Cost Effective Techniques used to Validate the Performance of the microK Resistance Thermometry Instrument with sub mK Uncertainty

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This article describes techniques used to validate the performance of the microK thermometry instrument, which was designed for use in secondary temperature calibration and high accuracy temperature measurement applications (to <0.4mK uncertainty). The microK is the first precision thermometer that can work with both resistance thermometers and thermocouples and provides sub mK uncertainties with SPRTs and mK uncertainties with thermocouples. With a resistance ratio accuracy of 0.4ppm and voltage accuracy of 0.25µV, validating the performance of the product represented a significant technical challenge. With a limited budget and operating mainly in a non-air-conditioned laboratory, cost effective ways were developed to validate the performance. It was possible to discriminate features in the linearity of the microK which were below 0.1ppm with relatively inexpensive equipment.

INTRODUCTION

The temperature scale is realised and disseminated using fixed point cells and standards platinum resistance thermometers (SPRTs). In addition, at higher temperatures, thermocouples are widely used as calibration transfer standards. The temperature measurement uncertainty required of a calibration laboratory will typically involve measuring resistance to better than 1ppm, (equivalent to 1mK with SPRTs) and voltage to better than 0.5µV (equivalent to 25mK for a gold-platinum thermocouple). These are measurement uncertainties that are comparable to those of a good electrical laboratory.

The microK-400 is capable of measuring resistance to better than 0.4ppm and voltage to better than 0.25µV. Validating the performance of the instrument and providing traceable production calibration involved producing standards that have extremely low uncertainties. The normal approach to this problem would perhaps be to work in an air-conditioned laboratory with Wilkins resistance standards maintained in a temperature controlled oil bath. Voltage standard would be a rack of zener references with calibrated attenuators. The cost of such a system was prohibitive and proved quite unnecessary. We were able to achieve measurement uncertainties for validating the performance of the microK below 0.1ppm for resistance using relatively inexpensive equipment and without operating in an air-conditioned environment. Production calibration is of course carried out in an air-conditional laboratory under a UKAS schedule.

RESISTANCE MEASUREMENT SYSTEM AND SOURCES OF ERROR

The most accurate temperature measurements involve measuring the resistance of an SPRT. The microK achieves this by measuring the voltage developed across the SPRT whilst it is connected (using a 4-wire technique) to a current source (Figure 1). The system then performs the same measurement on a reference resistor. The resistance of the SPRT is the ratio of the two measurements multiplied by the value of the reference resistor.

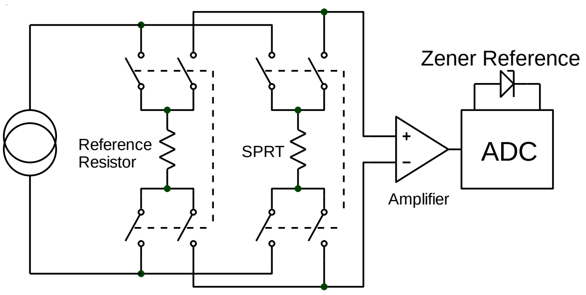

Figure 1: Resistance Measuring System

Thermal EMFs (EMFs generated as the result of dissimilar metals and temperature gradients) are a potential source of error when working at this precision. These can be eliminated when measuring resistance thermometers by taking two measurements (V_1 and V_2) and reversing the current (I) between them. Averaging the magnitude of the readings (i.e. calculating half the difference between the two readings) yields a result that is the voltage developed across the resistance R without any effect from the thermals EMFs (e):

$$V_1 = IR + e$$

$$V_2 = -IR + e$$

$$\frac{V_1 - V_2}{2} = \frac{(IR+e)-(-IR+e)}{2} = IR$$

The process of current reversal and averaging, together with true 4-wire resistance measurement has the effect of ensuring an intrinsically stable zero with time and temperature (the voltage at the Amplifier input when measuring a short-circuit will be the same whichever current direction is used). The process of averaging (the magnitude of) the measurements therefore yields zero, with uncertainty determined by the system noise.

