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Results of a blind comparison with NPL using the Ni-C-Cu cells

8.1. Introduction

The comparison of temperature scales between national measurement institutes (NMIs) is essential to ensure its worldwide equivalence of realization. Such comparisons are generally organized at the highest level of Metrology by the representative consultative committee for that unit, in this case it is that for thermometry. At high temperatures, such comparison has traditionally been performed by the exchange of artefacts such as high stability lamps or radiation thermometers. However, both of these artefacts have insufficient performance for modern requirements, unable to probe the claimed scale realisation uncertainties and/or the range realized by NMIs at a suitable level. For example, a high stability lamp has a 1,5 mm target size, only operates up to 1700 °C, it is not a blackbody so it is only suitable for use at the wavelength at which it is calibrated and is very fragile and hence must be hand carried. To overcome these problems, it has been proposed [27] that high temperature fixed points (HTFPs) be used as scale comparison artefacts. It has been objected that these fixed points had known temperature and so to negate that, [27] proposed that doped HTFPs, whose temperature was unknown, could be used instead.

The work of this thesis has been to test this hypothesis – and it would be incomplete without a comparison being performed. So, to test the suitability of the doped eutectic cells as comparison artefacts, a blind comparison was planned and executed with a top NMI. The term blind is important: the NMI in question, the National Physical Laboratory (NPL) of the UK, was not informed of the actual melting transition temperature of the doped eutectic high temperature fixed point cells. In fact the only information given to the participating laboratory was that the cells were based on the Ni-C eutectic cell and that the transition temperature was near that of Ni-C, the latter was needed to set up the NPL furnaces and measurement system. NPL was chosen to perform the blind comparison because of its extensive experience in HTFP research and world leading reputation in thermometry.

In preparation for the comparison the doped HTFPs were manufactured and characterized by Inmetro – these have been described in Chapter 6 above. After characterization the two doped eutectic cells, Ni-C-Cu #7 and Ni-C-Cu #8 were sent to NPL. The objective is to test how closely the temperature scales of NPL and Inmetro agreed. To ensure complete objectivity, NPL was given no guidance on temperatures or realization of the fixed points and NPL was to follow its common practice in measuring HTFPs with its own procedure and equipment.

The cells were posted to UK during November 2012. This incidentally checked that the cells are robust enough to be delivered through the regular mail service. No damage was reported during the circulation of the cells between Inmetro and NPL. All the measurements at NPL were made during December 2012. The cells returned to Inmetro in January 2013, where they were measured again, to check for any changes in their melting temperature, i.e. to confirm long term stability – these results are reported in Tables 12 and 14 in Chapter 6.

At NPL, the measurements were performed using their normal measurement procedure and equipment, which includes a linear pyrometer LP3 [61] (similar device to Inmetro), a Thermogauge furnace [62] and a Lenton three zone furnace [53].

The three zone furnace had its two end zones adjusted in such a way that a thermal gradient < 0,5 °C was obtained across the fixed point.

The thermal gradients were much higher in the Thermogauge furnace because it was a single zone furnace and the blackbody radiator was heated directly through passing a direct current through the walls of the cavity. In an attempt to reduce the thermal gradients in the Thermogauge furnace, five 5mm thick discs of graphite felt (grafelt®) separated by five discs of flexible graphite sheet (grafoil®) were installed around the front and rear of the eutectic cell. Obviously an aperture had to be made at the front of the insulation to view the blackbody. This approach improved the thermal gradient along the HTFP cell but it still remained at approximately 5 °C.

The NPL temperature scale is based on a standard copper fixed-point blackbody [63], which was measured periodically. The reference photocurrent of the LP3 demonstrated a stability of 0,03 °C throughout all the measurements. The routine scale realization uncertainty at the nominal temperature of 1330 °C was 0,18 °C (*k*=2), the largest contribution to the uncertainty being associated with the characterization of wavelength.

Five sets of data were taken with at least four melt freeze cycles performed each day. All the temperatures given for the cells were the point of inflection of the melting curve.

8.2. Measurements with the Ni-C-Cu #7 (4168 ppm) cell

This cell was measured in the two furnaces, in an attempt to investigate the effect of the furnace on the resultant temperature. In table 20 it is possible to see the Inmetro and NPL temperature assignments for the Ni-C-Cu #7 cell. A very good agreement between these measurements was achieved – showing that the scales as realized by NPL and Inmetro were < 0,1°C apart. There was a slight temperature difference between the first and last measurements performed at Inmetro, but well within their uncertainty of the realizations. It is important to note that, the measurements performed at Inmetro after the cell was measured by NPL, were performed with a broken blackbody cavity. As it was explained in Chapter 6, the blackbody cavity remained aligned and permitted the measurements. However, this breakage may have had a small effect on the emissivity and this could explain the temperature difference between the two sets of measurements.

