

Evidence for *s*-wave superconductivity in the β -pyrochlore oxide RbOs_2O_6

K. Magishi,^{1,2} J. L. Gavilano,¹ B. Pedrini,¹ J. Hinderer,¹ M. Weller,¹ H. R. Ott,¹ S. M. Kazakov,¹ and J. Karpinski¹

¹Laboratorium für Festkörperphysik, ETH-Hönggerberg, CH-8093 Zürich, Switzerland

²Faculty of Integrated Arts and Sciences, The University of Tokushima, Tokushima 770-8502, Japan

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We report the results of ^{87}Rb NMR measurements on RbOs_2O_6 , a member of the family of the superconducting pyrochlore-type oxides with a critical temperature $T_c=6.4$ K. In the normal state, the nuclear spin-lattice relaxation time T_1 obeys the Korringa-type relation $T_1T=\text{const}$ and the Knight shift is independent of temperature, indicating the absence of strong magnetic correlations. In the superconducting state, $T_1^{-1}(T)$ exhibits a tiny coherence enhancement just below T_c , and decreases exponentially with further decreasing temperatures. The value of the corresponding energy gap is close to that predicted by the conventional weak-coupling BCS theory. Our results indicate that RbOs_2O_6 is a conventional *s*-wave-type superconductor.

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Geometrical frustration of spin systems has recently attracted much attention because, instead of long-range magnetic order, novel ground states, including unconventional superconductivity, may be adopted.^{1,2} Pyrochlore-type oxides with tetrahedral networks of magnetic ions, the so-called pyrochlore lattice, are well known physical realizations of geometrically frustrated magnetic systems. This class of materials has recently received enhanced attention because superconductivity was found in $\text{Cd}_2\text{Re}_2\text{O}_7$ below $T_c=1$ K.³⁻⁵ The mechanism causing this superconductivity appears to be conventional and the physical properties are compatible with expectations based on the weak-coupling BCS theory.⁶ It was suggested that the onset of superconductivity might be related to an unusual structural phase transition.^{7,8} Another superconducting pyrochlore, KOs_2O_6 , seems to be a quite different case. This material crystallizes in the β -pyrochlore structure and exhibits superconductivity below $T_c=9.6$ K.⁹ In contrast to $\text{Cd}_2\text{Re}_2\text{O}_7$, KOs_2O_6 does not exhibit any structural phase transition near T_c . This suggests that the geometrical frustration persists to temperatures below T_c . The upper critical magnetic field H_{c2} appears to exceed the Pauli limit expected for conventional superconductivity. This observation was interpreted as a sign for unconventional superconductivity in KOs_2O_6 .¹⁰ The results of recent muon spin rotation (μSR) experiments were interpreted as to strongly suggest that the superconducting state of KOs_2O_6 is unconventional, characterized by gap nodes.¹¹ Analogous experiments invoking $\text{Cd}_2\text{Re}_2\text{O}_7$ (Refs. 12 and 13) revealed an isotropic gap for that material, instead.

Very recently, a ternary compound RbOs_2O_6 with the β -pyrochlore structure, the same as KOs_2O_6 , has been discovered.¹⁴ This compound exhibits superconductivity below $T_c=6.4$ K.^{14,15} The results of recent specific-heat¹⁵ and magnetic-field penetration depth measurements¹⁶ were claimed to indicate a conventional BCS-type behavior of this superconducting state. The zero-temperature upper critical magnetic field H_{c2} (~ 6 T), extracted from specific-heat measurements on RbOs_2O_6 , is lower than the Pauli limiting field,¹⁵ in contrast to the above cited claims for KOs_2O_6 . From the results of electrical resistivity measurements, however, the value of H_{c2} for RbOs_2O_6 at zero temperature was

claimed to be larger than the Pauli limit of 12 T.¹⁴ This estimated value for the Pauli limit may, however, be substantially modified toward higher values by spin-orbit interactions, as suggested by the results of band-structure calculations for the related pyrochlore-type oxides $\text{Cd}_2\text{Re}_2\text{O}_7$ and $\text{Cd}_2\text{Os}_2\text{O}_7$.¹⁷

In this paper, we present the results of magnetic susceptibility and ^{87}Rb NMR measurements on samples of polycrystalline RbOs_2O_6 in both the normal and the superconducting state. Our results support the view of conventional superconductivity in this material.

