Cryogenic thermometer calibration system based on triple points from D_2 to H_2O

P. D. Levine and B. Mellons

Primary Standards Laboratory, Lockheed Missiles and Space Co., Sunnyvale, California 94088

Utilizing a closed cycle helium refrigerator and sealed cells of pure gases, a system for realizing the International Temperature Scale of 1990 (ITS-90) from 24.56 K to 273.16 K was created. Once the behavior of a primary standard thermometer based on values obtained at fixed temperature points was established, the system was used for comparison measurements on a variety of thermometers. Only a slight increase in uncertainty above the primary level was introduced. A complete set of data for both a primary and a comparison calibration yielding accuracies to a few millikelvins is presented. Substitution of the deuterium triple point for the hydrogen triple point is explored. Overall measurement uncertainties are analyzed and areas for possible improvement noted.

INTRODUCTION

The primary interpolation instrument for ITS–90 from 13.80 K to 1234.93 K is the Standard Platinum Resistance Thermometer (SPRT). In the cryogenic temperature region (13.80 K to 273.16 K), a capsule–type SPRT is used. Primary calibration requires the determination of resistance ratios from measurements made at temperatures defined by the three–phase equilibrium state (triple point) of pure substances that occur at fixed temperatures and pressures. Properly constructed sealed cells ensure that such states can be realized repeatedly with highly reproducible results.

Two hydrogen vapor pressure points are also used to complete the ITS–90 to 13.80 K (triple point of hydrogen). Specific apparatus for making vapor pressure measurements is not readily available and is impractical in an industrial application. The ITS–90 formalism requires the hydrogen triple point to complete the scale to 24.56 K, Ref. (1). Thus, calibration utilizing triple points only is limited to a base temperature of 24.56 K, the triple point of neon.

Currently, the primary standards laboratory at Lockheed Missiles and Space Co., Inc. (LMSC) has available triple point cells of water, mercury, argon, nitrogen, oxygen, neon and deuterium. Neither nitrogen nor deuterium are recognized points, though both have been shown to be stable and reproducible references.

In this study, a thermometer calibrated on the International Practical Temperature Scale of 1968 (IPTS–68) was used to establish the hydrogen triple–point temperature by comparison (a triple point cell was not available). The thermometer resistance ratio at the IPTS–68 hydrogen triple point value was converted to the ITS–90 ratio and assigned the ITS–90 triple point temperature. In this way the subrange from 24.56 K to 273.16 K was realized.

In an effort to realize the scale entirely by fixed–point measurements, the possibility of using the deuterium triple point to replace the hydrogen comparison point in the ITS–90 equations was explored.

