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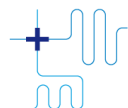
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ABSTRACT

A design and implementation of “in-cell” magnetic refrigeration to achieve sub-10 mK temperatures T in cryogen-free dilution refrigerators is presented. The ultra-low temperatures below 5 mK are attained in finite magnetic fields B up to 1 T. The holding time below 5 mK varies between about 3 and 30 h, depending on the final magnetic field after the demagnetization process. The developed technique can be used to study low dimensional devices at ultra-low electron temperatures in the high B/T regime.

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I. INTRODUCTION

Advances in cryogen-free technologies have greatly expanded the availability of millikelvin temperature platforms to a broad range of quantum research. However, cooling electrons in low-dimensional devices, such as graphene or GaAs heterostructures, is challenging due to weak electron–phonon coupling at these low temperatures. Compact and modular liquid ^3He immersion cells specially designed for cryogen-free refrigerators are routinely used at the National High Magnetic Field Laboratory’s (NHMFL or MagLab) High B/T Facility to ensure improved thermal coupling of the on-chip electrons within nano-sized low dimensional devices to the mixing chamber temperatures in the magnetic fields up to 14 T.^{1–5}

However, achieving temperatures below 10 mK on cryogen-free systems is difficult, as the cooling power relies on a pulse tube, which inevitably introduces mechanical vibrations at low frequencies that are challenging to isolate from the experiment. These vibrations, when combined with the magnetic field required for the demagnetization technique to achieve temperatures of about 1 mK,⁶ cause significant eddy current heating and, therefore, pose a significant experimental challenge for employing demagnetization in cryogen-free systems when compared to traditional wet refrigerators. Despite

these challenges, there are various types of nuclear demagnetization methods that have been implemented so far in cryogen-free systems in different scientific groups around the world.

Parallel nuclear demagnetization of external electrical leads.^{7,8} Cooling to temperatures below 10 mK is achieved by employing a series of demagnetization stages, where each measuring/control lead attached to a sample is routed through its own individual nuclear refrigerator. This arrangement removes the less efficient cooling path through an electrical insulator and replaces it with electronic Wiedemann–Franz cooling. However, the center of the final magnetic field is outside of the sample region, thus limiting the study of the sample to low magnetic fields unless a separate magnet is used for the experiment.

On-chip nuclear demagnetization.^{9,10} This method employs a minimal amount of refrigerant that is incorporated into a nano-electronic device. The refrigerant is connected electrically to the conduction electrons of the device, effectively bypassing the limitations of weak electron–phonon coupling and the inefficient thermal connection to off-chip wiring, particularly in high-impedance devices.

Nuclear demagnetization of an off-chip bundle.^{11–14} In this approach, the cooling below 10 mK and the thermalization of the device are treated as distinct processes, similar to traditional wet

cryogenic systems where the nuclear refrigerator and the magnet are separated from the experiment. Consequently, a tortuous electronic conduction path connects the refrigerant nuclei to the sample, posing challenges for achieving efficient thermalization of the sample down to the lowest temperatures. This challenge is particularly pronounced in the case of nanodevices, where the thermal link between the stage and the electrons within the device becomes notably weak.

Here, a different approach for a nuclear demagnetization experiment in a cryogen-free refrigerator is described, where powder/flake copper is suspended in the pure liquid ^3He used as the thermal media in an immersion cell. The combination of both the magnetic refrigerant and the pure liquid ^3He for thermalization of the sample¹⁵ and connecting leads^{16,17} facilitates the magneto-transport study of low-dimensional devices at ultra-low temperatures below 10 mK and in sizable magnetic fields, $1\text{ T} \lesssim B \lesssim 5\text{ T}$, corresponding to the final field after the demagnetization process. This approach was previously implemented in wet dilution refrigerators to study quantum fluids^{18,19} but to date has not been tested or deployed on cryogen-free platforms. The use of powder eliminates the conduction path along a copper bundle or wire, providing efficient thermal transfer directly to liquid ^3He through the large total surface area. The electrical conduction between the copper flakes is sufficiently low due to a stearate coating layer,¹⁹ thereby minimizing eddy current heating due to vibrations in the magnetic field. More specifically, the copper powder has a much stronger internal magnetic field of about 350 mT, Ref. 19, compared to only 0.4 mT in the case of bulk copper.⁶ This field provides a significant heat capacity to the copper nuclear spin system even at a zero external magnetic field, thereby protecting the cell from parasitic heat load and extending the hold times after the demagnetization process.

