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The NIST Low Temperature ITS-90 Realization and Calibration Facilities

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Abstract. Two facilities have been constructed at NIST for realizing and maintaining the ITS-90 below 84 K. The first facility is an integrated low temperature realization system that realizes the ITS-90 below 84 K in its entirety and which we refer to as the Low Temperature ITS-90 Realization Facility (LTRF). The second facility, known as the Low Temperature Calibration Facility (LTCF) is a multi-purpose low temperature facility for scale dissemination and maintenance and for realization of certain ITS-90 triple points in sealed cells. We present a description of the refrigeration, temperature-control, and resistance-measurement systems of the NIST LTRF and LTCF. For the LTRF, we also describe the realization cells and the copper block which contains them, the gas control system, and the pressure measurement system. The comparison and triple-point realization systems of the LTCF are likewise described. The design parameters of NIST fixed point cells as used in both facilities are given. The origin and purity of the NIST fixed-point materials as used in both facilities are given along with the isotopic content in the case of hydrogen.

INTRODUCTION

Below 84 K the International Temperature Scale of 1990 (ITS-90) [1] has four different overlapping definitions. The first three define the scale in terms of ^{3}He vapor pressure thermometry (0.65 K to 3.2 K), ^{4}He vapor pressure thermometry (1.25 K to 5 K), and interpolating constant volume gas thermometry (3 K to 24.5561 K). The fourth defines the scale in terms of standard platinum resistance thermometry with subranges 13.8033 K to 273.16 K, 24.5561 K to 273.16 K, and 54.35 K to 273.16 K. Calibration of standard platinum resistance thermometers (SPRTs) over these subranges requires the realization of four triple points below 84 K (argon, oxygen, neon and equilibrium hydrogen) and two e-H_2 vapor-pressure fixed points.

In order to realize and maintain the ITS-90 below 84 K, two facilities have been constructed at NIST. These facilities have been designed to realize and disseminate the ITS-90 using the guidelines published by the BIPM [2]. The first facility realizes the ITS-90 below 84 K in its entirety for calibration of NIST check thermometers; we refer to it as the Low Temperature ITS-90 Realization Facility (LTRF). The second facility, known as the Low Temperature Calibration Facility (LTCF), maintains the scale and calibrates customer thermometers against NIST check thermometers in a comparison block. A set of capsule-type standard platinum resistance thermometers (CSPRTs) and rhodium-iron resistance thermometers (RIRTs) are used as the NIST reference and check thermometers and are shared between the facilities.

Both facilities are used to realize triple points; the LTRF realizes them in open cells while the LTCF realizes them in sealed cells. Open cells have the advantage that their gases can be easily withdrawn for analysis or replacement. Also, open cells can be filled with different amounts of gas, enabling extrapolation to zero hydrostatic head. Advantages of sealed cells are their portability and better thermal contact between the solid/liquid interface and the thermometer being calibrated. Furthermore, the gas remains in a sealed cell when it is warmed to room temperature, giving less opportunity for cell contamination.

In this paper we present a description of the refrigeration, temperature-control, and resistance-measurement systems of the NIST LTRF and LTCF. For the LTRF, we describe the realization cells and the copper block containing them, the gas control system, and the pressure measurement system. For the LTCF, we describe the comparison block and arrangement for mounting sealed triple-point cells (STPCs). A summary of the fixed-point materials used in both facilities is provided in the final section.

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LOW TEMPERATURE FACILITIES

Common Features

The facilities each use a continuously operating ^{3}He refrigerator for cooling to the lowest temperatures. The coldest place in the refrigerator is a circular platform on which a container of liquid ^{3}He is mounted. The lowest temperatures achieved by this liquid ^{3}He platform have been about 0.5 K. The ^{3}He platform is located above the top of the realization/calibration system being cooled. A cylindrical copper shield surrounds the ^{3}He platform and realization/calibration system. It is thermally anchored to the platform on which the refrigerator's ^{4}He 1 K pot is mounted. An outer copper shield surrounds this shield and is in thermal contact with the cryogenic liquid used. For the refrigerator to operate, this cryogenic liquid must be ^{4}He and a vacuum must exist within the outer shield.

The ^{3}He refrigerator is generally only used for cooling below ~12 K. From ~2 K to ~12 K, the ^{3}He platform is cooled by ^{3}He gas flow, with the ^{4}He pot cooled to 1 K when necessary. Below ~2 K, the ^{3}He platform is cooled by pumping on liquid ^{3}He. For temperatures above ~12 K, cooling by thermal conduction from the cryogenic liquid surrounding the cryostat is used instead. For temperatures above 80 K, the cryogen is liquid N_2. Between 50 K and 80 K, the dewar containing the liquid N_2 is pumped on to solidify and cool the N_2 to a temperature below 50 K. For 50 K and below, freely vented liquid ^{4}He is used.

