A Comparison-Calibration Apparatus Covering the Range 500°C to 1000°C

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Abstract

A comparison-calibration apparatus covering the range 500°C to 1000°C was developed. The core of the apparatus is made from a three-well, high-temperature equilibration block. Copper was chosen for the equilibration block because of its thermal conductivity. At high temperatures, copper oxidizes, but this problem was solved by sealing the copper block with argon gas in a silica glass shell. For SPRT calibrations, platinum thimbles were used in the block to protect the SPRT elements from contamination. Test results show the apparatus has excellent horizontal and vertical temperature gradients, and is well suited for comparison calibration of resistance thermometers and thermocouples.

Introduction

Fixed-point cells and related equipment are widely used for the calibration of primary standard thermometers. Fixed-point calibrations provide very high accuracy and do not require reference thermometers. For calibrations of secondary standards, baths and furnaces are preferred because of their efficiency and simple operation. From 0°C to 500°C, baths using water, oil, and molten salt are satisfactory for calibrating secondary standards. A fixed-point calibration method using smaller fixed-point cells has been introduced for secondary calibrations in the range from 0°C to 660°C [1]. Though air fluidized-bed calibration baths may be used up to 1100°C, their temperature stability and uniformity are insufficient for precise calibrations [2].

We are currently developing a new precision platinum resistance thermometer (PRT) with an upper temperature limit of 850°C. Developing a comparison calibration apparatus that would reach 850°C was therefore essential. The calibration uncertainty requirement for our new PRTs at 850°C is better than 30 mK. Because no commercial comparison apparatus is available for such high temperatures, a new comparison-calibration apparatus covering the range 500°C to 1000°C was developed.

Apparatus Structure and Design

The core of the comparison calibration apparatus is made from a three-well, high-temperature equilibration block (Fig. 1). Copper was chosen for the equilibration block because of its excellent thermal conductivity. At high temperatures, copper oxidizes, but this problem was solved by sealing the copper block with argon gas in a silica-glass shell. The three silica-glass thermometer wells inside the equilibration block were sandblasted to prevent light piping and the wells were sealed to the protective shell. For SPRT calibrations, platinum thimbles were used in the block to protect SPRT elements from contamination. The platinum thimbles extended slightly beyond the depth of the thermometer well in the block. The equilibration block was assembled into a nickel case. Several radiation shields, comprised of thin inconel disks separated by ceramic fiber, were assembled on the top and bottom of the equilibration block.

The comparison-calibration apparatus also included a sodium heat pipe furnace, Hart Model 9115 (Fig. 2). A heat pipe behaves as a tube exhibiting very high thermal conductivity. Its very large thermal conductivity establishes a highly uniform temperature [2][3]. The sodium pipe's working temperature range is from 500°C to 1000°C. Three silica glass thermometer wells, which were sandblasted, extend from the top of the comparison block to the top of the furnace, and serve as thermometer guide tubes. Improved vertical uniformity is accomplished by additional radiation shielding using several thin inconel disks and ceramic fiber assembled on top of the nickel case.

