IMPROVEMENTS RELATING TO THE CALIBRATION OF THERMOMETERS

Abstract

ITS-90 specifies a series of temperatures which are used to define the scale. Supplementary Information to ITS-90 describes how these substances can be embodied into structures, known as fixed point cells that are useful to thermometrists.

At present, fixed point cells are used with apparatus such as cryostats, baths and furnaces. However, current furnaces for example are expensive. The ideal furnace employs a heat siphon which has thermal conductance 200 times better than copper to create isothermal conditions during phase transitions. It may not be possible for a furnace to use the working fluid of choice for safety reasons. There is often poor thermal conductance between the furnace and the fixed point cell such that a series of furnaces are required to enable the required temperature range to be achieved.

A patent application filed during September 2007 describes a combined metal clad fixed point cell and heat siphon, which when heated provides an isothermal environment for the metal within to change state. The outer wall of the cell becomes the inner wall of the heat siphon with cost as well as performance benefits. The device is called a “Siphonic Cell”. Siphonic Cells can be of Indium, Tin, Zinc, Aluminium, Silver, Gold or Copper.

A fixed point cell is not long enough to eliminate heat conductance along the standard platinum resistance thermometer calibrated in it. Currently, using long furnaces, heat shunts and reflective baffles an attempt is made to reduce these losses.

A second patent describes a device to compensate for the stem conduction problems caused when a thermometer under test is not sufficiently immersed into a fixed point cell. The device is called an “Immersion Compensator”.

Its advantage is that it is independent of the cell/heat siphon and so can be set to give perfect compensation for stem conduction. A combination of the two devices should offer improvements in thermometer calibration.

This paper describes the successful development of the above ideas into a working prototype and the results obtained during 2008 and 2009.

Introduction

The most accurate method of calibrating thermometers is in a series of fixed point cells as described in ITS-90.

To get the best results from the melt or freeze of a fixed point cell it needs to be placed in an apparatus which is gradientless. Unfortunately no fixed point cell is long enough to eliminate the stem conduction of the thermometer and so the apparatus and cell are designed with heat reflectors, thermal shunts and layers of insulation to reduce stem conduction in the thermometer during calibration.

The result is a complicated and expensive compromise, without proper scientific basis. During 2007 an attempt was made to separate the components and recombine them into a more logical and scientific solution.

Combining Cell & Apparatus

The ideal apparatus to surround a cell is a heat pipe or heat siphon. If the outer wall of a metal clad fixed point cell also became the inner wall of the heat siphon then a very simple structure of ideal thermal profile would result.

Firstly over 160 UKAS certificates evaluating quartz and metal clad cells were compared [1].

The analysis showed no detriment occurred to the metal in the cell provided the metal cladding was properly prepared. A literature search also confirmed the above [2, 3, 4, 5, 6, 7, 8].

A heat siphon provider was approached who was prepared to share his manufacture process. Perhaps not surprisingly the metals and metal preparation procedures were almost identical to those we use in preparing metal cladding for fixed point cells.

It therefore seemed probable that such combinations would work.

The concept was patented and called a Siphonic Cell (S.C.)

Working Fluids

Water in the Heat Siphon was suitable for indium and tin, caesium or potassium for zinc and sodium for aluminium.

Immersion compensation (I.C.)

The depth from metal surface to the bottom of the re-entrant tube is 180mm and this is inadequate for most SPRT's. The unit under test therefore needs to go through an isothermal zone above the cell set to the cells transition temperature. Called an Immersion Compensator this drilled and heated thermal block sits on top the Siphonic cell.

The total depth required can be calculated as follows;

An 8mm diameter thermometer with 35mm sensing length requires maximum immersion at around 420°C [9].

Traceable Temperatures (2) (P 136) shows an immersion of 28 diameters (K=2) gives 0.4mK error. The new design has a total immersion of 300mm offering an immersion error of less then 0.1mK – 420°C [10].

Losses

The Siphonic cell is dewar shaped and has no losses below the cell. After profiling the Immersion Compensator there are no losses above the cell. Extremely long plateaus with extremely close settings of the controller should be achievable.