Table 20: Summary of temperature measurements of Inmetro and NPL for the Ni-C-Cu #7 cell

Institute	ITS-90 Temperature (°C)	Uncertainty (*k*=2) (°C)
Inmetro	1328,41	0,22
NPL	1328,54	0,18
Inmetro	1328,52	0,22

In figure 37, the data taken at NPL in the two different furnaces is plotted. It is clear that the furnace has a small effect with the larger gradient furnace (the Thermogauge) yielding a slightly lower temperature (by between 20-50 mK), though not large enough to significantly affect the results of the comparison.

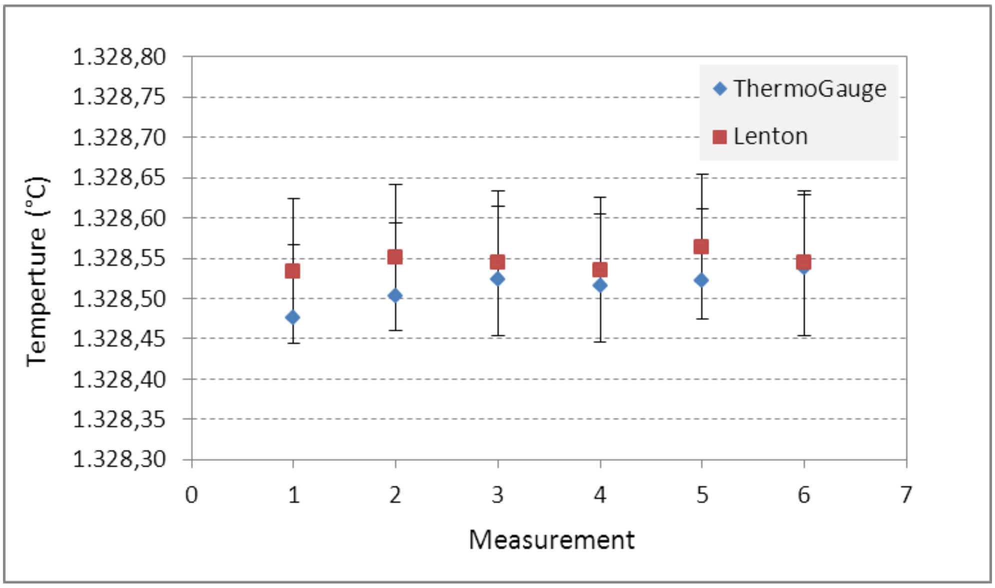

Figure 37: Effect of the furnace on the melting temperature of Ni-C-Cu #7

8.3. Measurements with the Ni-C-Cu #8 (7686 ppm) cell

This cell was measured only in the three zone furnace. Table 21 gives the temperatures measured by Inmetro and NPL for the Ni-C-Cu #8 cell. We can conclude that for this cell, the results are even better than those of cell Ni-C-Cu #7, with a temperature difference of only 40 mK between the NPL and Inmetro. Another thing to note is that the measurements performed at Inmetro gave almost the same results, before and after the measurements performed at NPL. This excellent agreement is probably because this Ni-C-Cu cell was not damaged. More importantly, these results demonstrate to a high level the stability of the Inmetro measurement system.

Table 21: Summary of temperature measurements of Inmetro and NPL for Ni-C-Cu #8

Institute	ITS-90 Temperature (°C)	Uncertainty (k=2) (°C)
Inmetro	1328,41	0,22
NPL	1328,45	0,18
Inmetro	1328,42	0,22

8.4. Furnace effects of the temperature realised for the Ni-C-Cu cells

Figure 38 shows the effect of the furnace on the resultant temperature of the high temperature fixed points. Looking at the Lenton furnace data, the difference between the No. 7 and the No. 8 cell is clear – the different doping levels have induced about 0,1 °C between the two HTFPs.