Traditionally, instruments of this precision use a bridge topology in which the device-under-test (DUT – in this case an SPRT) is connected in series with the reference resistor. The measurement system is then alternately connected to measure the voltage across the two resistances (Figure 2).

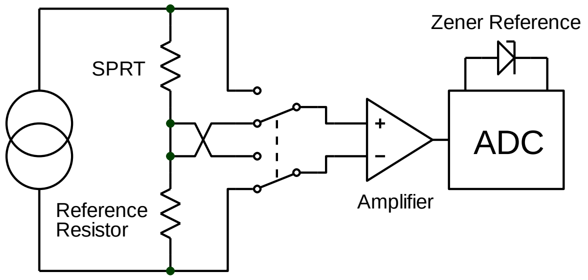

Figure 2; Conventional Bridge Topology

A significant source of error in such potentiometric bridge instruments is the common-mode rejection ratio of the amplifier. The common-mode signal at the input to the amplifier changes between the two measurements and will lead to an error at the input to the ADC.

The microK uses a substitution topology in which there is a single point of measurement in the system into which the SPRT and Reference Resistor are switched alternately (Figure 1). This means that the measurement system is also inherently stable at unity ratio since the voltages measured for a reference resistor and SPRT of the same value will be identical. There is, after all, no difference between these two measurements apart from the fact that they are taken at slight different times. The system noise will again determine the uncertainty of this unity ratio measurement.

Having employed a topology that provides inherent stability at both zero and full scale (assuming the reference resistor used has a value corresponding to the required full-scale) and confirmed this by testing, the main challenge was to verify the linearity to <0.1ppm uncertainty.

VOLTAGE MEASUREMENT SYSTEM AND SOURCES OF ERROR

The voltage measurement system is similar to the resistance system (Figure 3). In order to minimize the effect of thermal EMFs, the microK uses tellurium-copper connectors on the front panel and reverses the connections to the measurement system very close to these connectors. The voltage measurement system uses the same Amplifier and ADC as the resistance system. The linearity performance will therefore be the same. However, the zero stability of the voltage system will be determined by the performance of the components used to reverse the connections to the input terminals. The span stability will be determined by the stability of the zener reference used with the

ADC and also on the gain stability of the amplifier (Amplifier gain is not critical in resistance measurements because such measurements are inherently ratiometric).

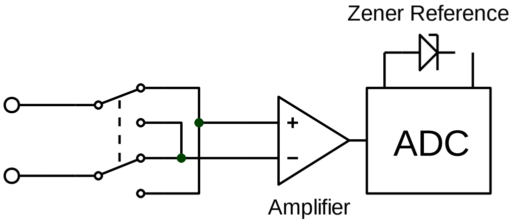

Figure 3; Voltage Measuring System

CHECKING MID-SCALE LINEARITY ERRORS

Measurement errors occur in both the Amplifier and the ADC. The more significant errors are essentially quadratic in form. A good example of this is the power-coefficient of the resistors used to set the amplifier gain. Typically, resistors have a linear temperature coefficient of resistance. However, since the power dissipated in the resistor is proportional to the square of the voltage across it, this leads to a variation in resistance that is quadratic with applied voltage. As discussed above, the microK will inherently read correctly at zero and at unity ratio. This has the effect of normalizing any quadratic errors at zero and unity so that the error function is a parabola with the maximum error at the mid-point (Figure 4).

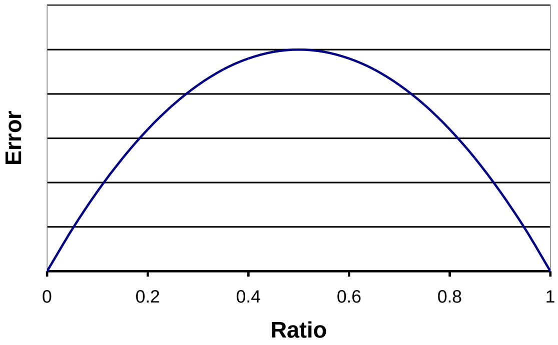

Figure 4: Typical Quadratic Error

The performance of the ADC in the microK was checked against the Josephson-Junction Array [1] at the National Physical Laboratory in the UK. This quantum standard is the country’s primary voltage standard and the measurements were limited only by the thermal EMFs in the test arrangement. These tests confirmed that the ADC was comfortably within specification and that the errors were substantially quadratic in form. The Josephson-Junction system is extremely costly to use and its absolute accuracy is not required to validate the linearity of the microK.

