Int J Thermophys (2009) 30:296–305
DOI 10.1007/s10765-008-0509-7

Current Work on Furnaces and Data Analysis to Improve the Uniformity and Noise Levels for Metal Fixed Points

D. I. Head · J. Gray · M. de Podesta

Abstract Ongoing work to improve the uniformity of vertically mounted furnaces, manufactured by Carbolite (e.g., Type TZF12/75—three-zone furnace capable of 1200 °C, with 75 mm inner bore) along the axis and across the working tube and/or equalizing block is reported. This involves adjusting the size of the end zones, the position of the control thermometers, and the use of cascade-control methods. Means regularly used at NPL to reduce electrical noise in some commercially available ac furnaces through a reduction in the voltage used to "fire" the heaters, and better use of thyristor controllers (by extending their cycle time) are described. The need to shield the controllers from local magnetic fields is described. With these measures, the electrical noise from ac furnaces can approach that of dc furnaces, without the large cost of a dc power supply. The application of new data analysis techniques (Allan deviation) will be shown to improve the representation of uninterrupted fixed-point traces (as used in ingot verification rather than PRT calibration). Reduction of statistical noise on the temperature measurements has been achieved for data on the freezing plateau by determining the statistically optimum averaging time. This shows that the statistical uncertainty in the determination of the temperature of a particular freezing plateau is less than 25 μK and that noise (drift) from other sources, possibly due to variations in room temperature, starts to become appreciable over periods longer than a few tens of minutes. The measurement of freezing and melting plateaux at this level is aided by the introduction of new ASL-F900 bridge(s), and quieter/larger standard resistor baths.

D. I. Head (✉) · J. Gray · M. de Podesta
National Physical Laboratory,
Teddington, Middlesex TW11 0LW, UK
e-mail: David.Head@npl.co.uk

Keywords Allan deviation · Controllers · Fixed points · Furnaces · Noise reduction

1 Introduction

There is interest in improving the measurement uncertainty obtained when using metal fixed points to realize the calibration temperatures of the International Temperature Scale of 1990 (ITS-90). Among the European national laboratories, the agreed temperature project #732 [1] under the auspices of Euromet seeks to improve the manufacture and realization of these artifacts. Typically, the three major components of the uncertainty budgets are impurity effects, the thermal uniformity of the fixed-point cell's environment, and the measurement noise. The effort at NPL to account for impurity effects using chemical analysis and MTDATA is reported elsewhere [2]. This article describes our efforts to improve the thermal environment and reduce the electrical noise in one type of furnace (commercially available from more than one source) that is regularly used in several national metrology institutes (NMIs). We refer particularly to Carbolite [3] furnaces of Type TZF12/75, which have a three-zone resistive wire heater wound around a ceramic tube of inner diameter 75 mm, that are capable of operation to a maximum of 1200 °C though operation above 1100 °C rapidly shortens their lifetime. As these furnaces were originally designed for quasi-industrial work (and are therefore cheaper than specialized metrology furnaces), they have neither been optimized for thermal homogeneity nor for minimal electrical noise. Here, we describe our methods to overcome these weaknesses. The ideas developed may be of help to furnace manufacturers or other users.

