

# **THERMAL CHARACTERISTICS OF THE NIST FIXED-POINT CELLS, FURNACES, AND MAINTENANCE BATHS OVER THE TEMPERATURE RANGE FROM 83.8058 K TO 1234.93 K**

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## **ABSTRACT**

The optimal realization of an International Temperature Scale of 1990 (ITS-90) defining fixed-point temperature, for an accurate calibration of a (high-temperature) standard platinum resistance thermometer [(HT)SPRT], critically depends on the thermal characteristics of the furnace or maintenance bath, the design of the fixed-point cell assembly, and the thermal contact of the (HT)SPRT with the liquid/solid interface of the fixed-point substance of the highest purity (>99.9999 wt. % pure). At NIST, several different types of furnaces and maintenance baths are used to realize the nine ITS-90 fixed points which cover the temperature range from 83.8058 K to 1234.93 K. The furnaces, maintenance baths, and fixed-point cells are designed to minimize the temperature difference over the length of the fixed-point cell crucible during fixed-point realization to less than 10 mK. Additionally, to insure that the (HT)SPRT is properly measuring the fixed-point cell temperature, the fixed-point-cell assemblies are designed such that the furnace or maintenance bath increases the immersion of the (HT)SPRT in the thermometer well of the fixed-point cell. At NIST, the immersion characteristic of the (HT)SPRT is tested by inserting the thermometer in 2 cm steps, starting at 10 cm above the bottom of the thermometer well. To exhibit proper immersion in a fixed-point cell, the (HT)SPRT is required to track the ITS-90 assigned value of the hydrostatic-head effect for the fixed-point substance over the bottommost 3 cm. Since different designs of thermometers may cause different immersion profiles, all models of (HT)SPRTs are tested for proper immersion in the appropriate fixed-point cell and furnace combination prior to use. In this paper, the furnace, maintenance bath and fixed-point-cell designs used at NIST are discussed. Examples of the furnace and maintenance bath temperature distributions as well as immersion profiles of different types of (HT)SPRTs in the different fixed-point cells, are given.

## **1. INTRODUCTION**

The Platinum Resistance Thermometry (PRT) Laboratory at the National Institute of Standards and Technology (NIST) uses several different types of furnaces, baths and fixed-point cells to maintain the International Temperature Scale of 1990 (ITS-90) fixed points over the temperature range from 83.8058 K to 1234.93 K to calibrate (high-temperature) standard platinum resistance thermometers (HT)SPRTs [1]. The ability of the (HT)SPRT to properly sense the inner liquid-solid interface temperature of the fixed-point substance is critically dependent on the interaction of the thermal characteristics of the furnace or maintenance bath, fixed-point cell, and the (HT)SPRT. To best achieve this requirement, the NIST furnaces, maintenance baths, and fixed-point cells are designed to minimize the temperature difference over the length of the fixed-point cell and maximize the immersion characteristics of the (HT)SPRT. Additionally, manufacturers design (HT)SPRTs with minimum axial heat transfer and maximum radial heat transfer to enhance the immersion characteristics of the (HT)SPRT. This paper provides information on the designs of the NIST furnaces, maintenance baths, fixed-point cells as well as the thermal characteristics of the furnaces. The results of immersion profile tests of various (HT)SPRTs in the PRT Laboratory fixed-point cells show that they all exceed the requirement for proper immersion.

## **2. MAINTENANCE BATHS AND FURNACES**

Three types of furnaces and three types of maintenance baths are used to realize the ITS-90 fixed-point temperatures in the range from 83.8058 K to 1234.93 K in the PRT Laboratory [2-7]. These furnaces and baths, either constructed at NIST or acquired commercially, were designed specifically to yield optimal performance with each fixed-point cell. As shown in Table 1, a furnace or bath is dedicated for use with each of the nine fixed-points that are required for the above temperature range.