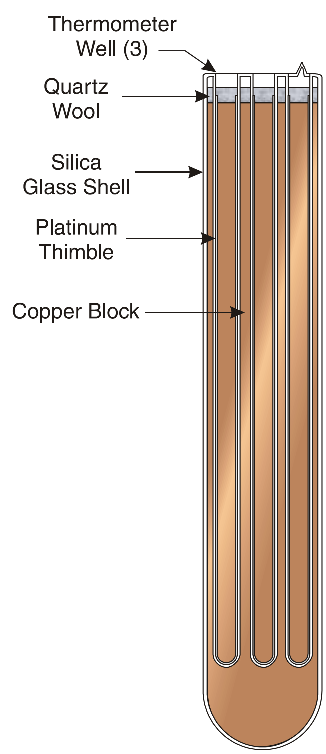

Fig. 1 Equilibration Block

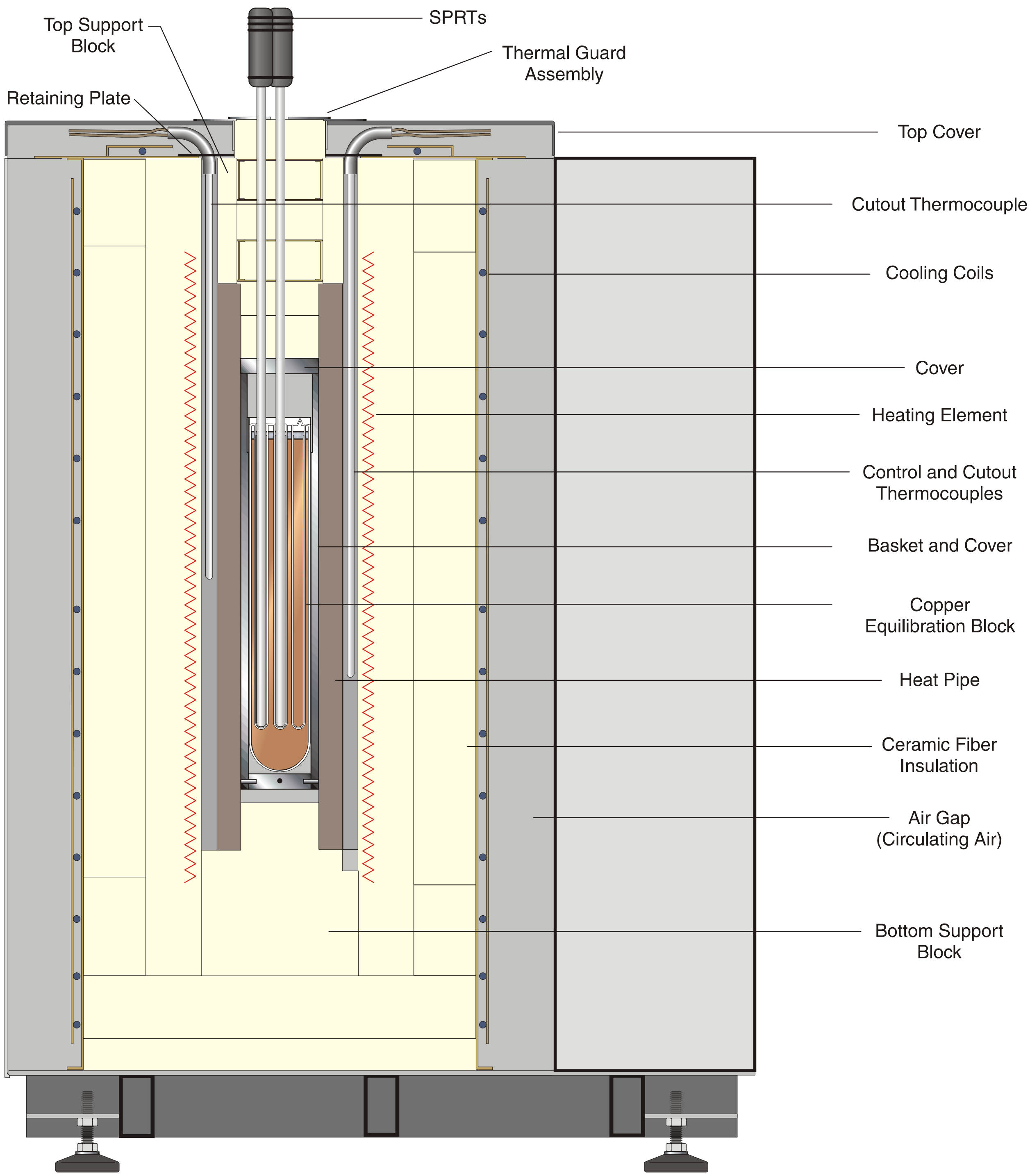

Fig. 2 Comparison-Calibration Apparatus

Testing and Results

Measurement Apparatus

Two Hart Model 5684 0.25-ohm high-temperature standard platinum resistance thermometers (HTSPRTs) were used to measure the temperature stability, vertical uniformity, and horizontal uniformity. HTSPRTs typically exhibit long-term stability better than 1 mK after 100 hours at 1085°C. A gold-platinum thermocouple, Hart Model 5629, was involved in the horizontal gradient test.

