

Normal and Anomalous Self-Heating in Capsule-type Resistance Thermometers in the Range 1 K to 273 K

W. L. Tew and C. W. Meyer

NIST, Gaithersburg, MD USA

Abstract. The normal self-heating coefficient for standard capsule-type resistance thermometers is gas-mediated over most of their designed range of use. This is readily modeled and calculated for thermometers with helium fill gas from known transport properties for temperatures above the superfluid-film transition at 1.3 K. Anomalous self-heating exists in certain capsules where air contamination has occurred, and this observed self-heating is compared with predictions from the thermal conductivity of gas mixtures. The experimental aspects of the self-heating measurements are described as well as certain aspects of the thermal transport modeling. Some associated interpolation errors are described and useful screening techniques are suggested for capsule thermometers over cryogenic temperature ranges.

Keywords: Self-heating coefficient; resistance thermometer; thermal impedance; rhodium-iron; standard platinum.

INTRODUCTION

Standard capsule-type resistance thermometers are necessary for realization and/or dissemination of the ITS-90 over its cryogenic ranges at the lowest achievable uncertainties. These include the capsule-type varieties of both standard platinum resistance thermometers (SPRTs) and miniature platinum resistance thermometers (MPRTs) for the range 13.8 K to 273 K, rhodium-iron resistance thermometers (RIRTs), and platinum-cobalt resistance thermometers (PCRTs) for the range 0.65 K to 24.5 K or 273 K. All these thermometers are designed for cryogenic service, filled with (10 to 30) μmol of helium heat-exchange gas, and hermetically sealed. The exchange gas is the dominant heat transport medium for exchanging heat with the environment in these designs. Power dissipation ΔP in the resistance element causes an elevated internal temperature difference ΔT_{sh} relative to the environment being sampled, commonly referred to as “self-heating”. The term ‘self-heating coefficient’ [1] as defined by $\eta = \Delta T_{\text{sh}} / \Delta P$ is used here, but alternative terminology is found in the literature such as the “thermal resistance” [2] or “thermal impedance” whose definitions are equivalent. Related terminology may also be found in this same context, such as “heat-transfer coefficient”, h , [3] or “dissipation constant”, but these are just the inverse of the self-heating coefficient used here.

The measurement of self heating of cryogenic resistance thermometers can be used to evaluate the efficacy of the thermometer installation and/or the integrity of the thermometer’s sheath and hermetic seals. A capsule thermometer with insufficient thermal

coupling to its environment will indicate a higher than normal self heating. A thermometer with a damaged seal which allows the He exchange gas to leak out of or into the capsule will also indicate higher than normal self heating under vacuum, but the effect may be reversible when He exchange gas is readmitted though backfilling the vacuum chamber. Long-term usage with smaller leaks also introduces the possibility of capsule thermometers with air-helium or even air-hydrogen mixtures. An understanding of what should be ‘normal’ self-heating behavior in these capsule-type thermometers over their full temperature range is therefore useful in evaluating these factors in practice.

The third law of thermodynamics requires that all heat transport coefficients such as $h = \eta^{-1} \rightarrow 0$ as $T \rightarrow 0$. Consequently, at low temperatures η exhibits divergent behavior such as $\eta \propto T^{-n}$, where typically $0.5 \leq n \leq 3$ in the temperature ranges of interest here. The larger values of n are observed at the lowest temperatures. The various exponents are associated with different heat transport mechanisms each of which will dominate in a different temperature range [4]. For gas-mediated self heating, the exponents are predictable from the temperature dependence of the thermal conductivity of the heat-exchange gas [5].

An exception to the rule that η increases with decreasing T occurs in cases where, in narrow regions of temperature, a phase change occurs in the thermometer fill gas. Such a case is observed in capsule RIRTs and PCRTs with ^4He fill gas near 1.4 K where a superfluid transition occurs in the condensed He film and η drops precipitously [6]. Heat is transferred away from the wire by evaporation and superfluid film flow preserves mass balance. This is a

normal self-heating feature in all He gas-mediated resistance thermometers.