2 Thermal Uniformity

Metal fixed points are typically realized in either vertical or horizontal tube furnaces (for contact and radiation thermometry, respectively). To improve the thermal uniformity around vertical fixed points, the ingot is normally supported within a metal block. Lack of thermal uniformity causes shorter plateaux, bad immersion results, and (in melting) manifests itself as early run-off [4] and "doughnuts" (i.e., where the measured temperature suddenly drops to(ward) the plateau value after melting is apparently completed), suggesting that solid metal has remained at the top, though the metal around the thermometer has melted. In extreme cases this may crack the cell, especially with metal of high expansion coefficient, e.g., aluminum. Although the block will ameliorate temperature variations from the heater nonuniformity, the effect of losses from the tube ends still dominates. One further way of reducing this is to separately control the end zones and use them as guard heaters. In the furnaces traditionally used by NPL, the heater wires are wound directly onto a ceramic work tube, which is its own insulator. The end zones each occupy 25 % of the heated length; 50 % of the length is the center zone. This ratio was chosen as it makes the balancing of the relative currents relatively easy, and allows for a five (as compared to six) lead configuration. (The center zone is fed from its center, and the current flows to the neutral points at the junctions with the end zones. This means that the high voltages of the zones are kept as far apart as is practical, which reduces leakage currents—everything becoming slightly conducting at higher temperatures.) A sensor, embedded in the furnace tube/windings, controls the temperature of each zone. In fact, nowadays a metal-sheathed thermocouple—often type N—is inserted into a small guide tube between the work tube and windings; the heater cross section is thus slightly egg-shaped rather than a true circle. The tips of the control sensors are normally placed at about the middle of each zone.

We are aware that in some other laboratories much longer single-zone furnaces have been used for fixed points. However, shorter thermometers are not then able to reach the bottom of the re-entrant well—including a commercially available standard thermometer (derived from the original Barber design at NPL). We are also aware that many dedicated furnaces are designed with the heater wound directly on the metal block. This will normally give improved performance, but is often harder to access for repair if the heater breaks. Also, it means that a more expensive—and possibly less conductive, e.g., Inconel—block has normally to be used at all times to cover all possible temperatures. Using a separate block within a ceramic tube enables one to change to or select a cheaper (and more conductive, e.g., aluminum alloy) block for lower-temperature work.

We used the remaining space in the control-sensor ceramic tube (control tube) to introduce a finer probe thermocouple (resolution 0.1 °C) so that we could measure the vertical profile of the heater. It was best to fully and slowly insert (to avoid upsetting the control sensors) the thermocouple and then make measurements on withdrawal. We found that, at high temperatures (e.g., above 660 °C), there were peaks in the temperature of 30 °C or more around the two zone junctions; see Fig. 1a. The exact value will depend on the contents of the working tube and their thermal conductivities, and on the operating temperature of the furnace. After our initial surprise (which was shared by some colleagues and manufacturers), we realized that this “double-humped camel” profile was to be expected. To keep the center of the end zones at the same temperature as the center zone, a much larger power is required. As the heater has a uniform winding, too much power would be provided between the end-zone control point and the junction to the center zone—causing the “hump” in temperature.

For some time now, we have been using fixed points to calibrate metal-sheathed thermometers. To overcome the increased heat leak, we have extended the length of our traditional fixed points, and the enclosing metal blocks. This has meant that the top of the blocks has invaded the “hump”, compromising the block’s effectiveness.

To improve the uniformity, the temperature of the end zone has been set lower (see Fig. 1b), but the best reduction is a function of the furnace temperature, and it is time consuming to measure and adjust the large offset required for each fixed-point temperature.

We also (crudely) measured around the circumference at a fixed height and appeared to find a variation of up to 2 °C, with the temperature coolest near the control tube and hottest opposite it. Making sure that the control tube was closed up at both ends (to stop any “chimney” drafts) had no effect. The heater leads go radially away from their take-off point, which traditionally coincides with the position of the control tube, and so we postulated that the heat leak along the leads to the outside is the cause of the lower temperature.

