

^{237}Np Mössbauer studies on actinide superconductors and related materials

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Abstract Actinide materials play a special role in condensed matter physics, spanning behaviours of itinerant d -electron and localized $4f$ -electron materials. This duality of the $5f$ electrons confer to actinide-based intermetallic compounds a broad variety of physical properties such as magnetic or multipolar ordering, heavy fermion behaviour, quantum criticality, unconventional superconductivity... ^{237}Np Mössbauer spectroscopy is a unique microscopic tool for gaining information on the electronic and magnetic properties of Np systems.

Keywords Mössbauer spectroscopy · Neptunium · Actinides · Superconductivity

1 Introduction

Superconductivity was first reported in 1942 for uranium metal (α -U) [1] and in 1958 for U compounds [2]: UCo, U₆Mn, U₆Fe, and U₆Co, with critical temperatures T_c , of 1.7, 2.3, 3.9, and 2.3 K, respectively. A new class of U superconductors emerged in the early 1980's with the discovery of U heavy fermion (HF) superconductors (SC): UBe₁₃ ($T_c = 0.85$ K) [3], UPt₃ ($T_c = 0.53$ K) [4], URu₂Si₂ ($T_c = 1.5$ K) [5] or UPd₂Al₃ ($T_c = 1.9$ K) [6]... Furthermore, in most of these systems, the SC phases coexist with antiferromagnetic (AF) correlations which have characteristic temperatures, usually the Néel temperature T_N , that are typically one order of magnitude larger

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than the corresponding SC critical temperatures T_c . SC was even shown to co-exist with ferromagnetism in e.g. UGe_2 ($T_c \approx 0.8$ K, $T_C \approx 30$ K at $p \approx 1.2$ GPa) [7] and $URhGe$ ($T_c = 0.25$ K, $T_C = 9.5$ K) [8]. HF-SC still remain a major challenge for condensed matter physics. The existence of HF-SC and its coexistence or proximity with magnetic order suggests that the conventional mechanism of phonon-mediated SC is inappropriate and that alternative mechanisms, like spin fluctuations, should be considered for Cooper pairing.

Beside the large number of U superconductors, it is worth noticing the lack of transuranium superconductors: neither Np nor Pu metals are SC and, until a decade ago, no Np or Pu-based SC was reported (with the exception of Np Chevrel phases $Np_{1+x}Mo_6Se_8$ ($T_c = 5.6$ K) [9], where $5f$ electrons are thought not to be SC). The recent discovery of $5f$ electrons HF-SC in $PuCoGa_5$ [10], $PuRhGa_5$ [11] and $NpPd_5Al_2$ [12] has therefore been a major advance. Furthermore, their critical temperatures— $T_c \approx 18.5$, 8.7 and 4.9 K, respectively—are the highest of all f -electron systems.

Mössbauer effect can be observed in a number of actinide isotopes, however only the 60 keV electric dipole (E1) transition of the ^{237}Np isotope is suitable for systematic investigations, combining reasonable experimental constraints and exploitable output parameters [13]. Indeed, the excellent Mössbauer resonance of the ^{237}Np nucleus makes it a unique microscopic tool for gaining information on the electronic and magnetic properties of Np systems [14]. One consequence, however, of the rather high energy of the ^{237}Np Mössbauer transition, is the experimental limitation to fairly low temperatures, where the Lamb-Mössbauer factor is large enough to observe a sizeable effect.

In this paper, we review ^{237}Np Mössbauer spectroscopy (MoS) studies of the Np analogues of selected actinide superconductors: U heavy fermions, Pu “1:1:5” and the only Np-based SC known so far, $NpPd_5Al_2$.

2 Neptunium analogues of uranium heavy fermion superconductors

2.1 $AnBe_{13}$

UBe_{13} , one of the rare U-based HF-SC that does not show any AF ordering in the normal state, has a cubic crystal structure, a critical temperature $T_c = 0.85$ K, and a huge Sommerfeld coefficient of the specific heat $\gamma \approx 1,100$ mJ mole $^{-1}$ K $^{-2}$ [3].