The following sections provide the details of the design, the assembly of instrumentation, and the outcomes of testing experimental cells, which allow the response of the sample to be monitored while both the temperature and magnetic field are slowly varied. Specifically, a pure liquid ^3He immersion cell with copper powder is constructed and tested in a Bluefors LD250 dilution refrigerator equipped with the 14 T superconducting magnet made by American Magnetics Inc. The data provide evidence for achieving temperatures of $2\text{ mK} \lesssim T \lesssim 5\text{ mK}$ in liquid ^3He in the magnetic fields of $B \sim 1\text{ T}$ for a time $t \sim 10\text{ h}$.

II. COPPER POWDER DEMAGNETIZATION CELL IN A CRYOGEN-FREE CRYOSTAT

The configuration of the low temperature stage is shown in Fig. 1(a). The cell, whose main outer components are made with standard polycarbonate, is entirely positioned in the bore of a superconducting magnet so the sample is in the maximum field region. The cell is held by the polycarbonate tubes (9.5 mm OD and 6.3 mm ID, not shown in Fig. 1) that are attached to the mixing chamber. Several annealed silver wires provide the thermal link to the mixing chamber through the aluminum heat switch shown in Fig. 1(b) (upper photo). The switch consists of four aluminum sheets thermally anchored to the copper leads using brass screws with molybdenum washers. The contribution of the thermal resistance of the aluminum oxide layer to pre-cooling times in high magnetic fields is discussed in the [supplementary material](#). The heat switch is located

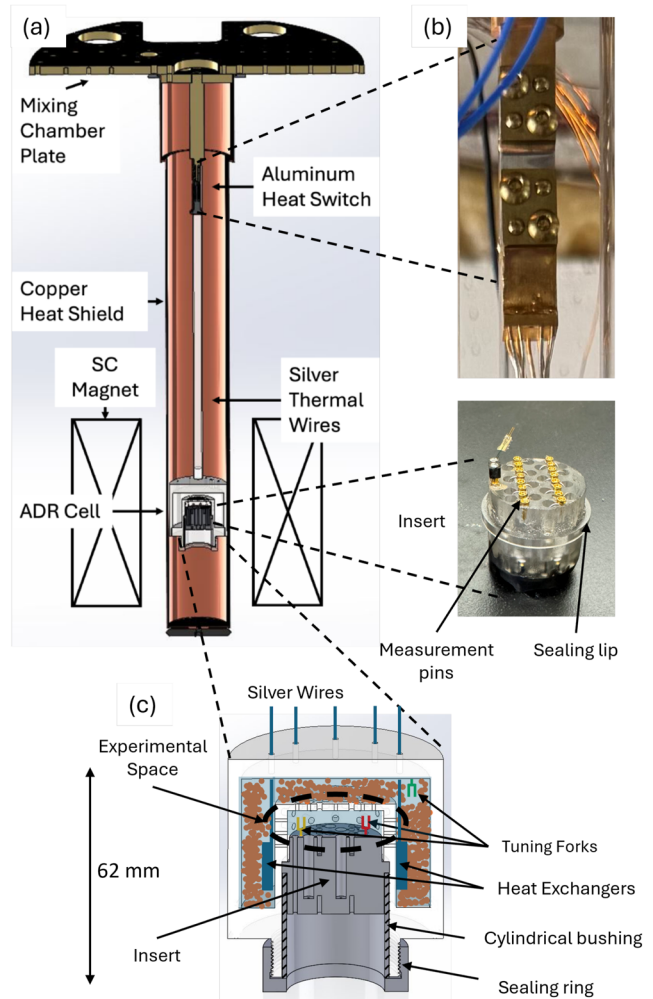


FIG. 1. (a) Layout of the cell and cryostat components. [(b), upper photo] The aluminum heat switch is thermally anchored to gold-plated copper at the top and gold-plated silver at the bottom using brass screws and Mo washers. [(b), lower photo] The insert with a connected tuning fork used for thermometry. A sample/device can be mounted on the insert with a standard 16-pin DIP socket. The pockets near each pin accommodate silver sinter heat exchangers for efficient thermalization. (c) Sketch of the cell incorporating copper powder in the “demagnetization” part. The experimental space is located in the center to provide additional thermal shielding. Three tuning fork thermometers are located in different parts of the cell to control the liquid He-3 level and its temperature.

in the fringe field region, so it switches during the ramping of the magnetic field at a center strength of about 4 T. The fringe field at the position of the heat switch is above 10 mT in this case; therefore, it is high enough to destroy the superconducting state of aluminum. The homemade heat shield is attached to the mixing chamber plate to protect the parts of the setup below the heat switch from radiation heat load from higher temperature parts of the cryostat. Importantly, no construction changes are made to the cryostat.