Other exceptions can occur in capsule thermometers in the region between 45 K and 70 K if air has contaminated the He fill gas. As temperature decreases, the air gradually condenses and the decreasing vapor pressure of the condensed air produces fewer collisions between the remaining gas-phase air molecules and the He gas atoms. The He fill gas can then more effectively transfer heat out of the device, so η decreases with decreasing T until the vapor pressure of the remaining condensed air is negligible [7]. In cases where significant air is present, the observed η may be large enough to produce noticeable deviations in otherwise smooth fitting functions used in finite-current calibrations. We refer to this type of behavior as ‘anomalous’ self-heating since it originates from a device defect (e.g., a leak or other source of gas contamination).

EXPERIMENTAL

A distinction exists between “internal” self heating (due to heat transfer within the sensor package) and “external self heating (due to heat transfer external to the sensor package). The respective self-heating coefficients for these are denoted η_{int} and η_{ext} . A capsule thermometer with exchange-gas dominated self-heating will exhibit negligible external self heating when the sheath is in direct contact with an ice bath, liquid nitrogen, or liquid helium near 4 K, but these nearly ideal cases are difficult to emulate in other more practical vacuum-well installations.

In practice some degree of external self-heating is inevitable in vacuum installations. The two effects act as series thermal impedances such that the total observed self-heating coefficient is the linear sum $\eta = \eta_{\text{int}} + \eta_{\text{ext}}$. The mechanisms responsible for η_{int} are solely a function of the thermometer package design and materials, while η_{ext} is in general installation dependent. The dominant contribution to η_{ext} is the contact interface between the sheath and well surfaces, mediated by thin layers of grease [7]. Best practices produce $\eta_{\text{ext}} \lesssim 10\%$ of η_{int} , but variable grease thickness and voids can lead to variability of $\pm 50\%$ in η between different installations.

Experimental values for η are readily obtained given by measurements of resistance $R(I)$ at two or more currents $I_2 > I_1$ allow a simple calculation using

$$\eta \cong \frac{R(I_2) - R(I_1)}{(I_2^2 R(I_2) - I_1^2 R(I_1))} \frac{dT}{dR} \quad (1)$$

Eqn. 1 presumes that joule heating contributions from portions of the current lead wires outside of the

four-terminal junction, but still inside the capsule, are negligible. This is a valid assumption for most capsule thermometers, with certain germanium resistance thermometers (GRTs) being possible exceptions.

Self-heating Data

We have collected self-heating data derived from 18 years of comparison and fixed-point calibrations using a variety of capsule thermometers from 0.65 K to 273 K. Figure 1 shows typical self-heating coefficient data for four capsule-type thermometers, all with Pt sheaths of 5.0 mm to 5.5 mm dia. and glass headers. The Standard Pt type 8164 is a classic helical-coiled wire design [8], RIRT types U and W [6] are bifilar coiled wire designs, and the Pt-Co type R800 [9] is another classic helical-coil design. These are all examples of what we consider “normal” self heating in that the data are consistent with exchange gas compositions of $\approx 100\%$ He.

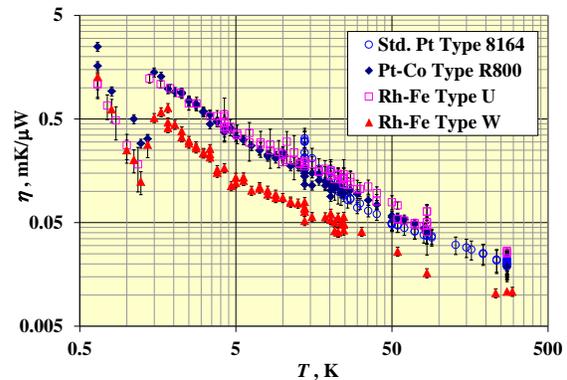


FIGURE 1. Normal self-heating data from 0.65 K to 273 K for four different varieties of capsule-type thermometers.

The measurement of η requires that the excitation levels are high enough to resolve the self-heating in the presence of temperature control fluctuations and measurement system noise. This typically requires self-heating levels which are 5 to 10 times higher than those of random fluctuations, but not all the data presented here were optimized in this way. The type 162D SPRTs are not treated here as these exhibit self-heating too low for routine measurements to resolve.

