponding fields. For comparison we have shown by a dotted curve in this figure the appearance of the $Tl^{205}$ absorption line in the enriched sample in a high field.

Figure 3 illustrates the manifestation in the line widths of the process of the smearing obliteration of the differences between the spins of the different isotopes as a result of the indirect exchange interaction.* Here we have shown the dependence of the second moment of the $Tl^{205}$ absorption line in the enriched sample on the square of the external field. The linear dependence of the second moment on the square of the field observed in the highest fields is due to the anisotropy of the Knight shift. The dotted line extrapolates this linear dependence into the weak field region. The dotted line shows the manner in which the second moment would vary if the spins of the two isotopes of thallium would remain different at all times. However, we see that in weak fields the second moment becomes smaller than it ought to be by approximately a factor of four. The origin of this effect can be easily understood: in high fields there exist nuclei of two different thallium isotopes and the exchange interaction between them gives a contribution to the second moment. In low fields all the spins become the same and, as is well known, the exchange interaction between identical spins gives no contribution to the second moment.

*We say that the spins are different if their gyromagnetic ratios are different.

## THEORY

For a quantitative description of the observed process of the merging of the lines and of the process of their narrowing we shall utilize the theory of magnetic resonance absorption developed by Kubo and Tomita.[5] It enables us to calculate the first and the second moments of the absorption lines in a sample containing two kinds of spins. The first moments describe the effect of the shift

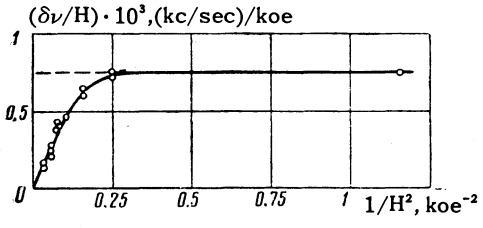


FIG. 4

of the lines from their initial position, while the second moments describe the line narrowing effect.

1. Following Van Vleck[8] we write the Hamiltonian for a system containing two different kinds of spins in the form

$$\mathcal{H} = -\hbar\gamma H\sum_i I_{iz} - h\gamma' H\sum_{i'} I_{i'z} + \sum_{i>k} B_{ik} I_{iz} I_{kz} + \sum_{i'>k'} B_{i'k'} I_{i'z} I_{k'z} + \sum_{i,\,k'} B_{ik'} I_{iz} I_{k'z} + \sum_{i>k} A_{ik}\mathbf{I}_i\mathbf{I}_k + \sum_{i'>k'} A_{i'k'}\mathbf{I}_{i'}\mathbf{I}_{k'} + \sum_{i,\,k'} A_{ik'}\mathbf{I}_i\mathbf{I}_{k'} = \mathcal{H}_z + \mathcal{H}_B + \mathcal{H}_A.$$

The unprimed quantities in this expression refer to spins of one kind, and the primed quantities to spins of the other kind. The first two terms describe the interaction with the external field H; the terms with the coefficients

$$B_{\alpha\beta} = -\tfrac{3}{2}(\gamma^2\hbar^2 r_{\alpha\beta}^{-3} + \widetilde{B}_{\alpha\beta})(3\gamma_{\alpha\beta}^2 - 1)$$

take into account the dipole and the pseudodipole[2] interactions; the terms with the coefficients

$$A_{\alpha\beta} = \tfrac{1}{2}(\gamma^2\hbar^2 r_{\alpha\beta}^{-3} + \widetilde{B}_{\alpha\beta})(3\gamma_{\alpha\beta}^2 - 1) + \widetilde{A}_{\alpha\beta},$$

take into account in addition to the dipole and the pseudodipole interactions also the isotropic exchange interaction. The subscripts $\alpha$ and $\beta$ can refer both to spins of one kind and to spins of the other kind. In all the expressions for the coefficients we assume that the gyromagnetic ratios of both kinds of spins are the same: $\gamma' = \gamma$; $r_{\alpha\beta}$ is the distance between the nucleus $\alpha$ and the nucleus $\beta$; $\gamma_{\alpha\beta}$ is the cosine of the angle between the vector $\mathbf{r}_{\alpha\beta}$ and the external field. We have omitted from the Hamiltonian all terms which lead to satellite absorption lines at frequencies that are multiples of the Larmor frequency, and we have not taken into account the Knight shift.

