Calibration

5943

Gallium Melting Point Cell

Users Guide

2004, Rev. 1, 5/11

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Introduction

The melting point of gallium (MPGa) at 29.7646 °C is one of the defining fixed points of the International Temperature Scale of 1990 (ITS-90). The MPGa is the phase equilibrium between the liquid phase and solid phase of pure gallium at a pressure of one standard atmospheric pressure (that is, 101.325 Pa). The temperature of the MPGa is defined to be 29.7646 °C by the ITS-90 making it an intrinsic temperature standard. The MPGa is highly reproducible. You can get almost the same temperature wherever and whenever you realize the MPGa. The differences among the different realizations might be well within a few tenths of a millikelvin if you carefully follow the instructions given in Supplementary Information for the ITS-90, Comite Consultatif de Thermometrie (CCT), and Bureau International des Poids et Mesures (BIPM) 1990.

For your convenience Fluke has developed a series of sealed cells and new techniques for the MPGa, which make it easier to realize the MPGa. The Model 5943 Stainless Steel-cased MPGa Cell is a new member of the Fluke MPGa sealed cells series. The Model 5943 reserves all of the benefits of the Model 5903, including extremely low uncertainty. Stainless steel is used as case material in the Model 5943 instead of the Pyrex glass used in the Model 5903. Therefore, the Model 5943 is much more durable compared with the Model 5903. The Fluke sealed cells will assist you immensely in realizing the MPGa in your laboratory.

The MPGa is an important fixed point for the calibration of a standard platinum resistance thermometer (SPRT). The ITS-90 specifies two subranges (for example, from 0 °C to 29.7646 °C and from -38.8344 °C to 29.7646 °C, in which SPRTs must be calibrated at the MPGa). Checking the stability of a temperature probe used mainly near room temperature at the MPGa is very convenient. There is widespread application for the melting point of gallium in biologic, environmental, oceanographic, geological, and energy research.

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 1. 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	N10140	Conforms to Relevant Australian EMC Requirements.
	DC	C US	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.**
- **Gallium is caustic. Consult the metal manufacturer’s MSDS (Material Safety Data Sheet).**
- **Federal law prohibits carrying this cell in the passenger section of a commercial airline. See MSDS in the back of this manual.**

⚠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

To contact Fluke, 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 2 below for the Model 5943 Gallium Melting Point Cell specifications.

Table 2. 5943 Specifications

Item	Range
Reproducibility	0.05 mK (0.00005 °C)
Cell Uncertainty (k = 2)	0.08 mK to 0.15 mK (0.00008 °C to 0.000015 °C)
Certification (k = 2)	0.1 mK
Purity of Gallium	99.99999 %
Cell Outer Diameter	38.1 mm
Cell Overall Height	250.1 mm
Entrant Well Inner Diameter	8.2 mm
Immersion Depth [1]	168.0 mm
[1] Depth is measured from the bottom of the entrant well to the upper surface of the gallium.	

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. The instrument operates safely under the following conditions:

- Temperature range: 5 °C to 35 °C (41 °F to 95 °F)
- Ambient relative humidity: 15 % to 50 %
- Pressure: 75k Pa to 106 kPa
- Mains voltage within ±10 % of nominal
- Altitudes less than 2,000 meters

Note

Vibrations in the calibration environment should be minimized.

Construction

The Model 5943 Gallium Melting Point Cell consists of a stainless steel outer shell, a stainless steel cap with an entrant well, a Teflon® cell body containing the high-purity gallium and a nylon lid with nylon entrant well (see Figure 1). Gallium expands on freezing by 3.1 % requiring the cell to have flexible walls. Teflon® is a suitable material from this point of view, but Teflon® is gas-permeable over time. Over a four-year period, completely sealed Teflon® cells drift about 0.5 mK under normal use. Therefore, a stainless steel outer case is used to keep gas tight in the cell.

