

FLUKE®

Calibration

5944/5945/5946/5947

Mini Metal Fixed Point Cells

Users Guide

2004, Rev. 1, 5/11

© 2004 - 2011 Fluke Corporation. All rights reserved. Specifications subject to change without notice.
All product names are trademarks of their respective companies.

Limited Warranty & Limitation of Liability

Each Fluke product is warranted to be free from defects in material and workmanship under normal use and service. The warranty period is one year and begins on the date of shipment. Parts, product repairs, and services are warranted for 90 days. This warranty extends only to the original buyer or end-user customer of a Fluke authorized reseller, and does not apply to fuses, disposable batteries, or to any product which, in Fluke's opinion, has been misused, altered, neglected, contaminated, or damaged by accident or abnormal conditions of operation or handling. Fluke warrants that software will operate substantially in accordance with its functional specifications for 90 days and that it has been properly recorded on non-defective media. Fluke does not warrant that software will be error free or operate without interruption.

Fluke authorized resellers shall extend this warranty on new and unused products to end-user customers only but have no authority to extend a greater or different warranty on behalf of Fluke. Warranty support is available only if product is purchased through a Fluke authorized sales outlet or Buyer has paid the applicable international price. Fluke reserves the right to invoice Buyer for importation costs of repair/replacement parts when product purchased in one country is submitted for repair in another country.

Fluke's warranty obligation is limited, at Fluke's option, to refund of the purchase price, free of charge repair, or replacement of a defective product which is returned to a Fluke authorized service center within the warranty period.

To obtain warranty service, contact your nearest Fluke authorized service center to obtain return authorization information, then send the product to that service center, with a description of the difficulty, postage and insurance prepaid (FOB Destination). Fluke assumes no risk for damage in transit. Following warranty repair, the product will be returned to Buyer, transportation prepaid (FOB Destination). If Fluke determines that failure was caused by neglect, misuse, contamination, alteration, accident, or abnormal condition of operation or handling, including overvoltage failures caused by use outside the product's specified rating, or normal wear and tear of mechanical components, Fluke will provide an estimate of repair costs and obtain authorization before commencing the work. Following repair, the product will be returned to the Buyer transportation prepaid and the Buyer will be billed for the repair and return transportation charges (FOB Shipping Point).

THIS WARRANTY IS BUYER'S SOLE AND EXCLUSIVE REMEDY AND IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. FLUKE SHALL NOT BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL, OR CONSEQUENTIAL DAMAGES OR LOSSES, INCLUDING LOSS OF DATA, ARISING FROM ANY CAUSE OR THEORY.

Since some countries or states do not allow limitation of the term of an implied warranty, or exclusion or limitation of incidental or consequential damages, the limitations and exclusions of this warranty may not apply to every buyer. If any provision of this Warranty is held invalid or unenforceable by a court or other decision-maker of competent jurisdiction, such holding will not affect the validity or enforceability of any other provision.

Fluke Corporation
P.O. Box 9090
Everett, WA 98206-9090
U.S.A.

Fluke Europe B.V.
P.O. Box 1186
5602 BD Eindhoven
The Netherlands

Table of Contents

Title	Page
Introduction.....	1
Before You Start.....	3
Symbols Used	3
Safety Information	4
How to Contact Fluke Calibration	4
Specifications	5
Environmental Conditions	5
Construction.....	5
Care and Handling Guidelines	6
Realization of the Fixed Point	7
Background Information.....	7
Vertical Temperature Gradient and Controller Accuracy Adjustment	12
Vertical Temperature Gradient Adjustment	12
Vertical Temperature Gradient Test Method	12
Controller Display Accuracy Adjustment.....	12
Procedure for Realizing the Freeze.....	12
Procedure for Realizing the Melting Point	14
The Correction for the Pressure Difference	15

List of Tables

Table	Title	Page
1.	The Defining Metal Freezing Points of the ITS-90, Pressure Constants, and Resistance Ratios	1
2.	Subranges of the ITS-90 and Freezing Points Required for Calibration	2
3.	International Electrical Symbols	3
4.	Specifications	5
5.	Summary of the First Cryoscopic Constants and the Estimated Effects of Impurities	7
6.	The Furnaces for Fixed Points and their Temperature Uniformity	12
7.	Coefficients for the Pressure Difference of Some Defining Fixed Points.....	15

List of Figures

Figure	Title	Page
1.	The Metal-cased Freezing Point Cell	2
2.	The Model 5944/5945/5946/5947 Metal-cased Fixed-point Cell	6
3.	Freezing Curve Comparison of One Cell	8
4.	Typical Freeze Point Cell Design Showing the Liquid-solid Interfaces	9
5.	9114 Furnace Interior with Freeze Point Cell, Cross Sectional View	10
6.	9260 Furnace Interior with Freeze Point Cell, Cross Sectional View	11
7.	A Typical Freezing Curve for the Zinc Cell	13

Introduction

The International Temperature Scale of 1990 (ITS-90) is based on a series of defining fixed points. At temperatures above 273.16 K, most of the fixed points are the freezing points of specified pure metals. Pure metals melt and freeze at a unique temperature through a process involving the absorption or liberation of the latent heat of fusion. A metal freezing point is the phase equilibrium between the liquid phase and solid phase of a pure metal at a pressure of one standard atmospheric pressure (101.325 Pa). The freezing points of indium, tin, zinc, aluminum, silver, gold, and copper are the defining fixed points of the ITS-90. The temperature values of these freezing points assigned by the ITS-90, the pressure effect constants and the resistance ratios at these fixed points according to the ITS-90 SPRT reference function are listed in Table 1.

Table 1. The Defining Metal Freezing Points of the ITS-90, Pressure Constants, and Resistance Ratios

Fixed Point	Assigned Temperature		Pressure Effect of Fixed Points		Wr (T90)	dW./dt (x 0.001)
	T ₉₀ (K)	t ₉₀ (°C)	dt/dP (10 ⁻⁸ K/Pa) [1]	dt/dh (10 ⁻³ K/m)		
FP In	429.7485	156.5985	4.9	3.3	1.60980185	3.801024
FP Sn	505.078	231.928	3.3	2.2	1.89279768	3.712721
FP Zn	692.677	419.527	4.3	2.7	2.56891730	3.495367
FP Al	933.473	660.323	7.0	1.6	3.37600860	3.204971
FP Ag	1234.93	961.78	6.0	5.4	4.28642053	2.840862
FP Au	1337.33	1064.18	6.1	10	--	--
FP Cu	1357.77	1084.62	3.3	2.6	--	--

[1] Equivalent to millikelvins per standard atmosphere.