The cell design is shown in Fig. 1(c). It contains two main parts, the “demagnetization space” and the “experimental space.”

The “demagnetization space” is filled with copper powder. It is initially compressed to about 30% packing fraction using a hydraulic press. The resulting blocks are then broken into smaller pieces and added to the cell. An array of annealed silver wires with silver sinter heat exchangers is placed symmetrically into the copper powder for initial pre-cooling of the copper powder and liquid ^3He before the demagnetization process. The “experimental space” is positioned at the center of the magnetic field region. The insert, shown in Fig. 1(c) as the gray part in the middle and also in Fig. 1(b) (lower photo), is designed to hold a sample/device in the “experimental space” using electrical pins secured to the insert with silver epoxy to make it compatible with a standard 16-pin DIP socket. A light layer of Cello-Seal[®] C601-100 vacuum grease is applied to the top edge of the sealing lip, and then the insert is positioned into the cell body by a polycarbonate cylindrical bushing, which is held in place by a polycarbonate threaded ring using light finger-only torque. Each measurement wire is equipped with its own silver sinter contact housed in a pocket on the insert, allowing for rapid thermalization of the electrons to the liquid ^3He bath. During this experiment, the two tuning fork (TF) resonators are used in the “experimental space” to measure the temperature of the liquid ^3He . Importantly, tuning fork thermometry does not depend on the applied magnetic field up to at least 14 T,²⁰ so the temperature measurements at different magnetic fields do not require recalibration of the thermometers.

In total, the cell contains about 66 g of copper powder with a surface area of about 61 m². The surface area of the silver sinter inside the cell is about 13 m². The amount of ^3He required to fill the cell is about 16 l of gas at room temperature. It is important to determine the total entropy of the nuclear spin system and liquid ^3He in the cell at the conditions prior to demagnetization. The entropy of non-interacting copper nuclear spins is given by⁶

$$\frac{S_{\text{Cu}}}{Nk_B} = \ln \left(\frac{\sinh(2x)}{\sinh(x/2)} \right) + (x/2)[\coth(x/2) - 4 \coth(2x)], \quad (1)$$

where $x = 0.54 [\text{mK T}^{-1}] \left(\frac{B}{T} \right)$ in the case of copper. The entropy of liquid ^3He is given by¹⁸

$$S_{\text{He}} = n \cdot 24 [\text{J K}^{-2} \text{mol}^{-1}] \cdot T. \quad (2)$$

The temperature dependence of the total entropy $S = S_{\text{Cu}} + S_{\text{He}}$ is shown in Fig. 2 in different magnetic fields. The arrows represent the demagnetization process from different starting conditions. It shows that the process would be efficient for the current cell configuration if initial temperatures below 20 mK could be achieved in a 14 T magnetic field, even if an additional heat load $Q \sim 10 \text{ nW}$ is present.

III. THERMOMETRY

The process of using tuning forks immersed in pure liquid ^3He to measure temperature involves tracking the linewidths of their resonances.²⁰ The tuning fork dimensions are characterized by their length L , thickness e , and width ℓ . The linewidth Δf reflects the hydrodynamic forces acting on the tuning forks based on the Euler–Bernoulli beam theory, which yields the expression^{21–24}

$$\Delta f_1 = f_0 \left(\frac{\pi \rho \ell}{4 \rho_q e} \right) \frac{\Gamma_{\text{Im}}(f_1)}{\left[1 + \left(\frac{\pi \rho \ell}{4 \rho_q e} \right) \Gamma_{\text{Re}}(f_1) \right]^{3/2}}, \quad (3)$$

