

NMR investigation of electron polarization effects in ternary lanthanum stannide

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An experimental investigation was made of NMR in ¹¹⁹Sn nuclei in a metallic Van Vleck paramagnet, (La_{1-x}Pr_x)₃Rh₄Sn₁₃ ($x = 0-0.15$). The NMR spectrum revealed satellite absorption lines of the tin nuclei located in the nearest coordination spheres of the rare-earth impurity. The spatial distribution of the electron polarization near the Van Vleck ion was determined and estimates were obtained of the hyperfine and exchange constants. Analysis of the experimental results suggests that the compound Pr₃Rh₄Sn₁₃ may be a very effective working substance for the attainment of ultralow temperatures by adiabatic demagnetization.

The idea of using Van Vleck paramagnets as working substances in experiments on nuclear adiabatic demagnetization was put forward in Ref. 1. The essence of the proposed method can be described as follows. An external static magnetic field H_0 applied to a sample polarizes the electron shell of a Van Vleck ion and induces a magnetic moment parallel to H_0 in the ground singlet state of the ion. The hyperfine interaction enhances the field at the nucleus by one to two orders of magnitude, which makes it possible to reduce significantly the entropy of the nuclear spin system of the sample using reasonable values of H_0 . The static magnetic field is then switched off adiabatically and the entropy of the spin system increases because of the spin-lattice relaxation processes, which cools the sample. Therefore, a large enhancement of the field at the nuclei and a short spin-lattice relaxation time are important properties of the working substance. The latter condition is satisfied automatically in metals because of the presence of conduction electrons. This is precisely why the proposed method has been implemented successfully in experiments on metallic Van Vleck paramagnets.² The lowest temperature which can be achieved by this method is limited by the nuclear magnetic ordering temperature.

Since the coefficient representing the enhancement of the field at the nuclei is governed by the hyperfine interaction and the nuclear magnetic ordering temperature is determined by the indirect exchange interactions of Van Vleck ions via conduction electrons, the information on these two characteristics can be obtained by the NMR method.

We shall report the results of an NMR investigation of a metallic Van Vleck paramagnet, (La_{1-x}Pr_x)₃Rh₄Sn₁₃ ($x = 0-0.15$).³ We were able to observe satellite lines of the NMR of ¹¹⁹Sn due to the spin polarization of conduction electrons and thus reconstruct the spatial distribution of the spin density near a Van Vleck ion and estimate the strength of the hyperfine and exchange interactions. We also considered the feasibility of using this compound as a working substance in nuclear adiabatic demagnetization.

1. EXPERIMENTAL RESULTS

The compounds (La_{1-x}Pr_x)₃Rh₄Sn₁₃ ($x = 0, 0.01, 0.025, 0.05, 0.15, \text{ and } 1.0$) were prepared in accordance with the recommendations given in Ref. 4. The NMR measurements were carried out using a continuously operating spectrometer at a frequency of 15 MHz in the temperature range 1.8–4.2 K. The signal/noise ratio was increased by crushing

the samples to form a powder with the characteristic particle size ($\sim 50 \mu\text{m}$) considerably smaller than the depth of penetration of the rf field.

1.1. Compound La₃Rh₄Sn₁₃

We observed a signal due to the magnetic resonance of the ¹¹⁹Sn nuclei consisting of two lines: a strong line with width at half-maximum of the peak $\Delta H = 20 \text{ G}$ and a shift $K_0 = 0.42\%$ and a weak line with width $\Delta H = 10 \text{ G}$ and $K_0 = 0.71\%$ (Fig. 1). The values of ΔH and K_0 were independent of temperature for both lines. Moreover, we determined the electrical resistivity ρ of a single crystal of La₃Rh₄Sn₁₃ using the standard four-contact method under dc conditions. The value of $\rho_{300 \text{ K}}$ was $185 \mu\Omega \cdot \text{cm}$, with $\rho_{300 \text{ K}}/\rho_{4.2 \text{ K}} = 8$. The compound La₃Rh₄Sn₁₃ is known to be a superconductor with $T_{c0} \approx 3 \text{ K}$ (Refs. 3 and 5). Measurements of the upper critical field H_{c2} of this sample as a function of temperature gave $(dH_{c2}/dT)_{T=T_{c0}} \approx 3 \text{ kG/K}$.

