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An Optimal Gallium Cell – Interoperability Between A Cell Made In England And Intercompared At NIST, To It's Reference Cell

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Abstract

An Open Gallium Cell of a design dating from 1973 and purity 99.999999% and made in England has been intercompared with NIST's Reference Gallium Cell.

After fully correcting for hydrostatic head the two cells were within 2µK of each other ±20µK. The intercomparison illustrates the International Operability of the Gallium Point and has subsequently reduced the UKAS uncertainties of the Gallium Point at the Northern Temperature Primary Laboratory (NTPL) from 0.45mK to 0.1mK.

Introduction

The Gallium Melting Point was developed and evaluated during the 1970's [1] and in 1990 became a new point on the International Temperature Scale (ITS-90).

Gallium is a metal used extensively in the semi conductor industry and is readily available in very high purity. Its melt temperature is 29.7646°C making it the most useful fixed point because it is close to room and blood temperatures.

Its operation is very simple requiring only body warmth to melt the metal and a refrigerator to freeze it.

Gallium expands 3% as it freezes and causes strain in the cell, thus Gallium is the only ITS-90 point which is a melt rather than a freeze point.

Other relevant properties are that the temperature decreases 2mK per Bar increase in pressure and decreases 1.2mK per metre beneath the Gallium's surface.

Gallium Purity

The Gallium Melt Point Cell takes a number of physical forms all of which have some flexibility to permit the expansion of the Gallium during freezing.

The material in contact with the Gallium is either Teflon or Nylon, neither of which will react chemically with the Gallium.

A Gallium Cell of 6N (99.9999%) purity will have a melt range of 1 to 2mK.
Gallium is available with 8N purity and so slopes of ±10 to 20µK are possible.

NIST in particular have explored the Gallium Point by evaluating 12 cells of 4 different designs and with 3 sources of metal.

All 12 Cells were within 20μK, and in consequence NIST claims ±40μK as its uncertainty of realization [2].

From an international operability point of view it should be possible to construct a Gallium Cell anywhere, and provided the metal is pure enough an intercomparison should agree within 20μK.

Open Gallium Cell

In 1996 the CCT published a document called "Optimal Realizations of the ITS-90". This was revised in 2000 and the latest version has reference CCT/2000-13 [3].

Referring to Gallium Cells it states that the cell must be open and either set to 1 Bar or its pressure measured and compensation made.

NIST have eliminated this source of error by running their Gallium Cells under a vacuum, simulating a Triple Point.

A Triple Point Cell is one which has solid, liquid, and vapor in equilibrium.

Realization of the Gallium Point at NTPL

Isotech's Primary Calibration Laboratory, the Northern Temperature Primary Laboratory (NTPL) has its own Reference Sealed Gallium Cell of 8N purity, traceable to NPL, England.

To evaluate the International Operability of the Gallium Melt Point, a Standard Gallium Cell of Henry Sostmann design was modified by inserting a small tube through the seal at the top of the cell.

This permitted the cell to be vacuumed, or pressure inside to be set to 1 Bar (the gas is 6N pure, dry Argon).

The Gallium Cell fits into an Automated Gallium Melter (figure 1), a small, 3 Zone Furnace, which automatically melts the Gallium over 15 hours and then refreezes it from the bottom upwards.

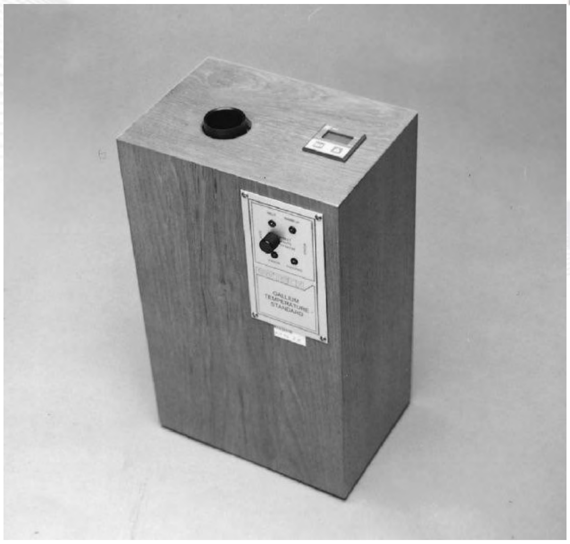

Figure 1. Automated Gallium Melter.