Having confirmed the quadratic form of the error function, attention was focused on ways to check the mid-scale error (the worst case point in quadratic errors) with uncertainty below 0.1ppm of range. This corresponds to 0.5µV at the ADC input (range ±5V).

Whilst multi-function calibrators are available with this sort of accuracy, they are very expensive and include capabilities that are just not required in this application. We therefore decided to use a constant current source and two good quality bulk-metal foil resistors to provide a mid-scale error check. The current source used was a Metron Designs I-REF2, originally developed for CERN. This provides a (traceable) constant 10mA and has the following key performance parameters:

Temperature Stability	±0.2ppmK^{-1}
First Year Stability	-1±3ppm/year
Output Resistance	>10GΩ

A resistance box was then made that allowed two 500Ω resistors to be connected individually or in parallel across the current source using a 4-terminal connection arrangement (Figure 5).

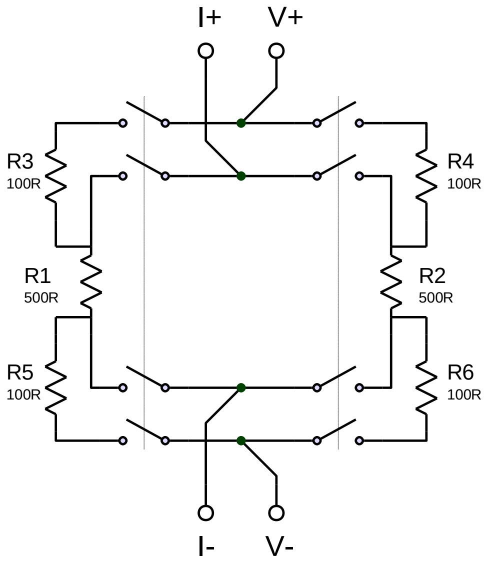

Figure 5: Resistance Box

When either switch is closed, the four-terminal resistance of the corresponding resistor (R1 or R2) can be seen at the four measurement terminals. When both switches are closed, a resistance nominally equal to the parallel combination of R1 and R2 is seen at the four measurement terminals. The function of R3 to R6 is to improve the precision with which the two four-terminal resistors are combined by sharing the inevitable voltage drops across the unwanted switch and wiring resistances between the resistance box’s potential terminals.

The equivalent circuit for the test box (with both switches closed) may reasonably be represented as:

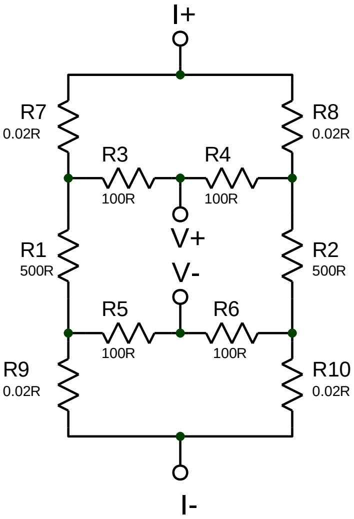

Figure 6: Equivalent Circuit for Resistance Box

Where R7 to R10 represent the unwanted resistances arising from switch contacts and wiring.

Ideally, the four-terminal resistance seen at the terminals is the parallel combination of R1 and R2. This only occurs if the system is completely symmetrical (R7 = R8, R1 = R2 and R9 = R10). Small imbalances in the values lead to a difference between the value at the terminals and value of the parallel combination of R1 and R2. This circuit is most readily analysed by assuming that the system is basically symmetrical and looking at the effect of any imbalances. The resistors R1 and R2 share the current approximately equally, so it is convenient to set up the network mesh with two current sources providing half the total current (Figure 7).

