Stem Conduction And Light Piping in ITS-90 Fixed Point Cell Assemblies At A UKAS Laboratory

J. P. Tavener & A. Blundell

Managing Director, Head of Primary Laboratory
Isothermal Technology Limited., Pine Grove, Southport, Merseyside, PR9 9AG, England.

Abstract. Standard Platinum Resistance Thermometers (SPRTs) with length-below-handle of only 480mm are regularly submitted for calibration at ITS-90 fixed points from –200 °C to +660 °C. The length of the thermometer limits the maximum size of fixed point cell that can be used to calibrate the thermometers. Stem conduction effects have been measured at zinc and aluminium temperatures in resealable cells. These have been quantified and eliminated by adopting a cell design with a very small connection between cell and gas supply.

INTRODUCTION

Figure 1 opposite shows three designs of fixed point cell.

In the first design (see figure 1a) the cell is sealed and immersed 200mm or more into apparatus that conditions it.

In the second design (figure 1b) the cell is resealable. The quartz crucible that holds the cell exits the apparatus and has a lid, reentrant tube, and is connected to a pure argon gas supply set to 101324 Pa.

In 1995 a third design of cell was described [1] in which only a thin quartz tube exits the apparatus, reducing the thermal path compared to the resealable cell, but permitting the cell's pressure to be measured and controlled (figure 1c). These cells are called Unsealed Sealed Cells (USC's).

In this paper the 3 designs have been investigated at the zinc and aluminium points because of inconsistent results obtained when comparing sealed to resealable cells.

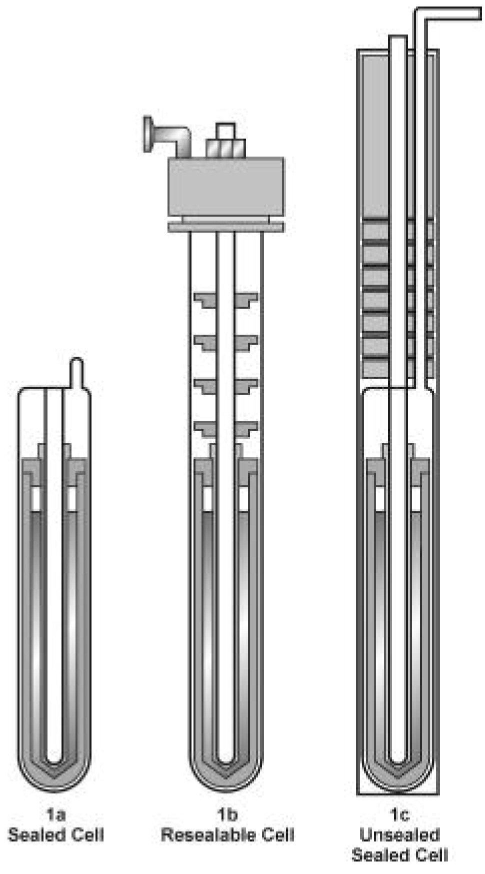


Figure 1. 3 Designs of Cell Assembly Suitable for Indium Through to Silver.

MOTIVATION

The motivation for this work evolved because there are many SPRTs of length 480mm, which are submitted for calibration up to the aluminium fixed point. The stem length of the SPRT dictates the maximum length of the resealable cell that can be used to calibrate the SPRT. Measurements suggested that stem conduction existed in these cells.

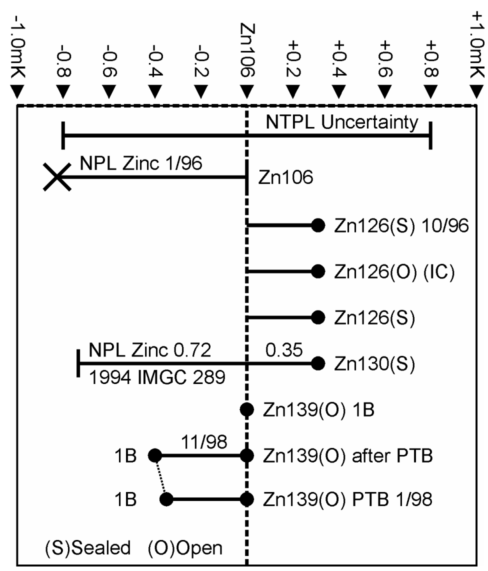
THE ZINC POINT

Initially Isotech's UKAS Primary Laboratory, Northern Temperature Primary Laboratory (NTPL) used sealed cells to calibrate SPRTs. The laboratory's reference sealed zinc cell (Zn 106) of 6N purity had been intercompared at the National Physical Laboratory (NPL), of England and was reported 0.84mK above NPL's Reference.