where $f_0 = \frac{\alpha_1^2 \ell}{4 \pi L^2} \sqrt{\frac{E}{3 \rho_q}}$ is the vacuum frequency and Δf_1 and f_1 are from the first resonance mode due to the hydrodynamic damping. The other constants are the densities of quartz (ρ_q) and ^3He (ρ), the Young’s modulus of quartz (E), and $\alpha_1 = 1.875$, which is the first positive root of $1 + \cos(\alpha_n) \cosh(\alpha_n) = 0$. In addition, f_0 and f_1 are related by the equation

$$f_0^2 = f_1^2 \left(1 + \frac{\pi \ell}{4 \rho_q e} \rho \Gamma_{\text{hydro}}^{\text{Re}}(f_1) \right). \quad (4)$$

Finally, the dimensionless hydrodynamic function is given by

$$\Gamma_{\text{hydro}}(f) = \Gamma_{\text{Re}} - i \Gamma_{\text{Im}} = 1 + \frac{4 K_1(\sqrt{i \text{Re}})}{\sqrt{i \text{Re}} K_0(\sqrt{i \text{Re}})}, \quad (5)$$

where K_1 and K_0 are first-order modified Bessel functions of the second kind, in addition to the Reynolds number $\text{Re} = \frac{\pi \rho f \ell^2}{2 \eta}$, where η is the dynamic viscosity.

This equation provides the viscosity of the liquid ^3He in which the tuning forks are immersed. Since the temperature dependence of liquid ^3He viscosity is well established, these functions are used to convert the measured viscosities to the temperature of the liquid in its monotonic region from 1 K down to 1 mK.²⁵

The tuning fork response is calibrated above 100 mK, using the resistive thermometer anchored to the silver wires at the top of the cell (Fig. 3). The red points are taken after setting the mixing chamber temperature to different values and allowing the cell to thermalize through the heat switch in the superconducting state over the course of 24 h. The thermometry model is then fitted against

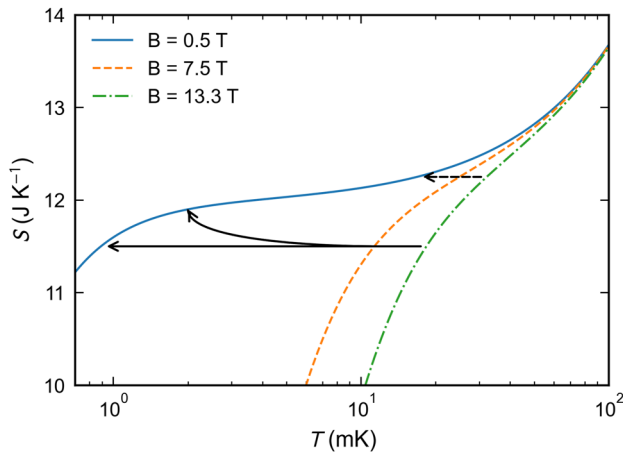


FIG. 2. Total entropy of the “liquid ^3He + copper powder” system calculated at different magnetic fields using cell parameters. As shown by the solid horizontal arrow, the demagnetization process is efficient when the cell is precooled below about 20 mK, if the applied field is 13.3 T. The curved solid arrow shows the demagnetization process when the system experiences an additional heat load $Q \sim 10 \text{ nW}$. The dashed arrow shows an inefficient demagnetization process when the starting temperature is too high. At smaller magnetic fields, the lower starting temperatures are required, as shown by the light orange line.

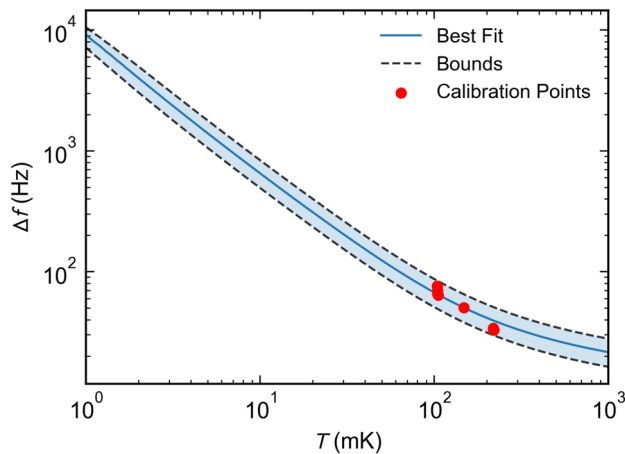


FIG. 3. Red points represent the measured linewidths of the tuning forks at known temperatures. The curve is the best fit from the hydrodynamic model, while the shaded region shows the uncertainty area, as described in the text.

these calibration points by adjusting the dimension of the tuning forks within a physically plausible range to provide the best fit and thereby establish the bounds of the calibration points. This adjustment is shown in Fig. 3 as a shaded region, which essentially defines the uncertainty of the temperatures.

