Positron lifetime measurements and electronic structure of CeNiSn

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Positron lifetime measurements have been carried out in CeNiSn, in the temperature range of 5 to 300 K. In the 300 and 150-K temperature interval the positron lifetime is seen to decrease from 178 ± 1 to 176 ± 1 ps. Below 150 K the lifetime shows a gradual increase and attains a value of 181 ± 1 ps at 35 K, below which the lifetime shows a small decrease. This behavior of the positron lifetime is seen to correlate with earlier measurements [Y. Uwamoto *et al.*, J. Magn. Magn. Mater. **104**, 643 (1992)] on the thermal expansion coefficient in CeNiSn, which has been understood in terms of electronic structure changes associated with the system transforming from an independent Kondo regime to a Kondo insulating state, via an intermediate mixed valent regime. Strong support for these electronic structure changes are provided by *ab initio* positron lifetime calculations which are seen to be in good agreement with the positron experiments.

I. INTRODUCTION

A special class of strongly correlated electron systems termed Kondo insulators have recently attracted considerable attention.[1,2] The number of systems that exhibit the Kondo insulating behavior are few and generally occur in cubic systems,[1,3] viz. SmB_6, $Ce_3Bi_4Pt_3$, YbB_{12}, FeSi, etc., in which the cubic structure leads to a simple band structure close to the Fermi energy E_F. The only exceptions in this category are CeNiSn and CeRhSb, which crystallize in the ϵ-TiNiSi orthorhombic structure.[3] The occurrence of the Kondo insulating ground state in CeNiSn has been under considerable debate. Early experiments using polycrystalline samples show[4] a transport gap, 5 K in magnitude, while measurements in single-crystal samples show[5] that the gap is anisotropic, being insulating along the *c* direction and metallic along the *a* and *b* directions. More recent resistivity experiments on very pure single crystals of CeNiSn indicate a metallic ground state[6] but specific-heat measurements, Hall measurements, and magnetic susceptibility experiments on both single and polycrystalline samples point to the presence of a gap close to E_F.[6–8] The CeNiSn system also exhibits anomalous thermal expansion[9] and temperature dependence of elastic constants.[10] The thermal expansion coefficients α_a and α_b and the elastic constants C_{11}, C_{22}, and C_{33} show anomalies at $\sim$150 and $\sim$10 K, respectively. These anomalies correlate with maxima in the resistivity in these samples seen along the three different crystallographic directions,[5] and can be understood[11,12] by considering that the system has two characteristic temperatures of the order of 150 and 10 K. The higher characteristic temperature is associated[11,12] with the system smoothly transforming from an independent Kondo to a mixed valent regime and the lower characteristic temperature with the opening of the Kondo insulating gap.

The formation of the Kondo insulating gap has been conventionally explained in terms of the formation of a local singlet in the strong-coupling limit,[1] or alternately due to the opening of an hybridization gap,[13,14] in the weak-coupling regime. Band-structure calculations[13] in the CeNiSn system suggest the presence of a semiconducting ground state with a band gap ($\sim$217 K), with the valence band comprising of Ce-5*d*, Ni-3*d*, Sn-5*p*, and Ce-4*f* electrons. Recent neutron-scattering experiments[14] provide evidence for the hybridization of Ce-4*f* and Ni-3*d* bands close to the Fermi surface. But photoemission measurements on this system seem to indicate that at the Fermi level, apart from the hybridization of the 4*f* electrons of Ce and the 3*d* electrons of Ni, there is also a dominant hybridization of the Sn 5*p* and Ce 4*f* electrons.[15] In the present experiments, we have investigated the Kondo insulating behavior in CeNiSn through a temperature-dependent study of positron lifetime. Supportive *ab initio* positron lifetime calculations are used to unravel the electronic structure changes responsible for the experimental observations. In may be mentioned that recent measurements in FeSi (Refs. 16 and 17) indicate the sensitivity of the positron lifetime to the opening of a hybridization induced Kondo insulating gap as a function of temperature.