A raised internal cell pressure was suggested as the cause of the high value.

To investigate this possibility and new cell of optimal design [2] was prepared using zinc with stated purity close to 7N (Zn 126) which was found to be 0.3mK above Zn 106. The Cell was unsealed and made into a cell close to the construction shown in figure 1c. The pressure inside the cell was controlled to 1 Bar ±4mb, despite this Zn 126 measured 0.3mK above Zn 106. See Table 1. These results suggest that NPL's zinc cell was low rather than the cell Zn 106 being high.

Table 1. Summary of Intercomparisons Made At The Zinc Point Against Zn 106.



PURITIES, RAOLTS LAW

One method of assessing the performance of fixed point cells uses a calculation based on the binary combination of each measured impurity, the sum of these elevations or depressions of temperature is used to assess the relation between the cell and ITS-90.

Some National Laboratories like this approach others do not accept the method at all.

In this paper purities are quoted as a relative indicator of a cell's performance, because as far as the authors know nobody has suggested that a less pure cell will perform better than a purer embodiment, i.e. realize a higher temperature.

Typically a 6N pure zinc cell will have a depression of temperature compared to a perfectly pure zinc cell of 0.5mK [2].

A cell of higher purity would be expected to have a higher temperature than a 6N pure cell.

Zn 130 was sent to IMGC, Italy who reported it 0.35mK above their cell – which implied that their cell is within 0.1mK of Zn 106 (table 1).

At this time our accreditation was changed, UKAS requiring us to have resealable cells in order to reduce our uncertainties. A cell of design 1b was constructed (Zn 139) of 7N pure zinc, and sent to PTB (Germany) for intercomparison, it measured 0.3mK lower than PTB's cell and on its return it also measured 0.3mK below Zn 106 (see table 2). This was a surprise since the zinc had the same purity as Zn 126. This established Zn 106 as being the same temperature as the PTB Cell.

Thus Zn 106 Cell seemed to be equivalent to other National Laboratories Realizations, except NPL.

Zn 126, because of its higher purity was 0.3mK above Zn 106. This left a puzzle, why Zn 139 the resealable cell although made with the same zinc as 126 measured 0.6mK below it and 0.3mK below Zn 106. Comparing the melt and freeze curves for these cells (see appendix) indicates that the cells maintain their purity.

Annually Zn 106 and Zn 139 are intercompared at NTPL. In 1999, the temperature difference increased from +0.3mK to +5.7mK.

Table 2. Differences in Temperature Between Secondary Reference, Sealed Zinc Cell Zn 106 and Open Primary Reference Cell Zn 139.

Date	Zn 139 Open Cell State	Direct Comparison DT (Zn 106 – Zn 139)	Standard Deviation (2d) ±mK
Feb 1997	Prior to going to PTB for intercomparison	0mK	0.13mK
Nov 1998	Following return from PTB	+0.34mK	0.28mK
Dec 1999	Following replacement of Zn 139 re-entrant tube with new tube (not fully sandblasted)	+5.7mK	0.17mK
Jan 2000	Following further sandblasting of the open cell (Zn 139) reentrant tube	+0.38mK	0.20mK
Feb 2001	No Change	+0.30mK	0.09mK
Feb 2002	No Change	+0.26mK	0.17mK

After 2 weeks of researching the cause, it was found that the reentrant tube of Zn 139 had been replaced because the original tube had a chip out of the top. Although the new tube had been sand blasted this had only been done partially.