Table 1. NIST furnaces and maintenance baths used to realize the ITS-90 from 83.8058 K to 1234.93 K

ITS-90 Fixed Point		T/K	Furnace/Maintenance Bath Type	ITS-90 Fixed Point		T/K	Furnace/Maintenance Bath Type
Ar	TP	83.8058	100 L Dewar	Sn	FP	505.078	three zone
Hg	TP	234.3156	40 L alcohol bath	Zn	FP	692.677	three zone
H <sub>2</sub> O	TP	273.16	43 L water bath	Al	FP	933.473	sodium heat pipe
Ga	TP	302.9166	single zone	Ag	FP	1234.93	sodium heat pipe
In	FP	429.7485	three zone				

TP = triple point      FP = freezing point

## 2.1 Maintenance baths

The argon TP cell is maintained in a commercially-available stainless-steel 100 L liquid N<sub>2</sub> Dewar with super-insulation [4]. The top of the Dewar was modified to allow the argon TP cell to be integrated into the apparatus as a single unit. The adiabatically-controlled TP cell can be maintained indefinitely as long as the Dewar contains a sufficient quantity of liquid N<sub>2</sub>.

The maintenance bath used for the Hg TP cells is a commercially-available stirred-liquid bath containing about 40 L of ethanol. In combination with the two-stage compressor system for cooling and the internal heater, the bath can be used over the range from 192 K to 373 K. The bath has a depth of 54.6 cm and can accommodate up to two cells. Additionally, two wells are provided for chilling SPRTs prior to insertion into the Hg TP cell. In this bath, a Hg TP cell can be maintained for at least one week.

The maintenance bath used for the H<sub>2</sub>O TP cell is a commercially-available stirred-liquid bath that contains 42.5 L of water and 0.5 L of ethanol. The Peltier-cooled bath can accommodate four H<sub>2</sub>O TP cells and two wells for chilling (HT)SPRTs prior to insertion into the H<sub>2</sub>O TP cell. By operating at 273.157 K, this bath can maintain a H<sub>2</sub>O TP cell mantle for at least six months.

## 2.2 Furnaces

There are three NIST-fabricated furnaces for realizing the Ga TP [8]. Each of the three furnaces is a large cylindrical block of aluminum with a central well for the Ga TP cell. A single-zone, dc-powered single-layer bifilar-wound heater on the aluminum block controls the temperature. The furnaces are designed to provide about a 1 mm annular space (filled with mineral oil) between the Ga TP cell and the wall of the well in the aluminum block. One of the three furnaces, which operates at 313.15 K, is used for the preparation of the outer liquid-solid interface of the Ga TP cell. In this furnace, the triple-point plateau will last approximately 13 hours. With the other two furnaces, which operate at 303.05 K, the triple-point plateau will last at least 6 months. An immersion heater operating at a temperature of 313.15 K is used to establish the inner liquid-solid interface of the Ga TP cell.

There are five NIST-fabricated furnaces available for realizing the In FP, Sn FP and Zn FP [2]. These automatically-controlled three-zone furnaces have three dc-powered heater zones (top, middle and bottom) with the top and bottom acting as guard zones to provide a uniform temperature environment over the vertical length of the fixed-point sample. All five of the furnaces and control systems [7] are interchangeable and direct comparison of fixed-point cells of the same type can be made with them. An auxiliary furnace is integrated into the furnace enclosure for heating the (HT)SPRT prior to insertion into the fixed-point cell. Freezing-point plateaus can be maintained for at least 16 h using these furnaces.

There are three NIST-fabricated, sodium-filled heat-pipe furnaces for realizing the Al FP and Ag FP [3, 9, 10]. These automatically-controlled high-temperature furnaces have two dc-powered heater zones for the heat pipe. One of the heaters extends slightly beyond the length of the heat pipe and the other heater, a plug heater, fits in the bottom of the open-bottom heat pipe. All three of the furnaces and control systems [7] are interchangeable and direct comparison of fixed-point cells of the same type can be made with them. Freezing-point plateaus can be maintained for at least 16 h using these furnaces. The two auxiliary furnaces, used either to heat the (HT)SPRT prior to insertion into the fixed-point cell or to anneal the (HT)SPRT, are placed in a single

enclosure [3, 5]. Each of the auxiliary furnaces contains a closed-end Pt protection tube placed between two closed-end fused silica tubes for protecting the (HT)SPRTs from metal ion contamination.