It is now well established that the positron lifetime measurement is an excellent probe for the study of electronic structure of solids.[18,19] The sensitivity of the positron lifetime τ, to either the localized or itinerant nature of the electron in the bulk of a solid, comes about through an enhancement factor in the electron density. This enhancement factor is of the order of 6 to 10 for itinerant electrons, while it is small and of order unity for localized core electrons.[18] It is therefore expected that a change in the nature of the electron from localized to itinerant character will have a significant effect on the measured lifetimes. For example, experiments across the valence transition in valence fluctuating systems, viz. SmS (Ref. 20) and Ce,[21] in which the localized *f* electrons in the insulating state gets delocalized under the application of pressure, giving rise to the metallic phase, did show a decrease in lifetime. However since these transitions were also associated with a decrease in volume it was difficult to unambiguously delineate the effects arising from electronic structure changes and lattice contraction.[20] The opportunity to study a pure electronic structure change without an accompanying lattice volume change was provided in the FeSi system which shows a Kondo insulating behavior as one lowers the temperature.[16,17] In positron lifetime measurements in the temperature regime where the system goes into the Kondo insulating state, a remarkable decrease in the positron lifetime was observed.[16] This was interpreted to arise as a consequence of the delocalization of an erstwhile localized *d* electron as it hybridizes with an *s* electron and doubly

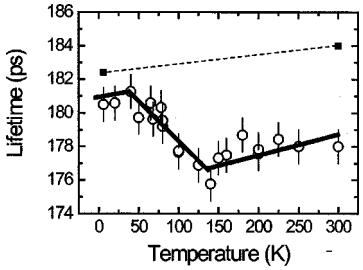

FIG. 1. Positron lifetime vs temperature in CeNiSn sample in the 5- and 300-K range (open circles). The solid line is guide to the eye. The expected variation in lifetime due to lattice expansion (see text) is shown as a dashed line.

occupies the lower more dispersed band, with decrease in temperature.[16,17] Here we report on the positron lifetime measurements done in CeNiSn in the 5- and 300-K temperature region and on the computation of the positron density distribution and positron lifetimes in this system.

II. EXPERIMENTAL DETAILS

CeNiSn samples were prepared by arc melting Ce (99.9%), Ni (99.99%), and Sn (99.99%) taken in stoichiometric proportions several times in a protective, flowing atmosphere of He gas using a tri-arc furnace. The loss of weight after the meltings was less than 1%. The arc melted buttons were vacuum sealed and homogenized, at 1000°C for 1 week. The homogenized samples were cut using a diamond wheel into slices $\sim$0.5-mm thick and $\sim$5 mm in diameter. Room temperature x-ray-diffraction (XRD) measurements were carried out using the Cu K_α radiation. The XRD patterns could be indexed to an ϵ-TiNiSi, orthorhombic structure and the sample was found to be monophasic, with lattice parameters $a=7.538$ Å, $b=4.598$ Å, and $c=7.617$ Å in good agreement with the values reported earlier.[14] The resistance versus temperature was measured in the 4- and 300-K regime in a dip-stick cryostat, with the sample mounted in a pressure contact assembly in a four probe geometry. As the temperature is lowered from 300 K, the resistance shows a gradual increase with decrease in temperature followed by a steep increase below 50 K. In the 5- and 50-K temperature interval the resistance data could be fitted to an Arrhenius behavior yielding a gap value of 6 K, in accordance with earlier studies on polycrystalline samples.[4] The positron lifetime measurements were made using a fast-fast coincidence spectrometer[20] having a time resolution of 260 ps. The positron source, ^{22}NaCl deposited on 2.5-μm Ni foil, was sandwiched between the disks of the CeNiSn samples in a copper sample cell which was mounted inside a JANIS make continuous flow cryostat, using which the temperature variation between 300 and 5 K was achieved.

III. RESULTS AND DISCUSSION

The measured lifetime spectra were best analyzed ($\chi^2 \leqslant 1.1$) in terms of a single lifetime component after taking into account the annihilations in the source foil.[16,20] The resulting temperature variation of the lifetime (τ), is shown in Fig. 1. It can be seen from the figure that the lifetime decreases from the 300-K value of 178 ± 1 to 176 ± 1 ps at 150 K. Below 150 K the measured positron lifetime shows an unmistakable increase to 181 ± 1 ps at 35 K, below which it again tends show a small decrease upto the lowest temperature of 5 K. This variation in τ over the temperature range was found to show a similar behavior in both the heating and cooling runs.

