I. INTRODUCTION

There has been a great deal of interest in noncentrosymmetric superconductors (NCS), instigated by the discovery of the noncentrosymmetric heavy-fermion superconductor CePt$_3$Si$_5$. The absence of a center of inversion in the crystal structure along with a nontrivial anti-symmetric spin-orbit coupling, leads to the intriguing possibility of a superconducting state with an admixture of spin-triplet and spin-singlet pairs. Despite intense theoretical and experimental efforts, the study of the physics of noncentrosymmetric superconductors remains a dynamic and active field.

One manifestation of unconventional superconductivity is the breaking of time-reversal symmetry (TRS). The magnetic moments associated with the Cooper pairs are non-zero for such superconductors. A local alignment of these moments produces spontaneous, but extremely small, internal magnetic fields. Muon-spin relaxation is thus an ideal probe with which to search for TRS breaking are LaNiC$_2$ and Re$_6$Zr$_6$ for Re$_6$Zr$_6$. The absence of a center of inversion in the crystal structure along with a nontrivial anti-symmetric spin-orbit coupling, leads to the intriguing possibility of a superconducting state with an admixture of spin-triplet and spin-singlet pairs. Despite intense theoretical and experimental efforts, the study of the physics of noncentrosymmetric superconductors remains a dynamic and active field.

Muon-spin relaxation can be used to accurately determine the magnetic penetration depth $\lambda$ and hence the temperature dependence of the superfluid density, yielding information on the symmetry of the superconducting gap. Muon spectroscopy studies have been performed on some of these compounds e.g. Re$_3$W, LaRhSi$_3$, LaPtSi$_3$, LaPdSi$_3$. However, no spontaneous fields were observed in the superconducting state in any of these materials. This indicates that TRS breaking is either undetectable or not present in the superconducting state of these compounds.

Recently a new family of superconducting materials has been discovered with the formula $RTX_n$ (where $R =$ rare earth, $T =$ transition metal, and $X =$ Si or Ge). These 113 materials can crystallize in either the noncentrosymmetric BaNiSn$_3$-type crystal structure (space group $I4mm$), or in the centrosymmetric LaRu$_2$Sb$_3$-type cubic structure (space group $Pm\bar{3}n$) shown in Fig. 1. Several 113 materials with a noncentrosymmetric structure such as CeCoSi$_3$, CeRhSi$_3$, CeIrSi$_3$, and CeIrSi$_3$ exhibit novel ground states. For example, at ambient pressure CeCoSi$_3$ becomes superconducting at 1.3 K, while CeRhSi$_3$ and CeIrSi$_3$ exhibit antiferromagnetic ordering at 1.5 and 5 K, respectively. The latter two compounds also reveal superconductivity under applied pressure. In CeRhSi$_3$ the superconducting transition temperature $T_c$ increases from 0.45 to 1.1 K between 0.4 and 2.3 GPa, while for CeIrSi$_3$ $T_c$ increases from 0.5 to 1.6 K between 1.8 and 2.5 GPa. In these compounds, the competition between the on-site (nonmagnetic) Kondo interaction and the oscillatory inter-site long-range magnetic interactions, known as Rudermann-Kittel-Kasuya-Yosida (RKKY) interaction, plays an important role in the observed and rather unusual physical properties, evidence for multiple gaps, or a significant admixture of a triplet component to the superconducting order parameter.
FIG. 1: (color online) Two possible crystal structures of the RTX$_3$ (113) compounds. Left is the noncentrosymmetric structure with the space group $I4mm$ and right is the centrosymmetric structure with the space group $Pmn3$. The light (blue) spheres are the $R$ atoms, the black spheres the $T$ atoms, and the dark (red) spheres the $X$ atoms.