We have omitted examples of MPRT [10] self-heating in Figure 1 because all of the devices of this type we have examined exhibit anomalous self heating to one degree or another. Figure 2 shows three examples of different self-heating behavior exhibited over the range 13.8 K to 84 K. In two cases, MPRTs A and B exhibit self heating consistent with a helium exchange gas but contaminated with small amounts of

air. The slight increase in $\eta(T)$ with temperature between 45 K and 70 K is created by the increasing density of air molecules as they sublime into the gas phase and cause increasing collisions with the He atoms. In the case of MPRT C, the observed self-heating is consistent with only air in the capsule, so that at ≈ 40 K all the gas is condensed with negligible vapor pressure and only the solid-state conduction through the lead wires remains to limit self-heating. The result is an increase of the observed η by a factor of more than 15 with decreasing temperature between 55 K and 32 K.

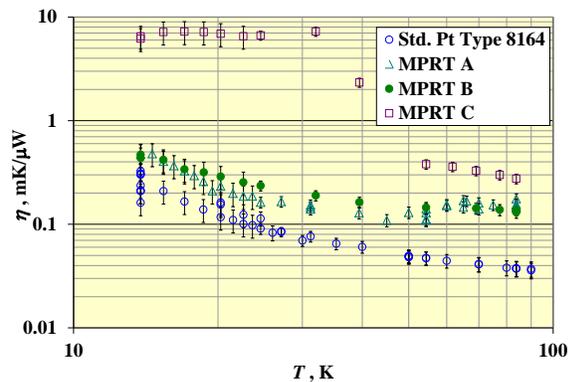


FIGURE 2. Anomalous self-heating characteristics of three MPRTs with variable helium and/or air content and an SPRT exhibiting ‘normal’ (i.e., He gas mediated) self heating.

For the data presented here, variations in $\eta(T)$ from one installation to the next are due to variable $\eta_{\text{ext}}(T)$. These variations often exceed the purely random measurement errors, which are typically 1 % to 5 % of $\eta(T)$. The combined standard uncertainties for most of these experimental values of $\eta(T)$ are ≈ 10 %. There are a few special cases, particularly SPRTs and MPRTs below ≈ 30 K, where parasitic heating effects and poor signal to noise causes systematic errors and additional measurement uncertainty with combined uncertainties of ≈ 25 % in $\eta(T)$.

THERMAL MODELING

In general, η depends on details of heat transport mechanisms through the sensor package and on the thermal details of the installation. In practice the observed η is a combination of the associated series and parallel thermal impedances such that a complete description of the net effect is complicated. Figure 3 shows a schematic diagram of a simplified thermal impedance network for a thermometer self-heating where T_{int} is the temperature of the sensing wire, T_{ext} is the external sheath temperature, and T_0 is the known temperature of a comparison or fixed point reference.

We treat the internal self-heating coefficient as a parallel combination of gas-mediated (Z_{gas}) and lead-wire-mediated (Z_{lw}) thermal impedances, where both are temperature dependent. The external self-heating is treated as a parallel combination of interfacial thermal boundary impedances. The sheath interface impedance $Z_{\text{ex-s}}$ is formed by the boundary between the thermometer sheath and the inner surface of the thermowell, as mediated by a weakly-temperature-dependent grease layer. The lead-wire interface impedance $Z_{\text{ex-lw}}$ is a combination of two series impedances: the lead-wire to copper-heat-sink interfaces and the thermal resistance of the external lead wires, which are also temperature dependent.

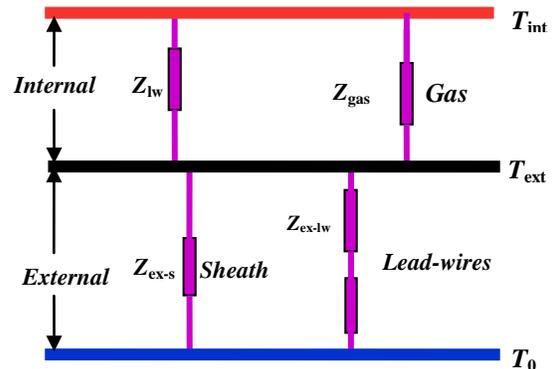


FIGURE 3. Simplified thermal equivalent circuit for internal and external thermometer self heating.