The resistance of the thermometer was measured with a digital data collection system, including two Hart Scientific Model 1590 Super-Thermometers. The Super-Thermometer achieves 1-ppm accuracy with an external standard resistor. Two Tinsley 1-ohm AC/DC standard resistors were used as the external resistors. The gold-platinum thermocouples were measured using a Keithley Model 2128 Nanovoltmeter.

Temperature Stability

The temperature stability of the comparison-calibration apparatus was measured at 500°C, 850°C and 1000°C. A comparison calibration can be completed in about 20 minutes. Three typical 30-minute curves at 500°C, 850°C, and 1000°C are shown in Fig. 3, Fig. 4, and Fig. 5 respectively. Test results demonstrated that the apparatus had excellent temperature stability. The peak-to-peak fluctuations within 30 minutes were less than 10 mK at 500°C, 20 mK at 850°C, and 25mK at 1000°C.

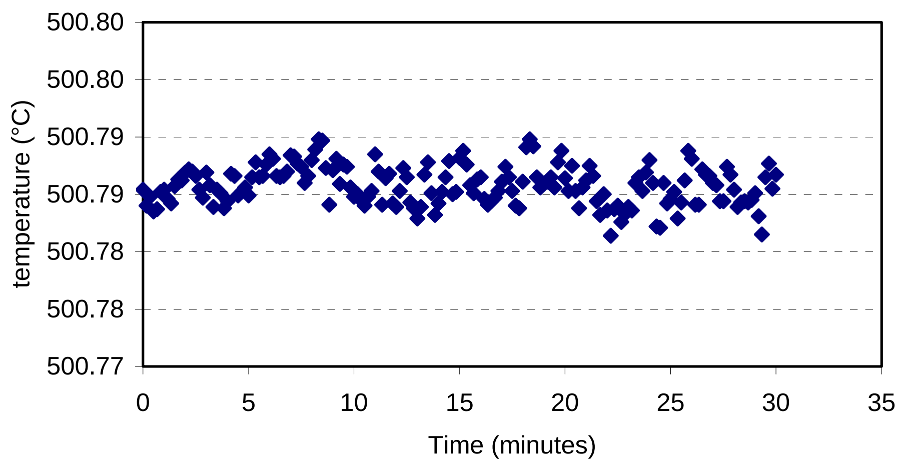

Fig. 3 Temperature stability at 500°C

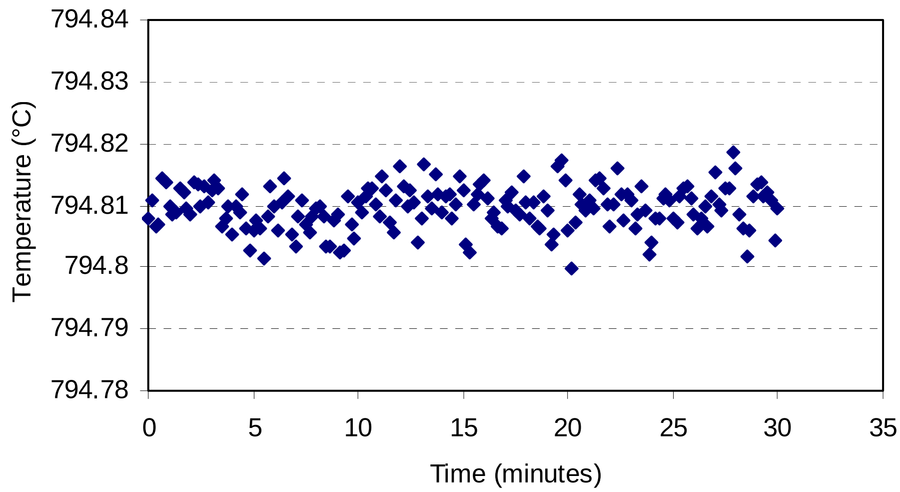

Fig. 4 Temperature stability at 850°C

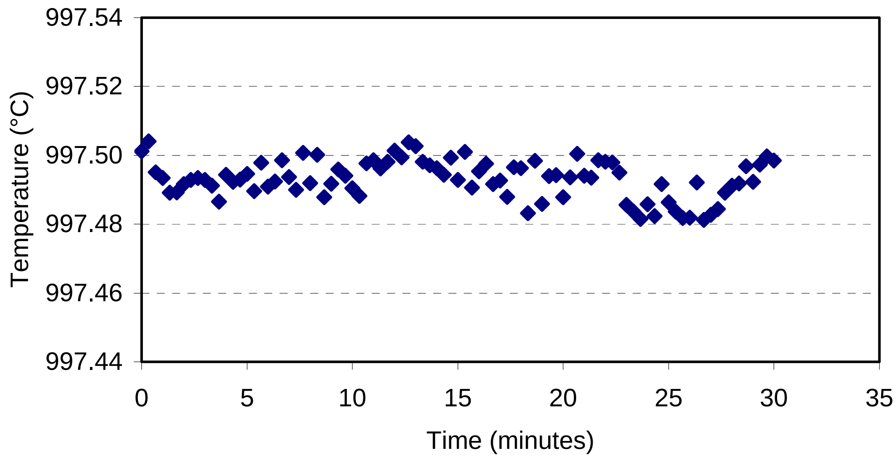

Fig. 5 Temperature stability at 1000°C

Horizontal Gradient

The horizontal gradient was measured using two 0.25-ohm HTSPRTs and two 1590 Super-Thermometers. The temperature differences among the three thermometer wells were measured as follows. First, the temperature difference between well #1 and well #2 was measured. Two HTSPRTs were respectively inserted into well #1 and well #2. Each HTSPRT was connected to a Super-Thermometer to allow the data to be recorded simultaneously. After the temperature stabilized, data was recorded for 10 minutes. The two HTSPRTs' positions were then reversed and the process was repeated. The average reading of the two HTSPRTs was calculated for each of the two wells as was the difference between the two averages. Repeating this method, the temperature difference between well #1 and well #3 was also measured. The temperature differences between the three wells at 500°C, 850°C, and 1000°C are listed in Table 1. The test results show the horizontal uniformity of the apparatus to be better than 1 mK at 500°C, 2 mK at 850°C, and 3 mK at 1000°C.