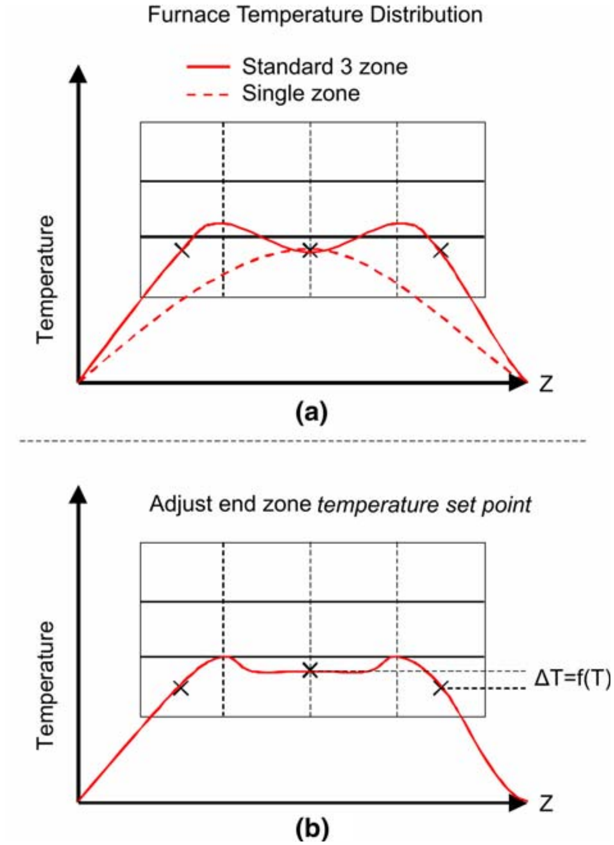


Fig. 1 (**a**) Temperature distributions in a standard single- and three-zone furnace, e.g., in a three-zone furnace under study, a peak of around 30 °C above the control temperature occurs at 660 °C and (**b**) reduction in temperature peaks ("humps") in a three-zone furnace, at one temperature, if the end-zone settings are reduced (the peaks can normally be totally removed by appropriate setting of the end zones, but often a small peak is left to ensure no overcompensation and to provide some additional heat to overcome any heat leak along the thermometer tube)

2.1 Improvement in Thermal Control

To improve the profile, we commissioned two manufacturers to produce a variant of their standard wire-wound tube furnace. We arranged that the control sensors for the end zones could be repositioned *in situ* (while the furnace was "on load") at any point from about half-way down the end zone to just within the center zone (see Fig. 2a). We also reduced the end zones to 1/8th of the heated length (so the center zone now stretched over 75 % of the heated length; see Fig. 2b. We also arranged that the take-off points for the heaters were positioned at 120° to the sensor control tube.

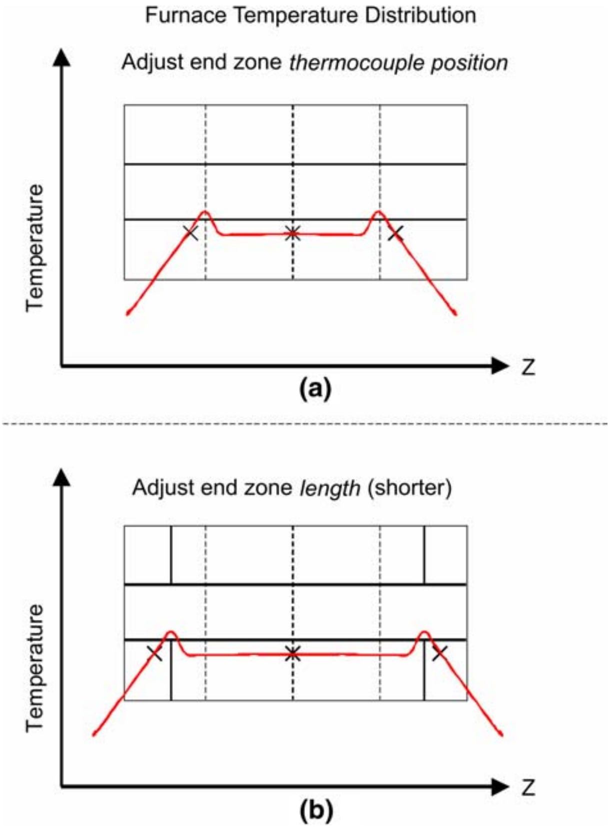


Fig. 2 (**a**) Temperature distributions in a three-zone furnace where the end-zone control sensors have been moved closer to the junctions with the center zone and (**b**) improved zone of uniformity when the length of the end zones are reduced and their control sensors are closer to the junctions with the center zone