Results

CCT/2000-13 "Optimal Realisations of the Defining Fixed Points of the ITS-90 that are used for Contact Thermometry" was chosen as the standard to which the results would be compared, as this document describes *"…techniques that should be used when it is desired to achieve realisations at the highest levels of accuracy and precision that can be expected with the best equipment presently available" [11].*

The same document describes desirable features of the melt and freeze curves.

"The desired features of (a melt) are constancy of the temperature of the plateau over 75 – 80% of the total (melt) curve to within 1mK.
…(during freezing) relative to the maximum of the freezing curve a depression of the fixed point temperature at 50% of the sample frozen of a few tenths of a milliKelvin.

…a 99.9999% pure sample might have depressions as follows; 0.5mK for In, 0.3mK for Sn, 0.5mK for Zn, 0.7mK for Al and 1.1mK for Ag.

A duration of the plateau of at least 10 hours."

"…the samples liquidus-point temperature obtained from a slow freeze and from a melt obtained following a fast freeze should agree within 0.2mK."

The cell should be sufficiently immersed to exhibit no stem conduction effects over at least 3cm from the bottom of the re-entrant tube.

The Siphonic cells with their Immersion Compensator were assembled into simple desktop apparatus 400mm high with built-in controllers, see figures 1 and 2. The assemblage is called an ITS-90 Isothermal Tower.

ITS-90 isothermal towers of In, Sn, Zn and Al have been evaluated. Eight to ten melts and freezes were made with various settings of the Siphonic cell and also the Immersion Compensator.

Plateau lengths could be varied by offsetting the Siphonic Cell's temperature from 30+ hours (offset 0.1°C) to 4 hours (offset 0.7°C).

In an additional test insulation was added above the immersion compensator. This made no measurable difference to the thermometers temperature but it did reduce the losses and hence power needed by the Immersion Compensator.

Total power consumption is less than 400 Watts.

	CCT/2000-13 Indium	**In Siphonic Cell**
Coincidence	0.1 to 0.2mK	✓
Length of plateau	$10H^+$	✓
80% melt	1mK	✓
50% freeze	0.5mK	✓
Purity	99.9999%	99.9999%

Conclusion: the Indium Siphonic Cell complies to the requirements of CCT/2000-13 for Indium.

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	CCT/2000-13 Tin	Sn Siphonic Cell
Coincidence	0.1 to 0.2mK	✓
Length of plateau	$10H^+$	✓
80% melt	1mK	✓
50% freeze	0.3mK	✓
'O' immersion effects	btm 30mm	✓
Purity	99.9999%	✓

Conclusion: the Tin Siphonic Cell complies to the requirements of CCT/2000-13 for Tin.

	CCT/2000-13 Zinc	Zn Siphonic Cell
Coincidence	0.1 to 0.2mK	✓
Length of plateau	$10H^+$	✓
80% melt	1mK	✓
50% freeze	0.5mK	✓
'O' immersion effects	btm 30mm	✓
Purity	6N	✓

Conclusion: the Zinc Siphonic Cell complies to the requirements of CCT/2000-13 for Zinc.

	CCT/2000-13 Aluminium	Al Siphonic Cell
Coincidence	0.1 to 0.2mK	✓
Length of plateau	$10H^+$	✓
80% melt	1mK	✓
50% freeze	0.6mK	✓
'O' immersion effects	btm 30mm	✓
Purity	99.9999%	✓

Conclusion: the Aluminium Siphonic Cell complies to the requirements of CCT/2000-13 for Aluminium.

Apparatus Used

670SQ/25.5, Serial No. 002.

microK 400 using its internal resistor Tempco .1ppm.

Ambient temperature varied between 8°C and 20°C during testing.

Discussion

The ITS-90 isothermal towers resulting from combining ITS-90 fixed points with a Heat Siphon have shown that they can perform to the highest expectations of CCT/2000-13. By considering the immersion requirements of the thermometer-under-test built in immersion compensation enables the thermometer stem conduction errors to be eliminated.

One concern of accreditation authorities is that cell and apparatus are separated during intercomparisons. ITS-90 isothermal towers are integrated:-

Cell, apparatus and stem conduction correction cannot be separated.

The new device is easier to use than current solutions.

They require a very small amount of power.

