

Crystal Growth, Structure, Magnetic, and Transport Properties of TbRhIn₅Willa M. Williams,[†] Long Pham,[‡] Samuel MaQuilon,[‡] Monica Moldovan,[§] Zachary Fisk,[‡] David P. Young,[§] and Julia Y. Chan^{*,†}

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Single crystals of TbRhIn₅ were synthesized using the flux growth method. TbRhIn₅ adopts the HoCoGa₅ structure type and crystallizes in the space group *P4/mmm*, *Z* = 1. Lattice parameters are *a* = 4.6000(6) Å, *c* = 7.4370(11) Å, and *V* = 157.29(6) Å³. Transport measurements show that TbRhIn₅ is metallic (*dρ/dT* > 0). A sharp antiferromagnetic transition is observed at *T*_N = 48 K in the susceptibility data for TbRhIn₅, which is highly anisotropic when the field is oriented along the *c* axis and *a*–*b* plane of the crystal and has an average effective moment of 9.72 μ_B/Tb³⁺.

Introduction

Heavy fermion intermetallic compounds exhibit exotic physical properties caused by the interactions between their *f* electrons and conduction electrons.^{1–10} Heavy fermions show normal metallic behavior at room temperature, while at lower temperatures, the conduction electrons begin to screen the magnetic moment, resulting in effective masses approximately 2 orders of magnitude higher than that of a free electron. Since the effective mass of an electron is proportional to the electronic specific heat (*γ*), a large Sommerfeld coefficient (> 100 mJ/mol K²) may be observed.²

Recently, these compounds have been reviewed and summarized.¹¹ Heavy fermion compounds are typically cerium-, ytterbium-, or uranium-based intermetallic compounds.^{5,12–14} The rare earth ions in intermetallic compounds are well separated, so that any direct exchange between two neighboring *f* shells is negligible.^{15,16} Because of their metallic nature, however, the magnetic interaction between two such ions can take place via the polarization of the conduction band electrons, as in the case of the elemental rare-earth metals. This Ruderman–Kittel–Kasuya–Yoshida (RKKY) indirect exchange interaction is responsible for cooperative magnetic ordering.¹⁵ The competition between Ruderman–Kittel–Kasuya–Yoshida (RKKY) interactions and the Kondo effect (the progressive screening of the magnetic moments by the conduction electrons at low temperatures) is important because the heavy fermion state is formed when the Kondo effect overcomes the RKKY interaction.¹⁷

CeMIn₅ (M = Co, Rh, Ir) is a special class of heavy fermion materials which show magnetic ordering and unconventional superconductivity at low temperatures.^{18,19}

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The coexistence of magnetism and superconductivity is quite unusual and, in fact, is magnetically mediated. Heavy fermion intermetallic compounds which show both magnetic ordering and superconductivity are of interest because they present the opportunity to study the competition or coexistence between the two mechanisms.

The crystal structure of CeMIn_5 ($M = \text{Co, Rh, Ir}$),^{20,21} which adopts the HoCoGa_5 -structure type,²² consists of alternating layers of CeIn_3 and MIn_2 layers stacked along the c axis. Bulk CeIn_3 is a heavy fermion antiferromagnet which exhibits pressure-induced superconductivity.²³ CeCoIn_5 ($\gamma \approx 290$ mJ/mol of Ce K^2)²⁴ under ambient conditions, has the highest superconducting transition temperature ($T_c = 2.3$ K) reported for any heavy fermion compound.¹⁸ The magnetization of CeCoIn_5 is highly anisotropic, exhibiting a weak metamagnetic transition around 4.2 T along the c axis, while it gradually increases along the a - b plane.²⁵ CeRhIn_5 orders antiferromagnetically at $T_N = 3.8$ K and becomes superconducting at 2 K upon the application of > 16 kbar of pressure with a $\gamma \approx 420$ mJ/mol of Ce K^2 .¹⁸ CeIrIn_5 , under ambient conditions, has the largest Sommerfeld coefficient for the series with $\gamma \approx 750$ mJ/mol of Ce K^2 .²⁶ The superconducting temperature of CeIrIn_5 is 0.4 K, however there is a resistivity drop at 1.2 K, of which there is debate about the mechanism responsible for the decrease in resistivity.¹⁸ Upon the application of pressure, the transition temperature at 0.4 K increases to a maximum value of ~ 1 K at approximately 15 kbar. The highest ordering temperatures reported for this class of compounds are those observed in GdRhIn_5 and GdIrIn_5 which order antiferromagnetically at Néel temperatures of 40 and 42 K, respectively.^{27,28} Reduced spatial dimensionality and magnetic anisotropy, as a function of the rare-earth element, have been observed in LnRhIn_5 ($\text{Ln} = \text{Ce, Nd, Sm, Gd}$). In an effort to further study the effects of magnetic anisotropy in Kondo systems, we were prompted to study TbRhIn_5 . In this