The samples were prepared from polycrystalline material of RbOs_2O_6 , synthesized from the starting materials OsO_2 and Rb_2O . The experimental details of the synthesis and the purification of RbOs_2O_6 are described elsewhere.¹⁸ The material was confirmed to have the correct structure by x-ray diffraction. Most of the reflections could be indexed on the basis of a pyrochlore unit cell with a lattice parameter $a=10.1137(1)$ Å. A small amount of OsO_2 (less than 5 %) was detected as an impurity. Our NMR experiments probe the local environment of the Rb ions. The Rb cations occupy the $8b$ site in the pyrochlore lattice, which provides a local environment with cubic symmetry.

The magnetic susceptibility $\chi(T)=M(T)/H$, where $M(T)$ represents the temperature-dependent magnetization, was measured upon cooling the sample at temperatures between 300 and 2 K in external magnetic fields μ_0H of 50 G and 2.94 T, using a SQUID magnetometer. The NMR measurements were performed at temperatures between 0.4 and 35 K in an external magnetic field of 2.9427 T using a standard phase-coherent-type pulsed spectrometer. The ^{87}Rb NMR spectra were obtained by fast Fourier transformation (FFT) of the spin-echo signals, following a $\pi/2-\pi$ rf pulse sequence. The nuclear spin-lattice relaxation time T_1 was measured by the saturation recovery method, where the spin-echo signals were measured after the application of a comb of rf pulses and subsequent variable delays.

Figures 1(a) and 1(b) show the temperature dependences of the magnetic susceptibility $\chi(T)$ of a powdered sample of RbOs_2O_6 . The superconducting transition is reflected in the onset of a large diamagnetic signal due to the Meissner effect

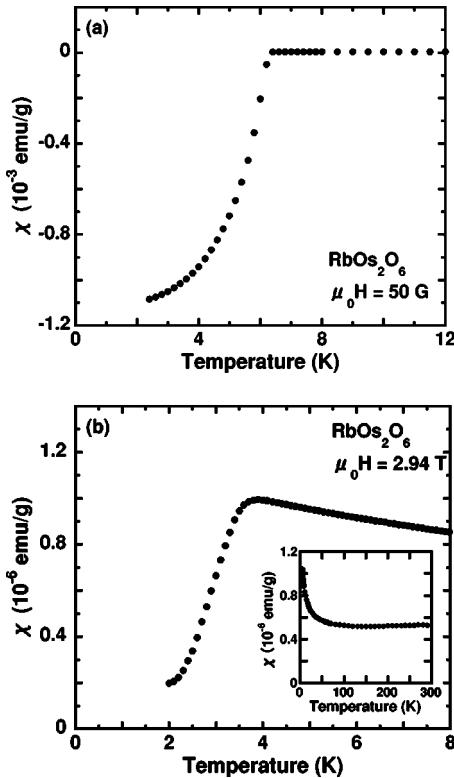


FIG. 1. Temperature dependences of the magnetic susceptibility $\chi(T)$ measured on a powdered sample of RbOs_2O_6 in external magnetic fields of (a) 50 G and (b) 2.94 T. Note the very different vertical scales. The sample was cooled in the field. Inset: Temperature dependence of $\chi(T)$ above T_c in an external magnetic field of 2.94 T.

[Fig. 1(a)]. In case of $\mu_0 H = 50$ G, $\chi(T)$ reflects the onset of diamagnetism at 6.4 K. As usual, increasing external magnetic fields shift the transition to lower temperatures.^{14,15} In an external magnetic field $\mu_0 H = 2.94$ T, the same that we used in our NMR measurements, the $\chi(T)$ data reveal the onset of superconductivity at $T_c = 3.8$ K [Fig. 1(b)]. This value is consistent with the results of previous specific-heat measurements.¹⁵ The inset of Fig. 1(b) shows the temperature dependence of $\chi(T)$ of RbOs_2O_6 in the normal state for $\mu_0 H = 2.94$ T. At temperatures exceeding 100 K, $\chi(T)$ is, to a good approximation, temperature independent. Upon cooling to below 50 K, the susceptibility increases gradually, such that $\chi(T) = \chi_0 + C/T$, where C is the Curie constant and $\chi_0 = 4.8 \times 10^{-7}$ emu/g is the temperature-independent susceptibility. For common metals, $\chi_0 = \chi_{\text{Pauli}} + \chi_{\text{Landau}} + \chi_{\text{shell}}$, where for free electrons $\chi_{\text{Landau}} = -\frac{1}{3}\chi_{\text{Pauli}}$. Assuming that the core-electron diamagnetism term χ_{shell} is negligibly small, $\chi_{\text{Pauli}} = \frac{3}{2}\chi_0$ and we can calculate the electronic density of states at the Fermi surface from $\chi_{\text{Pauli}} = \mu_B^2 D(E_F)$. Using this relation, we obtain $D(E_F) = 1.38$ states/eV atom. This value of $D(E_F)$ implies that the electronic specific-heat coefficient $\gamma = \pi^2 k_B^2 D(E_F) / 3 = 31$ mJ mol⁻¹ K⁻², in very good agreement with the value estimated from the result of specific-heat measurements.¹⁵ The Curie-type upturn of $\chi(T)$ at low temperatures is attributed to the presence of a small concentration of impurity moments. The effective paramagnetic mo-