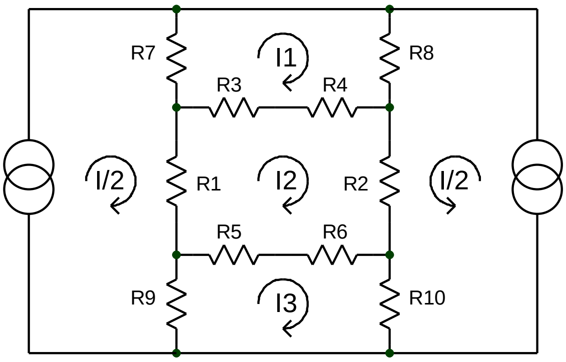

Figure 7: Equivalent Circuit

Although the circuit is quite simple, a full mesh analysis is somewhat tedious; the steps leading to the final equations are therefore not included in this paper. The difference between the resistance measured at the external terminals and the parallel combination of R1 and R2 is given by:

$$E = \frac{(R_7 - R_8)}{R_P}\left[\frac{(R_3 + R_4)K + R_3}{2(R_3 + R_4 + R_7 + R_8)}\right] + \frac{(R_9 - R_{10})}{R_P}\left[\frac{(R_5 + R_6)K + R_5}{2(R_5 + R_6 + R_9 + R_{10})}\right] + \frac{(R_1 - R_2)K}{2R_P} + \frac{R_1}{2R_P} - 1$$

Where:

$$R_P = \frac{R_1 R_2}{R_1 + R_2} \quad \left(\textit{the parallel combination of } R_1 \textit{ and } R_2\right)$$

$$K = \frac{\left[\frac{R_3(R_3 + R_4)}{R_3 + R_4 + R_7 + R_8} + \frac{R_5(R_5 + R_6)}{R_5 + R_6 + R_9 + R_{10}} - R_1 - R_3 - R_5\right]}{\left[R_0 - \frac{(R_3 + R_4)^2}{2(R_3 + R_4 + R_7 + R_8)} - \frac{(R_5 + R_6)^2}{2(R_5 + R_6 + R_9 + R_{10})}\right]}$$

$$R_0 = R_1 + R_2 + R_3 + R_4 + R_5 + R_6$$

These equations apply for any value of the resistors R1 to R10. The resistance box was made using Vishay bulk metal foil resistors for R1 and R2 and Tyco precision metal film resistors for R3 to R6. The specifications of the components used were:

R1-R2 = 500Ω ± 0.01%, 0.6ppmK^{-1}
R3-R6 = 100Ω ± 0.1%, 15ppmK^{-1}
R7-R10 < 0.02Ω

These components values allow the equations to be simplified. Care needs to be exercised in discounting terms, since we are looking for small second-order effects:

Since $(R_7+R_8) \ll (R_3+R_4)$ and $(R_9+R_{10}) \ll (R_5+R_6)$, K approaches:

$$K \to \frac{-R_1}{R_1+R_2}$$

We can see that the equation for E similarly reduces to:

$$E \approx (R_7 - R_8)\left[\frac{R_2R_3 - R_1R_4}{2(R_3+R_4)R_1R_2}\right] + (R_9 - R_{10})\left[\frac{R_2R_5 - R_1R_6}{2(R_5+R_6)R_1R_2}\right]$$

The validity of the approximation was confirmed by using an Excel spreadsheet with both the full and reduced equations. Discrepancies between the full and reduced equation (for example, from the effect of an imbalance in R1 and R2 alone) are below 1 in 10^{11} with the selected components.

The worst case error (R2, R3 & R5 high, R1, R4 & R6 low and with R8 and R10 set to zero) was calculated to be 0.04ppm. This is as a proportion of the parallel resistance, so as a proportion of each resistor (selected to generate full scale) the uncertainty is:

Uncertainty of Network << 0.02ppm of scale

With this number of variables, it is reasonable to use a statistical method for combining the contribution of the tolerances; this would significantly reduce the uncertainty associated with the parallel resistance realized. Also, the wiring in the resistance box was made symmetrically and with short, low resistance connections and measurements indicate that R7 to R10 were typically nearer to 2mΩ with a spread between the poles of the switch of less than 1mΩ. This gives another order of magnitude improvement in uncertainty.