2. In the case of high fields when two lines appear we choose (for notation see [5]):

$$\mathcal{H}_0 = \mathcal{H}_z, \qquad \mathcal{H}' = \mathcal{H}_A + \mathcal{H}_B.$$

In accordance with formulas (4.22) and (5.3) of the paper by Kubo and Tomita[5] we find by second order perturbation theory, for the case of spins $I = I' = \frac{1}{2}$, that in a polycrystalline sample the center of gravity of the line which was initially situated at the frequency $\nu = \gamma H/2\pi$ will be shifted by an amount

$$\delta\nu = -\frac{1}{4\pi(\nu - \nu')\hbar^2}\sum_{k'}(\widetilde{A}_{ik'}^2 - b_{ik'}^2),$$

$$b_{ik'} = -(\gamma^2\hbar^2 r_{ik'}^{-3} + \widetilde{B}_{ik'}). \qquad (1)$$

For the second moment with respect to the undisplaced position of the line we obtain in this approximation Van Vleck's expression:

$$\overline{\Delta\nu^2} = \frac{9}{20\hbar^2}\sum_k b_{ik}^2 + \frac{9}{45\hbar^2}\sum_{k'} b_{ik'}^2 + \frac{1}{4\hbar^2}\sum_{k'}\widetilde{A}_{ik'}^2. \qquad (2)$$

We shall see later that in our case $b_{\alpha\beta} \ll \widetilde{A}_{\alpha\beta}$, and, therefore, in making comparisons with experiment we can neglect the quantities $b_{\alpha\beta}^2$ in comparison with $\widetilde{A}_{\alpha\beta}^2$. Further, the expression obtained by Ruderman and Kittel[1] shows that $\widetilde{A}_{\alpha\beta}$ is inversely proportional to the cube of the distance between the nucleus $\alpha$ and the nucleus $\beta$, at any rate at large distances. Therefore, the summation in formulas (1) and (2) can be restricted to only the twelve nearest neighbors in the hexagonal close packed lattice of metallic thallium by taking $\widetilde{A}_{\alpha\beta} = A$, $b_{\alpha\beta} = b$ for the nearest neighbors, and $\widetilde{A}_{\alpha\beta} = b_{\alpha\beta} = 0$ in all the other cases. As a result of this we obtain (cf. [9]):

$$\delta\nu/H = -6\pi(A^2/h^2)\, f/(\gamma - \gamma')\, H^2, \qquad (1a)$$

$$\overline{\Delta\nu^2} = \tfrac{27}{5}(b^2/h^2)(1 - f) + 3(A^2/h^2) f. \qquad (2a)$$

Here f is the concentration of the "foreign," primed isotope.

The line displacement given by (1a) causes the second moment with respect to the center of gravity to be smaller than (2a) by an amount $(\delta\nu)^2$. However, this decrease is of the same order of smallness as the correction to the second moment which arises in fourth-order approximation of perturbation theory. We therefore obtain the total decrease in the second moment if we add to the above expression the correction evaluated in the fourth-order approximation (we note that all the third-order corrections vanish). As a result of this we have

$$\delta(\overline{\Delta\nu^2}) = \frac{1}{16(\nu - \nu')^2\hbar^4 N}\Big\{5\sum_{ik'}\widetilde{A}_{ik'}^4 + \sum_{ilk'\neq}[\widetilde{A}_{ik'}^2\widetilde{A}_{lk'}^2 - \widetilde{A}_{il}^2(\widetilde{A}_{ik'} - \widetilde{A}_{lk'})^2] + \sum_{ik'l'\neq}[4\widetilde{A}_{ik'}^2\widetilde{A}_{il'}^2 - \widetilde{A}_{k'l'}^2(\widetilde{A}_{ik'} - \widetilde{A}_{il'})^2]\Big\} + (\delta\nu)^2, \qquad (3)$$

where N is the number of unprimed spins. Summing this expression in the same manner as in the derivation of formulas (1a) and (2a) we obtain

$$\delta(\overline{\Delta\nu^2}) = 3\pi^2(A^4/h^4)(2f + 45f^2)/(\gamma - \gamma')^2 H^2. \qquad (3a)$$

3. In the weak field case we have only one resonance line, and we choose

$$\mathcal{H}_0 = -\hbar\gamma H\Big\{\sum_i I_{iz} + \sum_{i'} I_{i'z}\Big\},$$

$$\mathcal{H}' = \hbar(\gamma - \gamma')\, H\sum_{i'} I_{i'z} + \mathcal{H}_B + \mathcal{H}_A.$$

In accordance with formulas (4.22) and (5.3) of the paper by Kubo and Tomita[5] we find that the cen-

ter of gravity of the line will be displaced with respect to the frequency $\nu = (\gamma/2\pi)\,H$ by an amount

$$\delta\nu = -(\nu - \nu')\,f, \tag{4}$$

and the second moment with respect to the center of gravity will be given in a polycrystalline sample by

$$\overline{\Delta\nu^2} = \frac{9}{20h^2}\left\{\sum_k b_{ik}^2 + \sum_{k'} b_{ik'}^2\right\} + (\nu - \nu')^2 f\,(1-f). \tag{5}$$