In a typical installation of a capsule-type thermometer, $\eta_{\text{ext}} \ll Z_{\text{gas}} \ll Z_{\text{lw}}$, resulting in the net observed self-heating coefficient,

$$\eta \approx Z_{\text{gas}} \left(1 - \frac{Z_{\text{gas}}}{Z_{\text{lw}}} \right) + \eta_{\text{ext}}. \quad (2)$$

The gas-mediated self-heating Z_{gas} is the dominant mechanism and is the product of a geometric factor G and the inverse of the gas thermal conductivity $\kappa(T)$ or $Z_{\text{gas}} = G/\kappa(T)$. In the approximation that $Z_{\text{gas}}/Z_{\text{lw}}$ and η_{ext} can be neglected, it is sufficient to use published or calculated values for $\kappa(T)$ and one measurement of $\eta(T_0)$ (at a convenient reference temperature T_0) in order to determine G and hence infer values for $\eta(T)$ at all other temperatures.

Gas Thermal Conductivity Calculations and Literature Data

Johnston and Grilly [11] have measured the thermal conductivity of gas phase ^4He from 80 K to 300 K. Ubbink and de Haas [12] have made measurements from: 1.5 K to 4.26 K; 12 K to 15 K; and at 76.3 K. Taken together, those data are

consistent with a power law $\kappa_{\text{He}}(T) \approx T^n$ with $n \approx 0.65$ for $T > \approx 10$ K and $n \approx 1$ for $T < \approx 5$ K.

The thermal conductivity of gas phase He and its mixtures with other gases may also be calculated using the program REFPROP [13], which uses theoretical models to predict gas transport properties [14,15]. Figure 4 shows the results of REFPROP calculations of $\kappa(T)$ for He, Air, N₂, and several He-N₂ mixtures together with the He data from the literature.

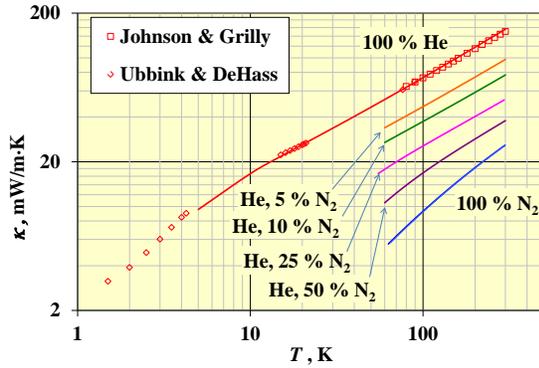


FIGURE 4. Lines: REFPROP values for $\kappa(T)$ of ⁴He and various mixtures with N₂. Air and 100 % N₂ are indistinguishable on this scale. Data Points: Literature values for ⁴He [11, 12].

The REFPROP calculations assume that the internal gas pressure is 33 kPa at 300 K. This assumption may be approximately valid for some, but certainly not all, capsule thermometers. However, the exact pressure is not important unless it is so low that the mean free path becomes comparable to the internal dimensions of the capsule. We assume that this condition is not relevant to any of the capsule thermometers treated here. This means we exclude those cases of a gross seal failure or otherwise negligible He gas content in the capsule. Uncertainties are estimated at 5 % in $\kappa(T)$ for $T > 10$ K.

Normal Self Heating

The REFPROP calculations of $\kappa(T)$ for 100 % He above 5 K may be used to compare normal self-heating data with the predictions of Eqn. 2, again ignoring the corrections from η_{ext} and Z_{lw} . Figure 5 shows two examples of self-heating data from 0.65 K to 273 K from two capsule thermometers of different designs. The solid lines from 5 K to 300 K are derived from $\kappa(T)$ calculations with the geometric factor G as the only free parameter. For the two cases shown in Fig. 5, $G = 0.03 \text{ cm}^{-1}$ and 0.013 cm^{-1} . The region between 1.3 K and 5 K can not be treated via REFPROP, but our data are generally consistent with the results of Ubbink and de Haas [12].

The solid lines shown in Fig. 5 from 0.5 K to 1.3 K are simple power law predictions of the form $\eta(T) = cT^{-3}$ with $c = (0.3 \text{ and } 0.6) \text{ mK} \cdot \text{K}^3 \cdot \mu\text{W}^{-1}$ respectively. Within the experimental uncertainties, the data in this range are consistent with the inverse T^3 dependence and the self-heating being limited by a Kapitza thermal boundary layer impedance [16]. In this case the boundary layer is presumably present between the superfluid ⁴He film and the inner surface of the capsules' Pt sheaths and/or Pt wire elements.