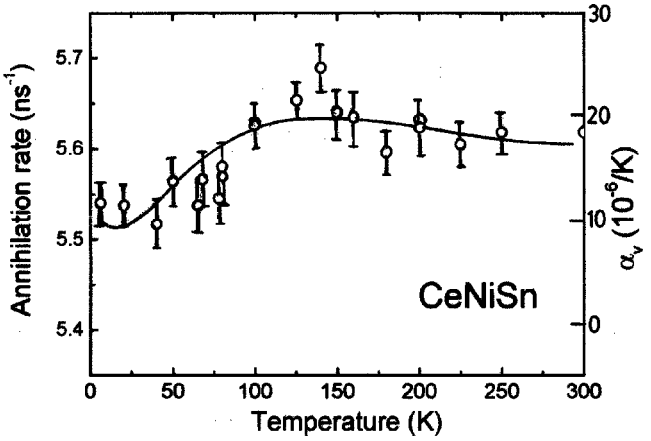

FIG. 2. Positron annihilation rate vs temperature in CeNiSn shown by open circles. The variation of the volume thermal-expansion coefficient α_v taken from Ref. 22 is shown as a solid line. The ordinate on the right has been offset vertically to show the correlation with the variation of the positron annihilation rate.

The small decrease in τ in the 300- and 150-K temperature regime, seen in Fig. 1, can be accounted for by thermal contraction of the lattice. The expected decrease due to thermal contraction, as estimated from theoretical calculations (indicated later in the text), using the lattice constants at 6 and 300 K (Ref. 14) is shown as a dashed line in Fig. 1. It can been seen from the figure that the experimentally observed slope of the positron lifetime in the 300- and 150-K temperature range and that obtained by the calculations are very similar, clearly indicating that in this regime it is lattice contraction that is responsible for the observed lifetime behavior. Below 150 K, the positron lifetime shows a clear increase deviating from the linear decrease expected from the lattice contraction. This behavior is therefore unusual and could arise from electronic structure changes occuring in this temperature regime. At this point we noticed that the increase of τ below 150 K and its saturation below 35 K seems to be similar to the changes observed in the elastic constants C_{11} and C_{33} and the thermal expansion coefficients α_a and α_c as a function of temperature.[9,10] In experiments on polycrystalline CeNiSn the variation of the volume thermal-expansion coefficient α_v has been obtained in the 4- and 300-K temperature interval.[22] When the annihilation rate λ, inverse of the measured τ, and α_v are plotted as a function of temperature a striking correlation is observed (cf. Fig. 2). It can be seen that α_v (bold line, Fig. 2) tracks the measured positron annihilation rate (open circles) fairly well. This can therefore imply that the changes observed by positron lifetime and the elastic constants could have the same physical origin. Such a correlation between α_v and annihilation parameters is found to be more generic and is seen in other strongly correlated electron systems.[23]

It is well known that anomalous thermal expansion is a characteristic feature of valence fluctuating systems and has its origin in the fact that the localized *f* level is pinned to the

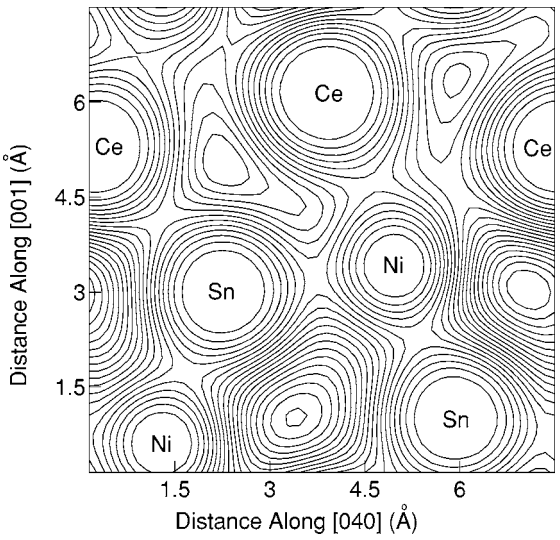

FIG. 3. The positron density distribution in the (040) plane. The position of the Ce, Ni, and Sn atoms are indicated. The maximum of the positron density is seen in the interstitial region between Ce, Ni, and Sn atoms. A uniform positron sampling of all the atoms is also indicated by the calculation of the partial annihilation rates (Ref. 29).

