

Improving fixed-point cell realization by modifying furnace heater shape

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Abstract. Recent changes to the SI make it possible to set up a primary temperature scale using established values for certain high-temperature fixed points. As the furnace used with the fixed points can itself have a significant impact on measurements, improving furnace temperature uniformity can help to reduce uncertainties. A thermal model was used to redesign heaters to reduce temperature gradients where the fixed-point cell is positioned in the furnace. A heater optimised for 1325 °C was compared to the standard one with a cobalt carbon high-temperature fixed-point cells, where the cell was installed in the middle, and also moved 10 mm to each end. The modified heater showed reduced melting range, improved plateau run-off and less sensitivity to fixed-point cell position. The improvements will reduce the uncertainties associated with this type of furnace.

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1 Introduction

Since May 2019 the kelvin has been defined in terms of the Boltzmann constant and its practical realization is given by the *mise en pratique* for the definition of the kelvin [1]. The guidance includes a category “primary radiometry by indirect means” which details fixed points, including high-temperature fixed points (HTFP), and can be used as the basis of an interpolated thermodynamic temperature scale that can extend to beyond 2500 °C.

It has been shown previously that such a scale can be set up in an ISO17025 accredited calibration laboratory [2] and that the uncertainties achievable can be comparable to those of a National Measurement Institute (NMI) [3]. That work was based on using a Thermo Gauge dual blackbody calibration source as a furnace to heat the HTFP, with the melting temperature of the fixed-point material used as the reference.

At present, the *mise en pratique* for the definition of the kelvin (MeP-K) specifies HTFP values of cobalt carbon (~1597 K), platinum carbon (~2011 K) and rhenium carbon (~2748 K) with low uncertainty, and palladium carbon (~1765 K) with higher uncertainty. The uncertainties include a significant component for what is often called the furnace effect [4,5]. Although the cause of this effect is not well understood, the uncertainty component is based on observed differences in HTFP values from using different furnaces and can be related to temperature gradients along the fixed-point cell [6]. It is therefore important that the temperature profile along the fixed-point cell should be as uniform as possible. Unlike other purpose-built furnaces, however, the Thermo Gauge was not designed to be used with HTFP, so it needs to be optimised for minimum temperature gradients along a fixed-point cell.

To achieve this, a thermal model was developed to predict the effect of changing the heater dimensions to improve the temperature uniformity. Modified heaters were manufactured and then compared to the standard heater for cobalt carbon, platinum carbon and rhenium carbon HTFP. Each was measured with the fixed-point cell at different positions in the heater tubes to assess the sensitivity to small differences when installing a fixed-point cell in a furnace.

2 Method

2.1 Matlab model

The model predicts the temperature distribution inside a Thermo Gauge 1" furnace. It can be used to make design decisions, such as wall thickness, with the aim of making the temperature distribution in the furnace as uniform as possible. The problem solved is the solution of the steady-state heat equation, including radiation and generation of heat by resistive heating, by a finite volume method. Matlab was used based on the method given in [7].

The model discretises the furnace as a set of volumes, and then uses the steady-state conservation of heat to state that energy in equals energy out for each volume. The energies in each case are either conducted or radiated to other volumes or result in the volume heating from the electric current. The conservation results in a system of the form $A(\mathbf{T}) \cdot \mathbf{T} = \mathbf{b}(\mathbf{T})$, which is solved using Newton's method.

The model reads in the thermal and electrical properties of each volume, the boundary condition temperatures of the environment and the water-cooled jacket and the electric current. The electrodes are made up of graphite and copper and were treated as lumped-elements, with thermal and electrical properties taken from published measurements [7]. The system, as is the actual furnace, is axisymmetric. The user specified heater profile and the size and arrangement of the insulation pieces are read in from text files and then the temperature profile is calculated for a given electric current.

