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Cernox® versus germanium cryogenic temperature sensor stability comparison over the 1 K to 27 K temperature range

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Abstract. Germanium resistance thermometers (GRTs) have played a crucial role in the dissemination of both the 1976 Provisional 0.5 K to 30 K Temperature Scale and the International Temperature Scale of 1990. GRTs combined a small physical package with a sensing element that possessed high temperature sensitivity and very high stability over thermal cycling and time. For thermometry applications, the best GRTs were fabricated with arsenic doping, and these devices were commercially available from multiple companies for nearly 50 years. At present, however, commercially available GRTs are nearly nonexistent as germanium crystal growers are reluctant to work with arsenic dopants. For applications below 30 K, a promising alternative is the Cernox® resistance thermometer (CxRT). CxRTs have characteristics similar to GRTs including small physical size and high stability. To date, no direct comparison of calibration stability between CxRTs and GRTs using an identical test protocol has been performed. In the present work, a group of 18 GRTs and 25 CX-1050-CU devices were calibrated against a combination of ten National Institute of Standards and Technology (NIST) or National Physical Laboratory (NPL) calibrated thermometers over the 1.2 K to 30 K temperature range, and subsequently thermally cycled slowly from room temperature to 1.2 K approximately once per week for 45 weeks. Following the 45 thermal cycles, both groups were recalibrated against 10 NIST/NPL calibrated thermometers. The data were analyzed in terms of temperature shift between the pre- and post-thermal cycling for both groups of thermometers. For GRTs, the results show a post- versus pre-thermal cycling calibration average stability of ±1 mK across the 1.2 K to 27.5 K temperature range with standard deviations ranging from 0.5 mK at 1.2 K to 1.5 mK at 27.5 K. For CxRTs, the results show a post- versus pre-thermal cycling calibration average stability of ±3 mK over the 1.2 K to 27.5 K range but with tighter low temperature grouping below 10 K. Additionally, the CxRTs exhibited calibration offsets for their first eight thermal cycles, but showed stability approaching GRT stability after the first eight thermal cycles suggesting additional initial thermal cycling would lead to a more stable CxRT.

1. Introduction

Germanium resistance thermometers (GRTs) have been widely used in cryogenics since the 1960s. The germanium sensing element can be doped to varying levels to produce temperature sensors useful as low as 20 mK or as high as 100 K. The seminal work by Blakemore showed that the best resistance versus temperature curves are achieved using germanium doped with n-type dopant arsenic and co-doped with p-type dopant gallium, with "best" defined as smoothest curve with minimal inflection points. [1] With their small size, high sensitivity, and excellent temperature stability, GRTs were used extensively in the 1970s, along with standards grade rhodium-iron resistance thermometers (RIRTs), in the development of the EPT-76 temperature scale. Given their importance to cryogenic thermometry, much research has investigated the stability of GRTs over thermal cycling and time. [2-9] An excellent review of the GRTs development and properties can be found at [10].

Currently, it is extremely difficult to obtain GRTs due to the difficulty in procuring arsenic-doped germanium crystals. At present, the best alternative to GRTs is the Cernox resistance thermometer (CxRT), first commercialized by Lake Shore Cryotronics, Inc. (Lake Shore) in 1992. [11] CxRTs were initially developed for use in supercollider applications that combined high radiation with high magnetic field environments, but they've also become a sensor of choice for aerospace applications involving cryogenic temperatures. Due to the long lifetime of these applications, CxRT temperature stability over thermal cycling and time has also been extensively investigated. [9, 12-15] As shown in Figure 1, calibration uncertainty increases with each step along the calibration chain from national laboratory primary standards to commercial thermometers. To date, GRT and CxRT stability have not been measured when both were subjected to the same conditions and recalibrated at the same level in the calibration chain. The present work examines the stability of GRTs and CxRTs operated under the same thermal cycling conditions for a one-year period with pre- and post-calibrations performed against the same national laboratory-calibrated thermometer standards.