A cylindrical copper shield is mechanically and thermally attached to the ^{3}He platform along its outside edge, making this platform the top plate of a cylindrical shield. For the LTRF, this is the innermost shield surrounding the realization system. For the LTCF, one additional inner shield surrounds the system, which is mounted underneath the ^{3}He platform and thermally decoupled through stainless-steel rods.

Each cryostat can be configured in two ways: the first for triple-point (TP) realizations and the second for temperature control (used with vapor-pressure and gas thermometry realizations and comparison calibrations). For TP realizations, the cryostat is arranged to perform calorimetry. Minimal thermal contact between the TP cell and the surrounding environment is desired. Therefore once the vacuum chamber has been evacuated, the only contact made is through thermometer leads, mechanical supports, and fill-tubes (LTRF only). The thermometer leads are thermally anchored to the inner shield and to the TP cells. The fill-tubes (LTRF) are also thermally anchored to the inner shield at the bottom fill-tube platform. The anchoring is made possible by a strip of copper foil connecting the platform to the shield. To minimize heat leaks to the TP cell during a TP realization, the inner shield is controlled at approximately the TP temperature using a heater attached to the shield. The sensor for the controller is an RIRT in thermal contact with the shield. Prior to a TP realization, the copper block (LTRF) or sealed cell (LTCF) is cooled below the TP temperature by introducing a small amount of He exchange gas into the cryostat. Once the block has been sufficiently cooled, the exchange gas is removed.

For the temperature-control configuration, a solid thermal connection (sheets of copper foil for the LTRF, stainless steel rods for the LTCF) is made between the realization/calibration system and the platform with the ^{3}He pot. The thermal connection between the inner shield and the fill-tubes (LTRF) is removed to ensure that the fill-tubes are always at a higher temperature than the VP cells. The realization/calibration system is heated with resistive wire wrapped around it. The sensor for the controller is an RIRT placed appropriately close to the heater. The resistance of the RIRT is measured with an AC resistance bridge, which gives a DC voltage output proportional to the deviation from balance. Temperature control is obtained using a PID controller. Temperature stability of better than 0.01 mK over several hours has been achieved with these systems.

The resistances of the thermometers being calibrated in these systems are measured with one of two commercial self-balancing AC bridges based on inductive voltage dividers (IVD). A bridge provides an output of the ratio of the unknown resistance to that of a standard resistor calibrated at NIST with an accuracy of 2×10^{-7} in the ratio. Two sets of standard resistors with nominal values of 1 Ω, 10 Ω, 100 Ω and 25 Ω are independently maintained. The standard resistor used for a particular measurement is usually that which gives a ratio closest to unity. The linearity of the bridges has been tested using both IVD-based instruments and network-based instruments [3].

Low Temperature Realization Facility

Realization Block

Descriptions of the LTRF and its accompanying measurement procedures, measurement uncertainties,

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and realization results have been published in Refs. [4-7]. The centerpiece of the LTRF is a cylindrical oxygen-free high conductivity (OFHC) copper block. This block, shown in Fig. 1, contains cells for realizing the entirety of the ITS-90 below 84 K: vapor-pressure (VP) cells for ^{3}He and ^{4}He, a ^{4}He interpolating constant volume gas thermometer (ICVGT), a vapor-pressure/triple-point cell for e-H_2, and TP cells for Ne, O_2 and Ar. The block is 31.8 cm high and has a maximum diameter of 10.2 cm. The block has horizontal wells located near the top and bottom for the insertion of RIRTs. In addition, there are four horizontal wells located in the middle of the block for the insertion of RIRTs and/or CSPRTs.

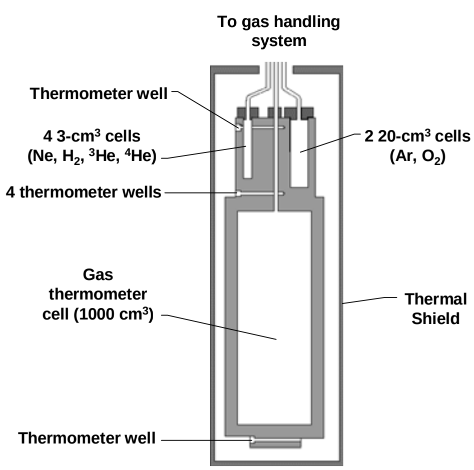

FIGURE 1 Copper block containing cells for realization of the ITS-90 below 84 K.