2.2 Thermal Profile Results

With the shorter end zones and therefore higher power density, we found that problems with earth leakage increased and, while the metal-sheathed control sensors have been traditionally grounded, we had to insulate and "float" the sensors to avoid earth leakage tripping the laboratory's breakers. If the sensor was placed in the vertical center of the (much shorter) end zone, the "hump" was higher than before (as might be expected). However, as the sensor was moved toward the zone junction, the height of the peak was steadily reduced. However, it is not totally eliminated, and still remains to be some degrees Celsius (at the heater/ceramic tube, which is able to sustain a larger temperature gradient than, say, a furnace with the heater wound directly onto a metal block). The hump could be further reduced as the control sensor "invaded" the center zone; however, as the time constant between the heater and control point inevitably increased, problems with oscillation in the control temperature of the end zone began to occur. (It may have been possible to re-tune the controller to overcome this, but this has not been attempted up to now.)

We had now greatly reduced the "hump" and moved it vertically further away from the "uniform zone" and further reduction may be counter productive—due to other requirements. The other major source of thermal nonuniformity in a fixed-point cell is the thermometer to be measured itself. The glass or silica sheath and the measurement leads go in an almost straight line between the fixed-point temperature and room temperature. It is not normally possible to clamp a heat sink onto the long-stem thermometer. Therefore, a certain amount of "overheating" in the top zone may be necessary to inject heat into the thermometer stem from the furnace, rather than taking heat out of the ingot through the thermometer. This is particularly the case for metal-sheathed thermometers or the commercial thermometer based on the Barber design. In these, the measurement leads are each individually cased in silica tubes that are "cranked," i.e., alternately bent side-to-side within the stem, to stop light piping. (While it has the disadvantage of increased heat leak, it does make the connecting wires much less prone to kinking, catching, and breaking on temperature cycling, and tends to make the thermometer slightly more robust.)

2.3 Cascade Control

Another advantage of heaters wrapped directly onto a metal block is that the control thermometer—usually placed within the block—is close to, but not directly in contact with, both the fixed-point cell and the heater, usually providing a short control response time. With our furnaces, the control point is near the heater, which can leave a gradient between the heater and the ingot. Alternatively, this gradient can be reduced by using a control sensor in the metal block that is inserted into the ceramic work tube; however, this means that the control time and the possibility of initial oscillation and overheat is greatly increased. To overcome this, we are investigating the use of "cascade control," in which a control sensor is placed both within our stand-alone block and within the furnace heater. The control of the block sensor is cascaded to the heater sensor. (This tends to cause the heater control power to vary more rapidly to ensure that the block remains unchanged, but careful tuning of the controller should normally reduce this. Modern controllers now allow for separate tuning at half a dozen temperature points or ranges—sufficient to individually cover the fixed-point temperatures of interest in the ITS-90.)

3 AC vs. DC

Some of the best measurement bridges for standard platinum resistance thermometers (SPRTs) are the F-series ac bridges made by ASL Ltd [5]. These operate at a frequency of 0.5 and 1.5 times the mains frequency. AC bridges are susceptible to picking up other electrical spikes and signals. As the SPRT is coupled to the furnace windings both capacitively and inductively, it can pick up electrical noise from the furnace. The furnaces we describe are also ac, with the power control achieved using thyristor firing of the heaters. Ideally, the furnaces would be dc powered—as is done in some measurement institutes; however, dc controllers are more complicated, can have large stray magnetic fields, and are much more expensive. DC controllers normally regulate the voltage amplitude. In theory, the ac amplitude could be varied rather than the ac duration (which is what happens in thyristor control). However, automatic control of the ac voltage is not readily available. With thyristors, the ac is switched on and off using “zero-crossing” thyristors—meaning that the waveform itself is not chopped, but the number of cycles “on” is controlled and the power is switched off as a zero is crossed. However, both the B and E fields cannot at the same time be zero, and the sudden *change* of one creates a noise spike across a range of frequencies. (We have noticed that the magnitude of the pickup of this spike can be surprisingly dependent on the rotational orientation of thermometers with respect to the furnace and the point the thermometer leads cross the circumferential edge of the furnace.)