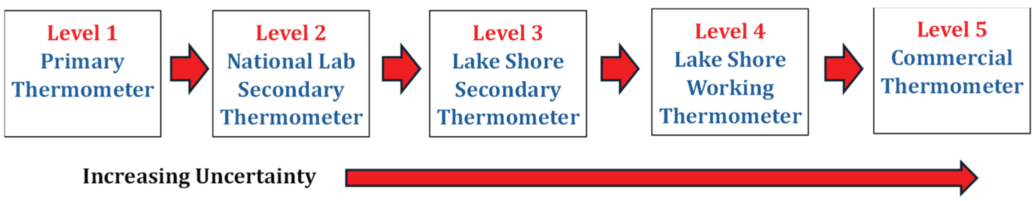

Figure 1. Temperature sensor calibration chain. Uncertainty increases with each increasing level.

2. Experiment

CxRTs and GRTs share many similarities including physically small compact size, negative temperature coefficient (NTC) resistance versus temperature behavior, and a resistance versus temperature response curve that can be tailored to maximize performance for a specified temperature range. As mentioned previously, germanium can be doped with arsenic and gallium to fabricate sensing elements optimized for the 1 K to 27.5 K temperature range. The CxRT sensing material consists of a matrix of conducting ZrN mixed with nonconducting ZrO manufactured through a sputtering deposition process. By controlling the ratio of ZrN to ZrO components, the CxRT resistance versus temperature response can also be tailored to maximize performance over the 1 K to 30 K temperature range. The CxRT model most closely matching the resistance and sensitivity of the GRT model GR-1400-AA is the model CX-1050 series. The resistance and sensitivity versus temperature characteristic for both models can be found at [11] and [15]. Assuming operation at 2 mV excitation utilizing a voltmeter with 0.01 μV resolution, the resulting GRT model GR-1400-AA temperature measurement resolution ranges from 2 μK at 1.2 K to 20 μK

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at 4.2 K to 80 μK at 27 K. The equivalent temperature resolutions for the CxRT model CX-1050 are slightly lower, but typically within a factor of two of the GR-1400-AA resolution. The CxRT model tested in this work is the model CX-1050-CU consisting of the model CX-1050-SD attached to a copper bobbin with leads wrapped around the bobbin and epoxied in place for heat sinking. Details of both the GRT and CxRT packaging can be found at [11].

Lake Shore's temperature traceability to the ITS-90 is maintained on a set of over 60 GRT, RIRT, and standard platinum resistance thermometers that have been directly calibrated (Level 3) by a national standards lab such as NIST in the US or NPL in the UK. On a yearly basis, LSCI completes a series of calibrations (informally called "standards" runs) where the everyday working standard thermometers (Level 4) used to assign temperature on commercial calibration runs (Level 5) are calibrated against a subset of thermometer standards that have been directly calibrated by national standards labs (level 3). For the last 45 years, LSCI has used the GRT model GR-1400-AA as the working standard thermometer over the 1 K to 27.5 K temperature range in their Temperature Sensor Calibration Facility. Over this time period, Lake Shore has accumulated a significant volume of data on the stability of GRTs acquired during these standards runs. To date, comparable CxRT stability data has not been acquired as their calibrations have always been performed at the commercial calibration level (i.e., Level 5). As a result, CxRTs could not be shown to perform as well or better than the GRTs they were calibrated against. In the present work, the working standards GRTs and CxRTs were calibrated directly against standards calibrated by national standards labs on the same standards runs (Level 4).

On a typical 1 K to 27.5 K standards run, a set of ten NIST/NPL calibrated thermometers consisting of a combination of GRTs, RIRTs, and SPRTs are chosen to define the ITS-90 scale to which the working standards thermometers are calibrated. Every temperature in this range is covered by a minimum of five NIST/NPL-calibrated thermometers. These devices, along with the everyday working standard thermometers, are loaded onto an OFHC copper calibration block mounted to a calibration station. During the run, resistance is measured for every device at approximately 40 temperatures spanning the 1.2 K to 27.5 K temperature range. At each temperature setpoint, the measured resistance for each NIST/NPL-calibrated thermometer standard is converted directly to temperature using the original curve fit provided by the calibrating national laboratory. The average temperature of all NIST/NPL calibrated thermometers defines the temperature at that setpoint.