All of the parts and material used to construct the cell were carefully cleaned to avoid any contamination to the high purity gallium. The high purity gallium was first sealed into the Teflon-nylon body in a pure argon (99.999 %) atmosphere. The pressure of argon in the body was adjusted to approximate 101,325 kPa at the melting temperature. The actual value of this pressure was recorded and is reported on the Report of Test for the cell. The correction for pressure difference from the standard value of 101,325 kPa is calculated according to the actual pressure in the cell (see the section “Correction for Pressure Difference”). The assembled Teflon®-nylon body was then encased into the stainless steel outer shell. The stainless steel top with the entrant well was joined to the stainless steel outer case using argon-arc welding. The assembled cell was then connected to a vacuum system and pumped for more than ten hours. During the pumping period, the cell was purged with high purity argon many times. Finally, the cell was filled with 99.999 % pure argon and sealed at almost the same pressure as that in the Teflon®-nylon body.

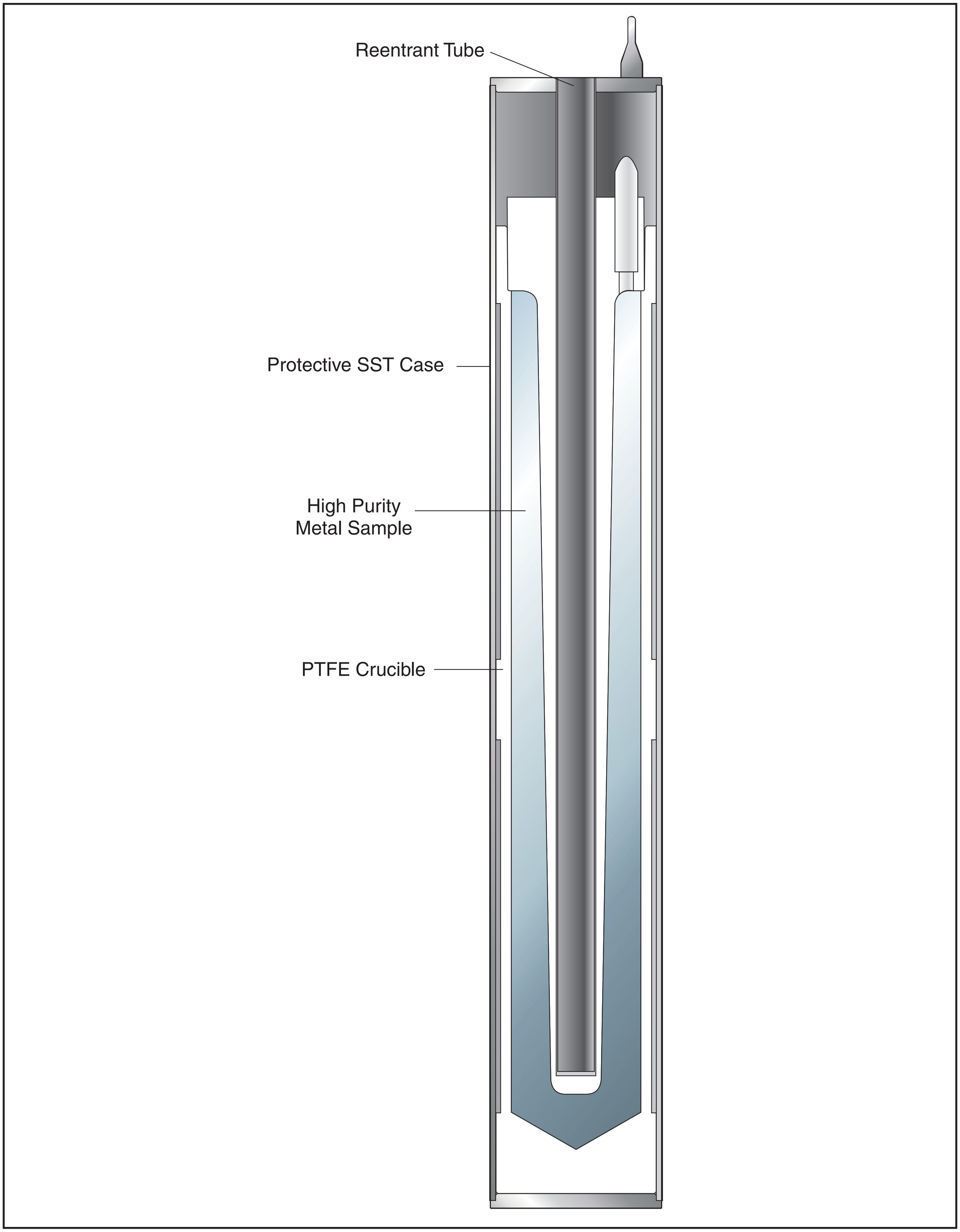

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Figure 1. Construction of the Gallium Melting Point Cell