The gas thermometer has been described in Ref. [5]. As shown in Fig. 1, it is composed of a cavity and a small fill-line in the block. Its entire volume is 1017 cm^3. The bottom of the gas thermometer consists of a circular OFHC copper plate. A groove is machined in the top of this plate for insertion of a gold O-ring that seals the gas thermometer when the plates are bolted together.

The cells for ^{3}He, ^{4}He, e-H_2 and Ne each have volumes of 3 cm^3. The e-H_2 cell contains about 0.5 cm^3 of FeO(OH) powder, which is used as a catalyst for the conversion of ortho-hydrogen and para-hydrogen to their equilibrium distribution. The O_2 and Ar cells have volumes of 20 cm^3. They each contain a vertical sheet of corrugated copper foil, folded and arranged to make five foil vanes projecting out from a the axis of the cell. This improves thermal contact between the periphery of the cell and its center.

The top of each TP and VP cell is covered by OFHC copper caps. All cell caps have vertical holes drilled through their center. Indium O-rings are used to seal the caps to the copper block. All parts of the copper block are plated with gold to give it a constant, uniform, and low emissivity.

The resistance thermometers used during the realizations are liberally coated with stop-cock grease to insure good thermal contact with the cell block. Their lead wires are soldered to tempering strips on the cell block to eliminate heat leaks to the thermometers.

Fill-tubes

All seven cells have vertical fill-tubes connecting them to the gas handling system. For most of their length these fill-tubes are made of stainless steel tubes of outer diameter 3.2 mm and wall thickness 0.13 mm. The fill-tube for the gas thermometer is directly over the center of the copper block, and the other six fill-tubes are located 6.4 mm away and distributed at equal angles. At 4 cm above the copper block all VP and TP fill-tubes are silver-soldered to stainless-steel connecting tubes of outer diameter 4.76 mm and wall thickness 0.89 mm. Each connecting tube is bent to position the bottom of the tube directly over its respective cell and silver-soldered into the cell cap.

The ICVGT fill-tube is 161.2 cm long and extends 2 mm into the ICVGT fill-line of the copper block. The fill-tube is silver-soldered to a fitting that is screwed to the top of the copper block and is sealed with an indium O-ring. The fill-tubes are partially slit in 4 evenly spaced locations along their length; copper-foil baffles are silver-soldered into these slits to prevent radiation from the top of the tubes reaching the cells. No special arrangements have been made for the fill-tube of the ^{4}He VP cell to prevent film-flow and refluxing of superfluid ^{4}He [8]. However, no effects from this (e.g. discontinuities in the ^{4}He VP at 2.1768 K due to the resulting pressure/temperature gradients) have been observed [4].

The seven fill-tubes are attached to eight copper platforms along their length. Seven holes are drilled through each platform for the fill-tubes to pass through; the fill-tubes are soft-soldered to the platform. Bakelite spacers are attached by epoxy to the outer edges of each platform at three equally spaced locations to provide thermal insulation from the throat of the cryostat. Each platform has a calibrated diode thermometer attached to it for measuring its temperature; these measurements are used to make aerostatic head corrections for the VP and ICVGT realizations and deadspace corrections for the ICVGT. All platforms also have copper wires attached to them to shunt them to cold locations on the cryostat for decreasing heat leaks down the fill-tubes.

Pressure Measurement

The pressure measurement system for the sample gases, shown in Figure 2, includes a gas-lubricated piston gauge (PG) used in absolute mode and a differential capacitance diaphragm gauge (DCDG). The DCDG is calibrated *in situ* against the PG using a constant pressure source in the cell. When making a pressure measurement, weights are placed on the PG so that when balanced, it generates a pressure within 200 Pa of the sample gas pressure; the difference is measured with the DCDG. An absolute CDG with a range of 133 kPa is attached to the manifold between the PG and DCDG and is used to help estimate the proper amount of weight to place on the piston gauge. A second DCDG is used to measure the difference between the pressure in the bell jar of the PG and that of a hard vacuum made by an ion pump. One PG is used for all cells, but each cell requiring pressure measurement is used with its own DCDG. The gas used between the PG and DCDG is 99.9999 % pure nitrogen. The lower limit of the PG is 1.38 kPa. When the pressure to be measured is below this, it is measured with the DCDG referenced against a vacuum produced by the turbomolecular pump. While the specified range of the DCDG is only 1.33 kPa, we are able to use it up to 1.38 kPa.