A total of 18 model GR-1400-AAs and 25 model CX-1050-CUs were chosen for this experiment. The chosen GRTs had an extended history of use as working standards in Lake Shore's calibration facility whereas the CX-1050-CUs were built especially for this test and completed the normal production assembly and test screening which included extended thermal shocking between room temperature and 77.35 K along with multiple thermal shocks to 4.2 K. Initially, both sets of devices were calibrated over the 1.2 K to 27.5 K temperature range against a set of ten NIST/NPL calibrated standard thermometers during the 2024 standards runs. This was the first calibration for the CxRT test devices. A detailed description of the calibration process can be found at [9]. Following this baseline calibration, the 18 GRT working standards were divided up among multiple temperature calibration probes and used as working standards for commercial calibrations over the course of the next year, averaging about one slow thermal cycle per week over the room temperature to 1.2 K temperature range. Likewise, 21 of the 25 CX-1050-CUs were divided among five calibration probes and underwent the same slow thermal cycling from room temperature to 1.2 K about once per week. These CxRT devices were measured as DUTs on each commercial calibration run over the course of the year. Four CxRTs were stored at room temperature as control samples with no thermal cycling. Each commercial calibration run takes one week to complete yielding approximately 45 runs per year after allowing for maintenance, standards runs, and company shutdown weeks between consecutive yearly standards runs.Following the one-year usage period consisting of approximately 45 thermal cycles, the 18 working standard GRTs and all 25 test CX-1050-CU devices were recalibrated against a set of ten NIST/NPL-calibrated thermometer standards on the 2025 standards runs.

3. Data and Results

In examining the GRT and CxRT stability, a proper starting point begins with an analysis of Lake Shore's ability to maintain the ITS-90 scale. For a standards run consisting of ten NIST/NPL calibrated thermometers, a typical intercomparison of these devices over the 1.2 K to 27.5 K is shown in Figure 2 with the thick, black dashed line indicating a ±1 standard deviation about the group mean.

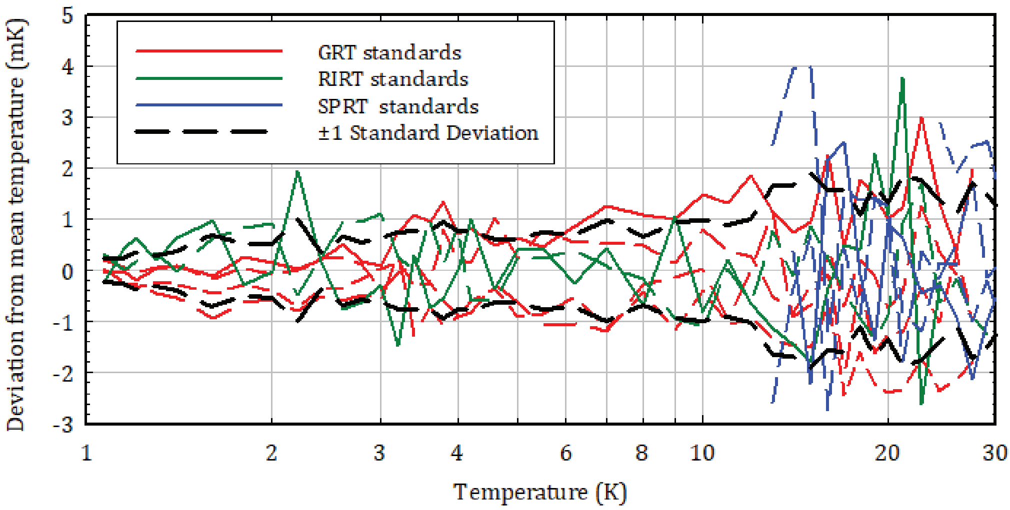

Figure 2. Agreement between 10 NIST/NPL calibrated standards on a typical standards run.