### 3. FIXED-POINT CELLS

All NIST-fabricated fixed-point cells contain appropriate substances of the highest available purity (>99.9999 wt. % pure) [6].

The NIST-fabricated Ar TP cell is designed to accommodate up to seven long-stem SPRTs from the top and up to six capsule SPRTs from the bottom of the cell [4]. A special capsule SPRT holder [2] with an outer diameter of 12 mm can be used to measure the resistance ratio of the thermometer in the 13 mm inner diameter central top well. The apparatus contains about 19.7 moles of liquid Ar with about 15.7 moles condensed into the cell giving the SPRTs an immersion depth of 10.9 cm. Helium gas is placed in the seven top-loading thermometer wells to increase the thermal contact of the SPRTs with the Ar TP.

The Hg TP cell is realized in a NIST-fabricated all stainless steel cell with a thermometer well extension [11, 12]. The cell, with its thermometer well extension, is placed in a stainless steel, insulation filled, outer jacket. Additionally, during the realization of the Hg TP, the outer jacket is evacuated to isolate the cell from temperature fluctuations in the maintenance bath and to increase the duration of the TP plateau. The cell contains 2.5 kg of Hg which provides an SPRT immersion depth of 15 cm. The thermometer well is completely filled with ethanol to improve the thermal contact of the SPRT with an inner liquid-solid interface of the Hg TP cell.

The commercially-acquired borosilicate glass H<sub>2</sub>O TP cell contains about 400 cm<sup>3</sup> of water. It has a nominal immersion depth of 26.5 cm for the (HT)SPRTs. The type A cell, which allows visual determination of the partial pressure of air in the cell, is the type normally used at NIST [13]. During use, the cell is completely immersed in its maintenance bath and its thermometer well is completely filled with water to improve the thermal contact of the (HT)SPRT with an inner liquid-solid interface of the H<sub>2</sub>O TP cell.

The NIST-fabricated Ga TP cells are of three different designs [8, 14,]. All three designs use virgin PTFE for the crucible and cap, but the thermometer well is either glass or nylon and the outer enclosure is either glass or nylon. The Ga TP cell is evacuated during the preparation of the TP and for the realization of the TP. The cell contains 1 kg of Ga giving an (HT)SPRT an immersion depth of 18 cm. The thermometer well is completely filled with mineral oil to improve the thermal contact of the (HT)SPRT with an inner liquid-solid interface of the Ga TP cell.

The appropriate metal in NIST-fabricated In FP, Sn FP, and Zn FP cells is contained within a graphite crucible, lid and thermometer well assembly [2]. The sample volume, after allowing for a 1 cm head space between the liquid metal and the underside of the graphite lid, is 149 cm<sup>3</sup>. The graphite assembly fits inside a precision-bore borosilicate-glass envelope with ceramic fiber blanket in the annular space between the graphite crucible and the borosilicate-glass envelope. A matte-finished borosilicate-glass (HT)SPRT guide tube, washed-ceramic fiber disks and two graphite heat shunts are installed above the graphite lid. The first heat shunt is placed approximately 3.2 cm and the second heat shunt is placed approximately 10.8 cm above the top of the graphite lid. The heat shunts are used to thermally temper the sheath of the (HT)SPRT and increase the immersion of the thermometer. The dimensions of the glass envelope, glass guide tube and heat shunts have been designed to fit snugly. The top of the glass envelope is sealed with a silicone rubber stopper that contains a modified compression fitting with a silicone rubber O-ring, for inserting and sealing the SPRT into the fixed-point cell, and a stainless steel gas filling tube for evacuating and backfilling the cell with an inert gas (Ar or He) to 0.25 kPa above the atmospheric pressure to prevent contamination of the metal. In these fixed-point cells, the immersion depth of an (HT)SPRT is 18 cm.