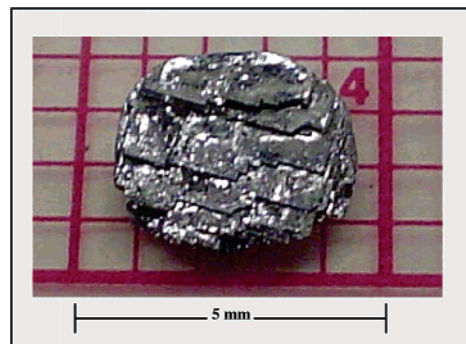


Figure 1. Aggregate of TbRhIn_5 (~ 4 mm).

Table 1. Crystallographic Parameters of TbRhIn_5

a (Å)	4.6000(6)
c (Å)	7.4370(11)
V (Å ³)	157.29(6)
cryst syst	tetragonal
Z	1
cryst dimension (mm ³)	0.075 \times 0.025 \times 0.025
space group	$P4/mmm$
θ range (deg)	2.5–30.0
μ (mm ⁻¹)	31.481
measured refls	742
independent refls	244
reflns with $I > 2\sigma(I)$	163
R_{int}	0.097
h, k, l	$\pm 7, \pm 5, \pm 11$
$^a R1$ [$F^2 > 2\sigma(F^2)$]	0.0432
$^b wR2$ (F^2)	0.0833
params	12
$\Delta\rho_{\text{max}}$ (e Å ⁻³), $\Delta\rho_{\text{min}}$ (e Å ⁻³)	3.24, -4.52
extinction coeff	0.0066(7)

$$^a R1 = \sum ||F_o| - |F_c|| / \sum |F_o|. \quad ^b wR2 = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}.$$

manuscript we compare the structure, transport, and physical properties of single crystals of TbRhIn_5 with other magnetic analogues, CeRhIn_5 , SmRhIn_5 , NdRhIn_5 , and GdRhIn_5 .

Experimental Section

Synthesis. Tb pieces, Rh powder, and In shot (Alfa Aesar), all with stated purities of $\geq 99.9\%$, were combined in an atomic ratio of 1:1:20. The starting materials were then placed into an alumina crucible and sealed in an evacuated fused silica tube. The sealed sample was then gradually heated from room temperature to 1373 K at a rate of 473 K/h for 2 h, then slowly cooled at 281 K/h to 923 K, at which point the excess flux was removed via centrifugation. Synthesis yielded aggregates of layered crystals exhibiting a metallic luster as shown in Figure 1.

Single-Crystal X-ray Diffraction. A $0.025 \times 0.025 \times 0.075$ mm³ single-crystal fragment was placed on a glass fiber and mounted on the goniometer of a Nonius Kappa CCD diffractometer equipped with Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). Data were collected at 293(2) K. Additional data collection and crystallographic parameters are presented in Table 1.

The structures were solved with the SHELXL software package²⁹ using CeRhIn_5 as a structural model. The atomic displacement parameters were treated anisotropically, and an extinction coefficient was applied to the data after a final least-squares cycle. The atomic coordinates and displacement parameters are provided in Table 2, and selected interatomic distances are listed in Table 3.

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Table 2. Atomic Positions and Thermal Parameters of TbRhIn₅

		<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} ^a (Å ²)
Tb	1a	0	0	0	0.014(3)
Rh	1b	0	0	1/2	0.015(2)
In1	4i	1/2	1/2	0	0.018(4)
In2	1c	0	1/2	0.3015(8)	0.017(3)

^a *U*_{eq} is defined as one-third of the trace of the orthogonalized *U*_{ij} tensor.

