Model K28C

Gallium Melting Point Cell
Operation and Maintenance Manual
Serial Number _____

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1. BACKGROUND	3
2. GENERAL INFORMATION AND OPERATING PROCEDURES .	3
3. TEMPERATURE CORRECTION FOR HYDROSTATIC HEAD	4
4. CELL GEOMETRY AND CONSTRUCTION	4
5. HANDLING AND MAINTENANCE	4
6. DIAGRAM OF CELL AND COMPONENTS	5
6.1 Diagram of Cell Handle	5



Gallium Melting Point Cell

1. Background

This report documents the fabrication, initial testing as well as the operating and maintenance procedures for Gallium Melting Point Cell. Information contained in this manual is considered by Pond Engineering Laboratories to be proprietary and is provided for use exclusively by the purchaser for instructional and maintenance purposes relative to the hardware delivered, any other use is prohibited.

2. General Information and Operating Procedures

On the ITS-90, the melting point (equilibrium state of solid and liquid phases at one standard atmosphere pressure) of gallium is assigned the value 302.9146K (29.7646 degrees C). Given that the first cryoscopic constant (as stated in NIST technical note 1265) of gallium is 0.00732/K, the liquidus point of an ideal gallium solution of 99.999 99% purity would be approximately 0.01 mK lower than that of 100% pure gallium.

The cell should be utilized in the melting mode because gallium may supercool as much as 25 to 75 degrees C. It is important to note that gallium expands approximately 3% in volume as it freezes and therefore care must be exercised to ensure that the freezing of the metal sample start at the bottom of the cell and progress toward the top of the cell. This precaution is necessary to minimize the possibility of bridging over the top of the cell with frozen metal sample and generating substantial hydrostatic pressures within the plastic crucible that could rupture the crucible. Pond Engineering Gallium Melting Point Cell Maintenance System, model K28M, provides positive cooling of the cell base using a thermoelectric cooling module as a means to accomplish this. However, any means of freezing the gallium cell melt is suitable provided measures are taken to be certain the cell is frozen from the bottom up.

Preparation of a completely frozen cell for measurements may be accomplished by placing the cell in a close fitting, air filled aluminum or copper block maintained at a temperature slightly below the melting point (approximately 29.5 degrees C) until the cell comes into thermal equilibrium within 0.1 degrees C of the block. After the cell has been warmed to a temperature slightly below the melting point, the block should be heated to a temperature slightly above the melting point (approximately 30.1 degrees C) and maintained for the duration of the melt. Measurements may be taken after approximately 20 minutes to 1 hour, after the cell temperature is observed to move onto the plateau, to ensure formation of an outer shell of liquid metal adjacent to the crucible wall. Pond Engineering Gallium Melting Point Cell Maintenance System, model K28M, provides a convenient means of controlling the cell environment for realization of the plateau, however, any means of maintaining the gallium cell melt is suitable provided measures are taken to protect the cell from excessively high temperatures (>100° C).

In order to realize the most accurate readings possible, an inner melt surrounding the thermometer well should be formed by inserting an electric heater probe (optional on the K28M system) into the well. Supplying sufficient electrical energy to form a shell surrounding the thermometer well (approximately 1 mm thick) is also essential. Pre heated rods or warm mineral oil (75 degrees C) may also be used to accomplish formation of the inner melt provided precautions are taken to protect the cell from excessively high temperatures (>100 C).

Following preparation of a cell for measurements, it is recommended that a pre heated monitoring thermometer be inserted into the central well to maintain the inner melt adjacent to the thermometer well. All subsequent test thermometers should be preheated before insertion into the cell to maintain the inner melt.



The user is referred to NIST technical note 1265, page 49 for additional background and detail on the realization and application.

3. Temperature Correction for Hydrostatic Head

Melting point conditions specified in ITS-90 are at one standard atmosphere pressure. Since this condition is maintained inside the cell only at the gas/ liquid interface, that occurs at the upper surface of the metal sample, a hydrostatic head correction must be applied as a function of the thermometer immersion depth. For gallium, the pressure effect on the melting point temperature is approximately -2.0 mK per atmosphere or -1.2 mK per meter of liquid metal (NIST technical note 1265, page 25).

Taking the sensing point of an SPRT as being approximately 2.5 cm above the tip of the thermometer, with the thermometer fully inserted into the central well, the hydrostatic head imposed by the liquid metal is approximately 19 cm. This results in a correction of -.23 mK which must be applied to the theoretical freezing point temperature to account for the static head pressure present at the sensing point of the thermometer. For all practical purposes, this correction results in a decrease in the temperature of 0.0002 degrees C and variability between cells is negligible.

4. Cell Geometry and Construction

These cells are constructed using a machined plastic crucible with a tight fitting lid and thermometer well and contain approximately 700 grams of high purity gallium metal with an initial purity of 99.999 99% or better. The crucible is surrounded by a rugged type 304 stainless steel outer envelope containing high purity argon gas. Cell envelope outside dimensions are 1.480 inches outside diameter by approximately 11.5 inches overall length. The thermometer well consists of .375" OD tubing with .020" wall and extends upward from the top of the envelope approximately 2.75 inches. During fabrication, each cell envelope is leak checked with a Helium Mass Spectrometer leak detector to verify leak rates less than 1X10E-9 standard cc/second air equivalent at high vacuum and back purged with high purity argon and sealed at a pressure of 1 standard atmosphere with the cell at the melting point temperature.

Attached to the end of this document is a copy of the material purity certificate for the metal sample used in fabrication of this cell.

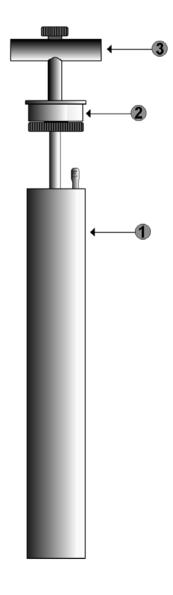
5. Handling and Maintenance

As previously mentioned, the fixed point cells are completely sealed and require no periodic maintenance. Handling precautions should be observed to generally store the cells in a vertical orientation.



6. Diagram of Cell and Components

REF.#	NAME	MAKE	MODEL
1	Gallium Melting Point Cell	Pond Engineering Labs.	K28C
2	Gallium Cell Top	Pond Engineering Labs.	K28C-TOP
3	Cell Handle	Pond Engineering Labs.	K18C-HND



6.1 Diagram of Cell Handle

REF.#	NAME	MAKE	MODEL
1	Tightening Knob	Pond	
		Engineering	
		Labs.	
2	Ferrule	Pond	
		Engineering	
		Labs.	