The appropriate metal in the NIST-fabricated Al FP and Ag FP cells is contained within a graphite crucible, lid and thermometer well assembly [5, 9]. The sample volume, after allowing for a 1 cm head space between the liquid metal and the underside of the graphite lid, is 149 cm<sup>3</sup>. The graphite assembly is placed inside a silica-glass envelope with a silica-glass thermometer well inserted into the graphite well. The silica-glass thermometer well is matte finished to prevent "light piping". Attached to the top of the silica-glass envelope is a matte-finished silica-glass pumping tube to evacuate and back fill the cell to a pressure of 101.3 kPa with

purified argon during use. The fixed-point cell is inserted into an Inconel protecting tube that contains a 0.5 cm thick ceramic-fiber cushion at the bottom. Above the silica-glass envelope, there is a 1 cm air gap, then 12 Inconel disks (radiation shields) separated by 1 cm long silica-glass spacers. The 18 cm space remaining above the top radiation shield is filled with disks of ceramic-fiber insulation. The radiation shields are used to thermally temper the sheath of the (HT)SPRT and increase the immersion of the thermometer. The matte-finished fused-silica thermometer guide tube extends about 0.5 cm above the top of the Inconel protecting tube. The pumping tube is used for evacuating and backfilling the cell with an inert gas to a pressure of 101.3 kPa. In these fixed-point cells, the immersion depth of an (HT)SPRT is 18 cm.

#### 4. THERMAL CHARACTERISTICS

The thermal characteristics of the furnaces and maintenance baths were studied with the fixed-point cells installed. The temperature stability of the furnace or bath was determined by placing the (HT)SPRT into the thermometer well of the appropriate fixed-point cell and measuring the temperature fluctuations overnight (15 h). The equilibrium temperatures over the vertical length of the thermometer well were determined at 2 cm steps. For these measurements, the temperature of the furnace or bath was set to control at approximately 2.5 K below the fixed-point temperatures of Ar, Hg, Ga, In, Sn, In, Al, and Ag and approximately 3 mK below that of H<sub>2</sub>O. The vertical temperature profile of the thermometer well was taken to closely approximate that of the furnace and maintenance bath during operation.

The ability of (HT)SPRTs of various manufacturer models to exhibit proper immersion in the NIST fixed-point cells was investigated. For the (HT)SPRT to be considered properly immersed (no stem-conduction effect influencing the measurements), the thermometer must track the ITS-90 assigned value of the hydrostatic-head effect over the bottommost 3 cm [15].

The measurement equipment used for the tests included a commercially-available ac resistance-ratio bridge operating at a frequency of 30 Hz, thermostatically-controlled (262.15 K  $\pm$  10 mK) ac/dc reference resistors and the (HT)SPRTs. A detailed description of the measurement system is found in references [3] and [7]. Measurements of the thermal characteristics of the furnaces and maintenance baths were made with an excitation current of 1.0 mA. Measurements of the ability of the (HT)SPRT to track the assigned hydrostatic-head effect in a fixed-point cell were made with two excitation currents of 1.0 mA and 1.414 mA for extrapolation to 0 mA. The 1 mA reading was repeated after the 1.414 mA reading to insure that the (HT)SPRT was at thermal equilibrium. The two 1 mA readings were required to repeat to within the equivalent of 0.01 mK for the measurements to be valid.