This unique design leaves most impurities at the top of the cell, far away from the thermometer's sensing length.

Using the cell and apparatus, a number of melts were performed (figure 2).

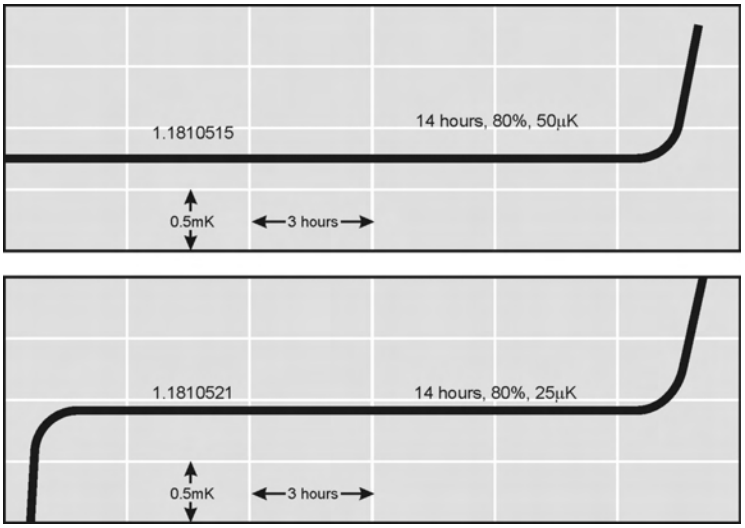

Figure 2. Ga 313 Re-sealable Cell.

It should be noted that, to get the flattest melt, the cell must melt both from the outside, as well as having Molten Gallium around the reentrant tube.

This can be done with a small heater, or warm water. Unfortunately, using this technique the beginning of the melt is lost.

For this reason melts were recorded without the reentrant tube being warmed to obtain complete melt curves.

International Intercomparison

NIST have the smallest uncertainty of realizing the Gallium Point because they researched the uncertainties using a series of 12 designs [2] and so the NTPL Open Cell and Apparatus were sent to NIST for intercomparison.

NIST performed a series of tests and intercomparisons which fully explored the cell (serial number Ga 313).

NIST reported on the cell's performance with and without heating the reentrant tube and operated as a melt point and under vacuum (figure 3).

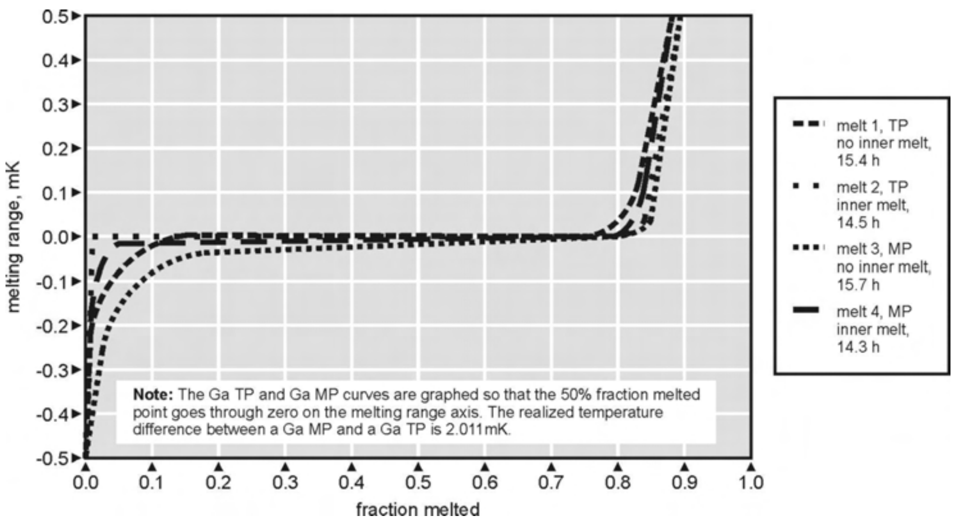

Figure 3. Melting Curves for Isotech Ga Cell (S/No. Ga 313).

Their analysis based on NTPL information that the immersion from Gallium surface to bottom of the reentrant well was 260mm. Showed that Ga 313 was just 40µK below their highest value Reference Cell (impurities generally depress a cell's temperature) (figure 4).