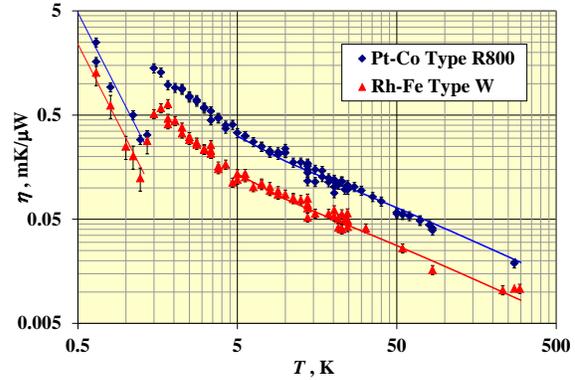


FIGURE 5. The self-heating coefficients for two capsule thermometers of different designs from 0.65 K to 273 K showing two normal self-heating regions dominated by: Kapitza boundary layer impedance below 1.3 K (solid lines – T^{-3} dependence); and He gas-phase self-heating above 5 K (solid lines - temperature dependence from REFPROP).

Anomalous Self Heating

We have examined self-heating in eight capsule thermometers that exhibit anomalous self-heating in the range 45 K to 70 K. In all cases where enough data exist, the anomalous behavior is easily understood as being a consequence of air contamination of the helium exchange gas in the capsule. While a detailed model describing the behavior in the anomaly region of 45 K to 70 K is beyond the scope of this work, it is straightforward to use the data in conjunction with REFPROP predictions to infer the degree of air contamination. As in the normal case, data below 45 K is used to yield the geometric constant G for the thermometer. Assuming a helium-air mixture, the data above 70 K are then used with the same G value to yield the gas composition inside the capsule.

Figure 6 shows a typical example of an obvious self-heating anomaly in a capsule RIRT from combined data sets taken over an 18 year time span. The self-heating below 45 K is assumed to be moderated by 100 % He gas and that above 70 K moderated by the helium-air mixture. REFPROP calculations are shown for both pure helium and a

nominal 75 % helium-25 % air mixture for a single geometric factor $G=0.017 \text{ cm}^{-1}$.

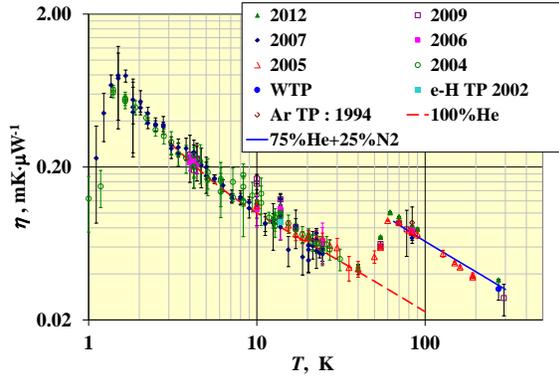


FIGURE 6. A compiled history of self-heating data from 1994 to 2012 for a NIST check capsule RIRT contaminated with air. The inferred concentration of air is approximately 25 % as indicated by the REFPROP calculations (dotted lines). Error bars are statistical standard uncertainties only.

DISCUSSION

Our study of 35 capsule-type thermometers has found 4 out of 11 RIRTs, 4 of 15 SPRTs, 1 of 4 PCRTs, and 5 out of 5 MPRTs with some degree of air contamination. All of the RIRTs studied exhibit stable calibration histories of 18 years, with no evidence of drift relative to our best available fixed-points. Given the observed levels of anomalous self-heating, their relative stability, and the age of these RIRTs (produced circa 1987-1988), it is unlikely that a leak led to the bulk of the air contamination (i.e. it was an original defect). Nonetheless, it is useful to examine the consequences of small leaks in capsule resistance thermometers in this context. We also consider the practical consequences of air contamination on thermometer calibrations and examine correlations in self-heating values as they relate to practical screening techniques for thermometers with air contamination.

Leaks in Capsule Thermometers

Two types of leaks may exist in capsule thermometers: 1.) physical leaks, such as micro-cracks in glass headers, in which both air and helium may pass primarily driven by pressure gradients; and 2.) permeation leaks, which primarily allow helium to diffuse through glass walls in the presence of a concentration or pressure gradient [17].