Another source of uncertainty would be the output impedance of the current source. The voltage at its terminals would change by 2.5V, so a 10GΩ output impedance would yield a change in current of 250pA. This in turn leads to a voltage uncertainty of 62.5nV across the 250Ω, which is 0.0125ppm of the 5V range. This uncertainty source can be reduced to negligible proportions simply by switching a 250Ω padding resistor in series with the current source when the resistors are connected in parallel. The current source then sees a constant 500 Ω load and its output voltage remains effectively constant.

The mid-scale error of the ADC was checked using this technique by measuring the voltage across the two individual resistors, the parallel combination and the voltage with the current disconnected. The latter reading was subtracted from the other readings in order to eliminate the effect of thermal EMFs and offsets. The measured value for the parallel combination was then compared with the computed value to derive the mid-scale error. The results for the first batch of ten microK-400 production units are shown in Figure 8:

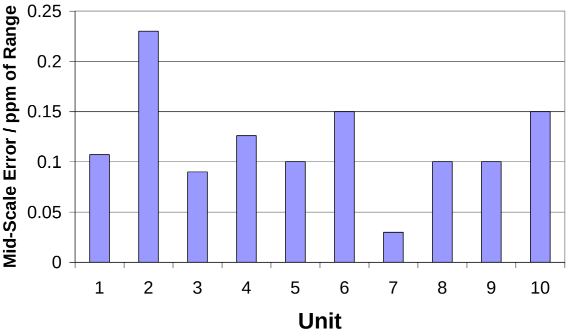

Figure 8: ADC Linearity Results

The mean mid-scale error is 0.12ppm with a standard deviation of 0.05ppm. These results confirm that the core ADC used in the microK-400 is comfortable within the 0.4ppm specification for the whole instrument.

A second resistance box containing two 10Ω bulk metal foil resistors was made in order to test the voltage measurement performance of the microK-400. When used with the 10mA current source this provided a 0 - 50 - 100mV voltage source. The other components used were unchanged, so that the worst case uncertainty at 50mV is 2ppm of value. As discussed earlier, this uncertainty can be expected to be much smaller and in practice, the overall measurement uncertainty will be limited by thermal EMFs (2ppm of 50mV being only 0.1µV).

USING AN RBC TO CHECK LINEARITY

The RBC (Resistance Bridge Calibrator) [2] was originally developed by Rod White of IRL (the New Zealand national laboratory). It is available as a commercial product from 2K Electronics.

The RBC contains four precision resistors that can be connected in 35 different series/parallel combinations and uses a similar but more sophisticated technique to the one described above (for combining two equal resistors) to ensure that the uncertainties arising from the switches are minimal. The specified accuracy is 0.1ppm; however, the RBC used in these tests has been compared with an ASL F900 resistance bridge (accuracy 0.02ppm) [3] and agreed with the bridge within 0.02ppm. Addition measures (detailed below) were taken in order to achieve this level of performance with the RBC.

The RBC is controlled using eight manual switches on the front panel. Early development work on the microK product used the RBC manually. A full 35 point measurement of a microK typically takes 2 hours with the RBC. With an increasing number of tests being required for ongoing development and production testing, we took the decision to automate the RBC.

The performance of the RBC is largely limited by the contact resistance of the switches. Since manual switches have much better on-resistance than relays, it was not possible to simply replace the front panel switches with relays. A system was therefore developed, using rotary servos designed for the remote controlled model market, to operate the RBC switches under the control of a PC. Software was then written to completely automate tests using the RBC.

This automation has enabled the engineering team to conduct a greater number of tests using the RBC since the cost (man-hours) is now minimal. Additionally, we are able to make better measurements. Tests now involve taking two sets of measurements, sequencing up through the RBC settings and then reversing the order for the second data set. Since the readings are taken at regular intervals, then by averaging the readings from the two data sets, any uncertainty sources that change linearly with time are effectively eliminated from the data set. This sort of measure would not have been acceptable for routine tests when using the RBC manually.