For the GRT and CxRT stability, the pre- and post-one year calibration calibrations were compared with the data analyzed as equivalent calibration shift, ΔT, calculated as

$$\Delta T(T) = [\, R_{final}(T) - R_{initial}(T)\,] \,/\, S_T(T) \qquad (1)$$

where $R_{initial}(T)$ and $R_{final}(T)$ are the resistances measured during the initial and final calibrations at temperature T respectively, and $S_T(T)$ is the temperature sensitivity, i.e. slope of the R vs T curve, in ohms/Kelvin at temperature T. As before, the temperature assigned at each temperature setpoint during the standards run is the average of all NIST/NPL-calibrated thermometer standards measured at that temperature. The measured data is curve fit using a cubic spline to adjust for temperature setpoint variations between the initial and final calibrations.

The recalibration plots for the 18 working standard GRTs are shown in Figure 3. Note that the oscillations in the recalibration plots can be an artifact of curve fitting. The group average calibration shift is shown in Figure 4 with error bars indicating a ±1 standard deviation about the average offset. Note the excellent stability with a group average within ±1 mK across the 1.2 K to

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27.5 K temperature range with a standard deviation ranging from 0.5 mK at the lowest temperature increasing to 1.5 mK at the upper temperatures. The GRT Level 4 calibration uncertainty is calculated by combining the ability to maintain ITS-90 along with instrumentation and other sources of error and is shown as the black dashed line in Figure 4.

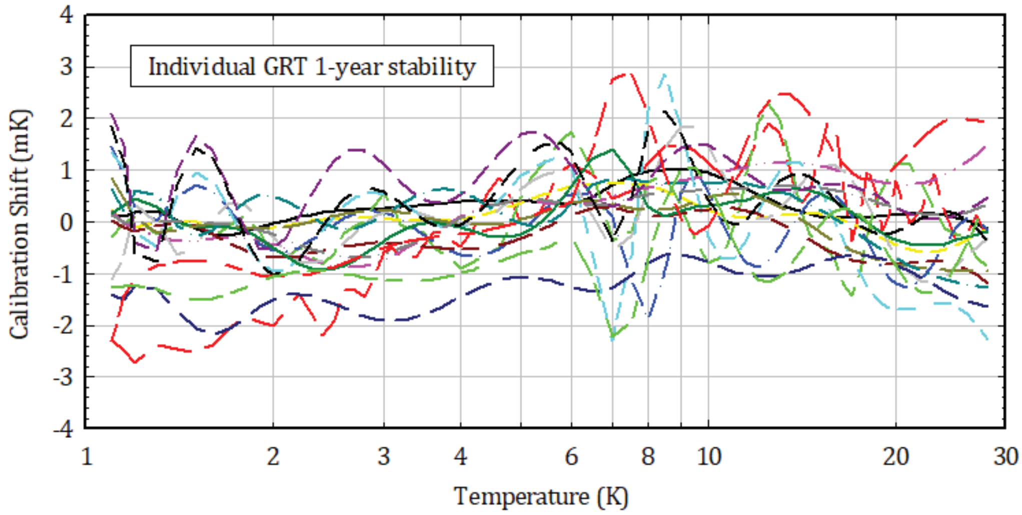

Figure 3. Individual calibration shift for each of the 18 GRTs.