In the first case, a gross leak is readily apparent once a capsule is placed in a vacuum, the exchange gas is removed from the capsule, and the observed self-

heating rises by a factor of 10 or more. More subtle effects may be observed in the case of very small physical leaks, in which only a small fraction of the He is replaced with air over multiple-year timescales. Changes to the self-heating anomaly provide a sensitive indicator of changes in gas composition. The ratio $\eta(70 \text{ K})/\eta(40 \text{ K}) = \kappa(40 \text{ K})/\kappa(70 \text{ K})$ varies from 0.7 to 1.08 as the gas composition changes from 100 % He to a 5 % air-He mixture. In contrast, a leak rate corresponding to 5 % of the helium at 33 kPa in a $V=0.7 \text{ cm}^3$ capsule volume removed in 1 year would be only $\approx 3 \times 10^{-10} \text{ cm}^3/\text{s}$ at standard conditions.

In the case of helium permeation, the helium gas content of a capsule would be expected to slowly decrease over time without any appreciable increase in air content. This would have no noticeable effect on $\eta(T)$ as long as the mean-free-path l_{He} for atomic collisions remains small compared to the capsule's radius, $d/2$. The hydrodynamic-to-molecular transition would correspond to a pressure of $\approx 8 \text{ Pa}$ at 300 K, or only 0.024 % $\approx 1/e^{8.3}$ of an initial 33 kPa gas charge. For a glass header of thickness $t=2 \text{ mm}$, area $A=1 \text{ cm}^2$ and a low permeation constant $K=1 \times 10^{-18} \text{ m}^2/\text{Pa}\cdot\text{s}$, (e.g. a lower limit for soda-lime glass) we estimate a time constant $\tau \approx tV/KAp_{\text{atm}} \approx 4$ years, and reaching the transition pressure in ~ 33 years. Some thermometers even older than this retain normal self-heating properties, implying the presence of mitigating factors.

Finite-current calibrations

Best practice in resistance thermometry is to extrapolate self-heating by power or I^2 to the zero-power condition. This effectively eliminates any self-heating effects in the calibration data. A more convenient and common practice in industry, however, is to calibrate to a finite current, in which case the self-heating and any associated anomaly become part of the calibration. Interpolation errors are mitigated if data are spaced in temperature with at least three points over the region of the anomaly and a fitting function has a sufficiently high polynomial degree to fit the shape of the anomaly. SPRT calibration according to the ITS-90, however, would not meet this criterion and interpolation in the region of the O_2 TP would be in error by amounts as large as $\eta(T)I^2R(T) - \eta(T_{\text{O}_2\text{TP}})I^2R(T_{\text{O}_2\text{TP}})$. For a 25.5Ω SPRT at $I=1 \text{ mA}$ and $G=0.03 \text{ cm}^{-1}$, the errors are $\approx -0.3 \text{ mK}$ and $+0.5 \text{ mK}$ at 40 K and 70 K respectively for a 50 % air-helium mixture. If no helium remains in the capsule, however, errors can be ≈ 10 times larger near 40 K.

Another criterion pertains to changes in gas composition. Besley [3] has commented that apparent instabilities in germanium resistance thermometers' (GRTs) finite-current calibrations above 40 K may in

fact be explained as changes in exchange gas composition rather than changes in the actual resistance characteristic of the GRT. We concur that larger leaks and variable gas composition are common in the miniature capsules of 3 mm diameter and smaller, which are used for MPRTs and GRTs.

Screening Thermometers for Air

Tests can be readily preformed to screen thermometers for air contamination without the necessity of making detailed measurements in the range of the self-heating anomaly. Based on REFPROP calculations of gas thermal conductivities, the ratio of self-heating coefficients for a mixture of 95 % He and 5 % air to that for 100 % He is approximately 1.6 for $T > \approx 100$ K. This represents a sufficiently large difference that, providing that external self-heating can be neglected, screening may be performed at the water triple point by simply comparing values for capsule thermometers to an established normal self-heating for each model of thermometer. If external self-heating is too large or variable in a WTP cell, then a ratio such as $\eta(40\text{ K})/\eta(70\text{ K})$ or $\eta(T_{\text{NeTP}})/\eta(T_{\text{ArTP}})$ can provide a more reliable indication of air content. Fig. 7 shows the degree of correlation of the later ratio with $\eta(T_{\text{WTP}})$ for a variety of Standard Platinum (SP), Rhodium-Iron (RI), Platinum-Cobalt (PC) and Miniature Platinum (MP) capsule models with standard uncertainties.