In order to reduce uncertainty, we were taking a number of readings (typically 30) for each RBC setting. However, as we increased the number of readings at each point, the standard deviations for the readings at each RBC setting did not appear to improve at the rate expected (the square-root of the number of readings). This was investigated by logging the readings for the RBC at a single setting over a 12 hour period. The readings were then decimated by calculating the rolling average with different numbers of readings in the average (specifically 1, 10, 30, 100, 300 and 1000). When the standard deviations for the decimated data were plotted against the reciprocal of the square-root of the readings used in the average, the results failed to follow the expected straight line (Figure 9).

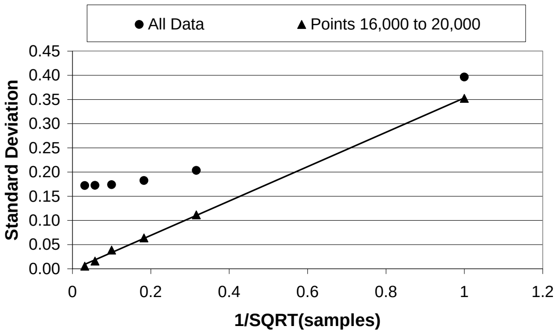

Figure 9: Noise versus Samples

Looking at the data as a function of time (Figure 10), we could see that there was a variation due to changes in the ambient temperature (the system was not operated in a temperature controlled environment. If we then performed the same analysis, but just using a sub-set of the data from readings 16000 to 20000, (where there is minimal change due to ambient temperature changes), we got a very good agreement with the predicted 1/(root samples) – see Figure 9:

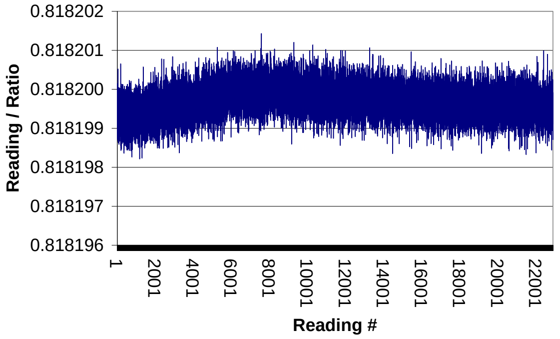

Figure 10: RBC Reading over 12 Hours

It is clear that as we increase the number of samples in order to reduce uncertainty caused by noise in the electronics, we increase the likelihood of uncertainties due to changes in the temperature of the resistors in the RBC and the reference resistor used (a Vishay bulk-metal foil resistor).

Since we were trying to operate in a laboratory without air-conditioning or good temperature control, we looked for other ways to improve our measurement capabilities with the RBC. The ambient temperature will affect the resistance of both the RBC and the reference resistor. A double skinned box was made for the RBC from 25mm thick expanded polystyrene, with a 1cm gap between the inner and outer boxes. Additionally, the reference resistor was potted into a cast aluminum box using thermally conductive epoxy. This was then placed into a stainless-steel vacuum flask normally used to store hot/cold drinks. The electrical connections were made using very fine (0.15mm diameter) enameled copper wires.

When this system was tested, we got a much better agreement with the expected form even when using the whole data set (Figure 11):

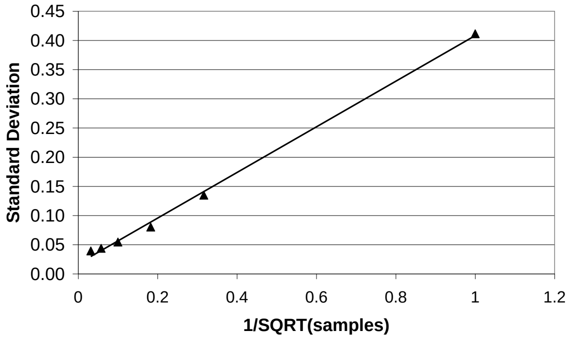

Figure 11: Noise versus Samples with Insulation

The data points are drifting away from the expected straight line as the number of sample increases, but this is much improved over the original results. These improvements to the test system cost less than $100 but allowed us to make measurement without investing in a costly air-conditioning system and oil maintenance baths and yet still achieve sub 0.1ppm uncertainty when checking the microK product.