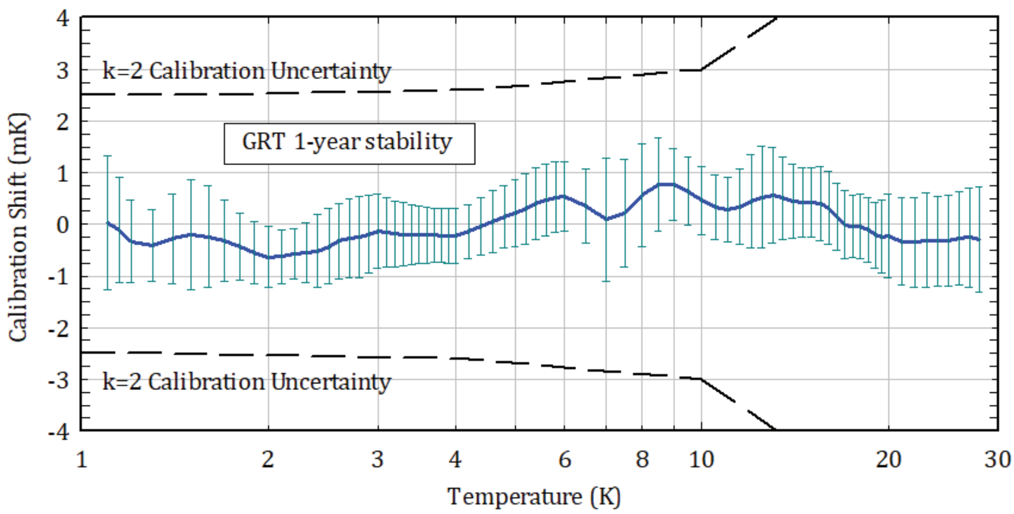

Figure 4. Average calibration shift of 18 GRTs with ±1 standard deviation error bars.

The individual CxRT recalibration comparisons are shown in Figure 5. The four control CxRTs that were stored at room temperature are also shown in this figure in dark red and show no discernible difference in behavior from the 21 CxRTs that were subjected to the 45 thermal cycles. The group average with ±1 standard deviation error bars is shown in Figure 6. These data show an initial offset of +1 mK at 1.2 K, rising to +2 mK at 8 K, and then decreasing to -2 mK at 27.5 K. Below 10 K, the CxRTs behavior is more tightly grouped than the GRTs with standard deviations that monotonically increase from 0.25 mK at 1.2 K, to 0.5 mK at 4.2 K, to 2.5 mK at 27.5 K. As before, the CxRT level 4 uncertainty is shown as the black dashed line in Figure 6.

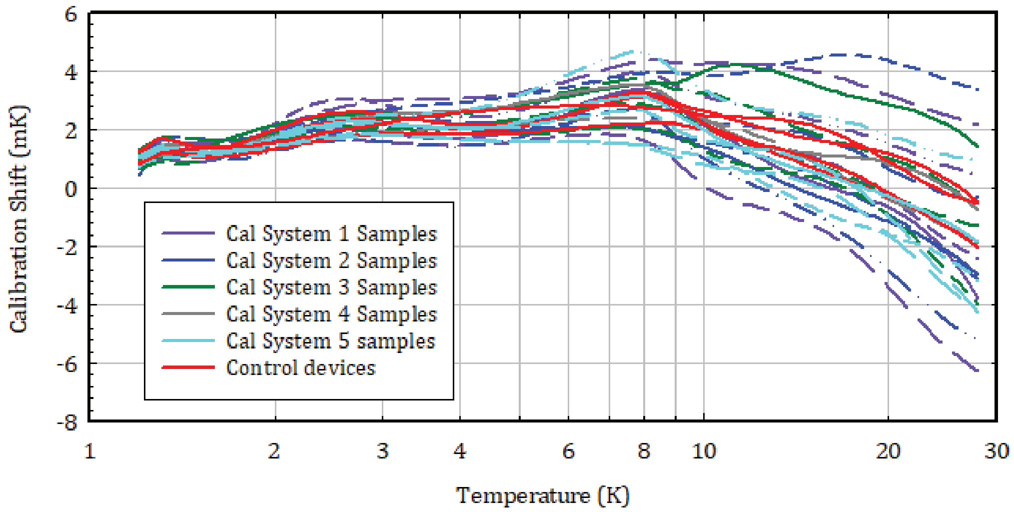

Figure 5. Individual calibration shift of 25 CxRTs. Four control CxRTs are highlighted in red.