1.2. Compound (La_{1-x}Pr_x)₃Rh₄Sn₁₃

An increase in the concentration x of the praseodymium impurity increased the width and reduced the intensity of the NMR line of ¹¹⁹Sn, which exhibited a shift $K_0 = 0.42\%$ (Fig. 2) and in the range $x > 0.05$ the line disappeared. The second line with $K_0 = 0.71\%$ became broader and its intensity decreased less, so that for a sample with $x = 0.15$ width was $\Delta H = 15 \text{ G}$ at $T = 4.2 \text{ K}$. A further increase in praseodymium concentration suppressed the second line also.

In samples with $x = 0.05$ at $T = 4.2 \text{ K}$ the NMR spectrum included not only these two lines, but also three weak satellite signals (identified by arrows *a*, *b*, and *c* in Fig. 3) shifted relative to the strong central line by $\Delta K_a = 0.81\%$, $\Delta K_b = 0.57\%$, and $\Delta K_c = -0.28\%$, respectively. Cooling shifted these signals away from the center (the satellite *c* became increasingly clear in the right-hand wing of the central line) and broadened them, so that the satellites *a* and *b* farthest from the main line disappeared in the noise background at $T < 3.0 \text{ K}$.

1.3. Compound Pr₃Rh₄Sn₁₃

We searched for the NMR of the ¹⁴¹Pr and ¹¹⁹Sn nuclei. No resonance absorption signals of these nuclei were observed in the range of static magnetic fields from 0 to 14 kG.

2. DISCUSSION OF RESULTS

2.1. Compound $\text{La}_3\text{Rh}_4\text{Sn}_{13}$

The compound $\text{La}_3\text{Rh}_4\text{Sn}_{13}$ has $Pm\bar{3}n$ cubic symmetry with lattice period $a = 9.693 \text{ \AA}$ (Ref. 6), confirmed by our x-ray structure analysis. In the unit cell the La^{3+} ions occupy three positions with $\bar{4}2m$ local symmetry and mutually perpendicular tetragonal axes. The tin ions are in two crystallographically inequivalent positions: twelve of them occupy the position with the monoclinic symmetry m and one ion is in the position with the symmetry $m\bar{3}$ which is close to cubic.⁶ The ratio of the integrated intensities of the two absorption lines observed in an NMR spectrum of $\text{La}_3\text{Rh}_4\text{Sn}_{13}$ (Fig. 1) was found to be close to 12. Hence, we concluded that the strong line is due to the ^{119}Sn nuclei located in the first of these two positions and the low intensity represents the nuclei in the second position.

It is known that the NMR frequency of nuclei in metals is shifted relative to its value in an insulator by an amount equal to the Knight shift (see, for example, Ref. 7):

$$K_0 = \frac{1}{2} (\beta/\beta_N) N(E_F) A, \quad (1)$$

where β and β_N are the electron and nuclear Bohr magnetons, $N(E_F)$ is the density of states of conduction electrons on the Fermi surface, and A is a constant of the hyperfine interaction between the nuclear moment and conduction electrons

We estimate the value of $N(E_F)$ for this compound using the familiar expression of Gor'kov⁸ for the coefficient of the electron specific heat, which is valid in the case of "dirty" superconductors:

$$\gamma = 2.2 \cdot 10^{-3} \rho_{4.2\text{K}}^{-1} (dH_{c2}/dT)_{T=T_{c0}}. \quad (2)$$

Substituting in Eq. (2) our values of the residual resistivity $\rho_{4.2\text{K}}$ and $(dH_{c2}/dT)_{T=T_{c0}}$, we obtain $\gamma \approx 95 \text{ mJ mol}^{-1} \text{ K}^{-2}$ or $N(E_F) \approx 0.5 \text{ eV}^{-1} \text{ spin}^{-1} \text{ atom}^{-1}$. Knowing the shift of the central line of the NMR spectrum, amounting to $K_0 = 0.42\%$, we can then apply Eq. (1) to estimate the constant of the hyperfine interaction of the nuclear moment of tin with conduction electrons: $A \approx 0.11 \text{ K}$. We assume that the larger Knight shift of the NMR line of the ^{119}Sn nuclei in the second inequivalent position was associated with the higher local density of states of conduction electrons at these positions.