Fermi level.[24] A simple physical interpretation[24] is that, as the localized f electron in the mixed valent state transfers into the conduction band, the $5d/6s$ electrons of the rare-earth ion move inwards to screen the f hole and consequently decrease the effective radius of the rare-earth ion resulting in a contraction of the lattice. This contraction further results in the d band shifting to lower energies which makes the f electron transfer into the conduction band more favorable. An increased creation of f holes ensues, followed by d electron screening leading to the anomalous behavior of α_v. Calculations of the positron lifetime in CeNiSn keeping these electronic structure changes in view has been attempted, and will be described in the following.

The positron density distribution and the positron lifetime in the solid is calculated following the method of Puska and Nieminen[25] as described in detail in Ref. 26. The calculational scheme consists of three major steps: (i) construction of the positron potential; (ii) numerical solution of the Schrodinger equation under periodic boundary conditions, and (iii) calculation of the lifetime, done by evaluating the overlap of the positron and electron densities. The potential experienced by the positron is obtained from a superposition of the atomic potentials and densities.[25] The solution of the Schrödinger equation has been done under periodic boundary conditions. The calculated positron density distribution in the (040) plane in CeNiSn is shown in Fig. 3. From this contour plot, it is seen that the positron density spans the entire unit cell and that the maxima occur in the interstitial regions between the metal atoms.

The positron annihilation rate λ, an inverse of the positron lifetime τ, was obtained as a sum of the core annihilation rate λ_c and valence annihilation rate λ_v. These are obtained by evaluating[25,26] the overlap of the calculated positron density $|\psi_+(\mathbf{r})|^2$, with the superposed core and valence electron densities $n_c(\mathbf{r})$ and $n_v(\mathbf{r})$, respectively, using the formulas

$$\lambda_c = 16\pi \int |\psi_+(\mathbf{r})|^2 n_c(\mathbf{r}) d\mathbf{r}, \tag{1}$$

$$\lambda_v = \int d\mathbf{r}[2 + 134 n_v(\mathbf{r})] |\psi_+(\mathbf{r})|^2. \tag{2}$$

In order to take into account the effect of lattice contraction on the positron lifetime, we use the lattice constants from neutron-scattering data[14] at 300 and 6 K. Using the electronic configurations for Ce, Ni, and Sn corresponding to the independent Kondo regime (cf. Table I), the lifetime value at 300 K is estimated to be 184.24 ps, which is seen to decrease to 182.41 ps at 6 K. This implies that only a small change of ~ 2 ps is expected due to lattice contraction and this is indicated as a dashed line in Fig. 1.

Before we proceed further, we would like to point out that the observed temperature dependence of lifetime cannot also arise due to positron trapping at defects.[25] This we rule out on several counts: (i) At first we note that the theoretically calculated lifetime of 184 ps is in good agreement with the experimentally measured τ of 178 ± 1 ps, indicating that the positron annihilates from the Bloch state (cf. Fig. 3). (ii) An increase in the positron lifetime with a decrease in temperature, seen in Fig. 1, could also arise if positron gets trapped at shallow defects.[18] However, such a possibility is unlikely, because in intermetallic systems like CeNiSn, vacancy-type traps are expected to be deep traps, as in metals.[18,19] Further, the increase of the positron lifetime due to thermally activated redistribution of the positron from the bulk to shallow traps is seen to have a sigmoidal behavior spread over a wider temperature range,[27] when compared to that seen in Fig. 1. Thus we believe that the role of defects in giving rise to the positron lifetime changes seen in Fig. 1 can be ruled out.

Having argued that the lattice contraction and defect trapping have no significant role to play in the observed variation of τ, we investigate as to how the temperature dependence seen in Fig. 1 could have come about due to electronic structure changes. Since the positron annihilation rate data shown in Fig. 2 tracks the variation of α_v, as a function of temperature we surmise that the origin of the lifetime changes and that of thermal expansion are correlated. We note that the localization model of Kasuya[12] satisfactorily explains the anomalous thermal expansion and elastic constant behavior[9–12,22] as the system makes a smooth transformation from an independent Kondo to a mixed valent regime. According to this model,[12] the electronic structure of the Ce atom changes from being predominantly trivalent to being in a mixed valent state with decrease in temperature. Therefore in the positron lifetime calculations carried out at 300 K, we consider the Ce atom to be in the trivalent state (electronic configuration: $[\mathbf{Xe}]\mathbf{4f^1}5d^2 6S^1$) and obtain a lifetime $\tau = 184.24$ ps, in good agreement with the measured lifetime value of 178 ± 1 ps at room temperature.