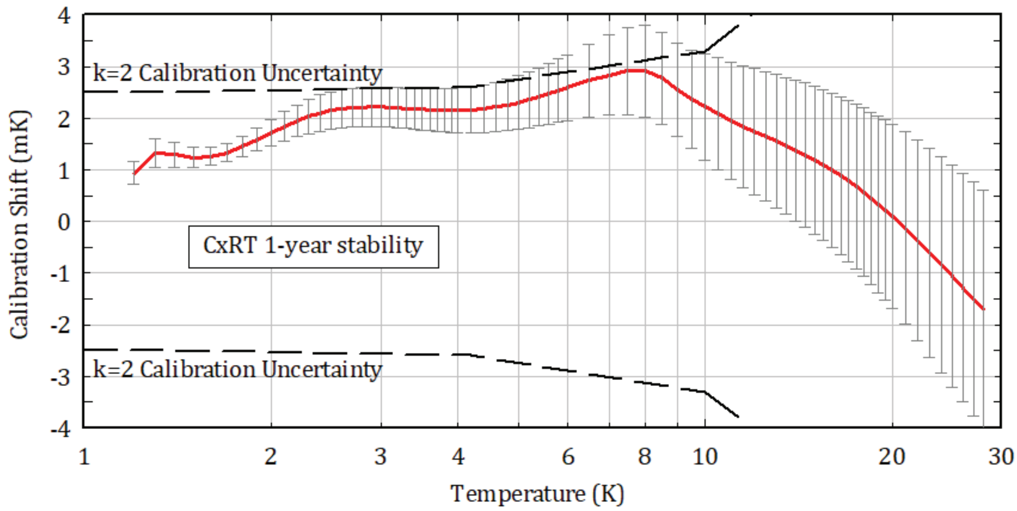

Figure 6. Average calibration shift of 25 CxRTs with ±1 standard deviation error bars.

While the CxRT stability was very good, especially below 10 mK, the results from Figures 5 and 6 show that the CxRTs are slightly less stable than the GRTs over the course of the test. Additional data acquired over the course of the year-long test provides insight into the pattern of the CxRT temperature shift with respect to thermal cycling. After the CxRTs' initial calibration, each CxRT was loaded onto a temperature calibration probe along with a GRT working standard thermometer. Over the course of the year-long test, each CxRT was calibrated as a DUT (Level 5) using that probe's unique GRT working standard and calibration electronics. For each CxRT, the data at temperatures 1.4 K, 4.2 K, 10 K, and 20 K were collected and analyzed in terms of temperature shift from the initial standards run calibration as a function of thermal cycle.

Figure 7 shows the average CxRT temperature offset at these four temperatures as a function of thermal cycle using the initial standards run calibration as the baseline These data show an immediate offset between the first standards run calibration and their first commercial calibration ranging from 0.75 mK at 1.4 K to +3.2 mK at 20 K. The initial offsets here are not completely unexpected as the CxRT calibrations will have any offset from the GRT working standard included, as well as offsets from different sets of electronics used for each calibration station. Additionally, the observed offsets are all within the uncertainty limits for a CxRT calibration at this level. More importantly, note that the CxRT devices stabilize after about eight thermal cycles. In Figure 8, the offsets of all subsequent calibrations are plotted using the eighth calibration as the baseline. After the eighth calibration, the CxRT stability approaches the GRT stability with a group average stability of ±1.5 mK across the 1.2 K to 27.5 K temperature range.