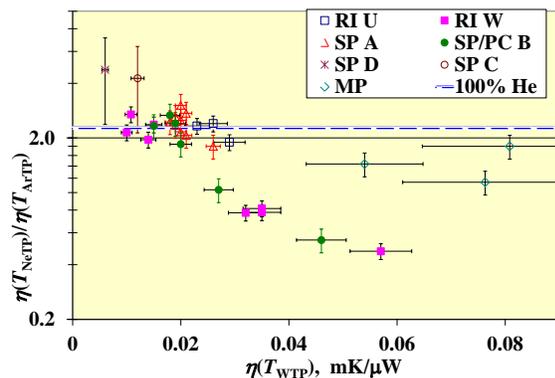


FIGURE 7. The ratio of self heating coefficients at the Ne TP to the Ar TP versus the coefficient at 273.16 K. The horizontal line at $\eta(T_{\text{NeTP}})/\eta(T_{\text{ArTP}})=2.28$ represents normal self-heating due to 100 % He exchange gas.

Summary

The capsule thermometer self-heating data we have examined indicate that relatively large self-heating anomalies can be observed in thermometers with air contamination. The air impurity concentrations are, in many cases, stable over an 18 year time span and do

not appear to influence zero-power calibration stabilities. Apparent instabilities in finite-current calibrations may be expected in capsules with variable air concentration. Finite-current SPRT calibrations exhibit small errors resulting from anomalous self-heating as long as some significant fraction of He remains within the capsule. Significant interpolation errors should be expected in those cases where the helium content has dropped to negligible levels.

ACKNOWLEDGEMENTS

We thank Eric Lemmon and Marcia Huber of NIST Boulder, CO for making REFPROP available for our use and providing valuable advice.

REFERENCES

- Nicholas, J. V. and White, D. R., *Traceable Temperatures*, 2nd Ed., New York: Wiley, 2001, 450 pp.
- Holmes, D. S. and Courts, S. S., in: *Advances in Cryogenic Engineering*, edited by P. Kittel, Vol. 41, New York: Plenum, 1996, pp. 1699-1706.
- Besley, L. M. and Hai, L., *Rev. Sci. Instrum.* **64**, 748-755 (1993).
- Yeager, C. J., Courts, S. S., and Davenport, W. E., in: *Adv. Cryo. Engin.*, edited by J. Waynert, Vol. 49A, New York: AIP, 2004, pp. 412-419.
- Hai, L. and Besley, L. M., *Rev. Sci. Instrum.* **64**, 741-747 (1993).
- Rusby, R. L., in: *Temperature, Its Measurement and Control in Science and Industry*, edited by J. Schooley, Vol. 5, New York: American Institute of Physics, (1982) 829-833.
- Ekin, J. W., *Experimental Techniques for Low Temperature Measurements*, New York: Oxford Univ. Press, 2006, 673 pp., Secs. 2.6 and 5.3 .
- Meyers, C. H., *Bur. Stand. J. Res.*, **9**, 807-813 (1932).
- Shiratori, T. and Mitsui, K., *Jap. J. Appl. Phys.* **17**, 1289-1290 (1978).
- Lucas, D. A., in: *Temperature: Its Measurement and Control in Science and Industry*", Vol. 4, H. H. Plumb, ed., Instrument Society of America, Pittsburgh (1972) 963-969.
- Johnston, H. L. and Grilly, E. R., *J. Chem. Phys.* **14**, 233-238 (1946).
- Ubbink, J. B. and de Haas, W. J., *Physica* **10**, 465-470 (1943).
- Lemmon, E. W., Huber, M. L., McLinden, M. O. NIST Standard Reference Database 23: Reference Fluid Thermodynamic and Transport Properties-REFPROP, Version 8.1, Nat. Inst. Stand. Tech., Standard Reference Data Program, Gaithersburg, 2009.
- Hands, B. A. and Arp, V. D., *Cryogenics*, **21** 697-703 (1981).
- Lemmon, E. W. and Jacobson, R. T., *Int. J. Thermophys* **25**, 21-69 (2004).
- Pollack, G. L., *Rev. Mod. Phys.* **41**, 48-81 (1969).
- Altomose, V. O., *J. Appl. Phys.* **32**, 1309-1316 (1961).