Its isostructural homologue $NpBe_{13}$ also displays a huge quasi-particle mass enhancement $\gamma \approx 900$ mJ mole $^{-1}$ K $^{-2}$, but orders AF at 3.4 K and is not SC down to 0.080 K [15]. ^{237}Np Mössbauer measurements have shown that the low-temperature properties were sensitive to Be content: samples with excess Be were not magnetic, whereas optimal samples had $T_N \approx 4.9$ K [16]. In the ordered state, the fit of the Mössbauer spectra requires two sites with different hyperfine fields ($B_{hf} = 241$ and 208 T) corresponding to Np ordered moments of 1.12 and 0.97 μ_B , respectively [17], and an intensity ratio $\sim 2:1$. These boundary conditions provided by MoS then allowed combined neutron diffraction experiments to determine the complex, modulated magnetic structure of $NpBe_{13}$ with wave vector $\mathbf{q} = (1/3, 0, 0)$, moments perpendicular to the propagation direction, and two sublattices with moments perpendicular to each other. The wave vector of the structure gave new information

on where to search for magnetic correlations in UBe_{13} [18]. However, subsequent investigations have revealed AF short-range magnetic correlations in UBe_{13} situated at $\mathbf{q} = (0.5, 0.5, 0)$ [19].

2.2 AnRu_2Si_2

URu_2Si_2 is a HF-SC with $T_c = 1.5$ K and $\gamma \approx 180$ mJ mole⁻¹ K⁻² that has a mysterious ground state below $T_0 = 17.5$ K [5] with a dipolar moment $m_U = 0.03 \mu_B$. The order parameter has not been identified yet, and the phase has been called “hidden order” (HO). With pressure, the ground state switches from the HO phase to an AF phase ($m_U = 0.4 \mu_B$), whereas SC is suppressed [20]. The application of high magnetic fields restores the HO phase [21].

It is worth noticing that ²³⁸U MoS measurements have been performed on URu_2Si_2 [22]. However, this rotational electric dipole (E2) transition leads to a vanishing isomer shift $\delta_{IS} \rightarrow 0$ and broad line width that severely limit the access to hyperfine interactions and the information gained. It is nonetheless worthwhile noticing that the hyperfine magnetic field observed at the U site is one order of magnitude higher than expected for such a small magnetic moment.

Combined ²³⁷Np MoS and neutron diffraction have shown that the NpRu_2Si_2 counterpart (tetragonal, like URu_2Si_2) is ordering at $T_N = 27.5$ K into an incommensurate structure ($\mathbf{q} = (0, 0, 0.86)$) with a maximum moment $m_{\text{Np}} = 1.5 \mu_B$ aligned along the \mathbf{c} -axis, and partial squaring at low temperatures [23]. The sizeable quadrupolar interaction ($e^2qQ \approx -66$ mm/s) contributes to make the Mössbauer pattern asymmetric and complex.

The intermediate systems $(\text{U}_{1-x}\text{Np}_x)\text{Ru}_2\text{Si}_2$ have also been studied and no SC has been detected by electrical resistivity measurements down to 1.5 K for all samples [24]. ²³⁷Np MoS have revealed very similar patterns for all concentrations from $x = 0.9$ down to 0.05: δ_{IS} , e^2qQ and B_{hf} are nearly insensitive to x . It is also worth mentioning the rapid squaring of the Np moments modulation as x decreases: already for $x = 0.9$, only two distinct hyperfine fields are observed and the main site represents 94% of the total intensity [25]. Finally, resonant x-ray magnetic scattering [26] have measured the ratio of the U and Np magnetic moments for $x = 0.1$ and $x = 0.5$ and, using m_{Np} from Mössbauer experiments, estimated $m_U \approx 0.4 \mu_B$. This value corresponds to the moment observed in the AF phase of the pure URu_2Si_2 under pressure [27], although doping with Np might be considered as applying a “negative pressure” since it increases the lattice parameters. However, the substitution of U by Np introduces a strong molecular field at the U sites as a result of the large moment of $1.5 \mu_B$ carried by Np. This molecular field acts on the U moment of $0.02 \mu_B$ to increase it by one order of magnitude. Figure 1a shows the ²³⁷Np Mössbauer spectra obtained for $x = 0.05$. Due to the very low amount of Np in this alloy, the spectrum required a full month recording and still displayed poor statistics, but the clear hyperfine magnetic splitting shows that the magnetic moment carried by Np is still $\approx 1.5 \mu_B$ at such a low Np concentration.