Since the resistive thermometer used for the calibration process loses sensitivity below 100 mK, the fits to the model are performed above this temperature, and then the hydrodynamic model is used to capture the temperature dependence of the linewidths down to about 1 mK.²⁰

IV. PERFORMANCE

Multiple cooling cycles have been performed in three processes: magnetized pre-cooling, demagnetization, and holding time. After applying a magnetic field of 13.3 T, the pre-cooling process takes about 4 days for the cell to reach 16 mK. At this point, the demagnetization process is expected to be efficient (Fig. 2). The magnetic field is then ramped down at 0.2 mT/s from 13.3 to 5 T and at 0.1 mT/s from 5 T down to the final fields. This process is quasi-adiabatic and shows continuous cooling down to 2 mK. The B/T ratio is nearly constant down to at least 5 mK (see the [supplementary material](#)). Following the end of the demagnetization, the cell warms from its base temperature over the course of 1–2 days depending on the final demagnetization field. The time traces of the inverse temperature are shown in Fig. 4.

Under a constant, post-demagnetization heat load $\dot{Q}$, the temperature T of the nuclear spins is to evolve with time as given by⁶

$$T^{-1} = T_{\min}^{-1} - \frac{\mu_0 \dot{Q}}{n \lambda_n B_{\text{eff}}^2} t, \quad (6)$$

where μ_0 and n are the vacuum permeability and the number of copper nuclei (in moles), respectively. The quantity $\lambda_n/\mu_0 = 5N_0\mu_n^2 g_n^2/4k_B$ is reported to be $3.22 \mu\text{J K T}^{-2} \text{mol}^{-1}$ for copper nuclei.⁶ The internal magnetic field of copper is higher than the value

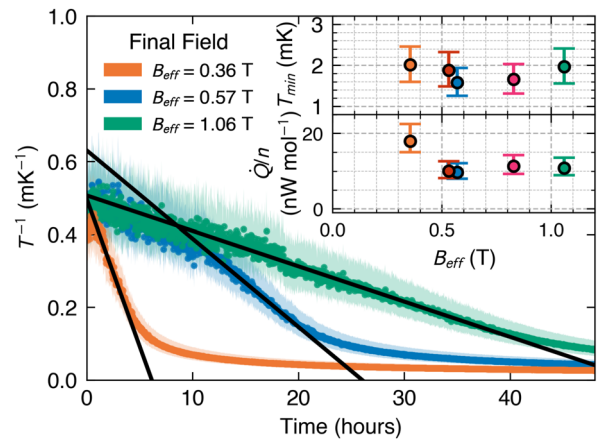


FIG. 4. Inverse temperature as a function of time at different magnetic fields. The slopes of the linear fits indicate a heat load of a few nW/mol. Inset: Minimum temperature and heat load per mole at different magnetic fields.

reported in bulk metal and has been measured to be ≈ 350 mT, which provides the effective magnetic field $B_{\text{eff}}^2 = B^2 + B_{\text{int}}^2$.^{6,19}

The measured $1/T$ values have a linear time dependence (Fig. 4, solid lines). Assuming that the liquid ^3He is well coupled to the electron temperature of the copper due to a high contact area, the minimum achieved temperature and the average heat load on the cell are calculated using Eq. (6) as shown in the inset of Fig. 4. This analysis suggests our cell has reached a minimum temperature of $\lesssim 2$ mK. Due to uncertainty in the calibration of the tuning fork thermometry, the uncertainties are ± 0.5 mK. Two demagnetization runs with final fields $B_f = 60$ mT and $B_f = 400$ mT have higher minimum temperatures than the adiabatic, constant B/T ratio estimate, and these outcomes are associated with the presence of higher heat loads near the end of the demagnetization process due to flux jumps in the main magnet at low current levels.²⁶