Care and Handling Guidelines

The Model 5943 Gallium Melting Point Cell is a delicate device. Care must be taken in handling, using, and transporting the cell. Keep the gallium in the cell in a solid state whenever possible.

⚠Caution

Use and store the product in the vertical orientation only.

The surrounding temperature should be at least 4 °C below the melting point (for example, 25 °C or lower).

Special care must be taken during transportation of the cell:

- Keep the gallium in the solid state.
- Store the cell in a vertical position.
- Keep the surrounding temperature lower than 25 °C.

If the instructions in this manual are carefully followed, the Model 5943 Gallium Melting Point Cell will provide years of accurate use.

Operation

Freezing of Gallium

The three methods for freezing the gallium cell, slow freeze, fast freeze, and automated freeze, are described in the following sections.

Slow Freeze Method

In order to freeze all the gallium in the cell, immerse the cell in an ice bath (or a bath, with a temperature close to the ice point). First, immerse only ¼ of the cell in the bath, 30 minutes later immerse ½ of the cell in the bath and another 30 minutes later immerse the ¾ of the cell in the bath. Because of the large super cool of gallium, even though the temperature of the gallium in the cell declines to a temperature near 0 °C, all gallium might still remain in the liquid state for a period of time. Therefore, monitor the temperature in the cell by using an SPRT. When the cell is placed in an ice bath, the temperature indicated by an SPRT in the cell decreases fast at first and then decreases slowly, at last the temperature stops decreasing. After a period of time the temperature raises, a signal that the gallium starts freezing. Finally, the temperature decreases again and all of the gallium is frozen. Once frozen, the cell may be transferred to a maintenance bath such as the Fluke Model 7011 (see Figure 2).