2.3 AnPd_2Al_3

The largest ordered magnetic moment ($m_U = 0.85 \mu_B$) in U HF-SC was observed in UPd_2Al_3 ($T_N = 14$ K; $T_c = 2$ K, $\gamma \approx 140$ mJ mole⁻¹ K⁻²) [28]. ²³⁸U Mössbauer was

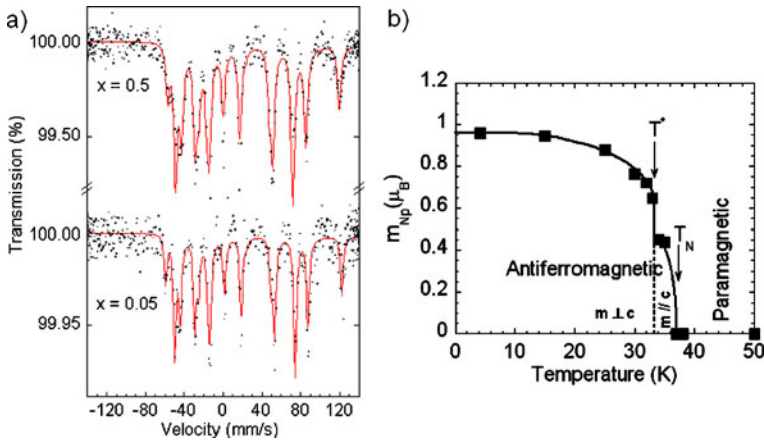


Fig. 1 **a** ^{237}Np Mössbauer spectra recorded at $T = 4.2$ K of $(\text{U}_{1-x}\text{Np}_x)\text{Ru}_2\text{Si}_2$ for $x = 0.05$ and $x = 0.5$. Notice the weak effect ($\sim 0.1\%$) for $x = 0.05$, one order of magnitude smaller than for $x = 0.5$ ($\sim 1\%$), scaling with Np concentration. Both patterns are similar and display similar hyperfine parameters. The fit assumes a single magnetic site (single value of B_{hf}). **b** Temperature dependence of the magnetic moment in NpRhGa_5 inferred from ^{237}Np MoS. The lines are guides for the eyes. The two magnetic transitions and the corresponding direction of the magnetic moments are indicated

also performed in this compound, but the magnetic transition is barely detectable (the line width increases from 50 mm/s to 60 mm/s at T_N) [22]. The hexagonal isostructural NpPd_2Al_3 parent compound is not SC down to 1.2 K [29]. ^{237}Np Mössbauer show that NpPd_2Al_3 orders at 38 K into a modulated magnetic structure with a maximum moment $m_{\text{Np}} = 1.67 \mu_B$ [30]. Measurements on $(\text{U}_{1-x}\text{Np}_x)\text{Pd}_2\text{Al}_3$ solid solutions reveal that δ_{IS} and e^2qQ are insensitive to x , but m_{Np} and T_N are much affected by chemical substitution: as x increases T_N first decreases from 14 K down to 4.5 K, and finally increases dramatically up to 38 K [25]. Electrical resistivity measurements [31] indicate the collapse of superconductivity when Np is substituted to U: for $x = 0.005$, the critical temperature is significantly reduced ($T_c = 1.5$ K) and for $x = 0.01$ it is no more observed (down to 1.2 K).