Polycrystalline samples of CaPtSi$_3$ and CaIrSi$_3$ were prepared by arc melting stoichiometric quantities of high purity Ca (5% excess of Ca to compensate for any weight loss), Pt/Ir and Si in a tri-arc furnace under an argon (5N) atmosphere on a water-cooled copper hearth. In order to minimize the loss of the Ca by evaporation, melting is done in two steps. In the first step, Pt/Ir are melted with Si. The observed weight loss during the melting of binaries Pt/Ir-Si is negligible. In the second step, Pt/Ir-Si binaries are melted with 5% excess of Ca. The sample buttons were melted and flipped several times to improve phase homogeneity.

### TABLE I: Lattice parameters of noncentrosymmetric CaPtSi$_3$ and CaIrSi$_3$ determined from powder x-ray diffraction data collected at 298 K.

|                | CaPtSi$_3$ | CaIrSi$_3$ |
|----------------|------------|------------|
| Structure      | Tetragonal  | Tetragonal  |
| Space group    | $I4mm$     | $I4mm$     |
| $a$ (nm)       | 0.42182(5) | 0.41957(2) |
| $c$ (nm)       | 0.9880(2)  | 0.98711(7) |

### II. EXPERIMENTAL DETAILS

#### A. Sample preparation

In this work we report on the properties of the new $f$-electron free 113 compounds, CaIrSi$_3$ and CaPtSi$_3$, both of which have the potential to be of great interest. CaIrSi$_3$ and CaPtSi$_3$ are NCS with superconducting transition temperatures of 3.6 and 2.3 K respectively and therefore do not require pressure to induce the superconducting state unlike their Ce analogues$^{35,36}$. Specific heat data are in general agreement with these superconductors being fully gapped, however, CaIrSi$_3$ appears to show a deviation from a pure $s$-wave gap, which has been suggested as evidence for a multiband or anisotropic gap$^{35}$. In the presence of Ir and Pt it is expected that spin-orbit coupling will be significant, strengthening the possibility that the mechanisms for superconductivity might not be entirely conventional in these materials. Therefore, it is timely and interesting to probe the superconducting state of CaIrSi$_3$ and CaPtSi$_3$ using muon spectroscopy. In this work, muon-spin relaxation is used to search for evidence of TRS breaking in these two superconductors. Muon-spin rotation is used to determine the temperature dependence magnetic penetration depth. Since $\lambda(T)$ is directly related to the superfluid density, the pairing symmetry can then be determined.

#### B. Sample characterization

Polycrystalline samples of CaPtSi$_3$ and CaIrSi$_3$ were prepared by arc melting stoichiometric quantities of high purity Ca (5% excess of Ca to compensate for any weight loss), Pt/Ir and Si in a tri-arc furnace under an argon (5N) atmosphere on a water-cooled copper hearth. In order to minimize the loss of the Ca by evaporation, melting is done in two steps. In the first step, Pt/Ir are melted with Si. The observed weight loss during the melting of binaries Pt/Ir-Si is negligible. In the second step, Pt/Ir-Si binaries are melted with 5% excess of Ca. The sample buttons were melted and flipped several times to improve phase homogeneity.

In order to confirm the superconducting transition temperatures of the samples, dc magnetic susceptibility measurements were made using a Quantum Design Magnetic Property Measurement System. Fig. 2 shows the magnetic susceptibility as a function of temperature in an applied field of 5 Oe. The observed superconducting transition temperatures $T_C$ for CaPtSi$_3$ and CaIrSi$_3$ are approximately 2.3 and 3.5 K respectively (see Table I). These transition temperatures are in good agreement with previously reported results measured by dc susceptibility on samples with the same composition$^{35}$. There is no evidence from the dc susceptibility data that the impurities present in our samples order magnetically or become superconducting. Since muon spectroscopy probes the full volume of the sample the results presented below are representative of the majority superconducting
The muons produced are implanted into the sample and with 4 out of 5 pulses going through the muon target.