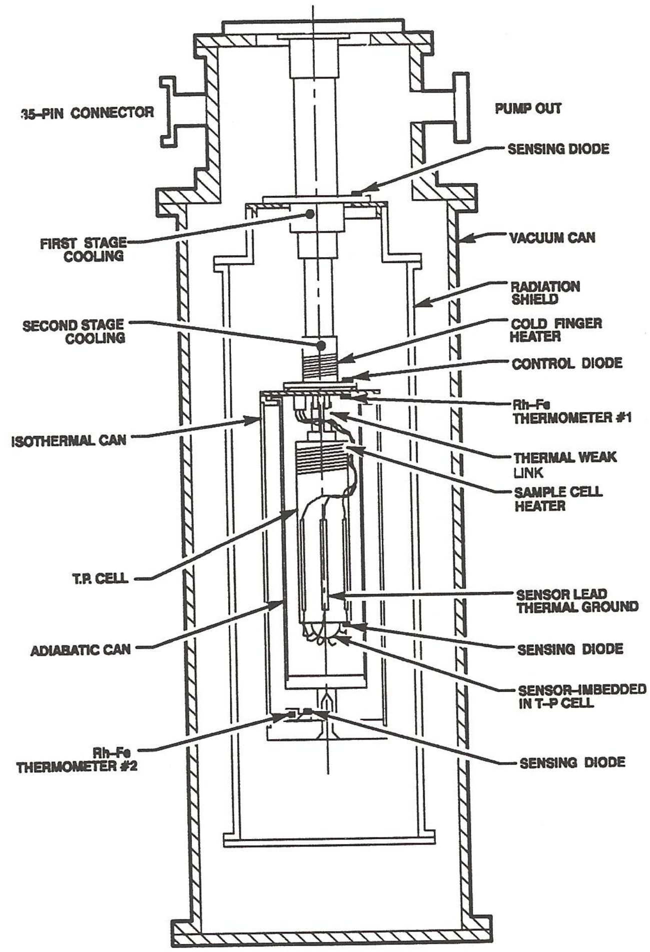

FIG. 1. Details of cryostat

APPARATUS AND EXPERIMENT DESIGN

The apparatus used for the gas triple points was built around a small–capacity, closed–cycle Helium–4 refrigerator. This device has two cold stages with base temperatures on stage 1 of 35 K and on stage 2 of 7 K under minimal load conditions. Concentric radiation shields are attached to each of the stages. The entire assembly is placed inside a 12.7 cm inner–diameter vacuum jacket. Electrical feed–throughs and vacuum plumbing are all at the top of the system.

The triple point cell under test, or the isothermal block for comparison measurements, is mounted to the second stage through a weak thermal link. The second stage shield also acts as a combination of isothermal/adiabatic enclosure. Figure 1 shows the arrangement for triple point measurements. Further details of the cryogenic system can be found in Ref. (2).

In order to make triple point measurements, a thermometer was mounted in a cell which was cooled to well below its triple point temperature. The refrigerator 2nd stage was then set to a temperature at or slightly below the triple point value. Once refrigerator control had stabilized, heat pulses of pre–set magnitude were applied to the cell at regular intervals to realize the triple point state (see Fig. 2). Data acquisition was automated and measurements proceeded under computer control until the melting plateau had become fully defined.

Comparison measurements were made using an isothermal blocked designed to fit the apparatus much like a triple point cell. The block was capable of holding three capsule SPRTs and was brought to the desired temperature by a combination of refrigerator control, judicious use of helium exchange gas and active heating. Drift rates of 0.5 mK/min or less were established during data acquisition to minimize gradients between thermometer positions in the block.

The experiment consisted of transferring values from a sensor calibrated at the National Physical Laboratory (NPL) of the U.K. to a single uncalibrated thermometer at temperatures

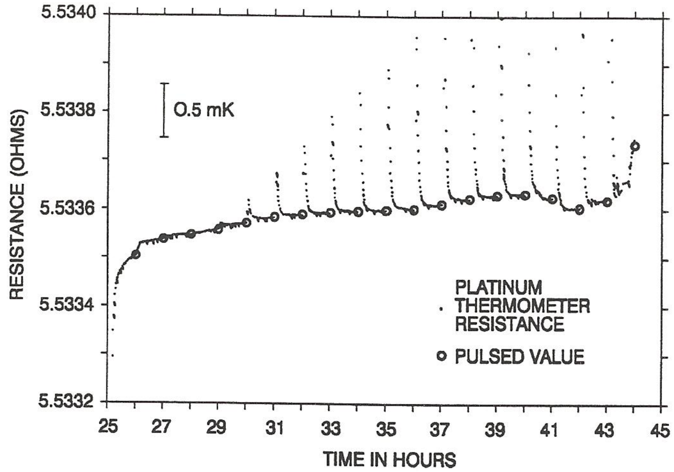

FIG. 2. Realization of the triple point state by pre–pulse equilibrium data

bracketing the triple point values. The NPL calibrated sensor had also been measured separately at the triple points available at LMSC, Ref. (3). Once this was accomplished, the uncalibrated thermometer was evaluated at the triple points and the results compared.