In the mixed valent state below 150 K, as the localized $4f$ electron is transferred to the conduction band and the $5d$ electron from the conduction band gets localized close to the Ce core to screen the $4f$ hole, the resultant $5d$ hole forms a bound state with the $4f$ electron.[11,12] *In effect both the* $4f$ *and* $5d$ *electrons acquire a very local character.* The electronic configuration of Ce in the mixed valent state can thus

TABLE I. A summary of positron lifetime calculations in CeNiSn corresponding to various electronic configurations. The electronic states treated as core electrons, in the calculation of τ is indicated in bold. [**Xe**], [**Kr**], and [**Ar**] denote the corresponding inert shell configurations. The lattice constants at 300 and 6 K (Ref. 14), viz. $a=7.542$ Å, $b=4.601$ Å, and $c=7.617$ Å and $a=7.500$ Å, $b=4.580$ Å, and $c=7.580$ Å have been used in the lifetime calculations.

Serial number	Regime	Electronic configuration	Calculated τ
1.	independent Kondo	Ce:[**Xe**]**4f^{1}**$5d^{2}6s^{1}$ Ni:[**Ar**]$3d^{8}4s^{2}$ Sn:[**Kr**]**4d^{10}**$5s^{2}5p^{2}$ lattice constant at 300 K	184.24 ps (Ref. 28)
2.	independent Kondo	Ce:[**Xe**]**4f^{1}**$5d^{2}6s^{1}$ Ni:[**Ar**]$3d^{8}4s^{2}$ Sn:[**Kr**]**4d^{10}**$5s^{2}5p^{2}$ lattice constant at 6 K	182.41 ps
3.	mixed valent	Ce:[**Xe**]**4f^{1}**$4f^{1}5d^{1}6s^{1}$ Ni:[**Ar**]$3d^{8}4s^{2}$ Sn:[**Kr**]$4d^{10}5s^{2}5p^{2}$ lattice constant at 6 K	190.08 ps
4.	Kondo insulating Ce-4f Ni-3d hybridization	Ce:[**Xe**]**4f^{0}**$4f^{1}5d^{1}6s^{1}$ Ni:[**Ar**]$3d^{9}4s^{2}$ Sn:[**Kr**]$4d^{10}5s^{2}5p^{2}$ lattice constant at 6 K	189.45 ps
5.	Kondo insulating Ce-4f Ce-5d hybridization	Ce:[**Xe**]**4f^{0}**$4f^{1}5d^{2}6s^{1}$ Ni:[**Ar**]$3d^{8}4s^{2}$ Sn:[**Kr**]**4d^{10}**$5s^{2}5p^{2}$ lattice constant at 6 K	181.90 ps
6.	Kondo insulating Ce-4f Sn-5p hybridization	Ce:[**Xe**]**4f^{0}**$4f^{1}5d^{1}6s^{1}$ Ni:[**Ar**]$3d^{8}4s^{2}$ Sn:[**Kr**]**4d^{10}**$5s^{2}5p^{3}$ lattice constant at 6 K	184.20 ps

be written as [**Xe**]**4f^{0}5d***$4f^{1}5d^{0}5d^{1}6s^{1}$, where $4f^{0}$ hole in the core of Ce atom is screened by the $5d^{*}$ electron of the surrounding atoms. In this process the latter acquires an f symmetry and has a small spatial extent. To maintain charge neutrality and to represent the fact that $5d^{*}$ electron that has attained an f character with a spatial extent similar to that of a $4f^{1}$ electron in Ce, the electronic configuration of Ce in the mixed valent state is written as [**Xe**]**4f^{1}**$4f^{1}5d^{1}6s^{1}$ for the purpose of performing the positron lifetime calculations (cf. Table I). Note that the electronic configuration of Ce in the independent Kondo regime was [**Xe**]**4f^{1}**$5d^{2}6s^{1}$. The electronic configurations of Ni and Sn in the mixed valent phase were taken to be same as that used for the positron calculation at 300 K. The calculated τ turns out to be 190.08 ps, using the measured lattice constant at 6 K (cf. Table I). The resultant increase in the calculated lifetime values is ~ 8 ps, which is in good agreement with the changes of τ seen in the experiment. Given that we can account for the overall increase, the observed gradual change in τ below 150 K can be attributed to be due to the smooth variation of the f electron count at Ce as a consequence of its transformation from the independent Kondo regime to the mixed valent regime, as is also reflected in the behavior of α_{v} (cf. Fig. 2). The origin of the increase in τ can be intuitively understood as follows: The $4f$ orbital radius which is ~ 5 times smaller than that of the $5d-6s$ orbital, is closer to the nucleus and is hardly sampled by the positron,[28] whereas the $5d$ electrons having a larger orbital radius have a significant overlap with the positron density. Thus the movement of the $5d$ electrons to screen the $4f$ hole, spatially close to the $4f$ level, reduces its contribution to the valence annihilation rate, resulting in an increase in τ, below 150 K.