On carrying out the summation in this case in the same manner as before we obtain

$$\overline{\Delta\nu^2} = 27b^2/5h^2 + (2\pi)^{-2}(\gamma - \gamma')^2 H^2 f\,(1-f). \tag{5a}$$

Similar formulas were obtained by a different method by Pryce.[10]

The magnetic field which separates the region of strong fields from the region of weak fields is given by the following expression[5]

$$H^* = \left\{\sum_{k'} \tilde{A}_{ik'}^2\right\}^{1/2} \Big/ 2\hbar\,(\gamma - \gamma') \approx \pi\sqrt{12f}A/h\,(\gamma - \gamma'). \tag{6}$$

## COMPARISON OF EXPERIMENT WITH THEORY

Our problem is to determine how well the expressions (1a) — (5a) describe the experimental data, and to obtain the values of the constants A and b, which describe the interaction of the nuclear spins in metallic thallium. A rough estimate for the indirect exchange interaction constant can be obtained directly from the data shown in Fig. 2. The lines due to the different isotopes begin to be resolved in fields of approximately 1.5 — 2 koe. If we assume that the difference in the energies of the Zeeman levels of the two isotopes in such fields is just of the order of the exchange energy, then we obtain for the constant A the estimate $A/h \approx 35 - 50$ kc/sec.

In order to justify the simplification introduced in obtaining formulas (1a) — (5a), whereby we neglected the quantity $b^2$ compared to $A^2$, we now obtain the value of the constant b, which characterizes the dipole and the pseudodipole interactions. This can be directly accomplished on the basis of the data shown in Fig. 3. As we have noted already, in the weakest fields when any difference between the gyromagnetic ratios of the spins of the different isotopes completely disappears, the value of the second moment is determined basically only by the dipole and the pseudodipole interactions, and is given by expression (5a). From this we obtain $b/h = 2.7$ kc/sec. The values of the second moment in weak fields in a sample of natural isotopic abundance and in the sample enriched in $Tl^{203}$ give for b/h respectively the values of 2.6 and 2.9 kc/sec.

From the data of Fig. 3 it is possible to obtain also a more accurate value for the constant A by making use of the following considerations. In the strongest fields the second moment is given by formula (2a) and must be constant. As we have noted already, the observed linear increase is due to the anisotropy of the Knight shift which was not taken into account in the derivation of this formula. It can be taken into account if we add to the right hand side of formula (2a) a term proportional to the square of the field. A sharp decrease in the second moment in weak fields compared to the value which would be given by the linear dependence is due to the disappearance from formula (2a) of the term $3(A^2/h^2)f$, i.e., of the contribution made to the second moment by the exchange interaction between the different isotopes. Without this term formula (2a) goes over into formula (5a) (if f is sufficiently small), which specifically describes the second moment in weak fields. Thus, by equating the quantity $3(A^2/h^2)f$ to the difference between that value of the second moment which is obtained by extrapolating the linear dependence into the weak field region and the actual value of the second moment we obtain $A/h = 36.0$ kc/sec. A similar procedure applied to the data obtained with the sample enriched in $Tl^{203}$ gives $A/h = 34.0$ kc/sec.

We shall now see how well the process of the merging of the lines is described by the theory. Formula (1a) referring to this is valid for fields $H > H^* \approx 1.4$ koe [this value is obtained from formula (6) by putting $A/h = 36$ kc/sec and $f = 0.3$]. Figure 4 shows the experimentally measured dependence on $1/H^2$ of the relative displacement $\delta\nu/H$ for the $Tl^{205}$ line in metal of natural isotopic abundance. The dotted line here shows the limiting value obtained in accordance with formula (4a) to which the value of $\delta\nu/H$ tends as $H \to 0$ [$\delta\nu/H = -(\gamma - \gamma')f/2\pi$]. The initial portion of this curve indeed turns out to be linear for fields $H \gtrsim 3$ koe. From its slope we find that $A/h = 36.5$ kc/sec.

The process of line narrowing shown in Fig. 3 is described by formula (3a) which is valid for fields $H > H^* \approx 500$ oe. [This value is obtained by means of formula (6) for $A/h = 36$ kc/sec and $f = 2.4 \times 10^{-2}$.] Figure 5 shows the dependence of $\delta\overline{(\Delta\nu^2)}$ on $1/H^2$. It can be seen that the initial rectilinear portion of this curve extends from high fields down to $H \approx 1.5$ koe. From its slope we obtain $A/h = 38.5$ kc/sec. Similar measurements in the case of $Tl^{203}$ yield $A/h = 35$ kc/sec.