At the ISIS facility, a pulse of protons with a full width at half maximum of ≈ 70 ns are produced every 20 ms, with 4 out of 5 pulses going through the muon target. The muons produced are implanted into the sample and decay with an average lifetime of 2.2 μs into a positron which is emitted preferentially in the direction of the muon spin axis along with two neutrinos. These positrons are detected and time stamped in the 64 detectors which are positioned either before, F, or after, B the sample for longitudinal (relaxation) experiments. The asymmetry A of the μSR time spectrum is then obtained as

\[ A(t) = (F(t) - \alpha B(t))/(F(t) + \alpha B(t)) \]

where \( \alpha \) represents a relative counting efficiency of the forward and backward detectors. Using these counts the asymmetry in the positron emission can be determined and, therefore, the muon polarization is measured as a function of time.

For the transverse-field experiments, a magnetic field is applied perpendicular to the initial muon spin direction and momentum. In this configuration, the signals from the instruments 64 detectors are normalized and reduced to two orthogonal components which are then fitted simultaneously.

Powder samples of each material were mixed with GE varnish and mounted onto silver holders. Any muons which stop in silver give a time independent background for ZF-μSR experiments and a non-decaying precession signal in the TF-μSR. The sample holder and sample were mounted into a helium-3 cryostat with a temperature range of 0.3 to 50 K. The samples were cooled to base temperature in zero field and the relaxation spectra were collected at fixed temperature upon warming while still in zero field. The stray fields at the sample position were canceled to within 10 mG by a flux-gate magnetometer and an active compensation system controlling three pairs of correction coils. The TF-μSR experiments were conducted in a range of applied fields from 50 to 600 Oe. The field was applied above the superconducting transition before cooling.

**III. RESULTS AND DISCUSSION**

**A. Zero-field muon-spin relaxation**

Firstly, let us consider the zero-field muon-spin relaxation results (see Fig. 3). The absence of an oscillation in the ZF-μSR asymmetry data at all temperatures for both samples confirms that there are no coherent magnetic fields, usually, associated with long-range magnetic order. In the absence of atomic moments, in CaTSi₃ the muon-spin relaxation is expected to arise from the local fields associated with nuclear moments. These moments are usually static on the timescale of the muon and are randomly orientated. In a case such as this the depolarization function can be described by a Kubo-Toyabe function. In Fig. 3 we can see that for both CaIrSi₃ and CaPtSi₃ the data is relatively flat and does not have the characteristic shape of the aforementioned Kubo-Toyabe function. This indicates that the fields from the nuclear moments are small. Moreover, the μSR signals for temperatures above and below the superconducting transition overlay and the depolarization rate is the same. This indicates that time-reversal symmetry is preserved, as would be expected in a conventional singlet superconductor, or at least any symmetry breaking field is not observable by μSR.
FIG. 3: (color online) Zero-field muon-spin relaxation spectra for CaIrSi$_3$ (upper panel) and CaPtSi$_3$ (lower panel) at temperatures above (open symbols) and below (closed symbols) $T_c$.

B. Transverse-field muon-spin rotation

Transverse-field muon-spin rotation can be used to determine the magnetic penetration depth $\lambda$. Figure 4 shows typical spectra with a transverse applied field of 100 Oe at $T = 0.2$, 1.1, and 1.95 K after being cooled through $T_c$. The TF-$\mu$SR spectra were fit as a sum of sinusoidally oscillating components, each within a Gaussian relaxation envelope,

$$G_x(t) = \sum_{i=1}^{n} A_i \exp \left( -\frac{\sigma_i^2 t^2}{2} \right) \cos (\gamma_\mu B_i t + \varphi),$$

(1)

where $A_i$ is the initial asymmetry, $\sigma_i$ is the Gaussian relaxation rate, and $B_i$ is the first moment for the $i$-th component of the field distribution. There is a common phase offset $\varphi$ and $\gamma_\mu$ is the muon gyromagnetic ratio. In these fits, $\sigma_n$ for the $n$-th component is set to zero and corresponds to a background term arising from those muons which are implanted into the silver sample holder producing an oscillating signal that has no depolarization, as silver has a negligible nuclear moment. Using Eq. (1) is equivalent to assuming a field distribution $P(B)$ within the sample given by

$$P(B) = \gamma_\mu \sum_{i=1}^{n} \frac{A_i}{\sigma_i} \exp \left( -\frac{\gamma_\mu^2 (B - B_i)^2}{2\sigma_i^2} \right).$$