Conclusion

The new device meets or exceeds the requirements of optimal realisations of ITS-90 (CCT/2000-13) for Indium, Tin, Zinc and Aluminium.

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Figure 1: Siphonic Cell & Immersion Compensator

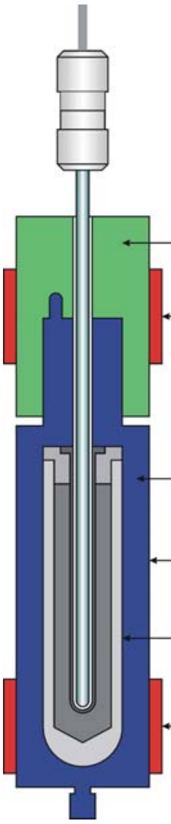

Figure 2: ITS-90 Isothermal Tower

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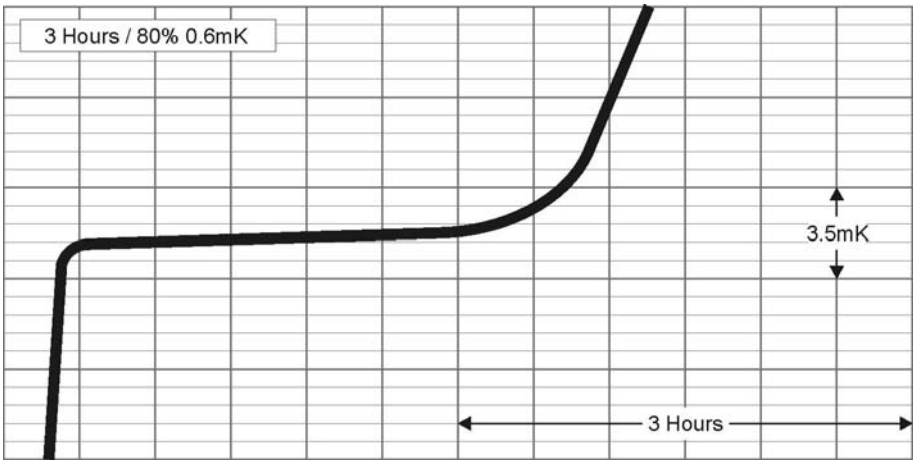
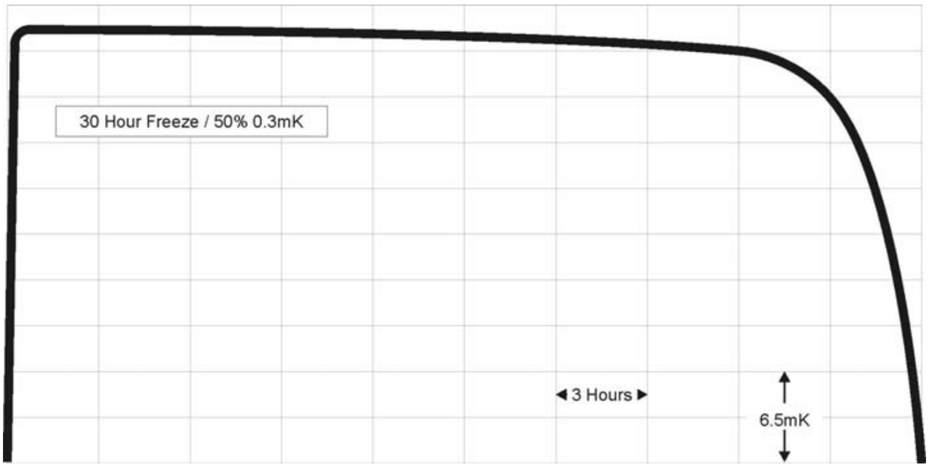
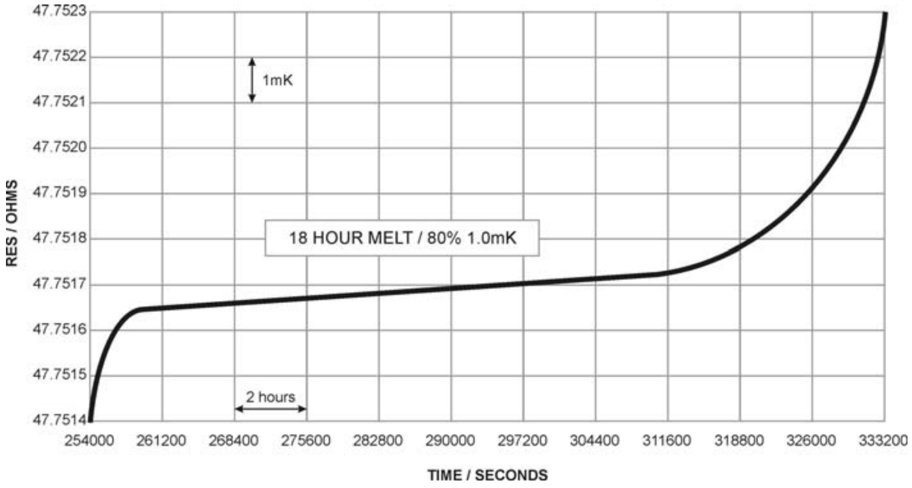
Graph 1: Siphonic In Fixed Point Cell Melt Plateau (26th April 2008)