The observed absorption lines has a near-Gaussian profile. An estimate of the contribution made by the nuclear dipole-dipole interactions to the width of the lines gives a value less than 1 G for each of the tin positions. Clearly, an important contribution to the observed line widths is made by the dipole-dipole interactions between the nuclear moments of tin and the weakly localized d electrons of rhodium. The difference between the line widths may be due to the different positions of the rhodium ions relative to the tin ions located at the inequivalent positions. Moreover, the large width of the lines of the tin nuclei located at the low-symmetry position may be due to the anisotropic Knight shift.⁷

2.2. Compound $(\text{La}_{1-x}\text{Pr}_x)_3\text{Rh}_4\text{Sn}_{13}$

According to the generally accepted theory of the exchange interactions between paramagnetic ions in a metal, developed by Ruderman, Kittel, Kasuya, and Yosida

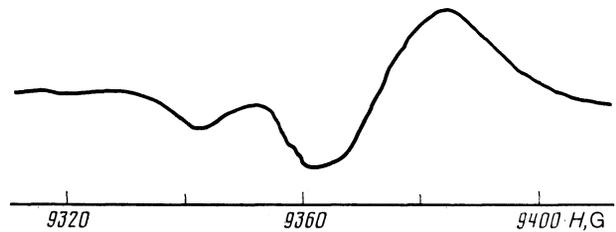


FIG. 1. Derivative of the NMR signal of ^{119}Sn in $\text{La}_3\text{Rh}_4\text{Sn}_{13}$ at $T = 4.2 \text{ K}$.

(RKKY theory),⁹ the Hamiltonian of the exchange interaction of a rare-earth impurity with conduction electrons in the crystal lattice of a metal is

$$H_{sf} = (g_J - 1) J_{sf} \mathbf{J} \cdot \boldsymbol{\sigma}, \quad (3)$$

where \mathbf{J} and g_J represent the angular momentum and the Landé factor of the rare-earth impurity ions; $\boldsymbol{\sigma}$ is the spin of conduction electrons; J_{sf} is the constant of the s - f exchange between the rare-earth impurity and conduction electrons. This interaction gives rise to a polarization of conduction electrons which is oscillatory and decays as r^{-3} :

$$P(k_{FR}) = P_0 F(2k_{FR}), \quad (4)$$

where

$$P_0 = (2/\pi) (g_J - 1) k_F^3 J_{sf} N(E_F) \langle J^z \rangle,$$

$\langle J^z \rangle$ is the average value of the z component of \mathbf{J} , k_F is the Fermi wave vector, r is the distance to the impurity, and

$$F(2k_{FR}) = [2k_{FR}r \cos(2k_{FR}r) - \sin(2k_{FR}r)] (2k_{FR}r)^{-4}.$$

In addition, the polarization of conduction electrons described by Eq. (4) shifts the resonance frequency of the i th nucleus in the crystal matrix by¹⁰

$$\Delta K_i = 6\pi K_0 z (g_J - 1) J_{sf} \chi(T) F(2k_{FR}r_i) / N_A g_J \beta^2 \quad (5)$$

[where z is the number of conduction electrons per atom, $\chi(T)$ is the molar susceptibility of the rare-earth impurity, and N_A is the Avogadro number], which results in broadening of the absorption signal. When the value of ΔK_i exceeds the width of the NMR line, additional satellite lines may appear in the spectrum.¹¹

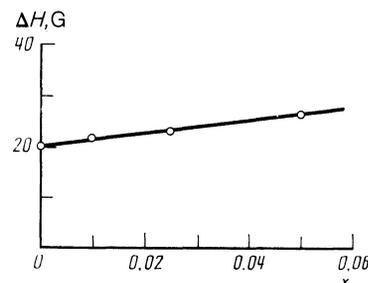


FIG. 2. Dependence of the width of the NMR line of ^{119}Sn ($K_0 = 0.42\%$) in $(\text{La}_{1-x}\text{Pr}_x)_3\text{Rh}_4\text{Sn}_{13}$ on the praseodymium concentration at $T = 4.2 \text{ K}$. The continuous line is calculated on the basis of Eq. (8).

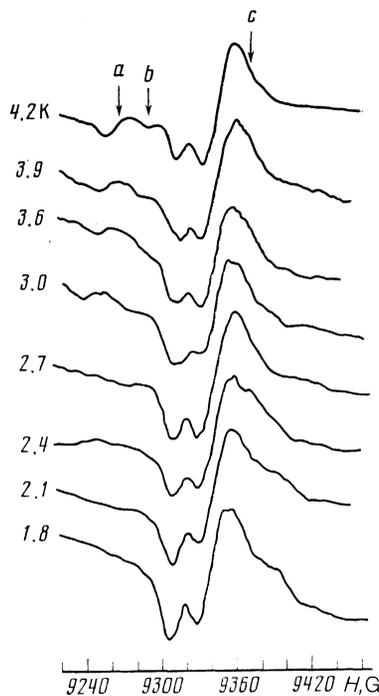


FIG. 3. Experimentally observed NMR spectra of ^{119}Sn in $(\text{La}_{1-x}\text{Pr}_x)_3\text{Rh}_4\text{Sn}_{13}$ ($x = 0.05$) at $T = 4.2\text{--}1.8$ K.