All of these fixed points are intrinsic temperature standards according to the definition of the ITS-90. Under controlled conditions these freezing points are highly reproducible. The variance among different realizations of a freezing point should be well within 1.0 mK for the freezing points of indium, tin and zinc; and within a few millikelven for the freezing points of aluminum, silver, gold, and copper. For your convenience, Fluke has developed a sealed cell design and new technique for the realization of the freezing points, which has made it easy to realize these fixed points.

Model 5944/5945/5946/5947 Metal-cased Fixed-point Cells are members of the Fluke Fixed-point Cell family (see Figure 1). The 5944/5945/5946/5947 fixed-point cells not only retain the merit of the lowest uncertainties of Fluke fused silica fixed-point cells (590X and 591X), but also provide a much stronger, more durable case. You will never worry about breaking the cell case again. The new cell can be shipped by conventional carrier eliminating the inconvenience of hand-carrying the cell from one place to another.



gpq001.bmp

Figure 1. The Metal-cased Freezing Point Cell

These freezing points are indispensable for the calibration of a standard platinum resistance thermometer (SPRT). Different sub-ranges require different sets of freezing points, as summarized in Table 2.

Table 2. Subranges of the ITS-90 and Freezing Points Required for Calibration

















Subrange	Freezing Points Required
0 °C to 961.78 °C	FP Sn, FP Zn, FP Al, and FP Ag
0 °C to 660.323 °C	FP Sn, FP Zn, and FP Al
0 °C to 419.527 °C	FP Sn and FP Zn
0 °C to 231.928 °C	FP In and FP Sn
0 °C to 156.5985 °C	FP In

Before You Start

Symbols Used

Table 3 lists the International Electrical Symbols. Some or all of these symbols may be used on the instrument or in this manual.

Table 3. International Electrical Symbols

Symbol	Description	Symbol	Description
	Hazardous Voltage		Off
	Hot Surface (Burn Hazard)		On
	Risk of Danger. Important information. See manual.		Fuse
	AC (Alternating Current)		Battery
	AC-DC		Conforms to Relevant Australian EMC Requirements.
	DC		Conforms to Relevant North American Safety Standards.
	Double Insulated		Conforms to European Union Directives.
	PE Ground		Do Not Dispose of this Product as Unsorted Municipal Waste. Go to Fluke's Website for Recycling Information.
CAT II	CAT II equipment is designed to protect against transients from energy-consuming equipment supplied from the fixed installation, such as TVs, PCs, portable tools, and other household appliances.		

Safety Information

Use this instrument only as specified in this manual. Otherwise, the protection provided by the instrument may be impaired.

The following definitions apply to the terms “Warning” and “Caution”.

- “**Warning**” identifies conditions and actions that may pose hazards to the user.
- “**Caution**” identifies conditions and actions that may damage the instrument being used.

⚠ Warning

To avoid personal injury, follow these guidelines:

- **DO NOT use this instrument for any application other than calibration work.**
- **DO NOT use this instrument in environments other than those listed in the Users Guide.**
- **Follow all safety guidelines listed in the Users Guide.**
- **Avoid leaving a PRT installed for an extended period of time which can cause the PRT handle to become hot.**
- **Calibration Equipment should only be used by trained personnel.**
- **Use the Product only as specified, or the protection supplied by the Product can be compromised.**
- **Do not use and disable the Product if it is damaged.**
- **Use this Product indoors only.**
- **Have an approved technician repair the Product.**

⚠ Caution

To avoid possible damage to the instrument, follow these guidelines:

- **Keep the cell clean and avoid contact with bare hands, tap water, or contaminated PRTs. If there is any chance that the cell has been contaminated, clean the quartz with reagent grade alcohol before inserting it into a furnace.**
- **Use the product in the vertical orientation only.**

How to Contact Fluke Calibration

To contact Fluke Calibration, call one of the following telephone numbers:

- Technical Support USA: 1-877-355-3225
- Calibration/Repair USA: 1-877-355-3225
- Canada: 1-800-36-FLUKE (1-800-363-5853)
- Europe: +31-40-2675-200
- Japan: +81-3-6714-3114
- Singapore: +65-6799-5566
- China: +86-400-810-3435
- Brazil: +55-11-3759-7600
- Anywhere in the world: +1-425-446-6110

To see product information and download the latest manual supplements, visit Fluke Calibration’s website at www.flukecal.com.

To register your product, visit <http://flukecal.com/register-product>.

Specifications

See Table 4 below for the Model 5944/5945/5946/5947 Metal-cased Fixed-point Cells specifications.

Table 4. Specifications

Model Number	5944	5945	5946	5947
ITS-90 Assigned Temperature	156.5985 °C	231.928 °C	419.527 °C	660.323 °C
Expanded Uncertainty k=2, Cell Itself	0.6 °C	0.8 °C	1.0 °C	2.0 °C
Expanded Uncertainty k=2, Used in 9260, Melting Curve	1.2 °C	1.6 °C	2.0 °C	4.0 °C
Metal Purity	>99.9999 %	>99.9999 %	>99.9999 %	>99.9999 %
Quantity of Metal	660 g	655 g	648 g	200 g
Outer Diameter of the Cell	41.3 mm	41.3 mm	41.3 mm	41.3 mm
Overall Height of the Cell	222 mm	222 mm	222 mm	222 mm
Inner diameter of the Re-entrant Well	7.8 mm	7.8 mm	7.8 mm	7.8 mm
Total Immersion Depth ^[1]	156 mm	156 mm	156 mm	156 mm

[1] The distance from the bottom of the re-entrant well to the upper surface of the pure metal.