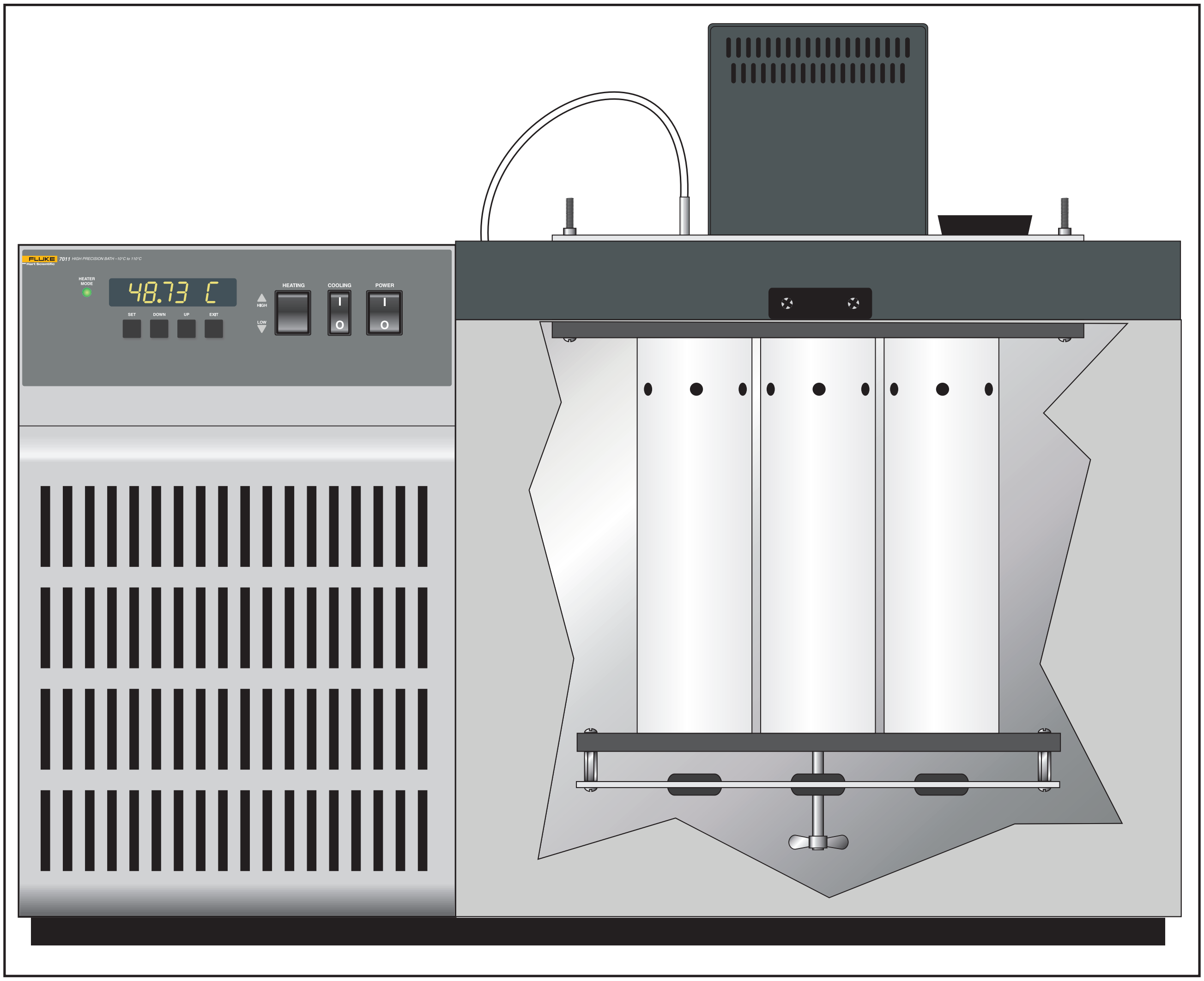

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Figure 2. Maintenance Bath Model 7011 with Gallium Cell Insert

Fast Freeze Method

The fast freeze method is based on the National Institute of Standards and Technology (NIST) Technical Note 1265. Start with the fixed point cell at room temperature. Use liquid nitrogen to super cool two or three solid copper rods for insertion in the entrant well. The copper rods should be only slightly smaller than the inside diameter of the entrant well. For example, the Fluke Model 5943 Gallium Cell has an entrant well with an inside diameter of 8.2 mm. The solid copper rods should have a diameter of approximately 7.0 mm. Insert the two or three super cooled copper rods into the entrant well successively to induce nucleation. As soon as the rods have been used to induce nucleation, place the cell in an ice bath for at least one hour or longer. Monitor the freeze with an SPRT. Once the freeze has been achieved, place the cell in a maintenance bath such as the Fluke Model 7011 (see Figure 2).

Automated Freeze Method

The Fluke Model 9230 Maintenance System is specifically for the Model 5943 Gallium Cell. The Model 9230 has a fully automated system for maintenance of the Model 5943. Refer to the Model 9230 Users Guide for complete information on how to use the maintenance device with the Model 5943 Gallium Cell.

Melting Plateau

Realizing the melting point can be performed in a bath or in the Model 9230 Maintenance System as described below.

Realizing the Melting Point in a Bath

A stable and long (a few days) melting plateau, during which a great amount of thermometers can be calibrated, is easy to obtain if you follow the instructions described below.

Put the cell into the maintenance bath at a temperature of 0.5 °C below the melting point. Fluke manufactures a Model 7011 bath with a special cell support and SPRT preheat holder for the purpose of maintaining the gallium cell (see Figure 2). Use silicone oil, 200.05 or 200.10, as the bath fluid. Light mineral oil, Fluke Model 5011 oil, can be used instead of silicon oil. Use the same oil in the entrant well to provide adequate heat transfer between the cell and the thermometer. The oil level in the well should be approximately the same level (52 mm below the top of the cell) as that of the gallium in the cell when the thermometer is in place in the well.