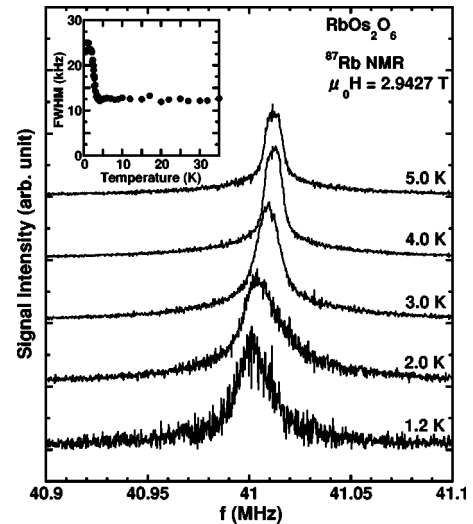


FIG. 2. Evolution of the FFT spectra of ^{87}Rb NMR in an external magnetic field of 2.9427 T between 1.2 and 5.0 K. Inset: Temperature dependence of the full width at half maximum (FWHM) of the signal.

ment deduced from the Curie constant is small, of the order of $0.1 \mu_B/\text{Os}$. Thus, the intrinsic behavior of $\chi(T)$ of RbOs_2O_6 in the normal state is that of a simple metal, consistent with the results of measurements of the Knight shift and the nuclear spin-lattice relaxation rate, to be discussed below.

Figure 2 shows the evolution of the FFT spectra of ^{87}Rb NMR at low temperatures. The spectrum contains a single resonance line. As described above, the Rb nuclei occupy highly symmetrical sites and thus, the influence of the quadrupole interaction is quenched ($I = \frac{3}{2}$ for the ^{87}Rb nucleus). The inset of Fig. 2 shows the temperature dependence of the full width at half maximum (FWHM) of the ^{87}Rb NMR spectrum. In the normal state, this width is of the order of 12 kHz and independent of temperature. In the superconducting state, the NMR spectrum broadens appreciably due to a distribution of local fields produced by the vortex lattice. The inhomogeneous broadening of the NMR line of type-II superconductors can approximately be calculated as $\Gamma \sim \phi_0 / \lambda^2 (16\pi^3)^{1/2}$, the square root of the second moment of the expected field distribution due to the vortices.¹⁹ With $\lambda = 4100$ Å as the zero-temperature London penetration depth¹⁶ and $\phi_0 = hc/2e$ as the flux quantum, we calculate $\Gamma \sim 5.5$ Oe ~ 8 kHz at $T = 0$ K. This value is close to the observed total enhancement of the line width of approximately 12 kHz.

Next, we consider the nuclear spin-lattice relaxation. Figure 3 shows the temperature dependence of $(T_1 T)^{-1}$. The T_1 measurements were made at the peak positions of the resonance signals, but the employed rf pulses were short enough to irradiate the entire NMR line. The observed magnetization recovery (data not shown) followed a single-exponential curve.

In the normal state, $T_1^{-1}(T)$ obeys the Korringa relation $(T_1 T)_n^{-1} = 0.117$ (s K)⁻¹, as expected for simple metals and indicating the absence of significant magnetic interactions in

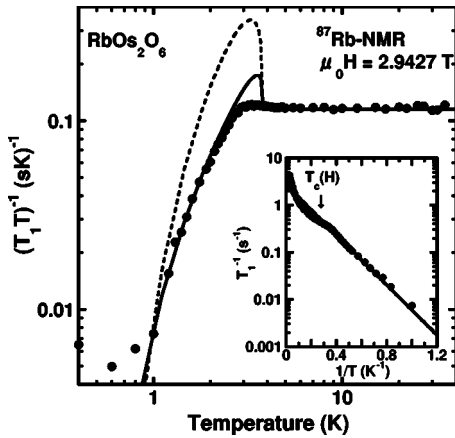


FIG. 3. Temperature dependence of $(T_1T)^{-1}$. Dotted and solid lines represent the BCS expectation for an isotropic and anisotropic gap, respectively (see text). Inset: Semilogarithmic plot of T_1^{-1} vs $1/T$. The solid line represents the relation $T_1^{-1} \propto \exp[-\Delta(0)/k_B T]$ with $\Delta(0)/k_B = 6.1$ K.