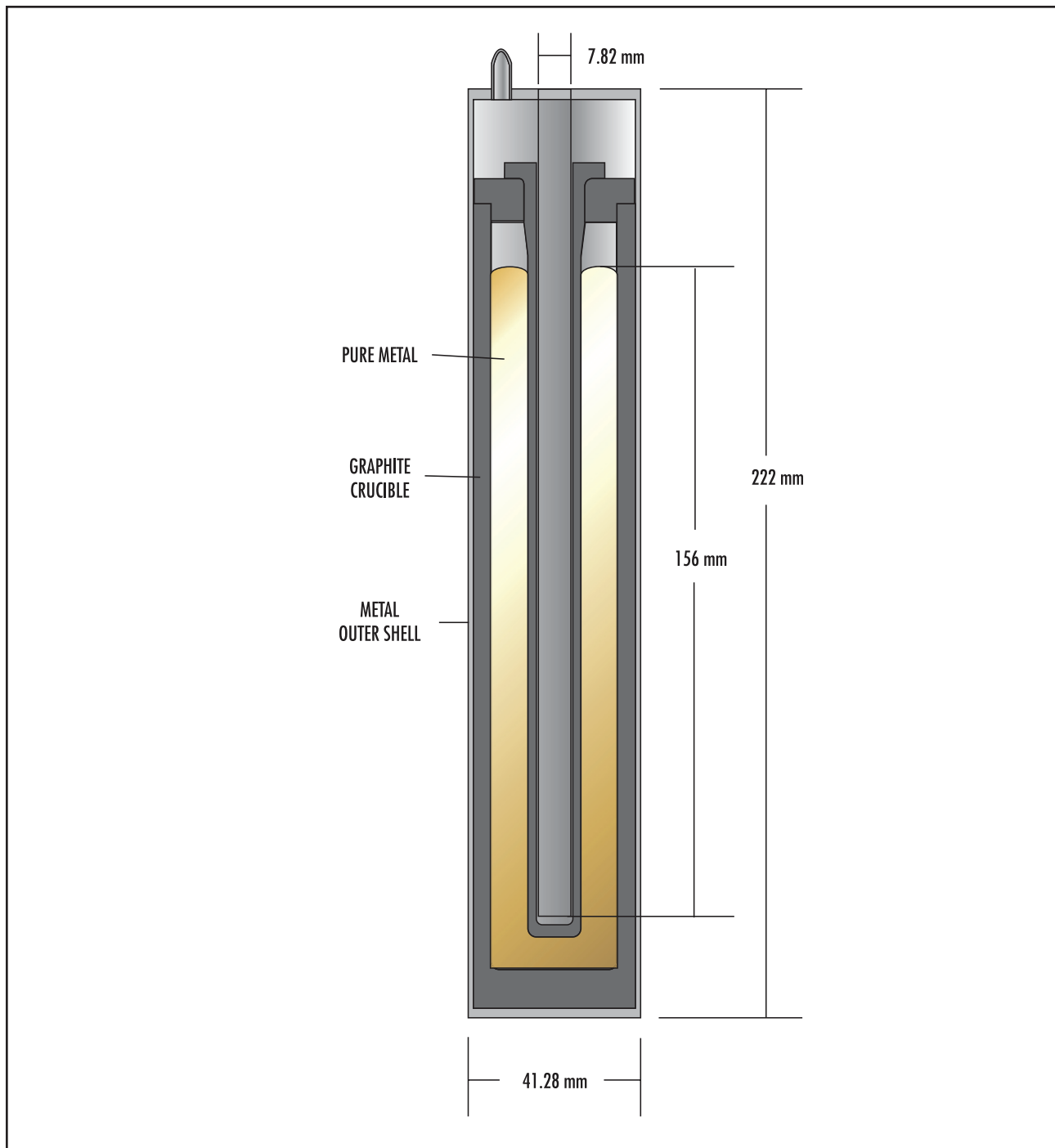
Environmental Conditions

Although the instrument has been designed for optimum durability and trouble-free operation, it must be handled with care. The instrument should not be operated in an excessively dusty or dirty environment.

Construction

The 5944/5945/5946/5947 Metal Freezing Point Cell is shown in Figure 2. An appropriate quantity of metal (See Table 4) with a purity of 99.9999+ % is melted into a graphite crucible with a graphite lid and re-entrant well. The impurity in the graphite is less than 5 ppm. All of the graphite parts are subjected to a high-temperature, high-vacuum treatment before loading the metal sample. It is important to avoid any possible contamination to the surface of the graphite parts during the manufacturing process. The assembled graphite crucible, with the high-purity metal, is then enclosed in a metal outer case and connected to a high vacuum system. The cell is drawn down to a proper pressure at a temperature near the freezing point for several days. During this period the cell is purged with high purity argon repeatedly to remove any contaminants. Finally, the cell is filled with 99.999 % pure argon and permanently sealed at the freezing point. The pressure of the argon in the cell at the freezing point is closely adjusted to 101,325 Pa and the actual value of the pressure recorded. A small temperature correction for the pressure difference can be made using the information in “The Correction for the Pressure Difference” section.

The metal-cased cell is design to be used not only in primary fixed-point furnace (Model 9114), but also in small portable furnace (Model 9260). The total depth of a portable furnace is only about 270 mm (10.6 in), allowing short probes to be calibrated by fixed point. On the top (the space above the fixed-point cell) of the furnace a minimum of 50 mm of thermal insulation is required to maintain good vertical temperature uniformity in the cell. That means that the total length of the cell is limited to about 220 mm. Therefore a total immersion depth into the pure metal is 156 mm (Figure 2). The total immersion depth (156 mm) is a slightly more than is available in the 591X quartz glass cell (140 mm), but less than that of 590X quartz glass cell (195 mm). We pay special attention to decreasing, or eliminating thermal conduction error during calibration at fixed points. Very thin metal tubing (0.01 in thickness) as the re-entrant well decreases the conductivity along the well to a minimum. The tight match between the metal well and the graphite well improves thermal conductivity in the radial direction, and decreases the thermal conduction error.



gpq002.eps

Figure 2. The Model 5944/5945/5946/5947 Metal-cased Fixed-point Cell

Care and Handling Guidelines

The 5944/5945/5946/5947 Fixed-point Cell is a delicate device. Great care must be taken in handling, using and transporting the cell. The metal case is very sturdy, however, the graphite crucible is brittle. It is suggested that the cell be kept in the vertical position as shown in Figure 2 for safety. However, putting a cold cell in horizontal orientation for a short period of time will not cause any damage. Up-side-down orientation might damage the cell. Store the cell in the vertical position and in a safe place. Always handle the cell with care.

The cell can be shipped by commercial carrier with the following preparation:

- Keep the cell in the vertical direction during transportation.
- Put the cell into the wooden case specially designed for fixed-point cell first, and then place the wooden cell case into a large package with foam around the wooden cell case.
- Avoid bumping, shock and strong vibration during transportation. Utilize Shock, Watch, and Tilt Watch indicators.

If the instructions in this manual are carefully followed, the Model 5944/5945/5946/5947 Fixed-point Cell will provide many years of accurate use.

Realization of the Fixed Point

As was mentioned in the Introduction, it is not difficult to realize a freezing point by using a Fluke completely sealed metal freezing point cell. In order to get the highest possible accuracy, a general understanding of the freezing process of an ideal pure metal is helpful.