With some care, distilled water can be used instead of oil as the bath fluid and heat transfer medium. Distilled water is cleaner and the viscosity of water is much lower than that of oil. The water must be kept as clean as possible. Long-term direct contact between stainless steel and dirty water causes the cell case to rust. The user should take the cell out of the bath and wipe the cell surface dry using clean tissue. When the cell is not used for a period of time, store the cell in the vertical position and in a clean place. The surrounding temperature should be below 25 °C. Before freezing the cell, remove all of the water from the entrant well using an injector or other tool. Never tilt the cell if you are not sure all of the gallium is frozen. If the temperature of the cell goes below 0 °C during freezing, the water in the entrant well freezes and might break the entrant well.

The procedure for realization of a melting curve in a bath is as follows. Make sure that all gallium is frozen before starting. Maintain the bath at a temperature of 0.5 °C below the melting point for at least 30 minutes. Raise the temperature to 1 °C above the melting point at a rate of 0.2 °C per minute. Maintain the bath at this temperature for 20 minutes. Then insert an 8 watt heater into the entrant well for four minutes. After the four minutes, decrease the bath to a temperature of 0.01 °C to 0.1 °C above the melting point. Take the heater out of the entrant well and insert a preheated thermometer into the well. When the thermal equilibrium between the thermometer and pure gallium is reached (approximately 20 minutes), start the measurements. See Figure 3 for the idealized liquid/solid equilibrium state of a gallium cell at the start of the melt. A typical melting curve is shown in Figure 4. If the bath temperature is set at 0.1 °C above the melting point, the melting curve will last for more than ten days. If the bath temperature is set at 0.01 °C above the melting point, the melting curve will last for more than two months.