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2.2 Optimisation

The model was used with the thermal properties of the materials provided by the furnace manufacturer and the supplier of the insulation pieces. The heater profile was altered by reducing the diameter of sections at either end to raise the temperature relative to the middle, and so flatten the temperature profile. The size and depth of cut was varied to minimise the temperature gradients along the 44 mm length of the NPL design of HTFP cells. The optimised heaters were made by the manufacturer. The modified heater for the cobalt carbon HTFP is shown next to a standard one in Figure 1.

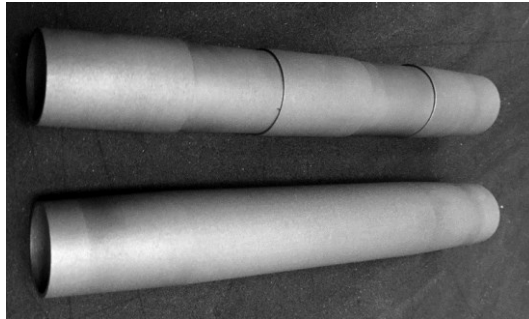


Figure 1. Heater optimised for 1324 °C (top) with standard heater (bottom)

2.3 Validation of the model

The model was used to determine the current required to heat the standard blackbody to a temperature, and that was compared of to the measured current at the same temperature. The gradients in the standard and modified heater were then compared. To do this, a dummy graphite crucible was used in place of a fixed point and an alumina tube through that and the insulation allowed a type R thermocouple to be moved along the central axis of the system at a nominal 1324 °C.

Finally, a cobalt carbon fixed-point cell was measured in both the standard and modified tubes, with the fixed-point cell at three different positions – the middle, 10 mm forward and 10 mm back – to test the sensitivity to how it is installed. Three melt and freeze cycles were made at each combination at different melting and freezing rates. The radiance temperature was measured using an IKE LP3 radiation thermometer calibrated in terms of ITS-90.

3. Results

3.1 Model validation

An initial check was made with the Thermo Gauge in its standard configuration as a calibration source. With a current of 340 A the measured blackbody temperature was 1200 °C. Using the model with the same 340 A current predicted a temperature of 1220 °C. The was considered to be good agreement.

The insulation used with the Thermo Gauge for high-temperature fixed points consists of alternating graphite felt and carbon-composite and is designed not to vignette any radiation thermometer that is compatible with the 25.4 mm furnace aperture and a 3 mm fixed-point cell aperture. The effect of varying the number of pieces was checked and we settled on five pieces each end.

Figure 2 shows the model of this arrangement at 310 A against the profile at nominally 1324 °C.

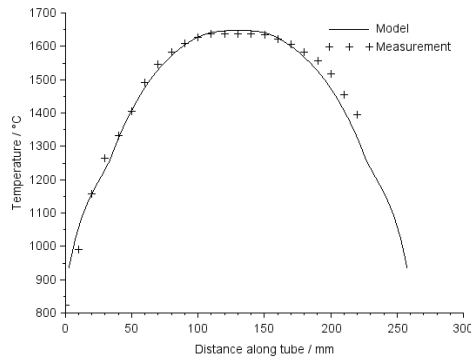


Figure 2. Comparing the furnace temperature profile predicted by the model and measured as it would be for cobalt carbon fixed point

Figure 3 shows how different length cuts are predicted to reduce the gradients. The design used, and shown in Figure 1, leaves the middle 60 mm as standard, then 50 mm at either end is reduced by 1 mm.

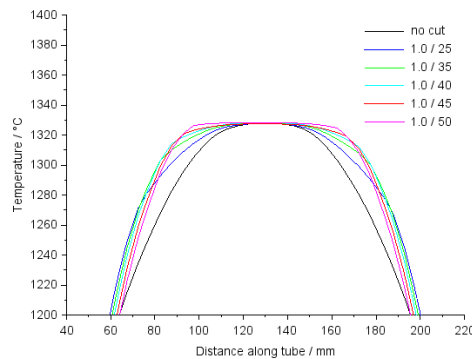


Figure 3. Effect of different cuts on the predicted furnace temperature profile: 1 mm deep and from 25 mm to 50 mm length

Figure 4 shows the measured improvement in uniformity with this design heater.