Gas-handling System

Above the cryostat, the gas-handling system is made entirely out of stainless steel. Each cell has its own manifold. The manifold connects the fill-tube to the pressure sensor (if present), gas source, vacuum system and a 3.2 L stainless steel flask used for storing the gas after its use. Each manifold has been baked out at 100 °C for a minimum of 24 hours. One vacuum system, which includes a turbomolecular pump, is used for all gases. A residual gas analyzer, located immediately above the turbomolecular pump, is used to confirm the purity of the gas samples and test for leaks.

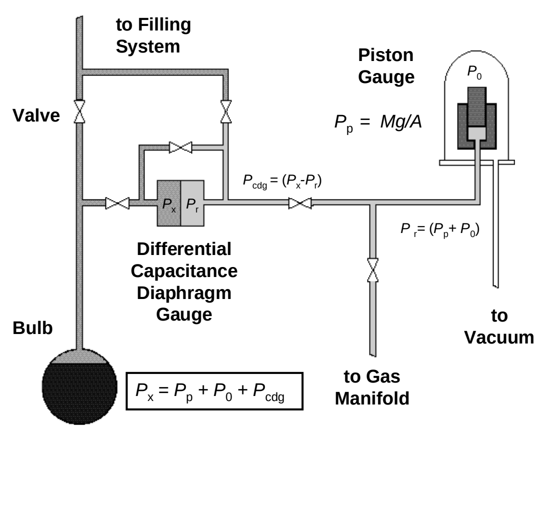

FIGURE 2. Pressure measurement system for the LTRF.

Low Temperature Calibration Facility

The LTCF described here completely replaces an older facility [9] at NIST which was decommissioned in 1993. The LTCF cryostat features a controlled platform at the lowest point of the ^{3}He refrigerator for use with either an isothermal comparison block or an adiabatic calorimeter. A schematic diagram of the two arrangements is shown in Figure 3.

Comparison Block

The LTCF uses an OFHC copper comparison block over the range 0.65 K to approximately 160 K for RIRT and CSPRT comparisons. In practice, dual-zone control is implemented at most temperatures above 24.5 K. The comparison block is approximately cubic, 56 mm on a side. It can accommodate 24 four-wire devices, up to 20 of which can be standard capsule thermometers. The thermometer wells are all vented horizontal bores of 5 mm to 5.7 mm diameters. Each pair of lead wires is thermally coupled to the block via varnished windings on two of eight separate tempering

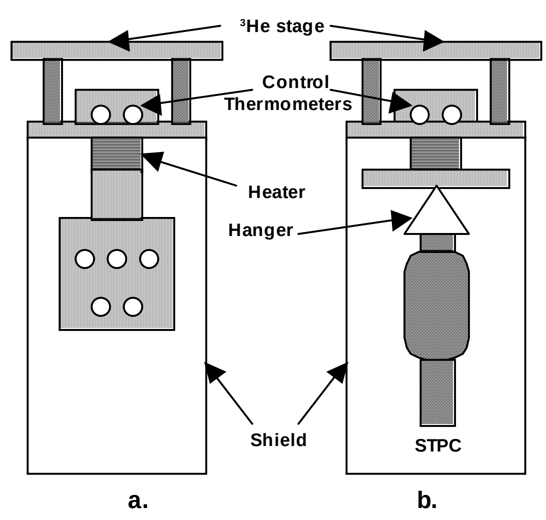

FIGURE 3. Controlled platform of ^{3}He system shown in both operating modes: a.) with block installed as an isothermal comparator. b.) with STPC arranged as a calorimeter.

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spools mounted on the block. The system is arranged so that no heating power is applied directly to the block. Rather, the mounting point for the block under an isothermal shield is heated in order to minimize induced thermal gradients in the block. The control thermometers are installed directly above the main heater (see Figure 3).

Calorimeter

The LTCF can be configured as a calorimeter for realizing triple points by removing the comparison block from the platform and replacing it with a structure for supporting STPCs. The calorimeter working volume is approximately 1.6 L, sufficient to hold up to three NIST STPCs. The wiring is thermally coupled to the adiabatic shield via tempering spools and can accommodate up to three thermometers and two heaters for each of three cells.

The calorimeter shield is controlled at a constant temperature generally within 1 mK of the triple-point plateau temperature. Hence, the system slightly departs from adiabatic conditions both above and below the melting plateau. There is no circulation of ^{3}He needed for any triple point realizations in the calorimeter, and all cooling is derived via conduction to the main bath. Approximately 5 mW of heating is applied to the shield to balance the available cooling power at 13.8 K when the main bath contains fully-vented liquid ^{4}He.