##### 4.1 Furnaces and maintenance baths

As shown in table 2, the temperature fluctuations of the maintenance baths for the Ar TP, Hg TP, and H<sub>2</sub>O TP cells did not cause the SPRT to vary more than  $\pm$ 2 mK over the 15 h test period. The temperature fluctuations in the Ga TP cell furnaces caused the largest temperature variation of  $\pm$ 10 mK. The temperature variations in the three-zone furnaces for the In, Sn, and the Zn FP cells caused instabilities that did not exceed  $\pm$ 7 mK. The two-zone heat-pipe furnaces for the Al and Ag FP cells caused instabilities that did not exceed  $\pm$ 9 mK.

The vertical temperature profile of the fixed-point cell thermometer well was determined by slowly inserting the (HT)SPRT in 2 cm steps over the length of the well. Five minutes per increment was allotted for the (HT)SPRT to equilibrate prior to measurement. As shown in table 2, the vertical temperature differences over the length of the thermometer well of the fixed-points cells in the maintenance baths, single-zone Ga TP furnace, three-zone furnace and the sodium heat pipe furnace did not exceed 2 mK, 7 mK, 8 mK and 9 mK, respectively. Figure 1 shows a mapping of the temperatures with vertical positions (0 cm to 22.5 cm) for the In FP, Sn FP and Zn FP cells used with three-zone furnaces and that for the Al FP and Ag FP cells used with the two-zone heat-pipe furnaces. Because of the greater depth of insertion of these cell crucibles into the furnaces, the measurements of the vertical temperature differences were made with the (HT)SPRT starting about 6 cm above the top of the graphite crucible (25 cm above the bottom of the thermometer well) in the thermometer guide tube.

##### 4.2 Fixed-point cells and (HT)SPRTs

Proper immersion of the (HT)SPRT was verified by measuring the (HT)SPRT resistance starting at 10 cm from the bottom of the thermometer well, then inserting the (HT)SPRT in 2 cm steps until 4 cm from the bottom, and then inserting the (HT)SPRT in 1 cm steps until the bottom of the thermometer well was reached. After changing the immersion depth of the (HT)SPRT, the SPRT was allowed to re-equilibrate at each step prior to

measurement. The immersion depth of the (HT)SPRT was calculated from the sensor midpoint to the height of the fixed-point material column during the fixed-point realization.

As shown in table 3, the different thermometer types that were tested all exceeded the minimum requirement for proper immersion by tracking the ITS-90 assigned hydrostatic-head effect over the bottommost 3 cm of the fixed-point cells. As expected, however, there are small differences between thermometer types. This is caused by the variation in the thermometer design to maximize radial heat transfer and to minimize axial heat transfer. The difference in the diameters of the thermometer and the well of the fixed-point cell may affect the immersion characteristics.