(2)

The second moment of the field distribution within the sample $\langle \Delta B^2 \rangle$ is

FIG. 4: Typical muon-spin rotation spectra for CaPtSi$_3$ in a transverse field of 100 Oe at temperatures of 0.2 K (upper), 1.1 K (middle) and 1.95 K (lower). The lines are fits to the data using Eq. (1) as described in the text.

FIG. 5: Field dependence of the muon depolarization rate at $T = 100$ mK for CaIrSi$_3$ (open symbols) and CaPtSi$_3$ (closed symbols). The lines are fits to the data using Eq. (4) as described in the text.
\( \langle \Delta B^2 \rangle = \left( \frac{\sigma}{\gamma_\mu} \right)^2 = \sum_{i=1}^{n-1} A_i \frac{\sigma_1^2}{\gamma_\mu} + (B_i - \langle B \rangle)^2, \)

where \( A_{\text{tot}} = \sum_{i=1}^{n-1} A_i \) and \( \langle B \rangle = \sum_{i=1}^{n-1} A_i B_i / A_{\text{tot}}. \) The superconducting component of the second moment \( \sigma_{sc} \) is then given by \( \sigma_{sc}^2 = \sigma^2 - \sigma_{nm}^2 \) where \( \sigma_{nm}^2 \) is the signal in the normal state due to the nuclear moments.

The spectra from the CaIrSi\(_3\) were best described by three oscillating functions whereas the spectra from the CaPtSi\(_3\) could be described by just two\(^{35}\). The field dependence of the superconducting depolarization rates \( \sigma_{sc} \) are shown in Fig. 6 As the field increases the depolarization rates decrease as may be expected for a superconductor when the applied field is a significant fraction of the upper-critical field \( B_{c2} \). The field dependence of \( \sigma_{sc} \) can be used to determine the magnetic penetration depth and to give an estimate for the upper-critical field (see Fig. 5). \( B_{c2} \) can be independently verified using other measurements. The \( \sigma_{sc} \) \( (B) \) data shown in Fig. 6 were fit using Eq. 4

\[
\sigma_{sc} \left[ \mu s^{-1} \right] = A \times (1-b)(1+1.21(1-\sqrt{b})^3)\lambda^{-2} [\text{nm}], \quad (4)
\]

where \( \lambda \) is in nm, \( b = B/B_{c2} \) is the ratio of applied field to the upper critical field, and \( A \) is a prefactor related to the structure of the flux-line lattice \( (A = 4.83 \times 10^4 \) for a hexagonal lattice\(^{40,41}\)). Assuming the penetration depth follows either a two-fluid model \( \left( \lambda^{-2} (T) / \lambda^{-2} (0) = \left[ 1 - (T/T_c)^{4} \right] \right) \) or can be described using the local (London) approximation for an s-wave gap superconductor (see below) gives penetration depths \( \lambda(0) \) of 448(6) and 150(7) nm for CaPtSi\(_3\) and CaIrSi\(_3\) respectively. The upper critical fields estimated from the \( \sigma_{sc} \) versus \( B \) data and from extrapolations to zero kelvin of the \( B_{c2} (T) \) curves determined from \( M (H) \) loops collected at temperatures above 1.5 K (data not shown) are in good agreement with those reported by Eguchi et al.\(^{35}\) from magnetic and transport data, although as in Ref. \(^{35}\) there is considerable uncertainty associated with these estimates. More comprehensive data sets down to low temperatures are required to accurately determine \( B_{c2} (0) \) for both materials.