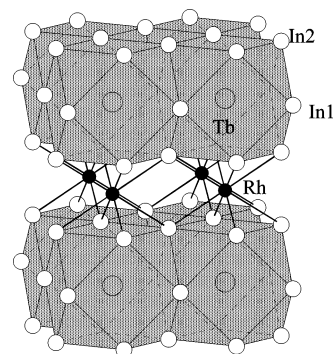
Table 3. Selected Interatomic Distances (Å) and Bond Angles (deg) of TbRhIn₅

within cuboctahedra		
In1–In2		3.2140(12)
In1–In1		3.2527(4)
Tb–In1 (×4)		3.2140(12)
Tb–In2 (×8)		3.2527(4)
In1–Tb–In1		90
In1–Tb–In2		60.960(8)
In1–Tb–In2		119.040(8)
In2–Tb–In2		88.330(2)
In2–Tb–In2		59.520(4)
In2–Tb–In2		120.480(4)
within rectangular polyhedron		
In2–In2 (<i>c</i> axis)		2.9470(3)
In2–In2 (<i>a–b</i> plane)		3.2562(7)
Rh–In2 (×8)		2.7316(9)
In2–Rh–In2		73.130(13)
In2–Rh–In2		65.200(2)

Physical Property Measurements. Magnetic properties were measured on single crystals using a Quantum Design (SQUID) magnetometer. The temperature-dependent susceptibility was measured in an applied field of 1000 G up to room temperature after being cooled to 2 K under zero magnetic field. Field-dependent magnetization data were also collected from zero field to 10 T at 2 K. The resistivity (down to 2 K) data were measured using a standard four-probe method with a Quantum Design physical property measurement system (PPMS) at ambient pressure. Specific heat data was determined using the thermal transport option on the PPMS. The heat capacity of the TbRhIn₅ was measured at zero field in the temperature range of 300–0.36 K. Single crystals of the nonmagnetic analogue, LaRhIn₅ which were used for heat capacity measurements, were also grown using the flux method at Los Alamos National Lab (LANL).

Results and Discussion

TbRhIn₅ is isostructural to the CeMIn₅ (M = Co, Rh, Ir) compounds which adopt the HoCoGa₅-type structure (*P4/mmm*).²² The structure consists of four atoms in the asymmetrical unit: Tb, Rh, In1, and In2 atoms occupying the 1a, 1b, 1c, and 4i Wyckoff positions, respectively. Figure 2 shows the crystal structure of TbRhIn₅ which consists of alternating layers of TbIn₃ cuboctahedra and RhIn₂ rectangular prisms that contain two independent indium sites, In1 and In2. The coordination of Tb in the cuboctahedra is 8-fold to In1 and 4-fold to In2 with distances of 3.2140(12) and 3.2527(4) Å, respectively. These distances are in good agreement with the Tb–In interatomic distances in the binary compounds, Tb₂–In and TbIn₃, in which the Tb–In distances range from 3.025 to 3.359 Å.³⁰

**Figure 2.** Structure of TbRhIn₅ consisting of layers of TbIn₃ cuboctahedra (gray), alternating with layers of RhIn₂, in which the Rh and In atoms are designated as black and white closed circles, respectively.

In CeCoIn₅, the cuboctahedra are elongated along the *c* axis, while a shortening of the *c* axis is observed in the Ir analogue. The ratio of Ce–In2/Ce–In1 in CeRhIn₅ is close to unity, indicating that the cuboctahedra are not distorted.³¹ The ratio of Tb–In2/Tb–In1 is 1.014, suggesting that the cuboctahedra in TbRhIn₅ are quite symmetrical.

The Rh atom is coordinated to eight In2 atoms and forms the edge of the neighboring rectangular prism. The Rh–In2 distance in TbRhIn₅ is 2.7316(9) Å and is comparable to the Rh–In2 distances of 2.7572(3) Å observed in LaRhIn₅, as well as the summation of the atomic radii for rhodium and indium.³¹ The In1–In2 and In1–In1 interatomic distances in TbRhIn₅ are 3.2140 (12) and 3.2527(4) Å, respectively, which are in good agreement with the values observed in RhIn₃³² and RhIn₃,^{33,34} ranging from 3.200 to 3.580 Å.