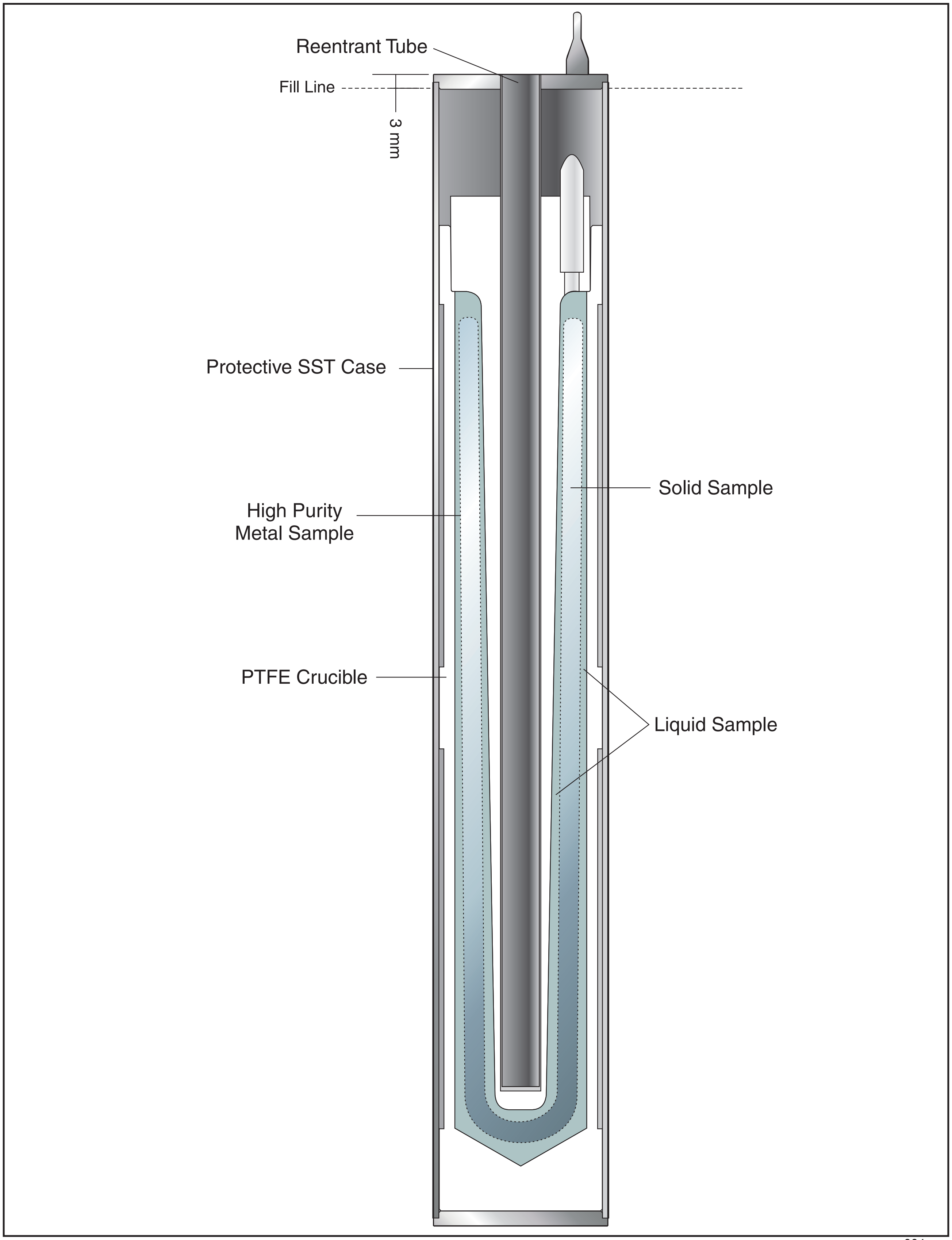

Figure 3. Idealized Liquid/Solid Equilibrium State of a Gallium Cell

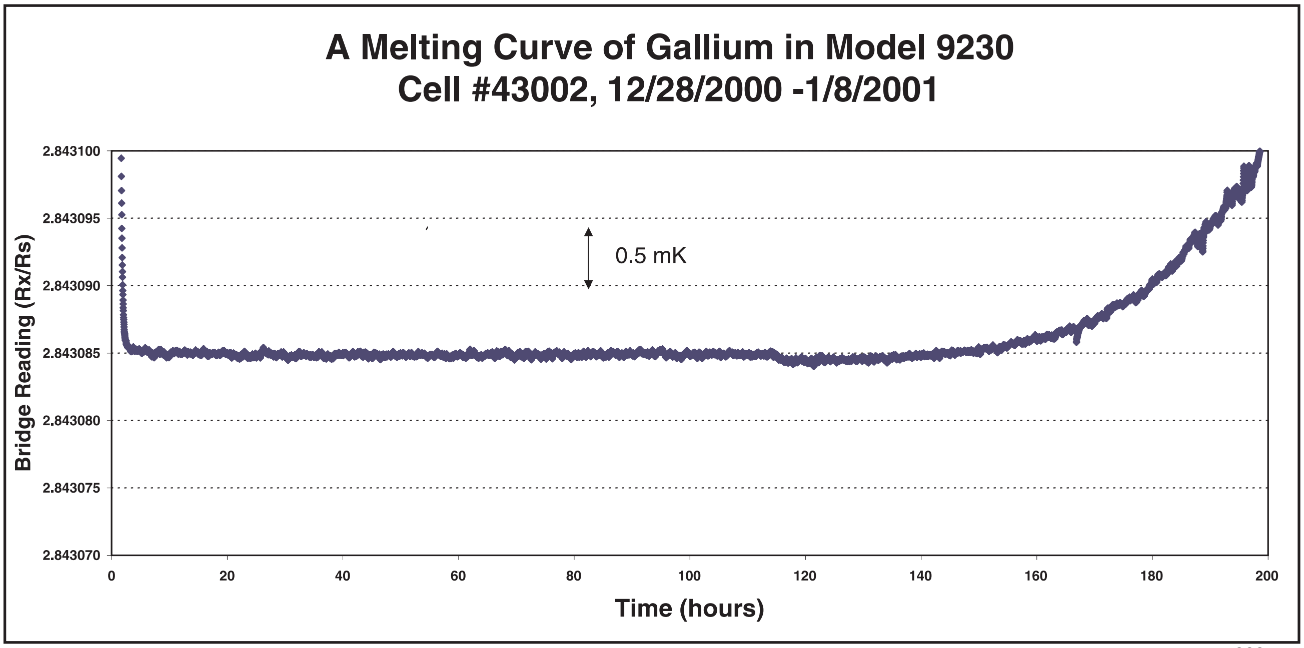

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Figure 4. Typical Melting Curve of Gallium