After re-sandblasting the reentrant tube, the difference between the cells reverted to +0.3mK (see table 2).

Based on this finding it was concluded that the drop in temperature between the sealed cells and Zn 139 made with the same zinc, was due to remnant stem conduction/light piping.

The purities remained as expected based on their melt and freeze curves (see appendix).

A simple model for stem conduction has been proposed by Nichols & White [3].

$$D\,Tm = (Tamb - Tsys)\,K\,Exp\left(\frac{-L}{Deff}\right)$$

D Tm is the stem conduction measured, where Tsys and Tamb are the system and ambient temperatures respectively, L is the depth of immersion of the Cell, Deff is the effective diameter of the cell and K can be assumed to be 1. Deff depends on the thermal resistance between the Cell and the system and on the heat capacities. If Deff is assumed to be double the cells diameter, [4], then in the case of a resealable zinc cell, for there to be less than 0.01 °C error at the top of the graphite crucible the cell should be 900mm long!

A resealable zinc cell is typically 500mm overall length with 35 to 50mm of that length outside the apparatus, this implies a very large error due to heat conduction.

In correspondence with Dr. White, he suggested that the careful construction of the cell's internals reduces the immersion required, but admitted that the remaining effects may not be small enough to ignore

DISCUSSION

The measurements above show that the apparent temperature of zinc in a resealable cell sufficiently short to calibrate 480mm long SPRT's can vary depending on the way the cell is sandblasted and in any case the measured temperature is depressed compared to a sealed cell.

When Zn 126 was opened and a construction like 1c was produced, it showed no change in temperature when compared to Zn 106 (a sealed Cell) showing that the 1c assembly created no measurable stem conduction or light piping, and produced a cell apparently 0.6mK above the temperature of a similar cell placed in a resealable package (figure 1b).

ALUMINIUM

Understanding that the resealable zinc cell construction was inadequate, it followed that the aluminium and silver cells would be even more affected. NIST had converted their cells to the 1c design before 1995. Most National Laboratories including Isotech's UKAS Primary Laboratory still use the conventional 1b design.

Our resealable aluminium cell of design 1b (A1 142), an optimal design with 0.2ppm Si as the only known impurity had been intercompared with NPL's cell and was reported 0.4mK above their cell.

Al 142 was used as a reference at NTPL to which various sealed cells were intercompared (see table 3).

The results again suggested that the sealed cells were over pressure and cells were opened and re-gassed without resulting change in temperature compared to Al 142 (see Al 106).

Table 3. Intercomparison of Various Sealed Aluminium Cells with Open Primary Reference Aluminium Cell Al 142.

Date	**Cell Serial Number**	**Direct Comparison DT (Test Cell -Al 142 Open)**	**Standard Deviation (2d) ±mK**
April 2000	Al 106	+6.0mK	0.20mK
June 2000	Al 106 (Following re-gassing and re-sealing cell)	+5.5mK	0.71mK
June 2000	Al 123	+6.2mK	0.72mK
June 2001	Al 124	+3.7mK	1.0mK
Aug 2001	Al 150	+3.0mK	1.0mK
Aug 2001	Al 149	+2.4mK	0.72mK
Sept 2001	Al 146	+4.8mK	0.42mK

During October 2001 an aluminium cell was made of construction 1c. The Cell, Al 148 had impurities 0.3ppm Ca, +0.1ppm Cu +0.1ppm Mg. When intercompared to Al 142 the resealable Cell it measured 3.2mK above it in temperature.

Various sealed cells were compared to Al 148 (see table 4)

The results clearly show that by changing to the 1c design, sealed cells agreed very closely to the reference Al 148.

The results also suggest that stem conduction/light piping contributes over 3mK at the Aluminium temperature, even for a well-sandblasted Resealable Cell which measured 0.4mK above NPL's Reference Cell.