RbOs₂O₆. Recently, it was reported that T_1^{-1} of ³⁹K nuclear spins in KOs₂O₆ (Ref. 20) exhibits an unusual temperature dependence in the normal state. This was interpreted as evidence for considerable antiferromagnetic correlations in the itinerant electron system. Assuming that both data sets are reliable, it must be concluded that the magnetic features of RbOs₂O₆ are quite different from those of KOs₂O₆.

In the superconducting state, T_1^{-1} reveals no clear coherence peak just below T_c ($=3.8$ K for $\mu_0 H = 2.94$ T) but drops sharply only below 3 K, i.e., at a temperature significantly lower than T_c . The maximum value of $(T_1T)^{-1}$ below T_c is only 4% larger than the value in the normal state. A similar result has recently been obtained by other workers.²⁰ This behavior is distinctly different from that of well-identified unconventional superconductors where T_1^{-1} drops sharply just below T_c . We argue that the superconducting state of RbOs₂O₆ is of conventional type and that the data between 3 K and $T_c(H)$ reflect a strongly reduced coherence peak in $T_1^{-1}(T)$. The coherence peaks for *s*-wave type-II superconductors are often reduced for various reasons. In the case of V₃Sn,²¹ it was argued that the application of an external magnetic field causes the observed reduction. The coherence peak may also be suppressed by finite lifetime effects on the quasiparticles due to, for instance, electron-phonon interactions.²² Fibich showed that an effective broadening of the electronic energy levels is brought about by absorption processes of thermal phonons. More accurately, the gap function has a (negative) imaginary part, which has the effect of removing the singularity in the electrical density of states at nonzero temperature. Although these lifetime effects are expected to be particularly important for strong-coupling superconductors, the mechanism was originally invoked in order to explain the only modest enhancement of T_1^{-1} below T_c in the case of Al. Fibich has derived the ratio T_{1n}/T_{1s} of the relaxation rates in the superconducting state to those in the normal state as

$$\frac{T_{1n}}{T_{1s}} = 2f(\Delta_1) \left[1 + \frac{\Delta_1(T)}{k_B T} [1 - f(\Delta_1)] \ln \left(\frac{2\Delta_1(T)}{|\Delta_2(T)|} \right) \right], \quad (1)$$

with

$$\frac{\Delta_2(T)}{\Delta_0} = C \left[\frac{\Delta_0}{\Delta_1(T)} \right]^{1/3} \left(\frac{T}{T_c} \right)^{8/3}, \quad (2)$$

where $\Delta_1(T)$ and $\Delta_2(T)$ are the real and the imaginary parts of the gap function, f is the Fermi distribution function, and C is a fitting parameter. Our simulations (data not shown) using Eq. (1) do not yield satisfactory results for the case of RbOs₂O₆, however, and we conclude that this type of reasoning is not adequate for explaining the only weakly developed coherence peak in $T_1^{-1}(T)$ in RbOs₂O₆.

In an attempt to elucidate the reduction of the coherence peak in T_1^{-1} below T_c , we tried to consider the effect of the applied magnetic field using the approach suggested by Goldberg and Weger.²³ Here, the basic assumption is that the total nuclear spin-lattice relaxation rate is the sum of two terms, where the first describes the relaxation in the normal-state vortex cores and the other captures T_1^{-1} in the remaining superconducting volume. Near T_c this leads to

$$(T_1T)^{-1} = (T_1T)_n^{-1} \frac{H\xi^2}{\Phi} + (T_1T)_{\text{BCS}}^{-1} \left(1 - \frac{H\xi^2}{\Phi} \right), \quad (3)$$

where $(T_1T)_n^{-1} = 0.117$ (s K)⁻¹. The coherence length is given by

$$\xi(T) = \frac{0.74\xi(0)}{\left(1 - \frac{T}{T_c(H)} \right)^{1/2}}$$

with $\xi(0) = 74$ Å,¹⁵ and $(T_1T)_{\text{BCS}}^{-1}$ represents the relaxation in the superconducting volume. Inserting the parameters for RbOs₂O₆, the first term on the r.h.s. of Eq. (3) turns out to be negligibly small, and hence for the field strength used in our experiments, the influence of the applied magnetic field in the manner described above plays no role in our problem. This may not be the case for higher applied magnetic fields, however.