Physical Properties. The temperature dependence of the magnetic susceptibility of TbRhIn₅ is shown in Figure 3 for the field (1000 G) both along the *c* axis and in the *a–b* plane. A large anisotropy in the susceptibility data is observed. A sharp antiferromagnetic transition appears at 48 K. Above *T*_N, the inverse susceptibility obeys the Curie–Weiss law and is well fit by $[1/\chi(T) = (T - \theta)/C]$ in the temperature range of 80–300 K. We find an average effective moment of $\sim 9.72 \mu_B/\text{Tb}^{3+}$ ion along the *c* axis and the *a–b* plane with Weiss temperatures of $\theta = -75$ and -5 K, respectively. The effective moment is in agreement with the full Hund's moment for Tb³⁺ which is $9.72 \mu_B$. The negative θ values indicate antiferromagnetic correlations, which are quite strong along the *c* axis.

Figure 4 shows the field-dependent magnetization of TbRhIn₅ at 2 K along the *c* axis and the *a–b* plane up to 7 T. The magnetization increases with field up to 7 T with induced moments at the maximum field equal to 1.18 and $0.88 \mu_B$ for the *c* axis and *a–b* plane, respectively, indicating

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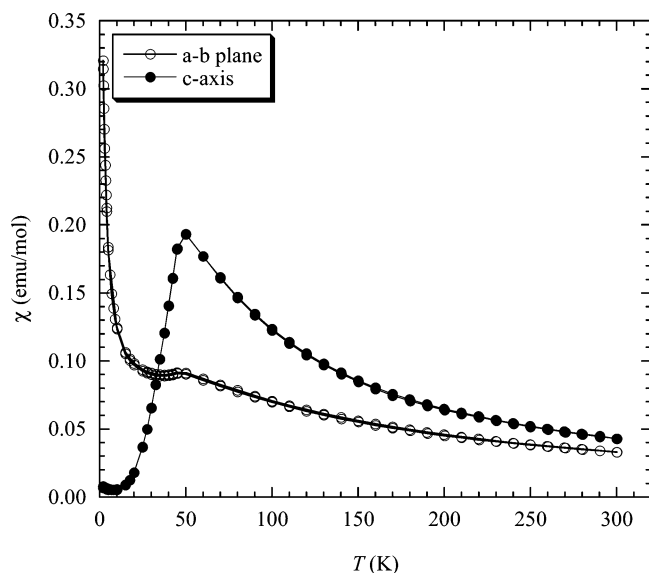


Figure 3. Temperature-dependent magnetic susceptibility (χ) of TbRhIn_5 along the a - b plane (open circles) and c axis (closed circles) measured at 1000 G.

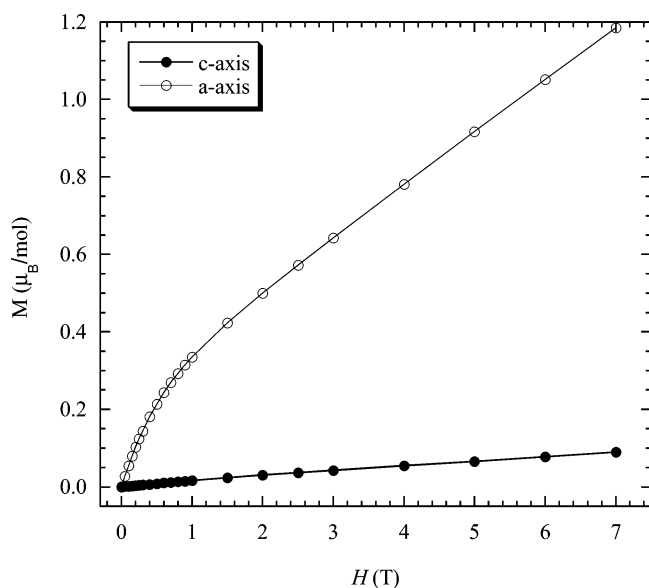


Figure 4. Field-dependent magnetization of TbRhIn_5 along the a - b plane (open circles) and c axis (closed circles) measured at 2 K.

that 7 T is not a significant field to induce saturation of the magnetic moments in TbRhIn_5 . These values are significantly smaller than the full Hund's saturation moment of $9 \mu_B$ expected for a Tb^{3+} ion.