A typical linearity result for a microK using the RBC (30 readings per RBC setting) is shown in Figure 12:

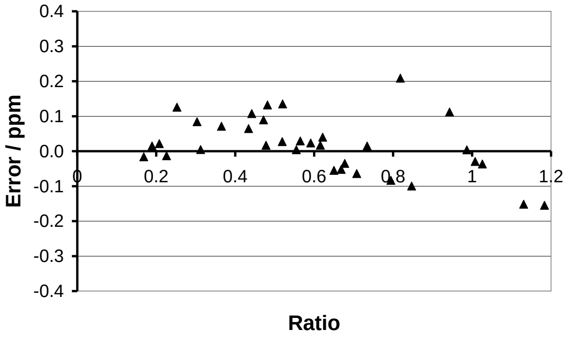

Figure 12: Typical RBC Linearity Check Result

The peak error values for the second batch of ten production microK-400 are shown in figure 13:

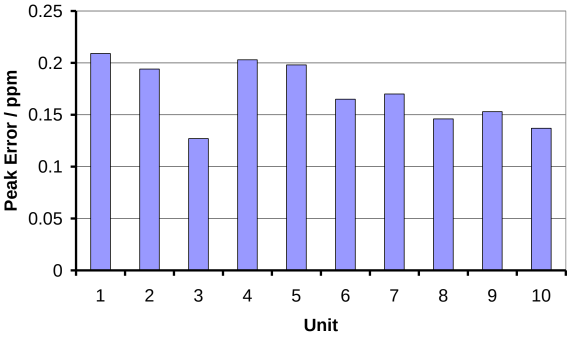

Figure 13: Peak Errors from RBC Checks

The mean of these peak errors is 0.17ppm with a standard deviation of 0.03ppm. These results confirm that the overall microK comfortably meets its performance specification.

PRODUCTION CALIBRATION

A similar approach has been employed when establishing the production test and calibration capability. The RBC is used to check the linearity of all units manufactured. The zero resistance performance is easily checked by applying a four-terminal short circuit connection to the microK. The unity ratio performance is confirmed by measuring two nominally identical resistors connected to two channels. These are then swapped over and the product of the two readings should then be unity (± root 2 of the specified accuracy). This test is performed on all permutations of the three input channels to ensure absolute confidence in the instrument prior to shipping. Finally the internal reference resistors (1, 10, 25, 100 and 400 ohms) are calibrated against Wilkins resistors that are maintained in an oil bath. These resistors have calibrations that are traceable to national standards and have a declared uncertainty of less than 0.05ppm, giving 0.07ppm calibration uncertainty.

The calibration of the microK's voltage ranges presented a more serious challenge, since we needed to provide a voltage source of 50mV with an uncertainty better than 0.25µV. Commercially available voltage sources struggled to meet this performance requirements and those that were the nearest proved to be very costly. In this case we again used the Metron Designs I-REF2. This has been calibrated at NPL (UK national standard laboratory) and is used with a range of Wilkins resistors maintained in oil baths to generate the required voltages. Using this approach, Isothermal Technology is able to achieve calibrations with an uncertainty of just 0.25µV.

CONCLUSION

Despite the very high specification of the microK instrument, it has been possible to develop inexpensive solutions that allow development testing and production calibration to be performed with uncertainties that would normally only be achievable using very expensive systems. Uncertainties of <0.1ppm for resistance ratio and 0.25µV for voltage have been realized.

REFERENCES

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2. D R White, K Jones, J M Williams and I E Ramsey, "A Simple Resistance Network for the Calibration of Resistance Bridges", IEEE Trans. Instrument Meas., IM-42, 5th Oct 1997, 1068-1074.
3. P Bramley, J Yewen and D Stanley. "A 20ppb Resistance Bridge for use in Thermometry" NCSLi, 2000.

ACRONYMS USED

ADC: Analog-to-Digital Converter
DUT: Device Under Test
EMF: Electro-Motive Force
RBC: Resistance Bridge Calibrator
SPRT: Standards Platinum Resistance Thermometer
UKAS: United Kingdom Accreditation Service