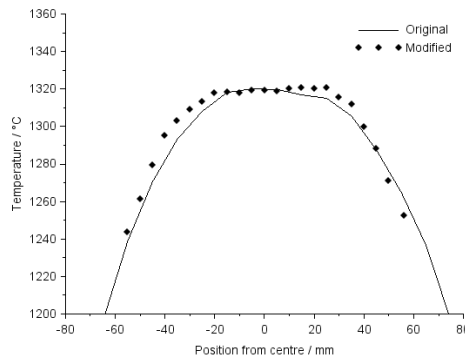


Figure 4. Measured temperature profile using the original and modified heaters at the temperature used for cobalt carbon fixed point

3.2 Fixed point measurements

A cobalt carbon fixed-point cell was measured in both the original and the modified heater, with the same insulation as modelled and used for profiling. Figure 5 shows melting and freezing curves with this fixed-point cell installed in the middle of the standard tube. Figure 6 shows the same sequence with the modified tube.

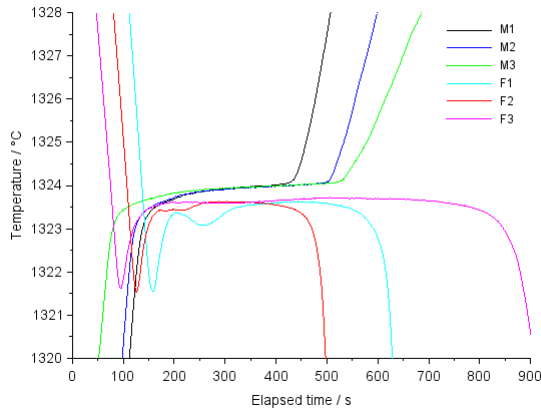


Figure 5 Three melt and freeze cycles of cobalt carbon in the middle of the standard tube

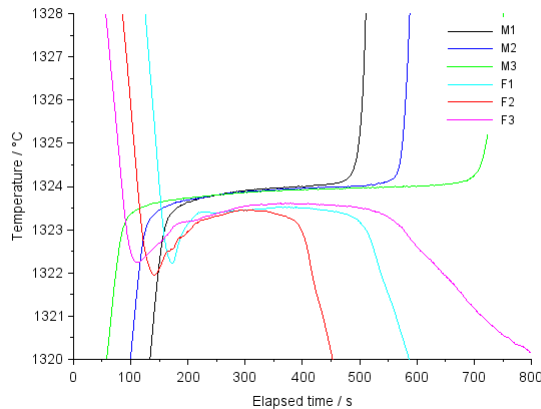


Figure 6 Three melting and freezing cycles of cobalt carbon in the middle of the modified tube

Melt 1 (M1) and freeze 1 (F1) were initiated with the furnace offset from the transition temperature by 20 °C, M2 and F2 by 15 °C and M3 and F3 by 10 °C.

The measurements were repeated, with the same furnace offsets, with the fixed-point cell moved 10 mm forward in the heater tube. The melting and freezing curves in the standard and modified heater tubes are shown in Figure 7 and Figure 8, respectively.

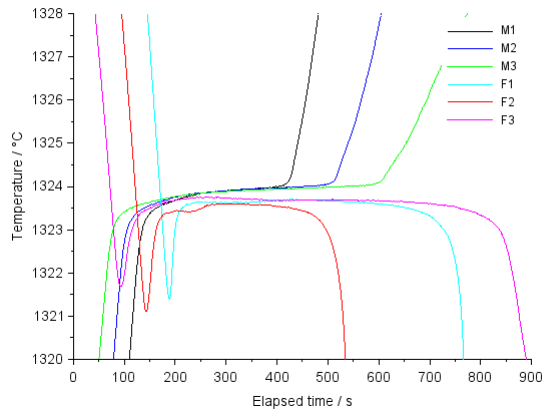


Figure 7. As Figure 5, with the fixed-point cell moved 10 mm forward in the standard tube