The temperature dependence of the electrical resistivity of a single crystal of TbRhIn_5 is shown in Figure 5. TbRhIn_5 is metallic ($d\rho/dT > 0$) and has a residual resistivity ratio (RRR) of 6. A kink in the resistivity is observed near the ordering temperature at 48 K, consistent with a reduction in the spin-disorder scattering. Above T_N , the resistivity increases linearly with temperature. The small downturn at 3.4 is caused by some residual In flux in the sample.

Figure 6 shows the temperature dependence of the specific heat, C_p , for TbRhIn_5 . At zero field, a large cusp is observed at ~ 48 K which is consistent with the antiferromagnetic transition observed in the susceptibility. The specific heat

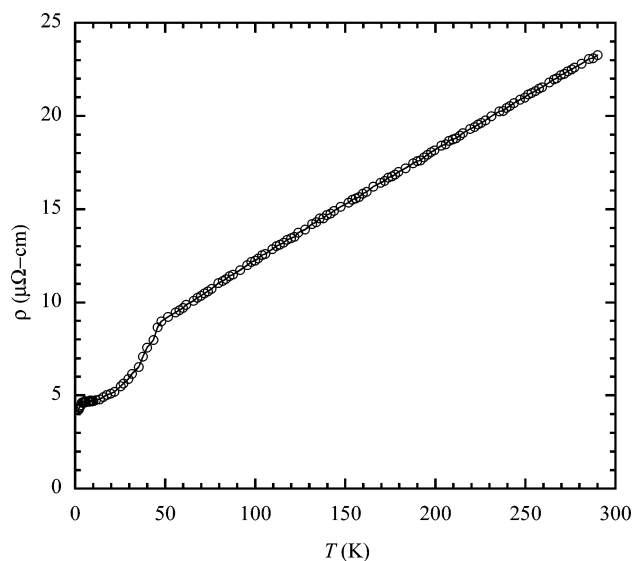


Figure 5. Temperature-dependent resistivity of TbRhIn_5 .

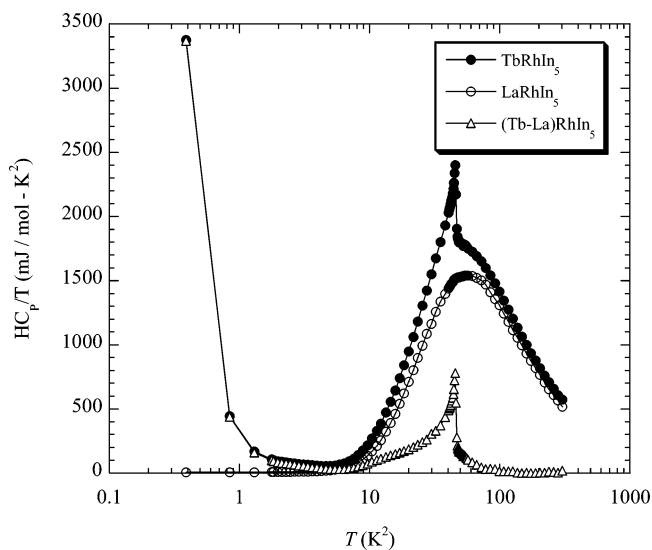


Figure 6. Specific heat of TbRhIn_5 (closed circles) and LaRhIn_5 (open circles). The f electron contribution of Tb is denoted with open triangles.

can be described by the equation $C_p = \gamma T + \alpha T^3$, where γ is the Sommerfeld coefficient and α is the phonon contribution to the total specific heat. The phonon contribution is negligible at low temperatures, which allows the electronic contribution to the specific heat to be determined experimentally. The f electron contribution to the specific heat, C/T_m (Figure 6), is obtained by subtracting the phonon contribution C/T of LaRhIn_5 . Since LaRhIn_5 does not contain any f electrons, it is a good approximation of the lattice contribution to the specific heat. The specific data in TbRhIn_5 is similar to other antiferromagnetic LnMIn_5 materials. Several mechanisms act simultaneously to produce the specific heat data as shown in Figure 6. There is a large nuclear Schottky contribution at low temperatures (below 2K). It results from the hyperfine interaction between the 4f electrons and the Tb^{3+} nuclei, which carry a nuclear spin moment of $I = 3/2$. There is a possible Schottky anomaly resulting from the crystalline electric field (CEF) at 11 K as shown in Figure 7, and there is a large peak at 48 K from