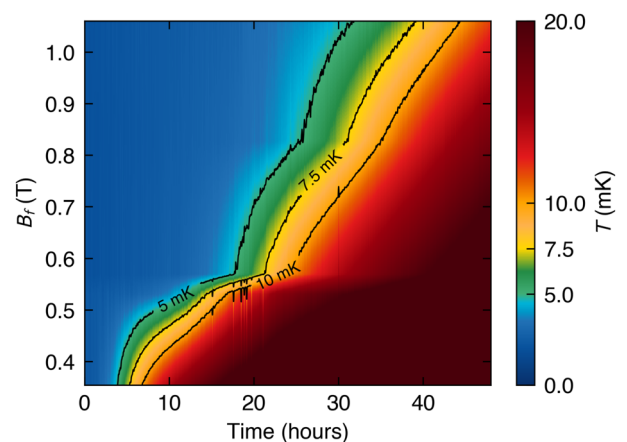


FIG. 5. Final magnetic field vs time contour plot of the temperature showing the performance/hold time during the post-demagnetization warm-up. The black lines show the equal temperature lines across the various runs.

A noteworthy outcome is that the heat load following the end of each demagnetization (Fig. 4, inset) does not change significantly with the final applied field, indicating negligible eddy current heating. This result directly reflects a key design objective of the cell and, therefore, strongly motivates further refinement and iteration to achieve improved performance.

Finally, there is a clear deviation from expected linear dependence when $1/T \lesssim 0.1 \text{ mK}^{-1}$ (Fig. 4). One possibility is that the plastic cell body is not being cooled efficiently and remains at temperatures higher than the mixing chamber, thereby leading to an additional radiation heat load. Presently, the source of this behavior is being investigated.

In Fig. 5, a color plot of the temperature time evolution after demagnetization to various final magnetic fields is shown. Voltage spikes in the magnet at low currents/magnetic fields²⁶ have caused shorter than expected hold times at final fields below 500 mT. The holding time below 5 mK and above 500 mT ranges from 18 to 30 h.

V. CONCLUSION

“In-cell” magnetic cooling below 2 mK has been successfully demonstrated using a cryogen-free refrigerator. Remarkably, no eddy-current heating is observed, a result of particular importance for pulse-tube-cooled systems. The prototype cell required only 66 g (1.04 mol) of copper, representing a substantial reduction relative to traditional bundle-stage designs that typically employ several kilograms of material. Crucially, this compact approach requires no modifications to the existing cryostat and, therefore, provides a practical route for cooling nanoelectronic materials and devices to sub-5 mK temperatures in finite magnetic fields. As a result, the accessible parameter space for ultralow temperature quantum experiments is significantly expanded, enabling broader adoption by the research community.

SUPPLEMENTARY MATERIAL

The supplementary material covers the adiabatic demagnetization process, which takes ~24 h. At the beginning, the liquid ³He cools along the expected adiabatic curve, but deviations emerge below 5 mK due to a combination of residual heat load on the cell and thermal decoupling between the liquid ³He and the copper refrigerant.

The supplementary material also covers the heat switch design. The original design used aluminum sheets clamped to copper or silver, while an upgraded diffusion-welded version was developed to reduce contact resistance from the aluminum oxide layer.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Alexander M. Donald: Data curation (equal); Formal analysis (equal); Software (equal); Writing – original draft (equal); Writing – review & editing (equal). **Nicolas Silva:** Data curation (equal); Investigation (equal); Writing – review & editing (equal). **Christopher J. Ollmann:** Conceptualization (equal); Data curation (equal); Investigation (equal); Writing – review & editing (equal). **Roch Schanen:** Data curation (equal); Writing – review & editing (equal). **Chao Huan:** Data curation (equal); Writing – review & editing (equal). **Sangyun Lee:** Writing – review & editing (equal). **Dominique Laroche:** Data curation (equal); Writing – review & editing (equal). **Richard P. Haley:** Conceptualization (equal); Data curation (equal); Writing – review & editing (equal). **Mark W. Meisel:** Data curation (equal); Validation (equal); Writing – review & editing (equal). **Rasul Gazizulin:** Conceptualization (equal); Data curation (equal); Investigation (equal); Supervision (equal); Writing – original draft (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are openly available in Zenodo at <https://doi.org/10.5281/zenodo.20286028>.

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