Table 4. Intercomparison of Various Aluminium Cells with Unsealed Sealed Cell Al 148

Date	**Cell Serial Number**	**Direct Comparison DT (Test Cell - Al 148)**	**Standard Deviation (2d) ±mK**
Oct 2001	Al 142 (open)	-3.2mK	0.81mK
Oct 2001	Al 146	-0.17mK	0.47mK
Oct 2001	Al 113	+0.1mK	1.7mK
Jan 2002	Al 149	+0.63mK	1.1mK

DISCUSSION

There has been a trend during the past 5 years for National Laboratories to move from the sealed cell design of 1a to the 1b design on basis that the pressure can be controlled.

It is observed that, unless the 1b design is long enough to eliminate it, stem conduction/light piping may cause large errors that can be variable depending on the type of sand blasting done on the quartz crucible and re-entrant tube.

The design 1c eliminates this source of error and gives the following advantages, improved thermal isolation of the sealed cell and the ability to set and measure the pressure.

For those laboratories who wish to convert a set of sealed cells, most qualified glass blowers would be able to unpick the seal at the top of the cell and join it to a small sandblasted quartz tube long enough to exit the apparatus.

CONCLUSION

The cells indium through to silver are being converted to design 1c at Isotech's Primary Laboratory. The evidence obtained at zinc and aluminium is convincing enough to justify the change to all cells although it is expected that the effect of stem conduction will be small at Sn and In.

Reported intercomparison between fully sealed cells and the 1c design show that concerns about pressure variations inside the sealed cells may have more to do with the 1b design than the sealed cell.

FURTHER WORK

During 2002/3 the unsealed sealed cells will be intercompared to cells of similar design at NIST. Thus at each fixed point 3 internationally intercompared cells will be available 1 of each design (1a, 1b and 1c). Further intercomparison at Isotech's UKAS Primary Laboratory will show the differences in temperature at each fixed point due to the design differences.

UNCERTAINTIES

Intercomparisons eliminate the largest sources of uncertainties. Of the remaining uncertainties the largest source is the reproducibilites of the SPRT's used. By careful selection from over 20 SPRT's two were chosen for each fixed point that have over the years been most stable at the temperature of interest.

The laboratory used 2 M.I. Bridges, an F18 and an F900 during the 6 years involved in the data gathering.

The fixed resistors were Tinsley Type 5685A Wilkins design held at 20 °C ±0.005 °C in an oil bath which was monitored during the tests. All resistors are recalibrated at NPL at 2-year intervals.

Each intercomparison was accompanied by melts and freezes, see appendix, and the procedures were generally in accordance with our UKAS accredited methods.

The furnaces used were Potassium Heat Pipe Dual Furnaces.

Considered expected components of type B (rectangular distribution uj = maximum error / $\sqrt{3}$)		**Uj/mK**
Electrical measurement	Max ±0.10mK	±0.06
Self-heating of SPRT	Max ±0.05mK	±0.03
SPRT repeatability	Max ±0.10mK	±0.06
Spurious heat flux	Max ±0.10mK	±0.06
Hydrostatic effect	Max ±0.02mK	±0.01
Plateau determination	Max ±0.05mK	±0.03
	Combined type B sum	**±0.11**

REFERENCES

[1] NIST Special Publication 260-124. Standard Reference Materials, Gregory F. Strouse.

[2] Optimal Realizations of the Defining Fixed Points of ITS-90 that are used for Contact Thermometry, CCT/2000-13.

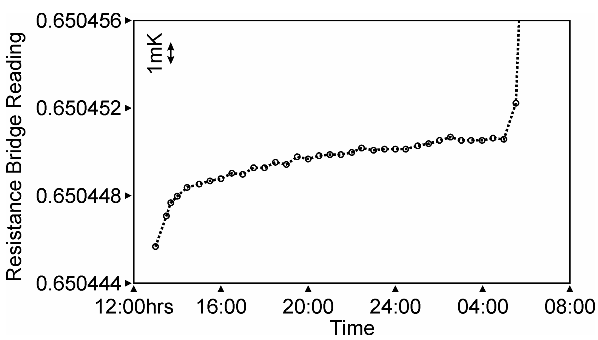
[3] Traceable Temperatures, J.V. Nichols & Dr. White, Wiley & Sons Ltd, ISBN 0-471-49211-4.