We assume that the three low-intensity signals we observed in the NMR spectrum of sample of a $(\text{La}_{0.95}\text{Pr}_{0.05})_3\text{Rh}_4\text{Sn}_{13}$ and identified by arrows a , b , and c in Fig. 3, belong to the nuclei located in the adjacent coordination spheres of the Pr^{3+} ion. In an analysis of the spin polarization in the direct vicinity of a paramagnetic ion we must allow first of all for the finite radius of the f shell of the rare-earth ion, since at distances within one unit cell it is already inconsistent to approximate the f shell of the Pr^{3+} ion with the radius $\rho = (\langle r^2 \rangle_{4f})^{1/2} = 0.55 \text{ \AA}$ (Ref. 12) by a point, as is usual in the RKKY model. Since the electron density in the f shell decreases fairly rapidly with distance,¹² we feel that it is quite reasonable to describe the spatial distribution of this shell by a Gauss law. We can then write down the following expression for $P(r)$:

$$P(r) = P_0$$

$$\int d^3r' \frac{\exp(-|\mathbf{r}-\mathbf{r}'|^2/2\rho^2)}{(2\pi)^{3/2}\rho^3} \frac{2k_F r' \cos(2k_F r') - \sin(2k_F r')}{(2k_F r')^4} \quad (6)$$

The expression (6) can be transformed to

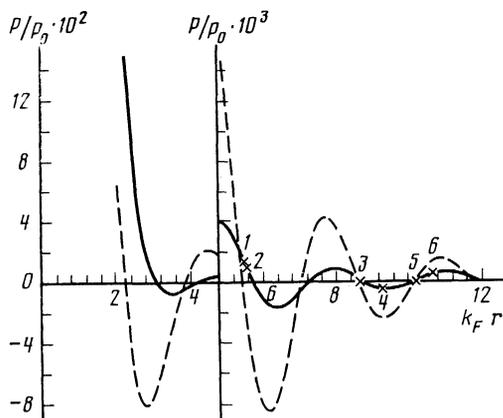


FIG. 4. Dependence of the polarization function on the distance to the magnetic impurity [calculated on the basis of Eq. (7)]. The dashed curve corresponds to $\rho = 0$ and the continuous curve to $\rho = 0.55$ and $k_F = 1.607 \text{ \AA}^{-1}$. The values of the polarization in the coordination spheres closest to Pr^{3+} ion are identified by crosses. The numbers alongside the symbols are the serial numbers of the spheres (Table I).

$$P(r) = \frac{(2\pi)^{-3/2}}{8} P_0 \frac{\exp(-r^2/2\rho^2)}{\rho r} \int_0^\infty dr' \frac{\exp(-r'^2/2\rho^2)}{r'^3} \cdot (2r' \cos 2r' - \sin 2r') \text{sh} \frac{rr'}{\rho^2} \quad (7)$$

Here, the distance to the impurity and the radius of the f shell are now in dimensionless units $k_F r'$ and $k_F \rho$. In the limit $\rho \rightarrow 0$, and also at large distances from the impurity, the form of this polarization function is identical with Eq. (4) and near an impurity allowing for the finite value of ρ reduces the amplitude and induces a phase shift of the polarization function (Fig. 4).

Calculation of the polarization at the tin nuclei located near the Pr^{3+} impurity requires the knowledge of the radii of the nearest coordination spheres and of the numbers of the nuclei in these spheres. The necessary data are presented in Table I. It should be pointed out that the tin ions located in the second inequivalent position (with the symmetry $m3$) form completely the third coordination sphere and the ions in the first position (symmetry m) form the remaining spheres. As pointed out in Sec. 2.1, the tin nuclei in the second inequivalent position (in the third coordination sphere) are characterized by a large Knight shift indicating a high density of the conduction electron states at this position. This should have resulted in a stronger broadening (compared with the central line) in the presence of a magnetic impurity. However, the experiments revealed the opposite. This was a clear indication that the third coordination sphere was located near one of the zeros of the polarization function. Since the usual range of variation was $k_F = 1\text{--}2 \text{ \AA}^{-1}$ (Ref. 13), we concluded that $k_F r_3 \approx 5.4\text{--}10.8$. In this

TABLE I.