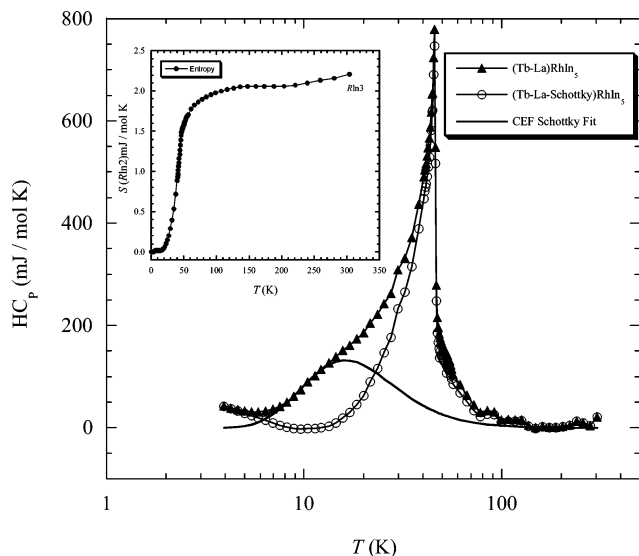


Figure 7. Specific heat of TbRhIn₅ after subtracting the lattice contribution (closed triangles), Schottky (line), and nuclear Schottky contributions (open circles). The entropy of TbRhIn₅ is shown in the inset.

the antiferromagnetic transition. The entropy is obtained by integrating C/T_m with respect to temperature. A value of $R \ln 3$ is recovered by the ordering temperature. Since the number of states, N , is determined by the entropy as $R \ln N$, this confirms that Tb is in a triplet ground state ($N = 3$).

In summary, TbRhIn₅ has been synthesized using flux methods and is isostructural to the well-studied CeRhIn₅. The magnetic moments of CeRhIn₅ form an incommensurate spiral along the c axis,^{26,35} and although CEF anisotropy energetically favors the moments to point along the c axis, the magnetic moments have been found to lie in the a – b plane.³⁶ Thus there may be competition between the two magnetic interactions, since we observe a 50% decrease in T_N for CeRhIn₅ in comparison to the parent compound CeIn₃.

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In contrast, the easy axis of magnetization in TbRhIn₅ ($T_N = 47$ K) is along the c axis; therefore, T_N is enhanced nearly 24% compared to that in TbIn₃ ($T_N = 36$ K).³⁷ In addition, the enhanced T_N indicates that RKKY interactions are more dominant than the Kondo effect in this compound because we observe more interaction between the uncompensated rare-earth ions. The magnetic susceptibility of GdRhIn₅ is only significantly anisotropic below T_N showing an easy axis of magnetization in the plane. Furthermore, CeRhIn₅ becomes superconducting under 16 kbar of mechanical pressure, but the superconducting state diminishes at ~ 25 kbar. The size of the atomic radii of Ce³⁺ versus Tb³⁺ decreases by $\sim 3.4\%$ because of lanthanide contraction. Multiplying 16 kbar by 3.4% gives an estimated molecular pressure for TbRhIn₅ of ~ 25 kbar, at which point the superconducting state diminishes in CeRhIn₅.²¹ The magnetic-ordering temperature of TbRhIn₅ scales in accordance with the de Gennes factor $[(gJ^2 - 1)][J(J + 1)]$ of LnRhIn₅ (Ln = Ce, Nd, Sm, Gd) for a ground-state multiplet, J , through the rare earths, with a T_N of 3.8–48 K for the Ce and Tb analogues. Although TbRhIn₅ is not a heavy fermion superconductor, it does have strong antiferromagnetic correlations resulting in an ordering temperature much higher than its heavy fermion analogue CeRhIn₅. It would be interesting to do a doping study by substituting Ce for Tb in TbRhIn₅ to observe how the heavy fermion superconducting state develops out of a strong antiferromagnet.

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Supporting Information Available: Crystallographic data in CIF format. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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