The x-axis is the previous freeze step before the melt step. This was adjusted because the melt temperature depends to a small extent on the previous freeze step and the magnitude of this needed to be investigated. The source of this effect is because the slower the freeze (i.e. the smaller the freeze step), the longer the time required to freeze the ingot – this allows the freezing to be performed closer to thermal equilibrium. If fast frozen large freeze step more disorder is introduced into the metallic lattice causing pre-melting and hence more rounded melt curves and lower temperatures. This is observed for both furnaces but the effect for the fixed point is very small less than 0,05 °C.

When the Thermogauge and the Lenton furnace data are compared for the Ni-C-Cu #7 cell it is clear that the Thermogauge yields a slightly lower temperature less than 0,05 °C for the HTFP cell than the Lenton furnace. This is the so called furnace effect and is largely due to the more uniform furnace introducing more stray thermal radiation (reflected from the blackbody) into the cavity than the less uniform Thermogauge.

Finally by comparing the Thermogauge data alone it is clear that the bigger melting step gives the lower temperature by about 0,02 °C. Although a very small effect this is very significant. The resultant temperature is lower for the higher melting step because of the fact that the ingot is melting more rapidly and hence the point of inflection moves downward in temperature – with more rapid breakthrough of molten material touching the blackbody tube and so contaminating the thermal radiation emitted. For the slower melt, the solid liquid interface remains intact for longer, allowing the melt to proceed more slowly and hence the point of inflection to be at a higher temperature.

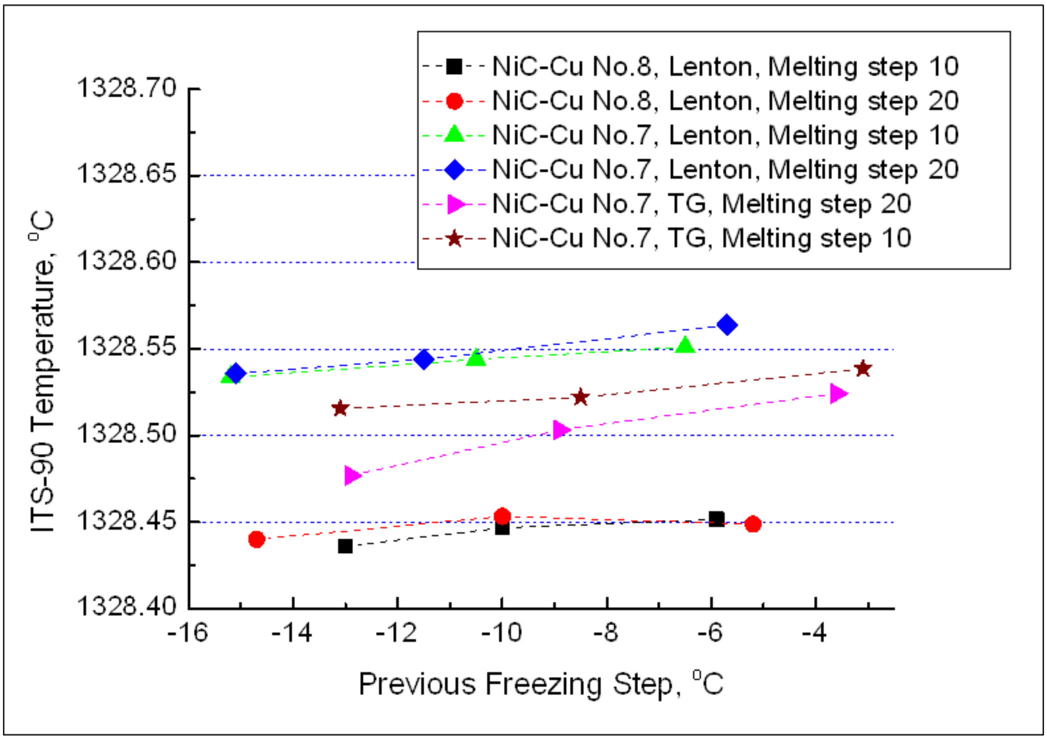

Figure 38: Different furnace effects on the realized temperature of the Ni-C-Cu cells

8.5. Summary of the chapter

The results of the comparison between Inmetro and NPL using doped HTFPs showed very low scale differences, demonstrating that the scales as realized by NPL and Inmetro at this temperature are in very good agreement.

The effect of furnace was investigated in great detail at NPL and its influence was found to be very small (<< 0,1°C), which does not negate the value of the results.

It is possible to conclude that the overall results show that HTFPs are very good comparison artefacts, probing individual laboratory scale realisations – which were not possible before, when lamps or pyrometers were used.