2.4 AnRhGe

In the wake of UGe_2 [7] and ZrZn_2 [32], URhGe displays the intriguing coexistence of ferromagnetism ($T_C = 9.5$ K), and superconductivity ($T_c = 0.25$ K)—for the first time at ambient pressure in a f -electrons system [8]. URhGe crystallizes into an orthorhombic structure and has strongly correlated $5f$ electrons ($\gamma \approx 160 \text{ mJ mole}^{-1} \text{ K}^{-2}$).

On the contrary, no SC was observed down to $T = 1.8$ K in NpRhGe , but it orders AF at $T_N \approx 21.5$ K [33]. In the paramagnetic state, ^{237}Np Mössbauer spectra consist of a broad line, revealing a weak quadrupolar interaction. At 4.2 K, the spectrum can be analysed in terms of a unique set of hyperfine parameters, but the line broadening as well as the enhanced intensity of internal lines and reduced intensity of external lines are typical of relaxation effects. The value of the B_{hf} corresponds to an ordered

magnetic moment $m_{\text{Np}} = 1.14 \mu_{\text{B}}$ and its Brillouin-like ($J = 1/2$) thermal variation suggests that the ground state of the Np ion is a magnetic doublet [34].

3 Neptunium analogues of Plutonium superconductors

3.1 AnCoGa₅

The first Pu-based superconductor, PuCoGa₅, with a critical temperature $T_{\text{c}} \approx 18.5$ K and a critical field $B_{\text{c}} \sim 74$ T, is unique among $5f$ systems and has been proposed as a bridge between two classes of spin-fluctuation-mediated SC: the known HF-SC and the high- T_{c} copper oxides [10].

Its Np counterpart NpCoGa₅ was found to order AF at $T_{\text{N}} = 46.5$ K [35]. The value of the magnetic moment inferred from the Mössbauer B_{hf} , amounts to $m_{\text{Np}} = 0.84 \mu_{\text{B}}$ at 4.2 K. Its variation with temperature follows a $J = 1/2$ Brillouin behavior, indicating that the ground state is a doublet, in agreement with the magnetic entropy released at T_{N} ($\sim R \ln 2$). The fact that e^2qQ measured in the ordered and paramagnetic states are about the same suggests that the Np moments are aligned along the c -axis which is the main component of the electric field gradient in the paramagnetic state (tetragonal symmetry). The value and direction of the ordered moment were later confirmed by neutron diffraction and the wave vector $\mathbf{q} = (0, 0, 1/2)$ determined [36].

3.2 AnRhGa₅

PuRhGa₅ is a SC with smaller critical parameters ($T_{\text{c}} \approx 8.7$ K and $B_{\text{c}} \sim 21$ T) than its Co analogue [11].

In analogy, NpRhGa₅ is an AF with lower T_{N} (37 K) [37] than NpCoGa₅. However, the magnetic phase diagram of NpRhGa₅ is more complex: it undergoes a second magnetic transition at $T^* = 32$ K, attributed to a reorientation of the magnetic moments from the c -axis to the basal plane, as evidenced by ²³⁷Np MoS (Fig. 1b).

4 Neptunium superconductor

4.1 NpPd₅Al₂

Recently, SC involving $5f$ electrons was observed for the first time in a Np compound, the paramagnetic HF NpPd₅Al₂ ($T_{\text{c}} = 4.9$ K and $\gamma = 200$ mJ mole⁻¹ K⁻²) with tetragonal symmetry [12]. This system thus represents a unique opportunity for the ²³⁷Np Mössbauer resonance. No magnetic hyperfine splitting is observed indicating that the Np ions do not carry any magnetic ordered moments [38]. δ_{IS} (-11.1 mm/s vs NpAl₂) is close to the value expected for ionic Np⁴⁺, but attributed rather to Np³⁺ (configuration 5f⁴), considering the influence of conduction electrons. The spectra recorded in the superconducting state are very similar to those taken in the normal state. However, a small increase in both δ_{IS} and e^2qQ is observed below T_{c} , indicating