Figure 1 also shows that below 35 K the increasing trend in the positron lifetime is arrested and τ shows a tendency towards a small decrease. Since it is this temperature regime that corresponds to the lower characteristic temperature[6–8,11] we think it worthwhile to examine if this change can be accounted for by the opening of the Kondo insulating gap. As mentioned in the Introduction, the band gap in CeNiSn could arise on account of the hybridization of the $4f$ levels of Ce with $3d$ levels of Ni at the Fermi surface[14] and/or its hybridization with $5p$ electrons of Sn.[15] Since the positron probes the three atomic species in CeNiSn with equal probability,[29] we calculate τ after hybridization of Ce-$4f$ with Ni-$3d$, Sn-$5p$, and Ce-$5d$ electrons, respectively (cf. Table I). According to the hybridization gap model, after the flat $4f$ band hybridizes with a dispersed conduction band, the two hybridized electrons doubly occupy the more dispersed

band. To implement this in our positron calculations for the specific case of hybridization between Ce-$4f$ and Ni-$3d$, we assume an electronic configuration of Ce in the Kondo insulating phase to be $[\mathbf{Xe}]\mathbf{4f^0}4f^15d^16s^1$ and that of Ni as $[\mathbf{Ar}]3d^94s^2$, viz. in effect we transfer the localized Ce $4f$ electron, with a flat dispersion, into the more dispersed Ni $3d$ level.[14] The electronic configuration of Sn remains unchanged at $[\mathbf{Kr}]\mathbf{4d^{10}}5s^25p^2$. The calculated value of τ for the lattice constant taken at the 6-K value was observed to decrease to 189.45 ps from the mixed valent phase value of 190.08 ps (cf. Table I). The calculated lifetimes corresponding to alternative hybridization scenarios, viz. when the $4f$ level of Ce hybridizes with the $5d^1$ level of Ce and that both the electrons now occupy a more dispersed $5d$-like band, employing the electronic configuration of Ce, Ni, and Sn as shown in Table I, we obtain a decrease in the calculated lifetime to 181.9 ps. If, however, we assume that the $4f$ level hybridizes with the $5p$ electrons of Sn (cf. Table I), we obtain a lifetime of 184.20 ps. Thus the small decrease observed in τ below 35 K in the experiment suggests that the hybridization of Ce $4f$ electrons with the $3d$ electrons of Ni is more likely than the alternative possibilities of hybridization with Ce $5d$ or Sn $5p$ electrons.

In summary, the behavior of temperature dependence of the positron lifetime tracks the variation of the thermal-expansion coefficient and the electronic structure changes used to explain the latter also account for the observed variation of τ. In the 300–150-K temperature range the system is in the independent Kondo regime with Ce in the trivalent state and the small decrease seen in the positron lifetime in this temperature range arises due to thermal contraction. The significant increase in τ, seen in the 150–35-K range is attributable to the smooth transformation of the system into the mixed valent state. Below 35 K, CeNiSn crosses over into the Kondo insulating state, arising due to the hybridization of Ce-$4f$ with the Ni-$3d$ electrons. The present positron lifetime experiments with support from theoretical calculations are in overall consonance with the theoretical prediction[30] that the mixed valent state is a necessary prerequisite for the opening of the Kondo insulating gap in dense Kondo systems.

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[28] Calculation of the positron lifetime by considering the $4f$ electron of Ce as a valence electron, viz. $[\mathbf{Xe}]4f^15d^26s^1$, results in a value of 183.75 ps. This implies that the overlap of the positron density with the $4f$ electron density is very small.
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