[4] Depths of Immersion, J.P. Tavener, Isotech Journal of Thermometry, Volume 9, Issue 2

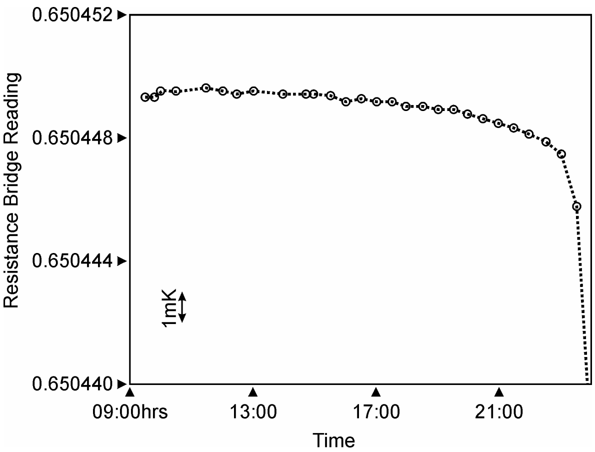
APPENDIX

Melts and Freeze Curves of Zn 106, and Al 142 showing 6N+ purity.

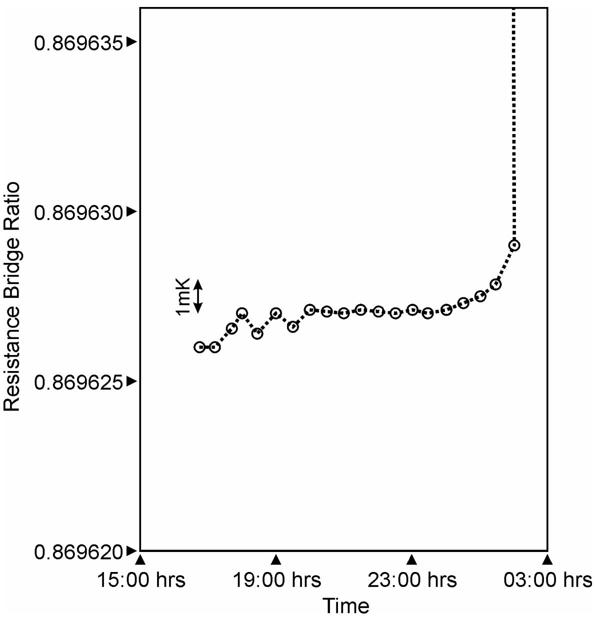
Zn 106 Melt



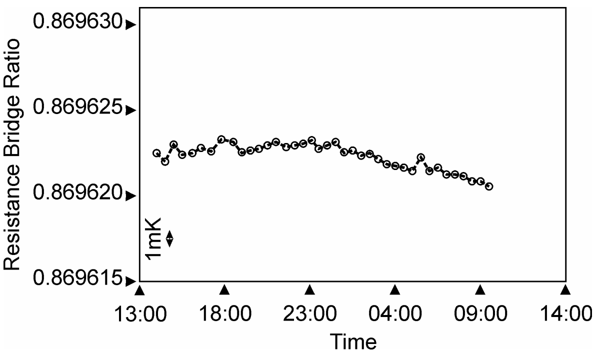
Zn 106 Freeze



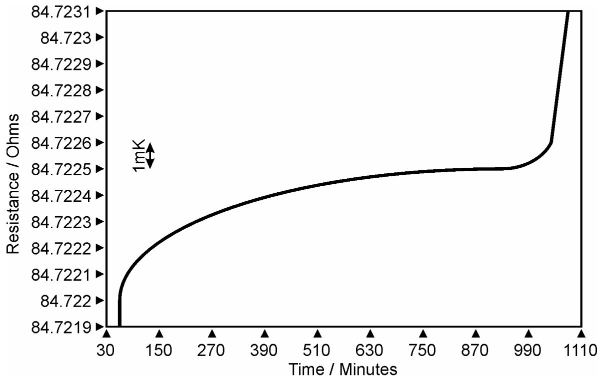
Al 142 Melt



Al 142 Freeze



Al 148 Melt



Al 148 Freeze