Background Information

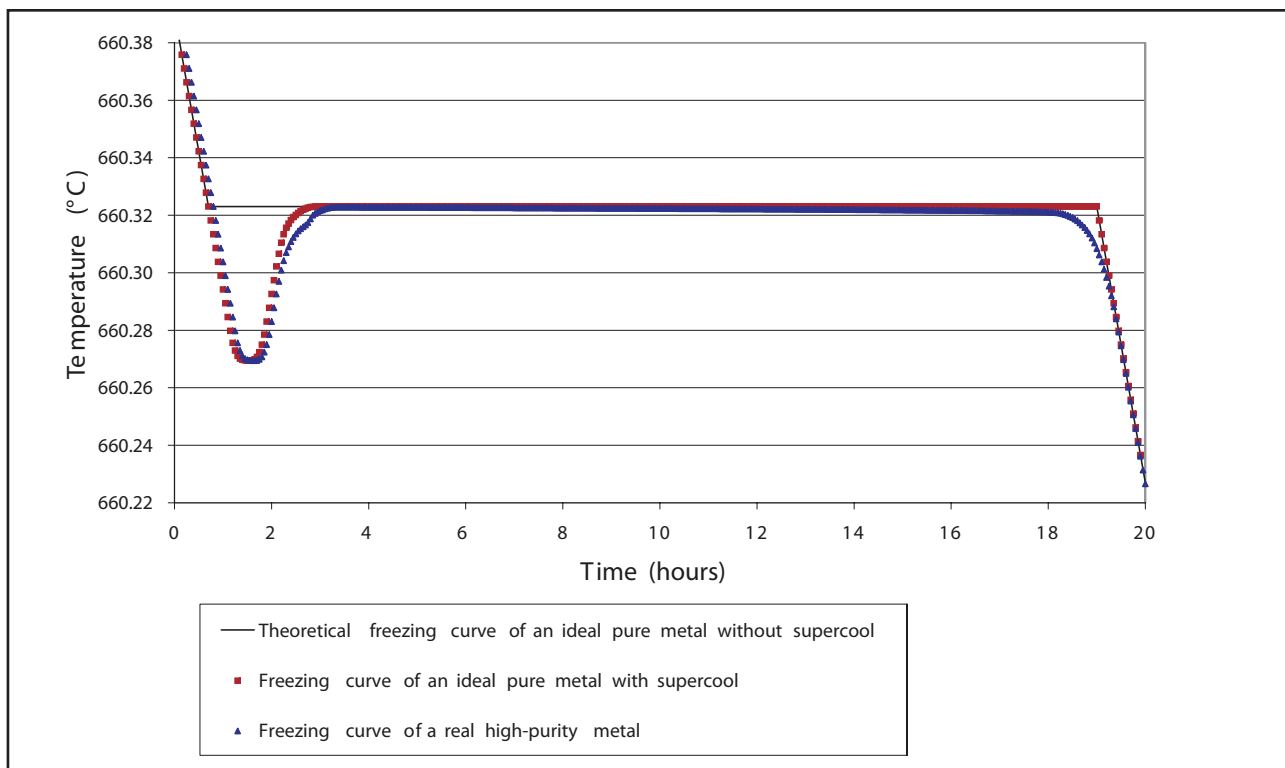
Theoretically the melting and freezing temperatures for an ideal pure metal are identical. However, with the introduction of impurities in the metal, the melting and freezing equilibrium points are usually slightly lower. The freezing plateau of an ideal pure metal is conceptually flat. The only exception is during the supercool. Impurities in the metal generally introduce a slightly negative slope to the plateau. Most of the different types of impurities will cause a drop in the freezing plateau. For example, gallium impurities in tin will cause a drop in the freezing plateau.

A few of the types of impurities can cause an increase in the plateau. For example, gold impurities in silver will cause the freezing plateau to increase. An extremely high purity metal, 99.9999 % or higher, behaves very closely to an ideal pure metal. Figure 3 shows the difference between a freeze of an ideal pure metal and a high-purity metal. The approximate effect of the impurity on the equilibrium point can be calculated using the first cryoscopic constant. This calculation is discussed in the Guidelines for Realizing the International Temperature Scale of 1990 (ITS-90).

For general uncertainty comparisons, the first cryoscopic constant, the metal purity requirement, and the difference in the liquidus point are outlined in Table 5. In a modern temperature standard laboratory using a SPRT, a temperature change as low as 0.01 mK (0.00001 °C) can be detected. Therefore, the best technique for realizing the freezing point with a real sample is one that measures a temperature nearest to the freezing point of the ideal pure metal. The beginning of the very slow freezing curve of a high purity metal is the closest temperature to the ideal freezing point which can be obtained in a modern temperature standard laboratory. A slow induced freezing technique was found to fit the purpose best (The detail of the technique will be described a little later). A very slow freeze allows enough time to calibrate a number of SPRTs in the beginning part of a single freeze.

Table 5. Summary of the First Cryoscopic Constants and the Estimated Effects of Impurities

Substance	1st Cryoscopic Constant	Impurity Level	Deviation from Pure Liquidus Point
Indium	0.00732/K	99.99999 %	-0.05 mk
Tin	0.00329/K	99.9999 %	-0.3 mK
Zinc	0.00185/K	99.9999 %	-0.5 mK
Aluminum	0.00149/K	99.9999 %	-0.7 mK
Silver	0.000891/K	99.9999 %	-1.1 mK
Gold	0.000831/K	99.9999 %	-1.2 mk
Copper	0.000857/K	99.9999 %	-1.2 mk



gpq003.eps

Figure 3. Freezing Curve Comparison of One Cell

The highest accuracy realizations are greatly facilitated by establishing a second, essentially static, solid-liquid interface immediately surrounding the re-entrant well. The induced technique mentioned above can generate two liquid-solid interfaces in the cell. When the pure metal has been melted thoroughly, maintain the furnace at 2 °C above the freezing point overnight. The next morning, decrease the furnace temperature to about 2 °C below the freezing point at a rate of 0.5 °C per minute. A SPRT inserted into the cell is used to monitor the pure metal temperature. As soon as the recalescence (indicated temperature stops to decrease and starts to rise) occurs, take the SPRT out of the furnace and insert two cold quartz glass rods into the cell in succession, each rod remains in the cell for about two minutes. Set the furnace to a temperature of about 0.5 °C below the freezing point. The cold quartz glass rods absorb heat from the pure metal, and a thin film of pure metal is frozen around the re-entrant well immediately. A continuous liquid-solid interface, as nearly as is practical, encloses the sensor of the SPRT being calibrated. Another liquid-solid interface is formed on the wall of the graphite crucible. In this situation, the outer interface advances slowly as the liquid continues to solidify. Ideally this generates a shell that continues to be of uniform thickness completely surrounding the liquid, which itself surrounds the inner liquid-solid interface that is adjacent to the thermometer well (Figure 4). The inner interface is essentially static except when a specific heat-extraction process takes place (for example, the insertion of a cool replacement thermometer). It is the temperature of the inner liquid-solid interface that is measured by the thermometer. Sometimes the inner liquid-solid interface is called the defining temperature interface.