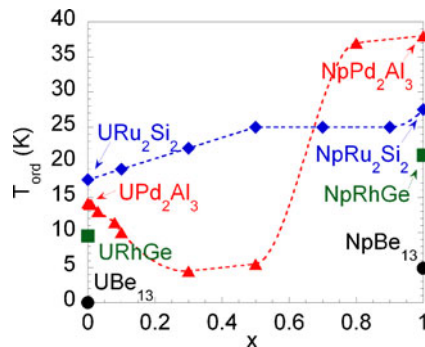
Table 1 ^{237}Np hyperfine parameters inferred from MoS

| Compound | δ_{IS} (mm/s) | e^2qQ (mm/s) | B_{hf} (T) | $T_{\text{N}}/T_{\text{c}}$ (K) | Reference |
|----------------------------|--------------------------------|-------------------|------------------------|------------------------------------|-----------|
| NpBe_{13} | 3.1 | 0 | 241/208 | 5 (AF) | [18] |
| NpRu_2Si_2 | 0.5 | 64 | 321/258/69 | 27.5 (AF) | [23] |
| NpPd_2Al_3 | 10.0 | 47 | 360/316/121 | 38 (AF) | [30] |
| NpRhGe | -2, 5 | 9 | 245 | 21 (AF) | [34] |
| NpCoGa_5 | 6.8 | 3.5 | 180 | 47 (AF) | [35] |
| NpRhGa_5 | 9.4 | 13.4 | 206 | 37 (AF) | [37] |
| NpPd_5Al_2 | -11.1 | 22.0 | 0 | 5 (SC) | [38] |

B_{hf} is given at 4.2 K, δ_{IS} and e^2qQ are given in the paramagnetic state

δ_{IS} is given versus the standard absorber NpAl_2

Fig. 2 Ordering temperature of U HF superconductors and their Np homologues. Data are also shown for $(\text{U}_{1-x}\text{Np}_x)\text{Ru}_2\text{Si}_2$ and $(\text{U}_{1-x}\text{Np}_x)\text{Pd}_2\text{Al}_3$ intermediate systems, as a function of the Np concentration x



that both the electronic density at the Np nuclei and the electric field gradient due to the $5f$ electrons are affected by the formation of Cooper pairs.

5 Summary

The hyperfine parameters inferred from ^{237}Np MoS in all reviewed compounds are summarized in Table 1. From the δ_{IS} values, it can be seen that all systems display the electronic configuration $5f^4$ corresponding to $J = 4$. The expected ordered moment for the free Np^{3+} ion would be $2.75 \mu_{\text{B}}$ (intermediate coupling), i.e. $B_{\text{hf}} = 591$ T. Kondo effect and crystal field effects have been invoked to explain the reduced values of the magnetic moments in these compounds (see e.g. [35] and [38]).

It is clear from Fig. 2 that the Np systems show much larger ordering temperatures than their U counterparts. This is in agreement with the general trend observed in actinide intermetallics, attributed to the shorter spatial extension of $5f$ orbitals in Np compared to U and, consequently, the weaker hybridization/delocalization of $5f$ electrons in Np.

The x -dependence of the ordering temperature in $(\text{U}_{1-x}\text{Np}_x)\text{Pd}_2\text{Al}_3$ is unusual, with a minimum for $x = 0.3$ (see Fig. 2). Interestingly, UPd_2Al_3 and NpPd_2Al_3 have different easy axis ($\mathbf{m}_{\text{U}} \perp \mathbf{c}$ and $\mathbf{m}_{\text{Np}} \parallel \mathbf{c}$) and, from $x = 0.3$ to $x = 0$, the magnetic moment carried by Np is continuously rotating from the \mathbf{c} -axis to the basal plane

[26]. Magnetic frustration may then explain the T_{ord} minimum observed at $x = 0.3$. The T_{ord} variation of $(\text{U}_{1-x}\text{Np}_x)\text{Ru}_2\text{Si}_2$ appears smooth and almost linear, although it should be noticed that for $x = 0$, the AF order vanishes and is replaced by the so-called “hidden order”.

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