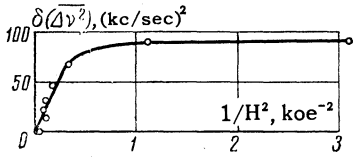


FIG. 5

The good agreement between the values of the indirect exchange interaction constant obtained from the different measurements testifies that the theory of Kubo and Tomita describes the situation sufficiently well. The linear dependence on $1/H^2$ predicted by the theory with respect to the shift $\delta\nu/H$ and the decrease in the second moment $\delta\overline{(\Delta\nu^2)}$ is confirmed by experiment, as can be seen from Figs. 4 and 5.

## DISCUSSION

Thus, as a result of the reduction of the experimental data we have obtained the following values for the constants describing the interaction between the nuclear spins in the metal: A/h = 36.0 kc/sec, b/h = 2.7 kc/sec. The ordinary dipole-dipole interaction constant in thallium is $\gamma^2\hbar^2a^{-3}$ = 1 kc/sec (a is the distance between nearest neighbors). Thus, it can be seen that the pseudodipole interaction is of approximately the same magnitude as the dipole interaction, and does not exceed 10% of the isotropic exchange interaction described by the constant A.

As we have noted already, Ruderman and Kittel [1] have obtained an expression relating the constant A to the properties of the conduction electrons, which in the case $I = I' = \frac{1}{2}$ has the form

$$\tilde{A}_{ik} = \Omega^2 m^* \langle a_k^2\rangle (8\pi r_{ik}^4)^{-1} [2k_m r_{ik}\cos(2k_m r_{ik}) - \sin(2k_m r_{ik})],$$

where $\Omega$ is the atomic volume, m* is the effective mass, $\langle a_k\rangle$ is the value of the hyperfine interaction constant between the nucleus and the electron averaged over the Fermi surface, $\hbar k_m$ is the momentum of the electron on the Fermi surface. Unfortunately, this expression was obtained under highly simplifying assumptions; in particular, it was assumed that the energy is a quadratic function of the momentum: $E = \hbar^2k^2/2m^*$. Apparently the last circumstance is completely excluded in the case of thallium. [11] Therefore, the estimates given below must be regarded more as an illustration, and we cannot assume that they have any serious significance at all.

As can be seen, the expression for A contains three unknown quantities: m*, $\langle a_k\rangle$ and $k_m$. However, we have at our disposal also the data on the Knight shift, in the expression for which obtained under the same restrictive assumptions these quantities appear in a different combination (cf., for example, [12]):

$$\Delta H/H = 3\pi\beta_0 \langle a_k\rangle m^*/h^2\gamma k_m^2,$$

where $\beta_0$ is the Bohr magneton. The method of averaging the quantity $a_k$ in this expression generally speaking differs from the method of averaging it in the expression for A, but for order of magnitude estimates this is not significant. According to our measurements, which agree well with the data of Bloembergen and Rowland, [2] $\Delta H/H = 1.52\%$. If for the value of the maximum Fermi momentum we take $k_m = \pi/a$, then we obtain $\langle a_k\rangle = 10^{11}$ cps, which exceeds the hyperfine interaction constant in the free atom by a factor 10, and m* = 0.2 m. But if we take m* = 1.2 m, which follows from data on specific heat, [14] we obtain $k_m \approx 5.5 \times 10^8$ cm$^{-1}$, and $\langle a_k\rangle = 10^{12}$ cps.

In conclusion we would like to note that there is an appreciable disagreement between our values of the constants and the values obtained by Bloembergen and Rowland [2]: A/h = 17.5 kc/sec, b/h = 5.5 kc/sec. In Fig. 4 we have shown by crosses those values of the second moment which are given in [2] for the sample enriched in $Tl^{205}$ which contained 1.3% $Tl^{203}$. The values of the second moment themselves agree very satisfactorily with the values obtained in the present work if we take into account the fact that in the evaluation of second moments fairly large errors can arise, particularly if the signal-to-noise ratio is not very great. An attempt might be made to explain the small systematic discrepancy which, apparently, nevertheless exists by means of additional line broadening due to the influence of the spin-lattice relaxation time $T_1$: Bloembergen and Rowland have carried out their measurements at 77° K and $T_1$ might not have yet reached very high values. However, these authors have made an error in the reduction of the results of their experiments. They assumed that the second moment obtained by extrapolating to zero field the linear dependence at high fields is determined only by the dipole and the pseudodipole interactions. But in fact, as has been discussed above, the indirect exchange interaction also makes an appreciable contribution to it. Their neglect of this circumstance caused them to grossly overestimate the magnitude of this pseudodipole interaction and to correspondingly underestimate the magnitude of the indirect exchange interaction.

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