To reduce the noise, once the fixed-point temperature is achieved, we power the furnace heaters at a reduced voltage using a wire-wound transformer, but still high enough to make the thyristors fire. Careful adjustment of the voltage means that the thyristors are on for around 90 % of the time, reducing the number of spikes but still enabling control. (This reduced voltage also has the benefit that the furnace cannot “run away” in temperature if the controller fails “on.”) One must be careful that the controllers are shielded from the transformers—mild steel is satisfactory—if they are in close proximity. This technique can reduce the noise amplitude by up to a factor of two across a range of ITS-90 fixed points. But a word of caution, if one only partly reduces the voltage, it is possible in some cases to slightly increase the noise. (We do not fully understand this, but we believe this is due to smaller voltages reducing the power delivered in each burst so that the thyristors are firing more, but not enough to be on for a full cycle, and it is only when the voltage is further reduced that there is a net gain. There is a trade-off between amplitude and frequency of firing.) For convenience, the controller power and heater power can be wired separately; see Fig. 3. (Manufacturers are advised to supply starred duplicate neutrals and to comply with modern regulation to avoid mains voltage appearing on the second plug, if only one is plugged in.)

Thyristor controllers can allow one to adjust the on/off cycle time, and we have found that this can be increased from the traditional fraction of a second to up to 30 s (e.g., for 66 % power, the voltage is applied for 20 s and then off for 10 s). This greatly reduces the number of spikes and the electric noise, normally halving the noise

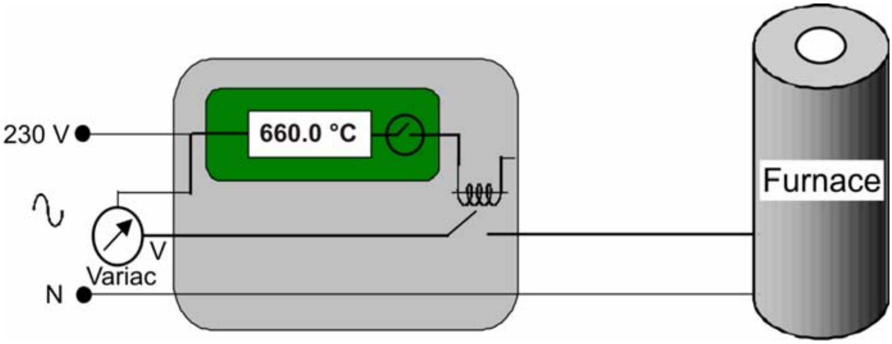


Fig. 3 Twin-socket controller with a reduced maximum voltage for the heater

amplitude. One cannot extend the time beyond this for our furnaces as it then becomes much longer than the natural time constant of the furnace and the ability to control the temperature is lost. In fact, due to the natural time constant of these furnaces, no gain in control appears to be achieved by having a cycle time of less than 20 s. (Note that the cycle time cannot be extended for all types of furnace as some may not be able to stay full power, e.g., where the cycle time is used to reduce the power if the supply voltage is too high.) The use of both voltage reduction and increased cycle time has an advantage over using only one of these techniques, but the effects are not multiplicative as each reduces the number of "spikes" that the other technique has to act on. The combination of all these interventions can reduce the effective noise of an ac furnace to close to that of a dc-powered furnace.