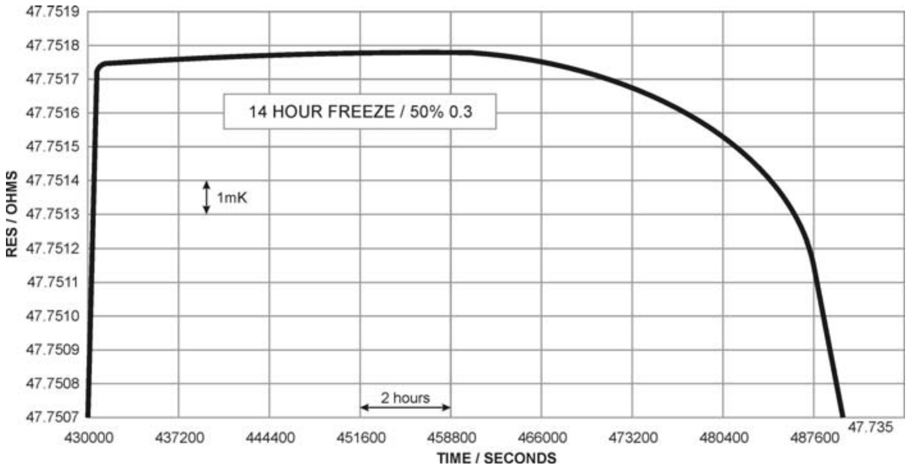
Graph 2: Siphonic In Fixed Point Cell Freeze Plateau (6th May 2008)



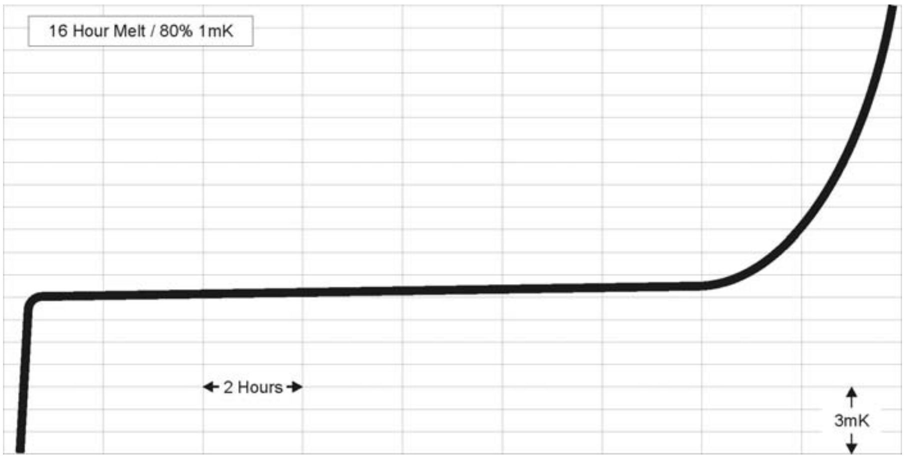
Graph 3: Siphonic Sn Fixed Point Cell Melt Plateau (27th November 2008)