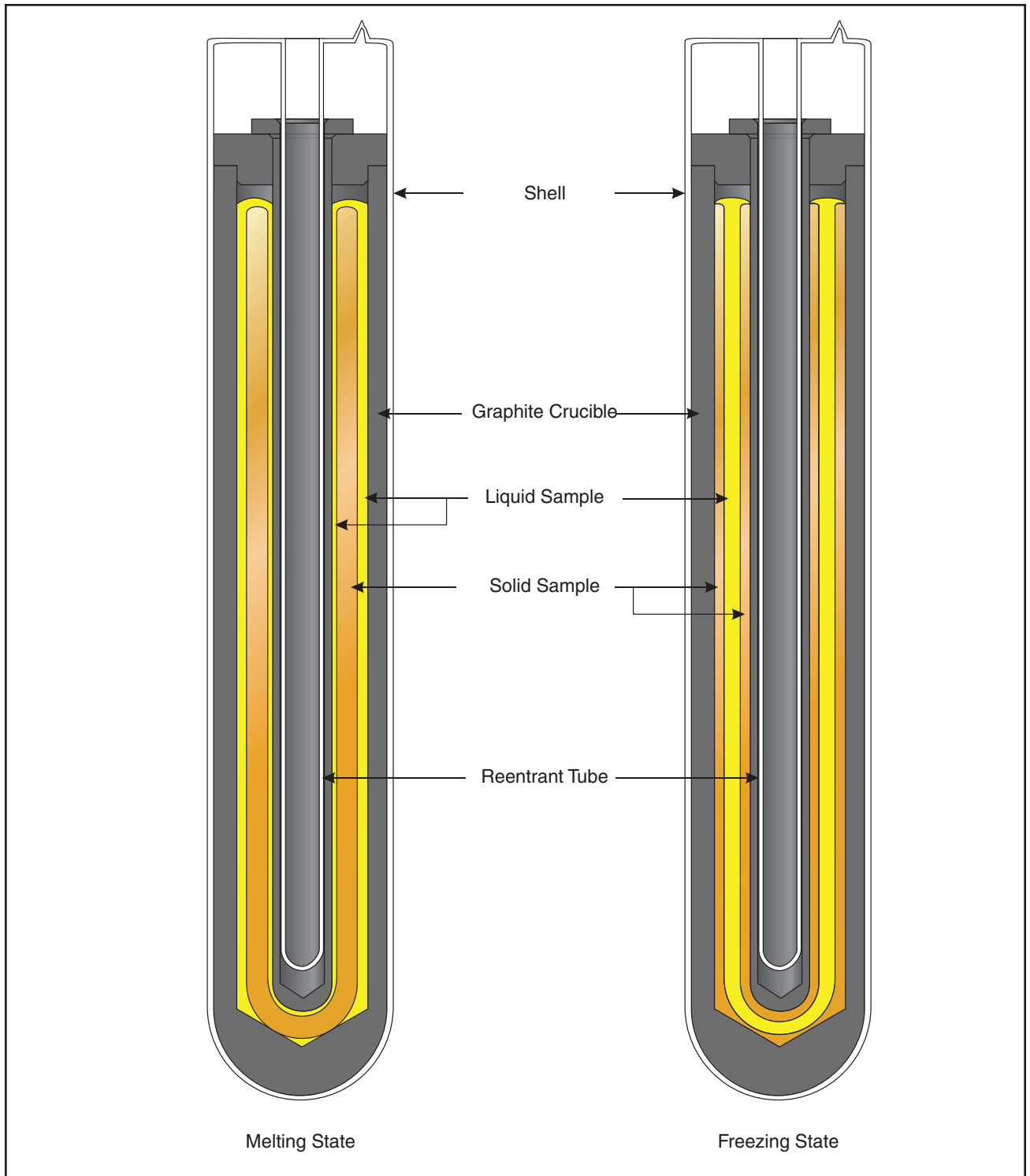
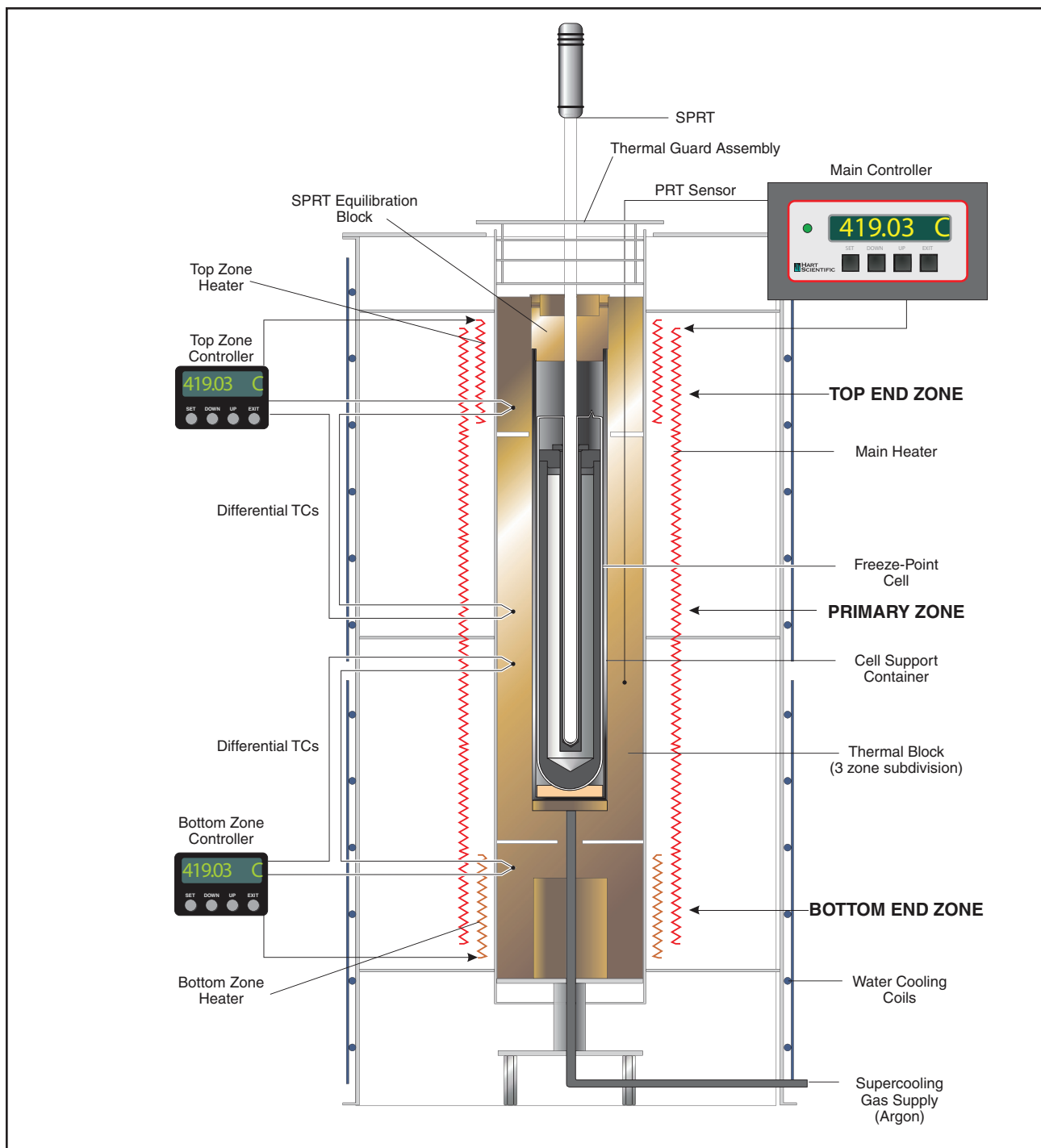