Sealed Triple-Point Cells

The NIST STPCs vary in design according to which substance they hold. For Ar cells, a series of all-stainless-steel (SS), 50 ml capacity cells were constructed in 1978 [10]. For O_2, a smaller series of 316 SS cells were constructed in 1985 [11]. For Xe, cells from the National Research Council of Canada have been used [12]. In the case of Ne, cells made from copper and SS were constructed at NIST in 1995 [13]. Most recently, a series of 40 ml cells for *e*-H_2 were constructed at NIST in 1999 made from copper and 316 SS using a similar design to that of the Ne series. All the *e*-H_2 cells use FeO(OH) (alpha) as the ortho-para catalyst.

SPTC sample amounts vary between 0.05 mol and 0.2 mol depending on the element. The method for filling an STPC with its sample has varied from cell to cell. In some cases the gas has been fully condensed into the cell as a solid at low temperatures before sealing. In other cases the filling has been accomplished in a high pressure gas phase at ambient temperatures. Sealing mechanisms have also varied from crimping stainless steel capillaries to collapsing copper tubing which is then fully welded shut. Of the oldest NIST/NBS cells still in use today, only one (Ar-NBS-7) has suffered a significant loss of its original gas charge.

All NIST STPCs are interfaced with the calorimeter via miniature 16-contact pin-and-socket connectors. Most of the cell heaters are metal-foil-on-polyimide bonded to the cell exterior with epoxy.

FIXED-POINT MATERIALS

With the exception of oxygen, all the fixed-point materials used for low temperature ITS-90 realizations at NIST are derived from commercial gas sources. The chemical purity has in some cases been provided by the manufacturer through a lot analysis. In the case of oxygen, the source gas (“NBS-O-83”) was originally synthesized by Furukawa [11] via thermal decomposition of $KMnO_4$. Table 1 provides a summary of the NIST low temperature fixed-point cells and their associated fixed-point materials.

TABLE 1. NIST fixed-point source materials.

Cell	Source†	Reported Chemical Purity
Ar-LTRF	MGI-Ar-89[14]	99.99999 mol %
Ar-NBS-1	ASG-74[15]	99.9999 mol %
Ar-NBS-3	ASG-74	99.9999 mol %
Ar-NBS-7	ASG-74	99.9999 mol %
O_2-LTRF	NBS-O-83[11]	>99.9999 mol %
O_2-PO-1	NBS-O-83	>99.9999 mol %
O_2-PO-3	NBS-O-83	>99.9999 mol %
Ne-LTRF	MGP-Ne-79[16]	>99.999 mol %
Ne-101	MGP-Ne-95[16]	<99.999 mol %
Ne-201	MGP-Ne-95	>99.9995 mol %
e-H_2-LTRF	MGI-H-89[17]	99.9999 mol %
e-H_2-211	MGP-H-99[18]	99.9999 mol %
e-H_2-212	MGI-H-89	99.9999 mol %
e-H_2-213	MGI-H-89	99.9999 mol %
e-H_2-214	MGP-H-99	99.9999 mol %
^{4}He-LTRF	MGI-He-89[19]	99.9999 mol %
^{3}He-LTRF	ISO-He-90[20]	99.995 mol %

In the case of hydrogen, the two NIST gas sources have been sampled from their gas cylinders and analyzed for deuterium (“D”) content relative to established isotopic reference materials [21]. Table 2

† The commercial gas sources mentioned here are included for completeness only and do not constitute an endorsement by NIST nor does it imply that they are necessarily the best available for this purpose.

lists the isotopic content of the two NIST hydrogen source gases.

TABLE 2. Isotopic content of NIST H_2 used in fixed point realizations since 1994 (k=2 uncertainties). [21]

Gas	δ_{VSMOW}‡	μmol D/mol H
MGP-H-99	–813 ‰ ± 4 ‰	45.8 ± 0.6
MGI-H-89	–706 ‰ ± 4 ‰	29.1 ± 0.6

‡ Relative depletion in D with respect to Vienna Standard Mean Ocean Water, ‰ = 10^{-3} .

SUMMARY

Two NIST facilities have been constructed for the combined objectives of realization and dissemination of the ITS-90 at temperatures below 84 K. The measurement results are linked through the exchange of check thermometers. The results of realizations and comparisons performed in these facilities are given elsewhere in these proceedings [22].

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