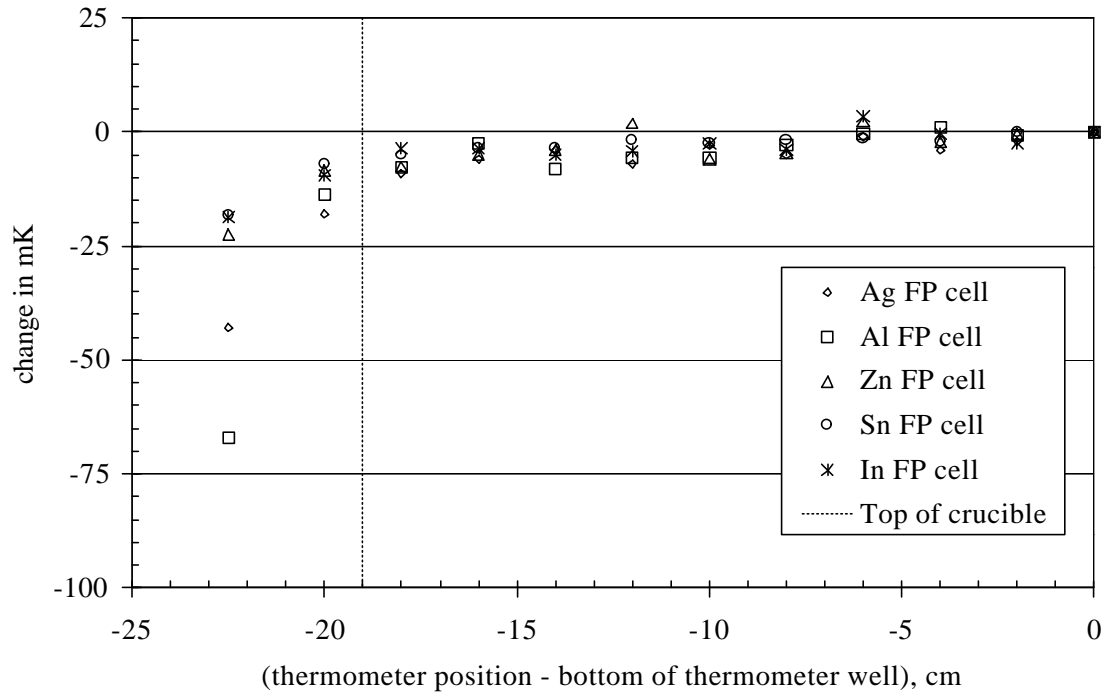


Figure 1. Vertical temperature profile for two-zone sodium heat pipe furnaces containing Ag FP and Al FP cells and for the three-zone furnaces containing the Zn FP, Sn FP and In FP cells. Measurements made with the furnace temperature 2.5 K below the freezing point of the fixed-point cell.

Table 2. Thermal characteristics of furnaces and maintenance baths used at NIST to realize the ITS-90 from 83.8058 K to 1234.93 K. Furnace and bath stability over 15 h and vertical temperature profile of thermometer well from 0 cm to 18 cm.

Fixed-Point Cell	Furnace/Bath Stability, $\pm$ mK	Thermometer Well Profile $\Delta T$ , mK	Fixed-Point Cell	Furnace/Bath Stability, $\pm$ mK	Thermometer Well Profile $\Delta T$ , mK
Ar TP	1	2	Sn FP	6	5
Hg TP	2	4	Zn FP	7	8
H <sub>2</sub> O TP	2	4	Al FP	8	8
Ga TP	10	7	Ag FP	9	9
In FP	4	4			

Table 3. Number of cm that certain (HT)SPRTs can track the hydrostatic-head effect in NIST fixed-point cells. Fixed-point cells were tested over the bottommost 10 cm of the thermometer wells. A minimum of 3 cm is required to overcome stem conduction loss.

Fixed-Point Cell	Winding and Former Type of SPRTs					Winding and Former Type of HTSPRTs				
	SLB-M	CH-M	CH-Q	BC	TC	SLB-Q	CH-Q	TC	BC	
Ar TP	5	5	5	4	5					
Hg TP	9	6	6	5	5					
H <sub>2</sub> O TP	9	8	8	7	6	7	7	8	8	
Ga TP	10	10	10	8	9	8	8	8	8	
In FP	10	8	8	7	8	9	8	8	7	
Sn FP	10	9	9	7	8	8	7	9	7	
Zn FP	9	9	9	6	8	7	7	8	6	
Al FP			7	5	6	7	7	8	6	
Ag FP						6	6	7	6	

SLB-M: single-layer bifilar-mica    CH-Q: coiled helix-fused silica    TC: twisted coil  
 CH-M: coiled helix-mica    BC: bird cage    SLB-Q: single-layer bifilar-fused silica

## 5. CONCLUSIONS

The results of measurements with various (HT)SPRTs of the thermal characteristics of the maintenance baths, furnaces, and fixed-point cells indicate that the calibration of (HT)SPRTs at the NIST PRT Laboratory will not contribute significant errors in the measurement of ITS-90 temperatures from 83.8058 K to 1234.93 K. This is evidenced in the ability of the various (HT)SPRT designs to exhibit proper immersion in the NIST fixed-point cells by tracking the ITS-90 assigned value of the hydrostatic-head effect over at least the bottommost 4 cm of the thermometer well.

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