Figure 4. Typical Freeze Point Cell Design Showing the Liquid-solid Interfaces

gpq004.eps

It is extremely important for the process described here that there is a very uniform, stable and controlled temperature environment enclosing the fixed-point cell. We have developed several designs of fixed-point furnaces to satisfy these requirements. The Model 9114 furnace has three independent heaters and controllers designed to be used for a temperature range up to 680 °C as shown in Figure 5.



gpq005.eps

Figure 5. 9114 Furnace Interior with Freeze Point Cell, Cross Sectional View

The 9260 small portable fixed-point furnace is much shorter than traditional furnaces and can easily be used on a table or bench (Figure 6). The furnace has a total height of 489 mm and an outer diameter of 209 mm, and it weighs about 17 kg. The 9260 permits simplified realization of either freezing or melting curves. Probes shorter than 430 mm (17 in) cannot be calibrated in the 9114 furnace. Probes at least 220 mm (9 in) length can be calibrated in the 9260 furnace. Three heaters are used to obtain uniform temperatures around the fixed-point cell. The main heater covers the furnace's entire length, while the top and bottom zone heaters cover only the upper and lower parts of the furnace, respectively. Only one controller is used in 9260. Software within the unit's controller is used to adjust the ratios of the three heaters. Using this technique, we can achieve temperature uniformity of ± 0.1 °C or better within the cell.

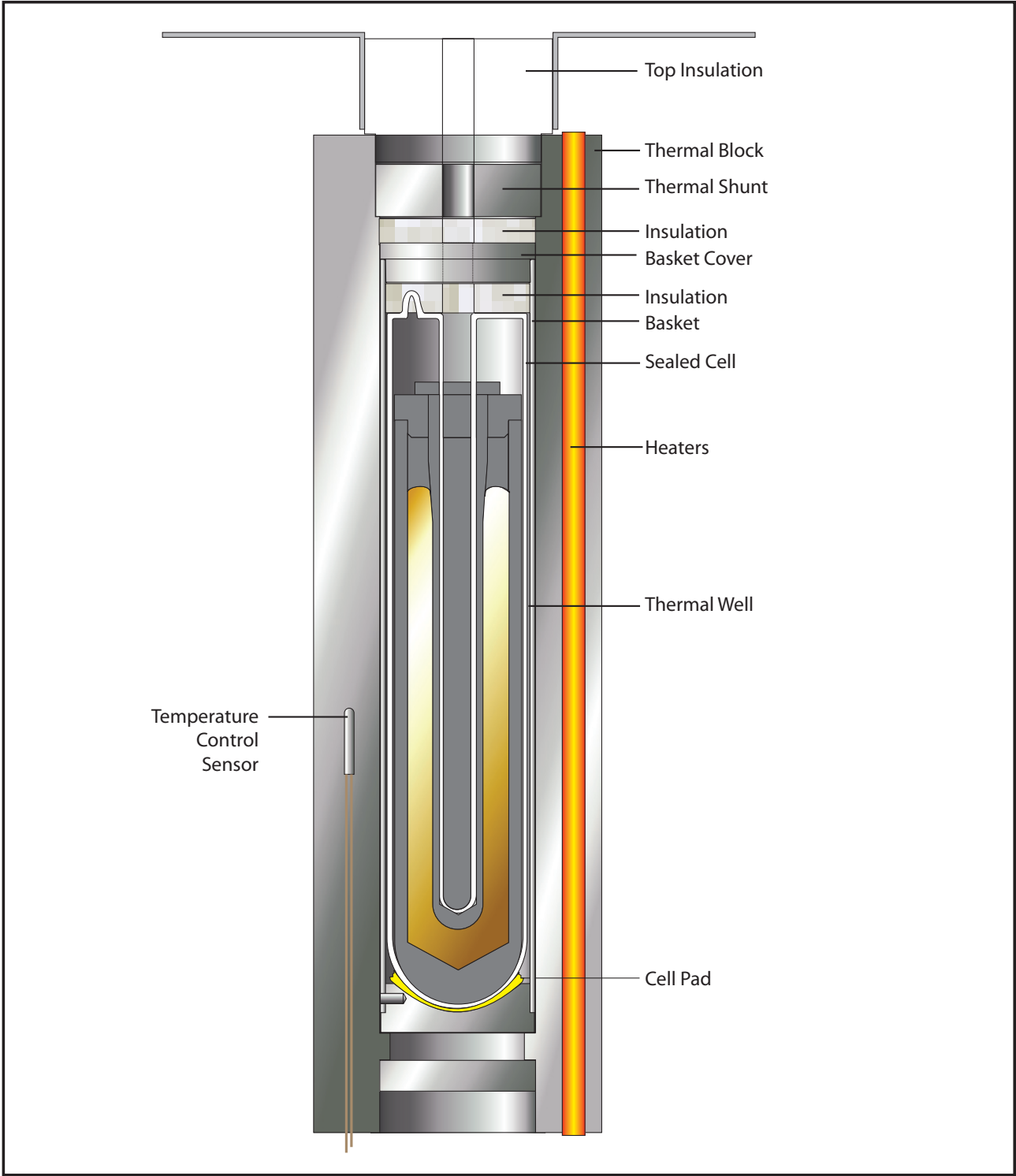


Figure 6. 9260 Furnace Interior with Freeze Point Cell, Cross Sectional View

gpq006.eps

Table 6. The Furnaces for Fixed Points and their Temperature Uniformity

Fixed Point	The Equipment Used	Temperature Uniformity
The freezing point of indium	Model 9114 furnace, three zones	± 0.01 °C
The freezing point of tin	Model 9114 furnace, three zones	± 0.015 °C
The freezing point of zinc	Model 9114 furnace, three zones	± 0.02 °C
The freezing point of indium	Model 9260 furnace, three zones	± 0.05 °C
The freezing point of tin	Model 9260 furnace, three zones	± 0.08 °C
The freezing point of zinc	Model 9260 furnace, three zones	± 0.10 °C

The cell should be put into the cell containment vessel before insertion into any furnace. Ideally each cell would be kept in its own unique vessel. Fiber ceramic insulation is placed in the bottom of the cell basket to protect the cell. Insulation is also placed on top of the cell for protection and to reduce heat loss.