The temperature dependence of the muon depolarization rate \( \sigma \) for CaPtSi\(_3\) and CaIrSi\(_3\) are given in Fig. 6 For both samples, the data shows a plateau and then decrease as the temperature is increased. \( \sigma \) then levels off at a temperature slightly less than \( T_c \). The depolarization rates at higher temperatures \( (T \geq T_c) \) are small, in agreement with the zero-field data discussed above. This depolarization \( \sigma_{nm} \), is associated with the nuclear moments. Below \( T_c \), the depolarization rates are related to the magnetic penetration depth (see Eq. 4) and therefore the structure of the superconducting gaps for the two materials can be investigated. After subtracting \( \sigma_{nm} \) from \( \sigma \) to give \( \sigma_{sc} \), as described above, \( \lambda \) can be calculated at each temperature, with a correction for the strong field dependence of the depolarization rates made using the \( B_{c2} (T) \) data from Ref. \(^{33}\) and Eq. 4

\[
\frac{\lambda^{-2} (T)}{\lambda^{-2} (0)} = 1 + 2 \int_{\Delta(T)}^{\infty} \left( \frac{\partial f}{\partial E} \right) \frac{E}{\sqrt{E^2 - \Delta^2 (T)}} dE, \quad (5)
\]

where \( f = [1 + \exp(E/k_B T)]^{-1} \) is the Fermi function. The temperature dependence of the gap is approximated by \( \Delta(T) = \Delta(0) \tanh [1.82(1.018(T_c/T - 1))^{-0.5}] \). As can be seen from Fig. 7 the temperature dependence of \( \sigma_{sc} \) for both samples is very well described by this isotropic s-wave model giving \( \Delta (0) = 0.81(1) \) and \( \Delta (0) = 0.38(1) \) meV, and BCS ratios \( \Delta (0) / k_B T_c \) of 3.8(2) and 5.4(2), for CaPtSi\(_3\) and CaIrSi\(_3\) respectively. The value for the BCS ratio is CaPtSi\(_3\) slightly higher that the 3.5 expected in the weak-coupling limit. The higher value obtained for CaIrSi\(_3\), along with the strong field dependence of \( \sigma_{sc} \) shown in Fig. 6 could be evidence of a strong-coupling and/or multigap behavior with each gap having a similar temperature dependence. Such a suggestion is consistent with the departure from a pure s-wave behavior seen in the specific heat of CaIrSi\(_3\), although the specific heat results for CaIrSi\(_3\) are generally well explained by a weak-coupling BCS theory\(^{25}\).

IV. SUMMARY

In summary, we have investigated the superconducting compounds CaPtSi\(_3\) and CaIrSi\(_3\) by using muon-spin...
relaxation and rotation. There is no evidence of time-reversal symmetry breaking in either material, at least within the sensitivity of μSR. The superconducting parameters determined from this study are summarized in Table III. The temperature dependence of the penetration depths for both materials are consistent with s-wave isotropic gaps. The BCS ratios place both materials in the intermediate to strong-coupling limit.

**TABLE II:** Superconducting parameters of noncentrosymmetric CaPtSi$_3$ and CaIrSi$_3$ determined from the dc magnetization and muon spectroscopy data.

|         | CaPtSi$_3$  | CaIrSi$_3$ |
|---------|-------------|------------|
| $T_c$ (K) | 2.30(5)     | 3.50(5)    |
| $\lambda(0)$ (nm) | 448(6)      | 150(7)     |
| $\Delta(0)$ (meV) | 0.38(1)     | 0.81(1)    |
| BCS ratio | 3.8(2)      | 5.4(2)     |

**FIG. 7:** Normalized inverse square of the London penetration depth (the superfluid density) $\lambda^{-2}(T)/\lambda^{-2}(0)$ versus the reduced temperature $T/T_c$ for CaPtSi$_3$ and CaIrSi$_3$. The lines are fits to the data as described in the text. The closed symbols and solid line are the data and fit for the CaPtSi$_3$, the open symbols and dashed line are the data and fit for CaIrSi$_3$.

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