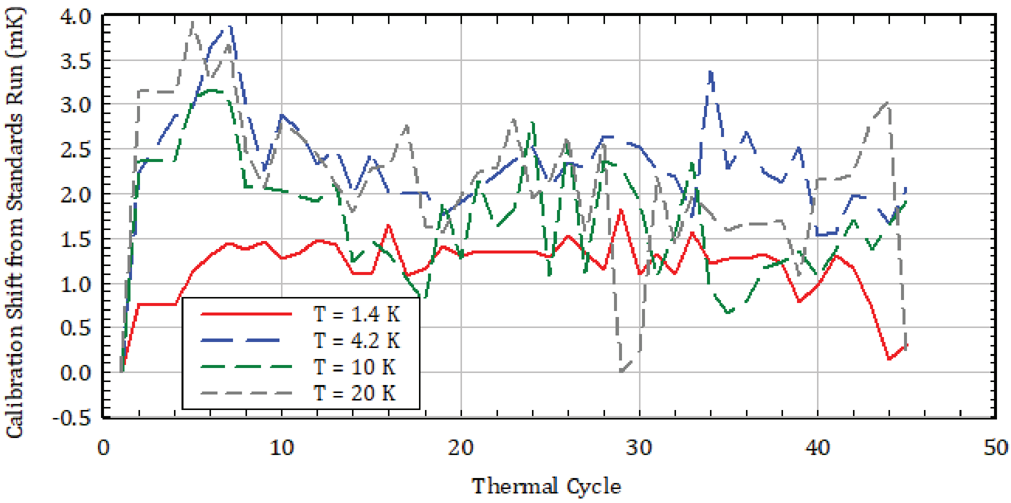

Figure 7. CxRT stability versus thermal cycle number for select temperatures.

4. Conclusions

This work has compared the temperature of stability of GRTs and CxRTs over the 1.2 K to 27.5 K temperature range when operated under the same conditions for a one-year period. Devices were calibrated directly against NIST/NPL calibrated thermometer standards for the pre- and post-calibrations. After a one-year period consisting of about 45 slow thermal cycles from room temperature to 1.2 K. The 18 GRTs remained stable to an average of ±1 mK over the 1.2 K to 27.5 K temperature range with a standard deviation ranging from 0.5 mK at 1.2 K to 1.5 mK at 27.5 K. The CxRTs exhibited a higher average stability of ±3 mK over the 1.2 K to 27.5 K but tighter low temperature grouping with a standard deviation ranging from 0.25 mK at 1.2 K, to 0.5 mK at 4.2 K, to 2.5 mK at 27.5 K. Further, the CxRTs displayed an immediate shift after their initial calibration, but after approximately eight thermal cycles, they showed a stability approaching GRT stability over the remaining thermal cycles in the test. The results suggest that the CxRTs become more stable after their eight initial thermal cycles to 4.2 K. Further work is in progress to test that conjecture.

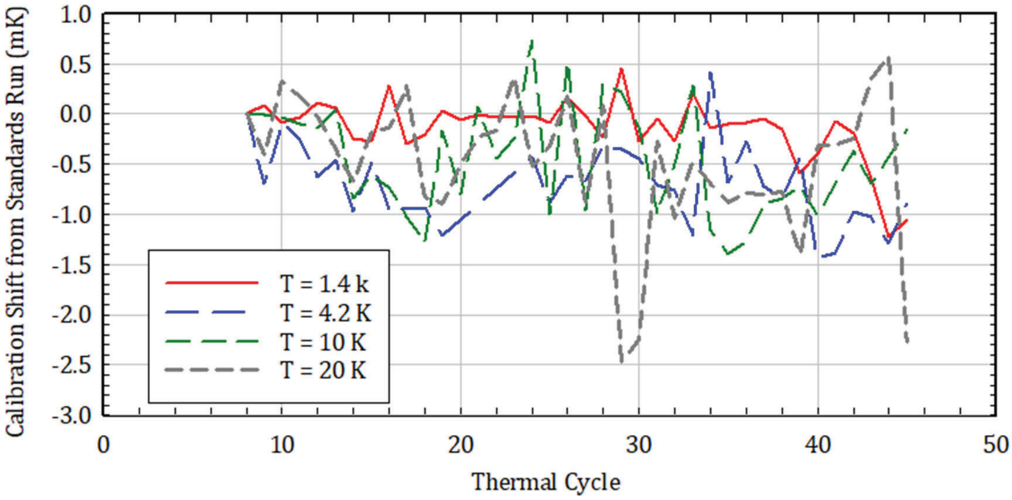

Figure 8. CxRT stability versus thermal cycle number for select temperatures.

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