Sphere No.	Distance from impurity, \AA	Number of tin nuclei in sphere	Sphere No.	Distance from impurity, \AA	Number of tin nuclei in sphere	Sphere No.	Distance from impurity, \AA	Number of tin nuclei in sphere
1	3.4	8	3	5.42	4	5	6.37	4
2	3.44	4	4	5.77	8	6	6.67	4

interval there were four zeros of the function $P(r)$ (Fig. 4). By placing the third sphere in turn at each of these four zeros and thus specifying the value of k_F , we could determine in each case the value of $P(r_i)$ for all the other spheres. The observed satellite lines could then be identified by comparing the ratio of their shifts ΔK_a , ΔK_b , and ΔK_c with the ratio of the polarization in the different spheres, because $\Delta K_i \propto P(r_i)$. A careful analysis of all possible situations gave an unambiguous result in the form of $k_F = 1.607 \text{ \AA}^{-1}$ and the conclusion that the nuclei located in the first, second, and fourth coordination spheres, respectively, contributed to the satellites a , b , and c . The satellites due to these spheres were no wider than the central line.

The dependence $\chi(T)$ describes the temperature dependence of the shift ΔK_i of a satellite [see Eq. (5)]. A determination of the temperature dependence of the susceptibility of $(\text{La}_{1-x}\text{Pr}_x)_3\text{Rh}_4\text{Sn}_{13}$ samples was reported in Ref. 3, where it was shown that $\chi(T)$ is entirely due to the influence of a crystal field of the tetragonal symmetry. An analysis of the crystal field parameters reported in Ref. 3 demonstrated that the investigated compound is a Van Vleck paramagnet with a singlet ground state $(0.616|4\rangle + 0.492|0\rangle + 0.616|-4\rangle)$ and a doublet $(0.974|\mp 3\rangle - 0.226|\pm 1\rangle)$ separated by 3.7 K. The other levels lie considerably higher on the energy scale. Calculation of the ion susceptibility based on these crystal field parameters shows that at helium temperatures we have $\chi_{\parallel}/\chi_{\perp} \sim 6$. Therefore, the position of each satellite line should exhibit a strong angular dependence and, moreover, since the Pr^{3+} ions can be located in the lattice at three positions with mutually perpendicular tetragonal axes, in the case of a powder specimen this should result in simultaneous observation of the same line in two extreme orientations and in the perpendicular case it should be twice as strong as in the parallel case. An analysis of the ratio of the intensities of the observed satellites and main absorption lines allowing for the concentration of the praseodymium impurity and the number of the tin nuclei in the respective coordination spheres supported the conclusion that the observed a , b , and c signals were due to the perpendicular orientation of the absorption lines of the ^{119}Sn nuclei located in the first, second, and fourth coordination spheres, respectively. The lines in the parallel orientation were not observed because of their low intensity.

An estimate of the exchange interaction between the Van Vleck ions and conduction electrons can be obtained from Eq. (5). Substituting the value of $\Delta K_a = 0.81\%$, calculated with the aid of Eq. (7), as well as the polarization in the first sphere $P(r_1)/P_0 \approx 1.4 \times 10^{-3}$ and also $\chi_1^{4.2\text{K}} = 0.47 \times 10^{-4} \text{ cm}^3/\text{g}$ and $z = 1.95$, we obtained the s - f exchange constant $J_{sf} \approx 0.13 \text{ eV}$.

The inhomogeneous broadening of the central line could be estimated from the relation¹⁴

$$\Delta H_{1/2} = (8\pi/3)n|J_0|\langle J^2 \rangle, \quad (8)$$

where $\Delta H_{1/2}$ is the line width at half-maximum, n is the concentration of the magnetic impurity (per unit volume),

$$J_0 = \pi(g_J - 1)N^2(E_F)J_{sJ}AE_F/2(k_F a_0)^3,$$

$a_0 = 4.85 \text{ \AA}$ is the distance between the nearest rare-earth ions in the lattice, and $\langle J^2 \rangle = \chi_{\text{atom}}^{\text{Pr}^{3+}} H/g_J \beta$. Since the NMR