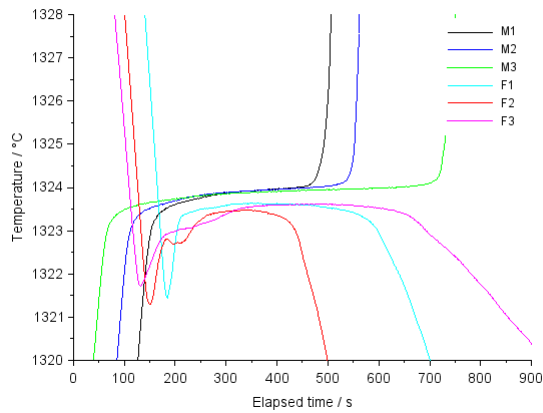


Figure 8. As Figure 6, with the fixed-point cell moved 10 mm forward in the modified tube

Finally, the measurements with the cobalt carbon fixed-point cell were repeated with the cell moved 10 mm backward in the heater, again with the same furnace offsets.

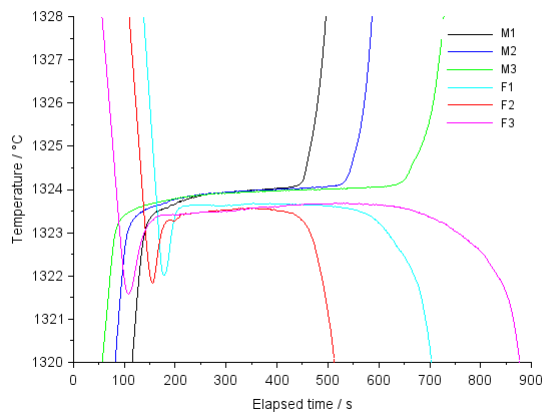


Figure 9. As Figure 5, with the fixed-point cell moved 10 mm backward in the standard tube

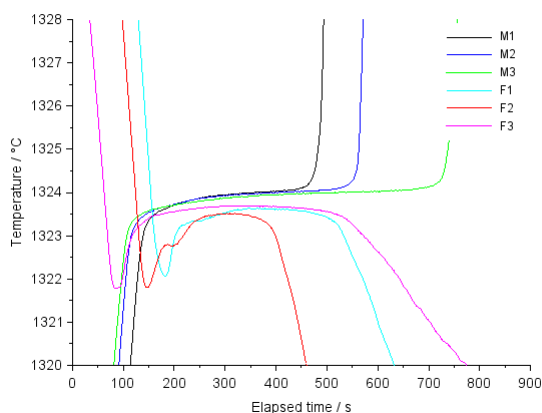


Figure 10. As Figure 6, with the fixed-point cell moved 10 mm backward in the modified tube

A common metric for assessing fixed-point cells is the melting range. This is found by extending the gradient at the point-of-inflection in the melting curve to the times at the maximum gradient before and after the melting plateau. The melting range is the difference in temperature at either end of this line.

Table 1 Melting range in degrees Celsius for the melting curves shown in Figure 5 to Figure 10

	Back	Middle	Front
	Standard heater tube		
M1	0.265	0.364	0.351
M2	0.264	0.335	0.288
M3	0.248	0.373	0.344
	Modified heater tube		
M1	0.241	0.318	0.341
M2	0.264	0.231	0.276
M3	0.237	0.289	0.225

4. Discussion

Melting range and irregularity in the plateau shape, such as non-sharp entry and slow exit (run-off), are aspects that are evaluated when assessing the quality of a fixed-point cell [8]. It is desirable to minimise any contribution from the furnace.