This particular experiment utilized only a single uncalibrated thermometer, as opposed to the maximum of two, in an effort to expedite the evaluation. The thermometers were of identical construction and were from the same manufacturer. A separate ac resistance bridge was used to monitor each sensor. This technique allowed for nearly simultaneous measurements to be made manually.

Realization of the ITS–90 requires obtaining a thermometer resistance ratio at the triple point of mercury. The cryogenic system cannot accommodate the current design of mercury cells so a separate apparatus was used.

Logistically speaking, it was actually simpler to obtain ratios at the mercury triple point directly rather than through comparison measurements. Certain capsule thermometers will not fit into this apparatus and will need to be evaluated through comparison measurements in a circulating oil bath and isothermal block. No data are currently available for the uncertainties in this transfer.

RESULTS AND ANALYSIS

Comparison measurements were carried out first. Sprt B56 was used to determine the block temperature on the IPTS–68 at several points on either side of the desired triple point value. The resistance ratio of the uncalibrated SPRT (B195) was recorded at each point and graphed as a function of the temperature indicated by B56. A straight line fit was made to the data and a resistance ratio obtained for the IPTS–68 triple point temperature from the fit. This ratio was then expressed in terms of ITS–90 and assigned the ITS–90 triple point temperature value (see column 2 of Tab. I).

Resistance ratios were then obtained for B195 in each of the available triple point cells (column 3 of Tab. I). Overall uncertainties in the realization of triple point temperatures were roughly 1 mK, at the equivalent of 3 sigma, for all but deuterium. The uncertainty in realizing the deuterium triple point temperature was around 4 mK (primarily due to the wider melting range), Ref. (3).

Previous work, using thermometer B56 for interpolation, has established the LMSC triple point cell temperatures to be as assigned by the IPTS–68 (to within the uncertainty of the NPL calibration), Ref. (3). The first column of Tab. I shows these temperatures as assigned by the ITS–90. It can be seen from column 4 that agreement between triple point and comparison measurements is to better than 2 mK for all but nitrogen. The nitrogen triple point temperature is not used to solve the ITS–90 equations and so will not affect the calibration directly. However, this point does serve to illustrate the magnitude of error which can occur.

TABLE I

Cell Type T (ITS–90)	Ratio B195 Comparison	Ratio B195 Triple Point	Delta Ratio (Equiv Temp)
Mercury 234.3146K		0.844228	N/A
Argon 83.8058K	0.216256	0.216261	0.000005 (1.03 mK)
Nitrogen 63.1504K	0.127763	0.127776	0.000013 (3.17 mK)
Oxygen 54.3584K	0.092183	0.092189	0.000006 (1.6 mK)
Neon 24.561K	0.008891	0.008893	0.000002 (1.2 mK)
Deuterium 18.7205K	0.003590	0.003591	0.000001 (1.6 mK)
Hydrogen 13.8033K	0.001544	N/A	est. 2.0 mK

Figure 3 shows a melting plateau at the neon triple point as defined by thermometer B195. Resistance ratio is shown as a function of percent of sample melted. It can be readily seen that the plateau is stable and the melting range is less than 0.5 mK. This curve is typical of what is obtained at the other points, with the exception of deuterium whose melting range is of the order of 1–2 mK.