At very low temperatures and high magnetic fields, yet another process, namely, spin diffusion, will play a dominant role. In this case, the observed relaxation rate T_1^{-1} will be dominated by fast processes in the normal core of the vortices. Note that due to the small value of ξ at low temperatures, finite-size effects may have to be taken into account in the vortex cores and the corresponding relaxation rate may not simply be given by $(T_1)_n^{-1}$ of the bulk in the normal state.

Below 3 K, T_1^{-1} decreases exponentially with temperature upon cooling. Below 1 K, where T_1^{-1} is less than 7×10^{-3} s⁻¹, the relaxation tends toward a temperature-independent value with decreasing temperature. This deviation may be caused by paramagnetic impurities and/or by relaxation via spin diffusion to the normal vortex cores, which are regions of fast relaxation. The onset of anomalous relaxation follows a more than an order of magnitude reduction of T_1^{-1} , indicating that the bulk of the sample ($\geq 95\%$) is indeed superconducting. Our results for the spin-lattice re-

laxation for $T < 1$ K were found not to be sufficiently reliable to allow for a detailed analysis. Because of the long T_1 's involved we see no simple way for improving their quality. Therefore, in our subsequent analysis, we refrain from discussing the data for $T \leq 1$ K. Likewise, the potentially interesting investigation of $T_1^{-1}(T)$ at much lower applied magnetic fields had to be abandoned because of poor signal-to-noise ratios.

The inset of Fig. 3 shows a semilogarithmic plot of T_1^{-1} vs $1/T$, which confirms the exponential decay of T_1 according to $T_1^{-1} \propto \exp[-\Delta(0)/k_B T]$ at temperatures well below T_c . The slope of the solid line implies that $\Delta(0)/k_B = 6.1$ K. With $T_c = 3.8$ K, we obtain $2\Delta(0)/k_B T_c = 3.2$. This is in fair agreement with the value of the conventional BCS theory in the weak-coupling regime, $2\Delta(0)/k_B T_c = 3.5$. This finding, however, should be considered with some caution because, as we shall see below, a more detailed inspection of the data suggests a considerable anisotropy in the gap parameter. Nevertheless, the observed thermally activated temperature dependence of T_1^{-1} provides clear evidence for a nodeless gap configuration and the remnants of the coherence peak just below T_c indicate a conventional s -wave-type pairing of the quasiparticles. This is consistent with previous interpretations of results of specific-heat¹⁵ and magnetic-field penetration depth measurements.¹⁶

In order to reproduce $T_1^{-1}(T)$ at intermediate temperatures below T_c , we fitted the data to the BCS model and assumed a distribution of energy gap amplitudes in the range between $\Delta - \delta$ and $\Delta + \delta$ across the Fermi surface.²⁴ The solid line below T_c in the mainframe of Fig. 3 represents the result of a calculation using $2\Delta(0)/k_B T_c = 3.5$ and $\delta/\Delta(T) = 0.5$. In this way, we obtain good agreement with the experimental results at temperatures between 1 and 3 K. The calculation cannot reproduce the behavior just below T_c quantitatively. The distribution of the energy gap can be accounted for by incorporating a k -space anisotropy of the conventional s -wave gap,²¹

$$\Delta(H, \Omega) = \langle \Delta(H) \rangle [1 + a(\Omega)], \quad (4)$$

where Ω is the solid angle in k space, $\langle \Delta(H) \rangle$ is the mean gap value over all orientations in k space, and $a(\Omega)$ is the anisotropy function satisfying the condition $\langle a(\Omega) \rangle = 0$. Our value for $\delta/\Delta(T)$ implies $\langle a^2(\Omega) \rangle = 0.25$. This anisotropy can substantially affect various thermodynamic quantities. For instance, it has been shown^{25,26} that it renormalizes the ratio $\Delta/k_B T_c$ as $\Delta/k_B T_c = (\Delta^0/k_B T_c^0)(1 - \frac{3}{2}\langle a^2 \rangle)$, where $\Delta^0/k_B T_c^0$ is the ratio in the absence of anisotropy. In our case, since $2\Delta/k_B T_c = 3.2$ and $\langle a^2(\Omega) \rangle = 0.25$, it follows that $\Delta^0/k_B T_c^0 = 5.1$, a substantially enhanced value with respect to the expectations for the BCS theory in the weak-coupling limit, and raising some questions about the validity of the approximation. A refined quantitative discussion of the NMR data obviously requires additional efforts in numerical calculations.