Graph 4: Siphonic Sn Fixed Point Cell Freeze Plateau (27th November 2008)



Graph 5: Siphonic Zn Fixed Point Cell Melt Plateau (24th February 2009)



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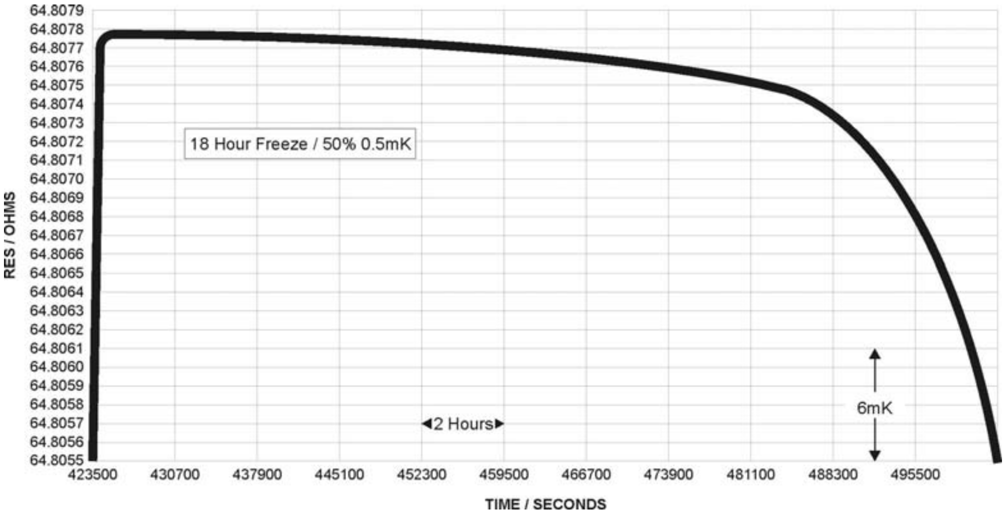
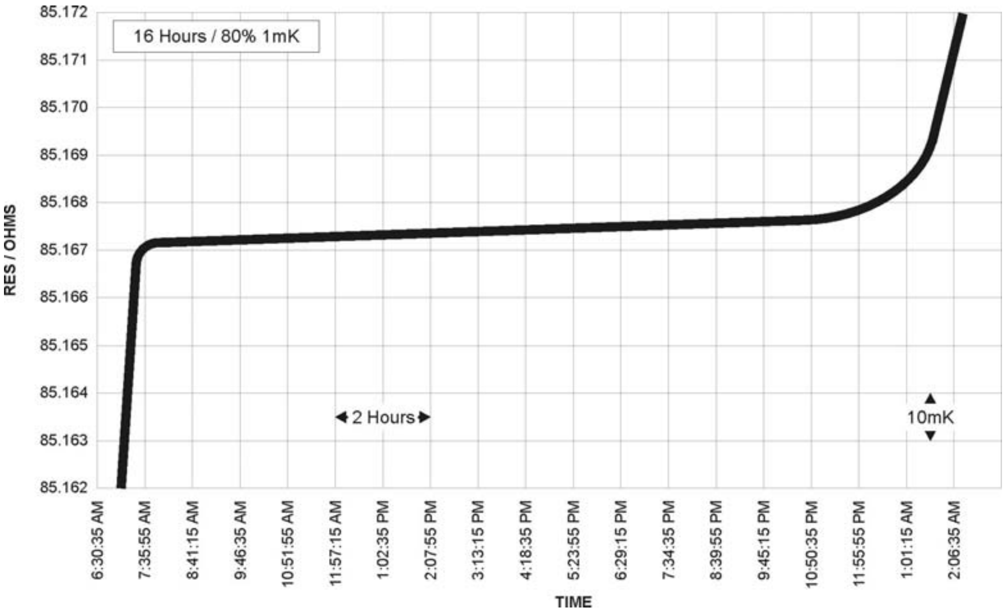
Graph 6: Siphonic Zn Fixed Point Cell Freeze Plateau (25th February 2009)



Graph 7: Siphonic Al Fixed Point Cell Melt Plateau (14th April 2009)



Graph 8: Siphonic Al Fixed Point Cell Freeze Plateau (15th April 2009)