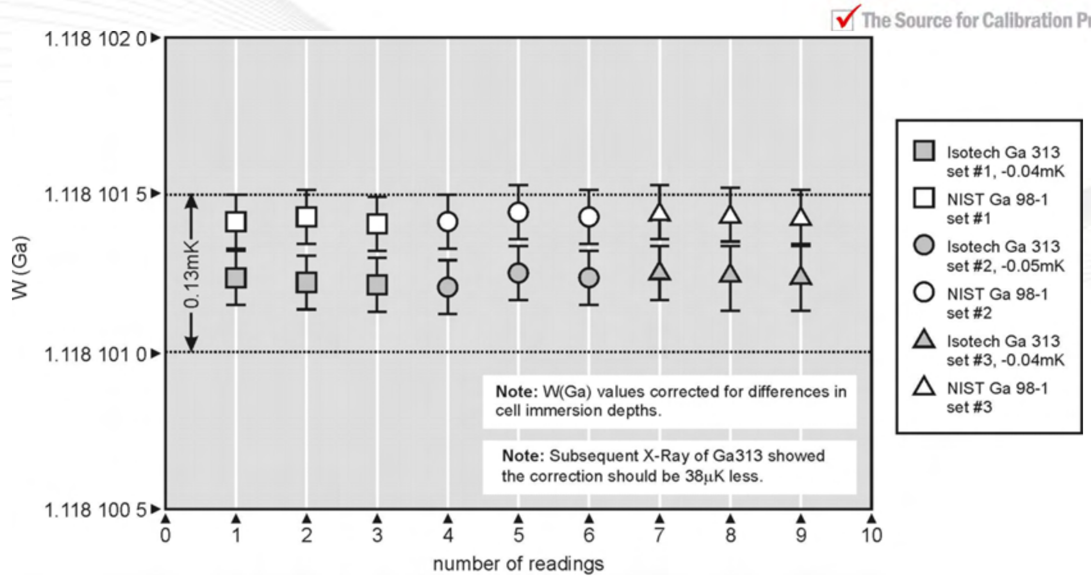

Figure 4. Direct Comparison of Isotech Ga TP Cell (S/No. Ga 313) with NIST Reference Ga TP Cell (Ga 98-1).

On its return to NTPL the cell was x-rayed and the actual depth was found to be 230mm deep and not 260mm as NIST was informed. This correction added 36µK to the temperature making the difference only 4µK. Further, as the cell melts, the Gallium shrinks 3%, which reduces the Hydrostatic head correction still further. Measurements were made after 20 to 30% of the metal had melted, reducing the difference to just 2µK.

This is well within the 20µK spread of results from NIST's 12 cells, and very close to their highest temperature cell.

Discussion

The intercomparison has shown that an artifact (the Gallium Cell) can be fabricated in 1 part of the world, and will have exactly the same temperature as one made thousands of miles away, with a different design and radically different apparatus.

UKAS Reduce NTPL Uncertainties at the Gallium Point

Our previous 2 sigma uncertainty for the verification of Gallium Cells was ±0.45mK this was based on our traceability to NPL who's realization at the Gallium Point is ±0.34mK, 2 sigma.

After inspecting the results of our intercomparison at NIST UKAS offered and NTPL has accepted a revised uncertainty of ±0.1mK, 2 sigma.

Conclusion

NIST's realization at the Gallium Point is 40µK 2 sigma.

An intercomparison between an 8N pure Gallium Cell produced in England and NIST's selected Reference Cell showed agreement within the 20µK, 2 sigma of the intercomparison uncertainty. Confirmation of the International Operability of the Gallium Point.

The consequent uncertainty of Ga 313 to ITS-90 using root sum of squares is 45µK, 2 sigma just 5µK more than NIST's own realization.

References

1. Henry Ernst Sostmann., *Thermometric Fixed Points and the Development of the Gallium Melting Point,* Isotech Journal of Thermometry, Vol. 8 No.1.

2. Gregory F. Strouse., *NIST Realization of the Gallium Triple Point,* Tempmeko '99 Proceedings © NMi 1999.

3. *Optimal Realization of the Defining Fixed Points of the ITS-90 that are used for Contact Thermometry.,* CCT/2000-13., CCT Working Group 1.