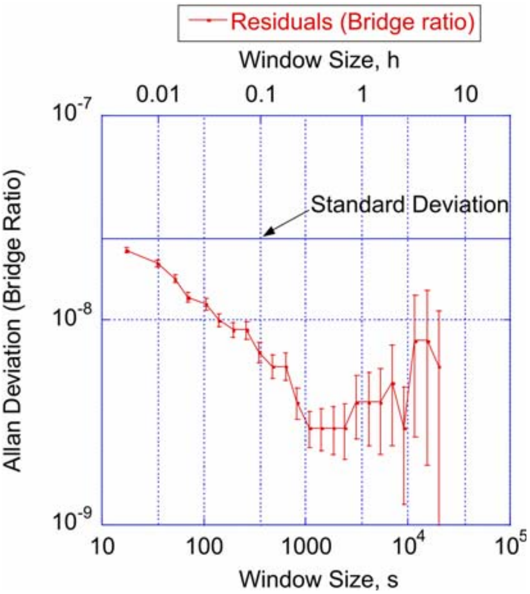
While the thermometer is located inside a furnace, there is also the standard resistor that is located within some form of temperature enclosure—often a liquid bath. In the UK, the reference temperature used for standard resistors is usually 20 °C, historically convenient in the UK. However, it does mean that a cooler as well as a heater is needed to maintain this temperature within a laboratory environment. We have noted that thyristors are used in the heater control of some cheaper baths and that the degree of noise pickup depends on whether it is a 10 Ω or 100 Ω resistor (NPL uses the "Wilkins" design) and repositioning the more susceptible resistor away from the heater will reduce noise. Baths with plastic control cases also seem to produce more noise, presumably as there is less shielding. Due to restricted access, we have not applied the furnace techniques to the resistor bath—physical movement suffices. These effects are quite visible using an ASL F18 bridge. With the arrival of two ASL F900 bridges, we chose to reduce noise input by assembling/purchasing larger Peltier-driven baths to make the best use of the increased resolution of the new bridges. Attempts at using an uninterruptible power supply (UPS) (designed for PC back-up) to run an ASL F18 was found to increase noise. The manufacturers informed us that our UPS caused a difference in phase or working frequency, abrogating some of the F18's noise cancellation circuits. We were advised that a more expensive UPS with phase and frequency locking will help, but we will not be able to test this until we move to our new laboratories. When doing noise reduction tests, it is best to use the F-bridges in the manual mode so that one sees the actual noise (via the rear analog output) rather than seeing the bridge's attempts to follow the noise in the automatic mode, which causes an additional "balancing noise". In the manual mode, one also has a higher signal resolution.

We have also noticed that, when doing a water triple point to produce the resistance ratio (W-value) for each fixed-point measurement, our traditional use of a large vacuum-insulated ice bucket still provides a noticeably quieter environment than the commercial compressor-driven water-cell baths that we have used so far.

4 Best Averaging of Other Measurement Noise

As the thermometer and standard resistor are connected by leads to a resistance bridge, itself connected to the mains power supply, there are several other external sources of noise that can be added to the measurement signal. To overcome these during a

Fig. 4 Plot of Allan deviation against averaging time, showing the reduction to a floor and subsequent worsening of the deviation with longer averaging time