Realizing the Melting Point in the 9230 Maintenance System

The user should be familiar with the following details before using the Model 9230 Maintenance System. The Model 9230 is designed to automate the gallium cell melting process.

This melting process is accomplished through the use of the Auto Program, a microprocessor-controlled program that determines set temperatures, scan rates, duration times, Peltier module configurations, and melt heater controls. By controlling these parameters in the proper order, a temperature profile can be maintained in the block assembly that assures a uniform melt, maintains the melt over long periods of time, properly refreezes the cell and keeps the cell ready for the next melt cycle. The only attempt in describing the melting process in this manual is to provide a simple overview. For complete details on realizing the melting point in the 9230 Maintenance System see the Model 9230 Users Guide. In the Auto Program, the Model 9230 stabilizes at 29.27 °C to allow the gallium cell to equalize. After equalization, the program initiates the melt. The block temperature is set to initiate a melt on the outer surfaces of gallium within the cell. At the appropriate time the unit beeps and the display flashes “Htr on” to indicate that the inner melt heater has been turned ON. This allows the user to place the inner melt heater in the entrant well of the cell to initiate the inner melt. Once the inner melt is initiated, the heater is removed from the cell. The unit automatically de-energizes the heater at the appropriate time. After the outer melt of the cell has been appropriately initiated, the unit changes into the maintenance mode and the block temperature resets to 29.86 °C. After 30 to 60 minutes of stabilization time, the unit is ready for use. The duration of the melt plateau varies depending on usage, but lengths of five days or longer is achievable.

The Correction for the Pressure Difference

The melting point of gallium, as mentioned earlier, is defined as the phase equilibrium between the liquid phase and solid phase of pure gallium at a pressure of 101,325 Pa (P_0). The actual pressure, P, in a gallium cell may not be exactly the standard value. During the course of manufacturing the fixed-point cell, it is easier to seal the cell if the pressure in the cell is a little lower than room pressure. The actual pressure value in the cell just at the melting point is measured at Fluke so that the correction for the difference of the pressure can be made later Table 2. of the ITS-90 gives the data of dT/dP for all the defining fixed points. For the melting point of gallium dT/dP = -2.0 x 10^{-8} °C/Pa. Therefore, the actual equilibrium temperature t' can be calculated using the following equation.

Actual Equilibrium Temperature Equation

$$t' = 29.7646°\text{C} - \left[\frac{2.0 \times 10^{-8} \left(P - P_0 \right) °\text{C}}{Pa} \right]$$

Furthermore, during the measurement at the melting point, the sensor of an SPRT is usually placed at an average height, which is 0.143 m (0.168 m to 0.025 m) lower than the surface of the gallium and where the pressure is higher than that at the surface due to the static head. The correction, calculated according to the data given in Table 2 of the ITS-90, is -0.000172 °C. The equilibrium temperature at an immersion depth of 0.143 m can be calculated using the following equation.

Pressure Correction Equation

$$t' = 29.7646°\text{C} - \left[\frac{0.000172°\text{C} - 2.0 \times 10^{-8} \left(P - P_0 \right) °\text{C}}{Pa} \right]$$

Pressure Correction Example

The pressure of argon, P, at the melting point in the gallium point cell is 91,325 Pa as given on the calibration certificate.

$$t' = 29.7646°\text{C} - \left[\frac{0.000172°\text{C} - 2.0 \times 10^{-8} \left(-10000 Pa \right) °\text{C}}{Pa} \right]$$

$t' = 29.7646\ °\text{C} - 0.000172\ °\text{C}$

Thus:

$t' = 29.764428\ °\text{C}$