Figure 5 to Figure 10 show consistently sharper run-off in the melting curves using the modified furnace tube. That is, there is a better-defined change from where there is still solid ingot in the fixed-point cell to where the metal is all liquid. This can be seen more clearly by plotting the gradient, shown in Figure 11 and Figure 12. After an initial rise, the gradient drops as melting starts and stays low until melting is completed. There has been a clear improvement: the modified tube results show a better-defined end of melting meaning the melting temperature can be determined with lower uncertainty.

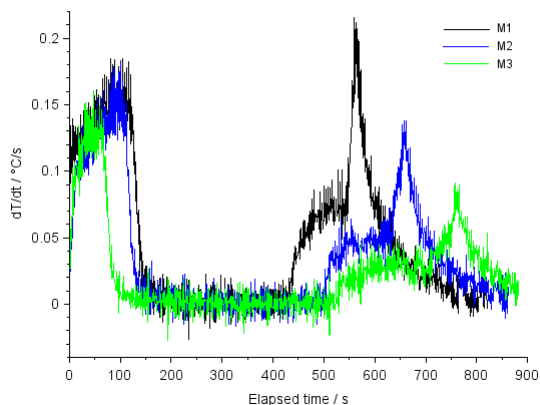


Figure 11. Gradients of the melting curves shown in Figure 5

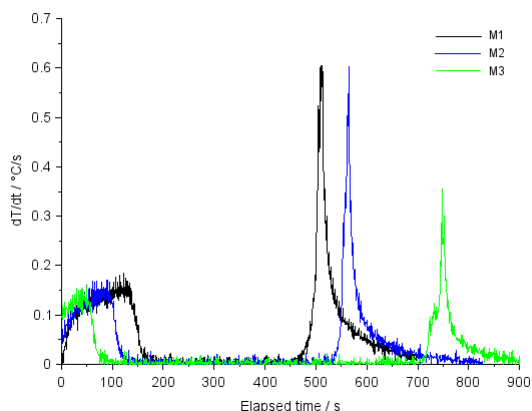


Figure 12. Gradients of the melting curves shown in Figure 6

This improvement in regularity of the melting plateau with the modified heater was also seen with the fixed-point cell moved forward and backward.

As well as regularity of the plateau, the melting range is also taken as an indicator of the quality of a fixed-point cell realization [8], smaller being better. Table 1 shows that the melting range is reduced for a given furnace offset using the modified heater.

High-temperature fixed points rely on using the melting transition since the freezing is depressed by kinetic effects when the eutectic alloy liquid separates into two solid phases. It is noticeable that in these results the standard tube shows freezing curves that look much better than those using the modified tube. There seems to be an inverse effect whereby as the melting curves lengthen and improve, the freezing curves shorten and degrade. It might be expected that improving temperature uniformity would improve both melting and freezing, but not in this case. When furnace gradients are very good, less than 0.5 °C along the crucible, well defined melts and freezes are seen at the same time [9]. Therefore, although the Thermo Gauge uniformity has been improved the furnace is still affecting the fixed-point cell. It is known that if there is a gradient along the fixed-point cell then, for melting, it is better to have it hotter at the back of the fixed-point cell [10]. It may therefore be that with gradients hotter in the front the standard heater tube is improving the freezing

at the expense of the melting curve. This would suggest that as the cell is moved, the cell in the standard tube should start to have shorter melts and longer flatter melts. Looking at the freeze curves in Figures 5, 7 and 9, and at the melting ranges in Table 1, this does appear to be happening.

5. Conclusion

A Matlab thermal model has been used to design a heater for a Thermo Gauge furnace that works with the cobalt fixed-point cell design and insulation arrangement used by NPL. The heater shows improved fixed-point cell realisation with lower melting range and sharper run-off than for the standard heater. The modified heater also has less sensitivity to positioning of the fixed-point cell in the furnace.

The improvement means that the Thermo Gauge furnace is better suited for setting up a high temperature scale in line with the *mise en pratique* for the definition of the kelvin. As this type of furnace is found in many calibration laboratories, this could be a cost-effective route to having reduced uncertainties in laboratories that have previously been dependent on standards calibrated at an NMI.

Acknowledgement

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