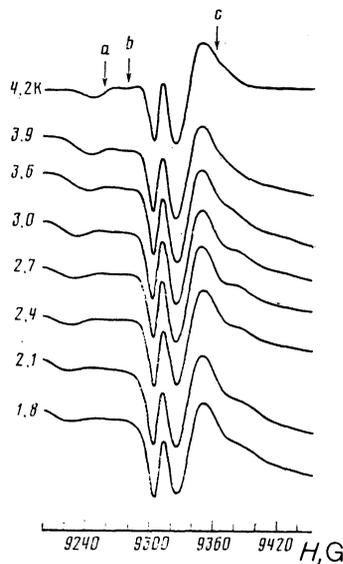


FIG. 5. Computer simulation of the NMR spectrum of ^{119}Sn in $(\text{La}_{1-x}\text{Pr}_x)_3\text{Rh}_4\text{Sn}_{13}$ ($x = 0.05$).

line of ^{119}Sn is nearly Gaussian in the absence of a magnetic impurity, this "Lorentzian" mechanism of the broadening of the Gaussian line profile transforms it to a Voigt profile.¹⁵ A numerical calculation shows that in that in the case of the line width $\Delta H = 20 \text{ G}$ observed for the "pure" compound a correction of the (8) type is 7 G for $x = 0.05$ and $T = 4.2 \text{ K}$, and this estimate is close to the value actually observed (Fig. 2).

The whole analysis of the experimental results described above was calibrated by comparing the observed spectrum (Fig. 3) with the results of its computer simulation. The line profiles of all the absorption signals were assumed to be Gaussian. A qualitative agreement with the experimental results was obtained only for $k_F = 1.607 \text{ \AA}^{-1}$ and by attributing the satellite lines a , b , and c to the first, second, and fourth coordination spheres, respectively (Fig. 5).

2.3. Compound $\text{Pr}_3\text{Rh}_4\text{Sn}_{13}$

We shall now estimate the enhancement of the external magnetic field at the nuclei of Pr^{3+} :

$$\alpha = 1 + K, \quad (9)$$

where

$$K = (A_J/N_A g_J \beta g_N \beta_N) \chi \quad (10)$$

($g_N = 1.64$ is the nuclear g factor and $A_J = 52.4 \text{ mK}$ is the hyperfine interaction constant of the Pr^{3+} ions—see Ref. 16). Substituting the calculated values $\chi_{\perp} = 0.47 \times 10^{-4} \text{ cm}^3/\text{g}$ and $\chi_{\parallel} = 2.8 \times 10^{-4} \text{ cm}^3/\text{g}$ (at $T = 4.2 \text{ K}$), we obtain $K_{\perp} = 7.2$ and $K_{\parallel} = 43.6$. In the limit $T \rightarrow 0$ the population of the magnetic doublet located 3.7 K above the ground state of Pr^{3+} is negligible and the susceptibility becomes of purely Van Vleck type, where $\chi_{\perp}^{\text{VV}} = 1.18 \times 10^{-4} \text{ cm}^3/\text{g}$ and $\chi_{\parallel}^{\text{VV}} = 0.46 \times 10^{-4} \text{ cm}^3/\text{g}$. The coefficients representing the enhancement of the external magnetic field at the nuclei of the Pr^{3+} ions are then $\alpha_{\perp} = 19.3$ and $\alpha_{\parallel} = 8.2$ near absolute zero. These values exceed the corresponding parameters of

PrNi₅ (Ref. 17) for which the lowest temperature was obtained by the adiabatic demagnetization method.¹⁸ Moreover, since the exchange interactions in Pr₃Rh₄Sn₁₃ make practically no contribution to the susceptibility,³ whereas in the case of PrNi₅ this contribution is ~20%, we can expect the nuclear magnetic ordering temperature T_c of the compound we have investigated to be even lower than that estimated for PrNi₅ ($T_c \approx 0.32$ mK is given in Ref. 17). A similar estimate, obtained for Pr₃Rh₄Sn₁₃ on the assumption that the exchange interactions contribute no more than 5% to the susceptibility, gives $T_c \lesssim 0.19$ mK. Consequently, we can expect the compound Pr₃Rh₄Sn₁₃ to be very effective in the attainment of ultralow temperatures by the adiabatic demagnetization method.

Our estimates of K_{\perp} and K_{\parallel} at liquid helium temperatures show that for rf field frequencies ~15 MHz we can expect the appearance of the NMR absorption line of the ¹⁴¹Pr nuclei in fields of the order of several hundreds gauss. The signal was not observed, apparently because in the investigated range of temperatures the low-lying magnetic doublet was still populated; hence the fluctuation contribution of the ¹⁴¹Pr ions to the width of the NMR line was so large that the line was smeared out. This was probably also the reason why the NMR signal of the ¹¹⁹Sn nuclei was not observed either.

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