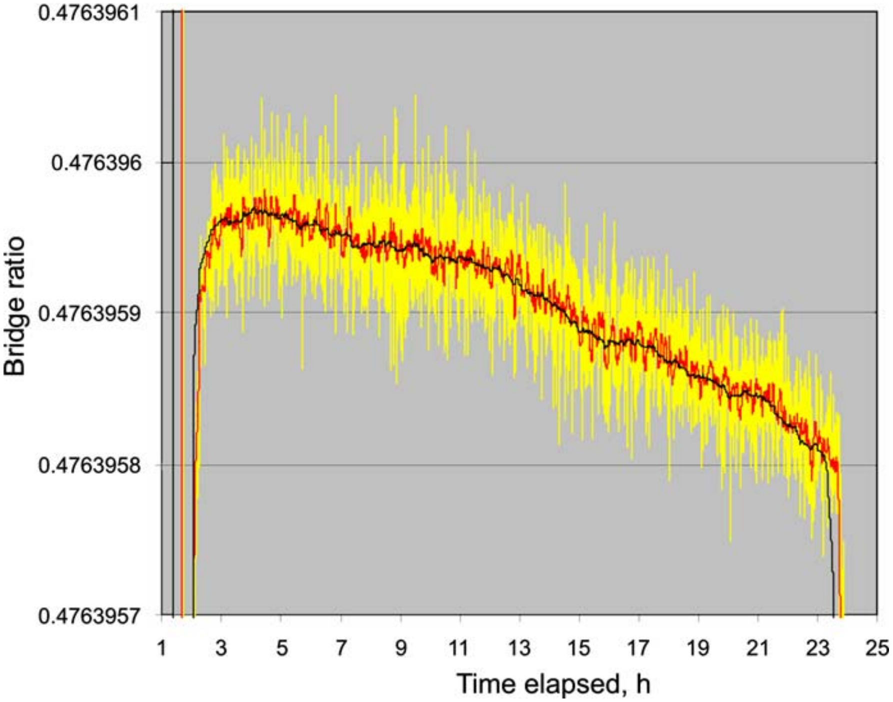


Fig. 5 Plots of a Sn freeze showing noise reduction by longer averaging intervals, i.e., averages of 1 point, 16 points, and 100 points, the latter about the time given by the Allan deviation minimum as each point takes 17 s. Horizontal lines show a temperature change of approximately 0.1 mK

fixed-point measurement, the readings are usually averaged over a longer period of time. What is the longest period of time one can do this and get an improved signal? An upper bound on this time can be obtained by calculating the Allan deviation of the measurements [6]. This is done by calculating the standard deviations of data sets of increasing length and then plotting these standard deviations against data set length. As the length increases, there is initially a fall in standard deviation, suggesting an improvement in the average value. However, once the length goes beyond a certain point, no further improvement occurs—in fact there is often an *increase* in the standard deviation. The initial minimum in standard deviation gives an upper limit on the length of time over which one should average. In some of our fixed-point measurements, we have found this to be no more than a quarter of an hour; see Fig. 4. (At 17 s per point, averaging more than 100 points makes no improvement.) We suggest that other cyclic effects, such as the air conditioning, might be the cause of longer-term drift that compromises any further averaging. In Fig. 5, we show the effect on a fixed-point trace of three levels of averaging, including one corresponding to the Allan Deviation minimum.

5 Conclusion

Using shorter end zones, better positioning of control sensors, reduced switching voltages, increasing the switching cycle time, tuning the controllers with the possibility of cascade control, ensuring the heat leak along the power leads is kept from control sensors, reducing noise input from the standard resistor, employing state-of-the-art ac bridges, and optimizing the amount of signal averaging leads to a much improved signal from the realized ITS-90 metal fixed point.

Acknowledgments Former NPL staff, notably Maurice Chattle, introduced the use of transformers to reduce the ac power voltage. Karen Alston carried out several of the early furnace-profile measurements. The work described in this paper has been funded by parts of several contracts over past years from the National Measurement System of the UK Department of Trade and Industry.

References

1. EUROMET 732: *Towards more Accurate Temperature Fixed Points*; Coordinator E. Renaot, LNE-INM/CNAM, 61 rue du Landy, 93210 La Plaine Saint Denis, France
2. D.I. Head, H. Davies, J. Gray, P. Quested, in *Proceedings of TEMPMEKO 2007*. Int. J. Thermophys. doi:10.1007/s10765-008-0440-y
3. Carbolite Furnaces—Parsons Lane, Hope, Hope Valley S33 6RB, UK, E-mail: info@carbolite.com
4. J. Ancsin, Metrologia **40**, 36 (2003)
5. ASL—Automatic Systems Laboratories, 275 King Henry's Drive, New Addington, Croydon, Surrey, CR0 0AE, UK, Tel: +44 (0) 1689 800799, E-mail: sales@aslltd.co.uk
6. D. Allan, IEEE Trans. Instrum. Meas. **36**, 646 (1987)