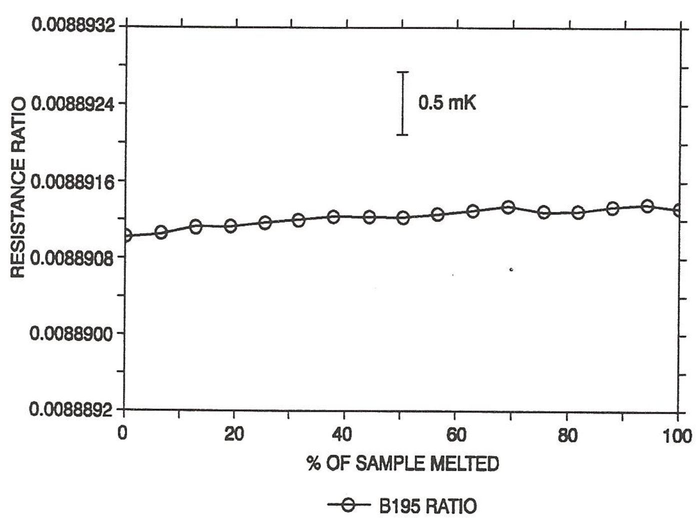

FIG. 3. Melting plateau of neon as realized by PRT B195

The SPRT defined scale does not extend below the hydrogen triple point. In order to establish this point by comparison, measurements slightly below 13.81 K were required. These temperatures were determined by linear extrapolation of the B56 calibration. Figure 4 shows resistance ratios for B56 and B195 at IPTS–68 temperatures above and below 13.81 K. Temperatures below 13.81 K are extrapolated.

It can be seen that both thermometers exhibit very similar, linear behavior. Thus, linear fits were used to establish the hydrogen triple point resistance ratio of B195. The resistance ratio obtained for B195 at 13.81 K on the IPTS–68 was converted to an ITS–90 ratio and assigned the value of 13.8033 K in

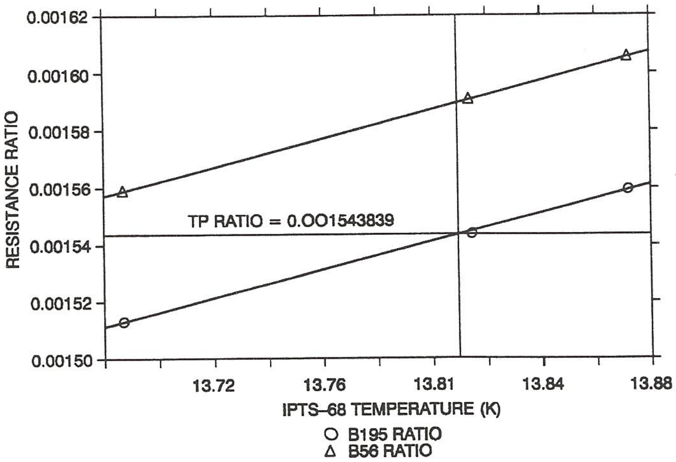

FIG. 4. Determination of hydrogen triple point by comparison measurements

order to produce an ITS–90 calibration. The ratio conversion is as follows:

$Ratio(T_{68})=R(13.81\ K)/R_0$; $Ratio(T_{90})=Ratio(T_{68})*(R_0/R_{TP})$

where R=thermometer resistance at a temperature T, $R_{TP}=R(273.16\ K)$, and $R_0=R(273.15\ K)$.

Interpolation curves were generated using the ITS–90 equations from fixed point data and also from comparison measurements in an effort to determine overall uncertainties for comparison calibration. Figure 5 shows the differences in resistance ratio over the entire range of calibration for values obtained by fixed point versus comparison measurements on PRT B195. The maximum ratio difference corresponds to less than 2 mK in interpolation.

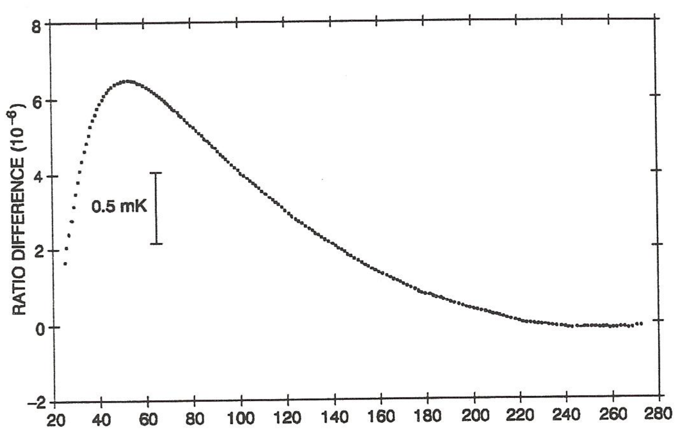

FIG. 5. Interpolation difference between curves generated by fixed point vs. comparison data