Vertical Temperature Gradient and Controller Accuracy Adjustment

Vertical Temperature Gradient Adjustment

The vertical temperature gradient has to be within the required specification before a fixed point cell sample is melted. Otherwise, the cell may break. Therefore, the vertical temperature gradient should be measured each time the cell is installed in a furnace, and checked at least every six months. The Model 9114 and 9260 Furnaces are three-zone furnaces, and their temperature gradient can be adjusted through the top zone and the bottom zone heater.

Vertical Temperature Gradient Test Method

Set the furnace temperature 5 °C below the melting point of the sample. For example, the melting point temperature of Zinc is 419.527 °C; the furnace temperature should be set to 414.5 °C. Maintain the temperature for at least 4 hours (stabilizing) after the display temperature of the furnace reaches the set point temperature. The SPRT is moved up 120 mm (4.7 in) from the bottom of the thermometer well and then back to the bottom. The SPRT was moved in 40 mm (1.6 in) increments and measurements taken at each depth. After every move and before each measurement, a two-minute stabilization period was allowed. The vertical temperature should meet the specification of the furnaces. Ideally the temperature uniformity of 9114 and 9260 should meet the uniformity listed in Table 6. If the furnace does not meet the requirement, the vertical temperature gradient should be adjusted through the top and bottom heater.

Controller Display Accuracy Adjustment

The absolute accuracy of the instrument might change after adjusting the temperature gradient, therefore it is important to check it after performing the vertical gradient adjustment. The offset in accuracy should be considered or corrected during the realization of fixed points. The controller accuracy should be checked after the sample is fully melted in every realization of a fixed point. Please refer to the respective furnace Users Guide for a calibration procedure. A calibrated SPRT should be used to measure the controller accuracy.

Procedure for Realizing the Freeze

All of the freezing points of metal-cased cells are realized manually in a similar way.

1. Insert the cell with the cell containment vessel carefully into the furnace.
2. Place as much thermal insulation material as possible onto the top of the fixed-point cell (in the cell containment vessel and on the top of the vessel).
3. Set the temperature of the furnace about 5 °C higher than the freezing point. Allow all of the metal to melt completely.
4. After all metal is completely melted, the furnace is set at a stable temperature about 2 °C above the freezing point overnight.

5. The next morning, the furnace temperature is decreased slowly (about 0.1 °C per minute). In order to monitor the pure metal sample temperature, a SPRT is inserted into the cell. The temperature of the metal sample decreases to less than the freezing point before recalescence. The amounts of supercool are different from metal-to-metal.
6. After recalescence remove the thermometer from the furnace immediately and insert two cold (room temperature) quartz rods or tubes into the fixed point cell one by one, keep each rod in the cell for two minutes.
7. Set the furnace at a stable temperature of 0.5 °C below the freezing point.
8. Insert the preheated SPRT to be calibrated into the cell. Preheat the SPRTs to be calibrated at a temperature of about 2 °C above the freezing point for thirty minutes before inserting them into the cell.
9. It will take 30 to 40 minutes to get equilibrium between the SPRT sensor and pure metal sample. After equilibrium reached you can start measurements.

This procedure provides a very stable, long freezing plateau that typically lasts for more than twenty hours. The changes in temperature in the first half of the plateau are usually within ± 0.2 mK to ± 0.3 mK. A typical freezing curve is shown in Figure 7.

Many SPRTs can be calibrated in a single freezing plateau. When multiple SPRTs are to be calibrated from a single freeze, preheat of SPRTs to be calibrated is very important.

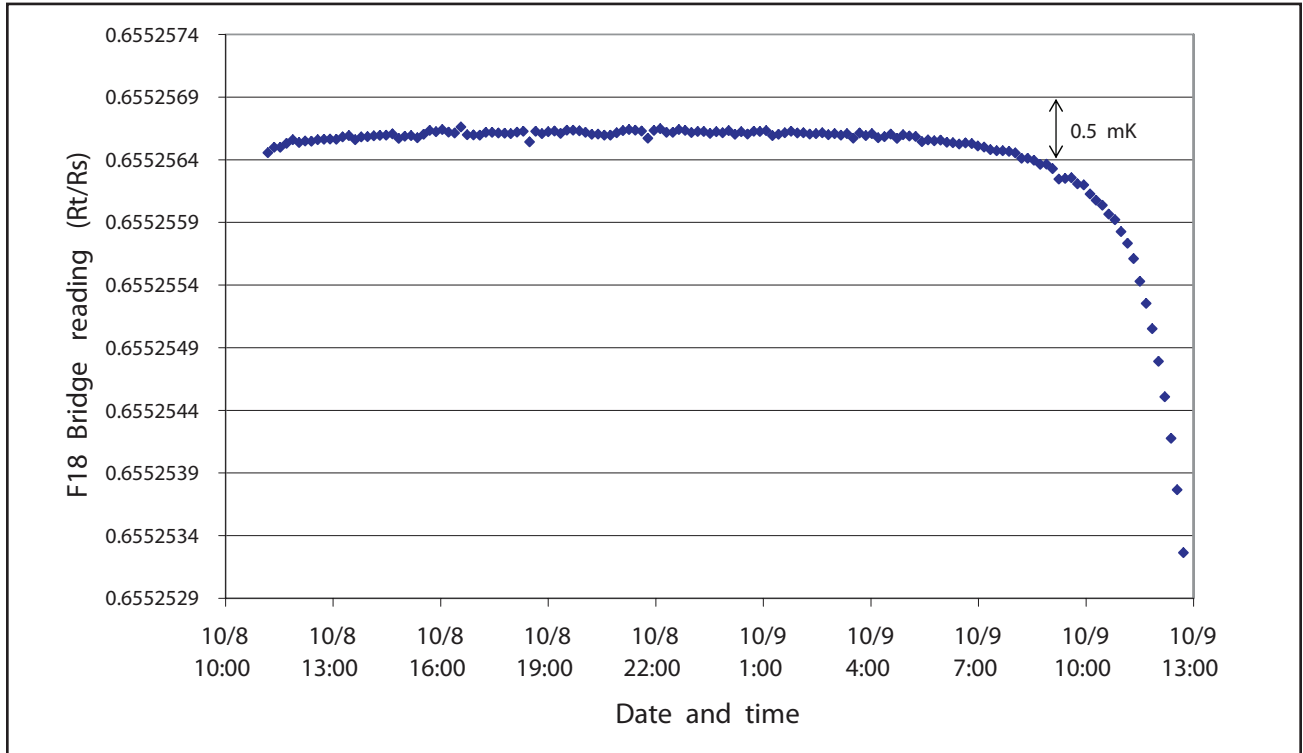


Figure 7. A Typical Freezing Curve for the Zinc Cell

gpq007.eps

Procedure for Realizing the Melting Point

The melting point of metal-cased cells are realized manually in a similar way.