Figure 4 shows the temperature dependence of the Knight shift of the ^{87}Rb NMR signal. The Knight shift is a measure of the uniform magnetic susceptibility of the conduction electrons, seen at a particular nuclear site. In general, the Knight shift consists of a T -independent orbital part and a

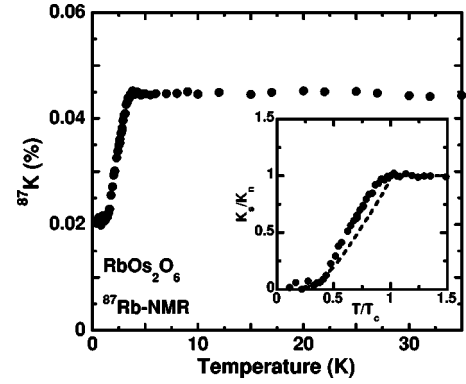


FIG. 4. Temperature dependence of the Knight shift of the ^{87}Rb NMR signal. Inset: Temperature dependence of the Knight shift K_s , normalized by its normal-state value at T_c . The dotted line is a calculation based on the conventional BCS theory with the same parameters as those employed in the analysis of T_1^{-1} below T_c .

spin part. In the normal state of RbOs_2O_6 , the Knight shift is very small, about 0.045%, and practically independent of temperature, reflecting the influence of the temperature-independent contribution of the electronic spin susceptibility. In the superconducting state, the diamagnetic shift H_{dia} is estimated²⁷ as

$$-\frac{H_{c1} \ln(\beta d / \sqrt{e} \xi)}{\ln(\lambda / \xi)}$$

With $\xi = 74$ Å,¹⁵ $\lambda = 4100$ Å,¹⁶ $\beta = 0.381$ for the triangular vortex lattice, and $d = 285$ Å as the nearest-neighbor vortex-lattice spacing in 2.9427 T, we calculate $K_{\text{dia}} = -0.004\%$. The observed Knight shift variation below T_c is much larger than K_{dia} , thus reflecting the spin-singlet-pairing of the quasiparticles. The residual line shift at very low temperatures is of the order of 0.020%.

The inset of Fig. 4 shows the temperature dependence of the Knight shift K_s normalized by its value at T_c . Here, the residual line shift attributed to orbital effects has been subtracted from the raw data. The dotted line represents the result of a calculation using the conventional BCS model with the same parameters and the same gap distribution as those in the previously discussed analysis of $T_1^{-1}(T)$ below T_c . Because the calculation does not agree with the experimental data, the deviation of the temperature dependence of the Knight shift below T_c from the BCS expectation needs further examination.

The ratio $K_\alpha = S/T_1 T K_s^2$ in the normal state provides a useful measure for the importance of electron-electron magnetic correlations.^{28,29} The parameter

$$S = \frac{\gamma_e^2 \hbar}{\gamma_n^2 4\pi k_B}$$

and γ_e and γ_n are the electronic and nuclear gyromagnetic ratios, respectively. Depending on the value of K_α being much smaller or larger than unity, substantial ferro- or antiferromagnetic correlations in the itinerant electron systems are significant. If we assume that the residual line shift $K(T=0)$ is due to the orbital contributions, K_α is estimated to

be 4.6, providing some evidence for the existence of antiferromagnetic electron–electron correlations.

We present and discuss the results of ^{87}Rb NMR measurements on the superconducting pyrochlore-type oxide RbOs_2O_6 . In the normal state, the nuclear spin-lattice relaxation rate T_1^{-1} obeys the Korringa-type relation $(T_1 T)^{-1} = 0.117(\text{s K})^{-1}$ and the line shift is independent of temperature. In the superconducting state, T_1^{-1} reveals a very much reduced coherence peak just below T_c , and eventually decreases with a thermally activated behavior upon further

cooling. The $T_1^{-1}(T)$ data can qualitatively be explained by the BCS model considering some anisotropy of the gap function. In spite of some remaining numerical inconsistencies, we claim that our NMR results imply that the superconducting state of RbOs_2O_6 is characterized by singlet pairing of the electrons and that the gap function exhibits a conventional *s*-wave-type symmetry; with some *k*-dependent variation of the amplitude, however.

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