The measurement system was identical for both triple point and comparison measurements. A complete analysis for this system appears in Ref. (3). Drawing on this analysis and the observed differences in interpolation curves, an overall uncertainty of 5 mK is estimated for comparison calibration. Future work will revise and formalize this estimate as a more comprehensive data base is created.

SCALE REALIZATION USING DEUTERIUM

The deuterium triple point has proven to be stable and reproducible. As such, it was of interest to investigate the feasibility of substituting it for the hydrogen triple point, as that point is not available in this lab. The deuterium value was substituted for hydrogen in the ITS–90 equations to generate an interpolation curve from fixed point data. The ITS–90 value of the deuterium triple point was ascertained from the published difference curves, Ref. (4), and measurements made using thermometers calibrated on the IPTS–68.

Figure 6 shows the difference in resistance ratio from 24.6 K to 273.16 K between the interpolation curve obtained by substituting the deuterium point for hydrogen and the curve generated in the ITS–90 prescribed fashion. As might be expected, the differences are extremely small.

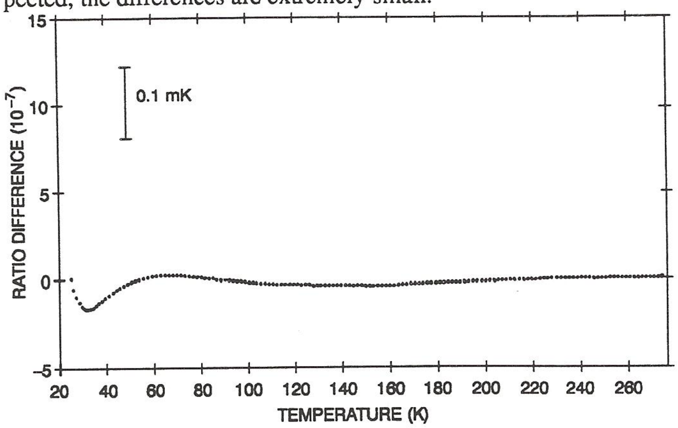

FIG. 6. Differences in interpolation on ITS–90 from 24.6 to 273.16 K when D_2 is substituted for H_2 triple point

CONCLUSIONS AND FUTURE PLANS

A viable system for comparison calibration of cryogenic PRTs has been successfully demonstrated. Utilizing in–house fixed points and an externally calibrated SPRT the ITS–90 can be realized between 24.56 K and 273.16 K. By substituting the deuterium for hydrogen triple point the scale can be realized entirely by fixed point determinations. Differences in interpolation of less than 0.5 mK from the formally prescribed fashion are observed. Thus, the system need not rely on external thermometer standards.

Standards created through fixed point calibration have been used for comparison measurements in the same system. Accuracies to 3 mK or better were achieved for such comparisons at the fixed points. This is a slight increase over the 1 mK achieved by direct fixed point determinations.

Immediate plans at LMSC involve transferring fixed point values to two thermometers from a standard created in–house. These thermometers will then be measured directly in the fixed point cells and a similar analysis done as above. In addition, the ITS–90 vapor pressure points will be transferred through the use of an externally calibrated platinum or Rhodium–Iron thermometer to realize the full scale from 13.8033 K.

Though transfer accuracies appear good, all transfer values are higher than direct fixed point measurements. An attempt will be made to determine the cause. Comparison measurements will be automated to improve efficiency and hopefully, accuracy. The possibility of transferring calibration to Rhodium–Iron thermometers at a number of points in the SPRT range and then linking to superconducting fixed points to extend the scale further will also be explored.

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