Note

In order to optimize the Melting Point Realization, it is recommended to preheat all probes before inserting them into the metal-cased cell. Probes can be preheated in a preheat well (not available on all furnaces) or annealing furnace operating at the same temperature of the metal-cased cell.

Note

For new setup or installation, it is recommended that the melt plateau be monitored with an SPRT and recorded throughout the entire plateau. Doing so establishes the length of the plateau.

1. Set the temperature of the furnace about 5 °C below the freezing point.
2. After the furnace temperature reaches the setting temperature, wait for 30 minutes for stabilization and equilibration.
3. Set the temperature of the furnace 0.8 °C higher than the freezing point with a scan rate of 1.5 °C per minute.
4. After the furnace temperature reaches the set temperature, insert the check standard SPRT (if available) into a pre-heat well or annealing furnace (see note above).
5. Wait for 30 minutes for the fixed-point cell to equilibrate and transition into the melt plateau.
6. Insert the check standard SPRT into the cell.
7. It will take 30 to 40 minutes to achieve equilibrium between the SPRT sensor and the pure metal sample. After equilibrium is reached, measurements can be made.
8. It is recommended to measure the check standard again to end the plateau.

The Correction for the Pressure Difference

This is the procedure used in the Fluke lab with the Fluke Sealed Fixed-point Cells. Other procedures are sometimes employed in industry. Except for a few triple points, the values of temperature assigned to the defining fixed points by ITS-90 correspond to the temperatures at the standard atmospheric pressure 101.325 kPa. The actual pressure in a cell may be not exactly the standard value. During the course of manufacture of a fixed-point cell, it is easier to seal the cell if the pressure in the cell is a slightly lower than the room pressure. The actual pressure in the cell exactly at the fixed point was measured at Fluke. This actual argon pressure in the cell at the freezing point is provided on the Report of Test, or Certification, enabling calculation of correction for the difference in pressure. During measurement at a fixed point, the sensor of a SPRT is usually placed at a height which is “h” meters lower than the upper surface of the pure metal and where the pressure is higher than that at the surface due to the static head. ITS-90 gives all of the necessary coefficients for the calculation of the correction caused by the pressure difference, which are summarized in Table 7.

Table 7. Coefficients for the Pressure Difference of Some Defining Fixed Points

Substance	Assigned Value of Equilibrium Temperature T Kelvin (K)	Temperature with Pressure, p K1; dT/dp (10 ⁻⁵ mK/Pa)	Variation with Depth K2 : dT/dh (mK/m)	Approximate dW/dt
Argon (T)	83.8058	25	3.3	0.004342
Mercury (T)	234.3156	5.4	7.1	0.004037
Water (T)	273.16	-7.5	-7.3	0.003989
Gallium (M)	302.9146	-2.0	-1.2	0.003952
Indium (F)	429.7485	4.9	3.3	0.003801
Tin (F)	505.078	3.3	2.2	0.003713
Zinc (F)	692.677	4.3	2.7	0.003495
Aluminum (F)	933.473	7.0	1.6	0.003205
Silver (F)	1234.93	6.0	5.4	0.002841
Gold (F)	1337.33	6.1	10	—
Copper (F)	1357.77	3.3	2.6	—
(T) – Triple Point (M) – Melting Point (F) – Freezing Point				

The correction of temperature caused by the difference in pressure can be calculated by using the following equation:

Equation 1: Pressure Dependent Temperature Correction

$$\Delta t = (P - P_0) \times k_1 + h \times k_2$$

P: the actual pressure of argon in the cell exactly at the fixed point temperature

P₀: the standard atmospheric pressure. For example, 101.325 kPa

$$k_1 = \frac{dT}{dP}$$

$$k_2 = \frac{dT}{dh}$$

h: the immersion depth of the midpoint of the sensor of a SPRT into the metal used for the fixed point

The immersion depth of the midpoint of a SPRT sensor in a Fluke 5944/5945/5946/5947 Fixed-point Cell is approximately 131 mm (the distance from the bottom of the central well to the

surface of liquid metal is about 0.156 m). The actual pressure of the argon at the freezing point in the cell, p , is provided in the Report of Test or Certification. The temperature correction, Δt , can be calculated using Equation 1.

Example:

The pressure of argon at the freezing point in the zinc freezing point cell S/N Zn-45001 is 86,100 Pa (86.10 kPa) as given in the Report of Test. k_1 and k_2 for the freezing point of zinc can be found in Table 7, $k_1 = 4.3 \times 10^{-5} \text{ mK / Pa}$ and $k_2 = 2.7 \text{ mK / m}$. The average immersion depth is 131 mm for most of standard platinum resistance thermometers. Therefore, use Equation 1 to calculate Δt . Substituting values into Equation 1:

$$(86.100 \text{ kPa} - 101.325 \text{ kPa}) \frac{4.3 \times 10^{-5} \text{ mK}}{\text{Pa}} + 0.131 \text{ m} \frac{2.7 \text{ mK}}{\text{m}} = -0.65 \text{ mK} + 0.3 \text{ mK}$$

Consequently:

$$\Delta t = -0.301 \text{ mK}$$

Hence, the actual temperature of a sensor of a SPRT at the point of total immersion during a freezing plateau in the cell is calculated using Equation 2.

Equation 2: Calculation of the Actual Temperature, t_1

$$t_1 = t + \Delta t$$

Therefore:

$$t_1 = 419.527 \text{ }^\circ\text{C} - 0.000301 \text{ }^\circ\text{C} = 419.5267 \text{ }^\circ\text{C}$$

where t is the defining fixed point temperature, (for example, 419.527 °C for the freezing point of zinc).

The resistance ratio, W_{Zn} , for the particular cell exactly at the freezing point of zinc can be calculated using the following equation. The value for dW/dt is taken from Table 7.

Equation : Calculation of W_{Zn} for the exact defining fixed point temperature.

$$W_{Zn} = W(t_1) - [\Delta t] \frac{dW}{dt}$$

Substituting values:

$$2.568917300 - (-3.01 \times 10^{-3}) \times (3.495 \times 10^{-3})$$

Thus the W_{Zn} for the cell is:

$$W_{Zn} = 2.56891835$$