Table 1. Temperature differences among the three thermometer wells

	Well #2 to Well #1		Well #3 to Well #1		Well #3 to Well #2	
Run	A	B	A	B	A	B
500°C	-0.39 mK	-0.17 mK	+0.18mK	-0.12 mK	+0.57 mK	+0.05 mK
850°C	-0.44 mK	-0.44 mK	-0.98 mK	+0.77 mK	-0.54 mK	+1.21 mK
1000°C	+0.89 mK	-0.89 mK	+0.90 mK	-2.60 mK	+0.01 mK	-1.71 mK

The two HTSPRTs used in these measurements were identical in design and element lengths. This reduced the effect of vertical temperature gradients. Different element lengths could affect the calibration accuracy of the apparatus by introducing vertical temperature gradients into the calibration uncertainty.

In order to test the probable effect of different element lengths, horizontal gradients were also measured using an HTSPRT and a gold-platinum thermocouple. The element length of the HTSPRT was 40 mm, and the spring sensor of the gold-platinum thermocouple was 3 mm. Both the HTSPRT and the gold-platinum thermocouple were inserted into the bottom of the thermometer wells. Using the method described above, the temperature differences among the three wells were measured. The HTSPRT was then kept in the bottom of its well, while the sensor of the gold-platinum thermocouple was raised 40 mm from the bottom of its well. The temperature differences among the wells were then measured again. Comparing the results from the two methods, we found that the horizontal temperature gradients were similar to those measured by the two HTSPRTs if both the HTSPRT and the gold-platinum thermocouple were placed in the bottom of the thermometer wells. However, if the HTSPRT was placed in the bottom of a well and the gold-platinum thermocouple was raised to 40 mm from the bottom of a well, the differences among the three wells increases, but is still better than 6 mK at 850°C, as shown in Table 2.

Table 2. Temperature differences among the three thermometer wells using one HTSPRT and one gold-platinum thermocouple at 850°C

	Well #2 to Well #1	Well #3 to Well #1	Well #3 to Well #2
Both HTSPRT and gold-platinum TC at the bottom	-1.0 mK	-1.9 mK	-0.9 mK
HTSPRT in bottom, gold-platinum TC was raised 40 mm from the bottom	-5.5 mK	-4.2 mK	+0.7 mK

Vertical Gradient

The vertical gradient was measured using two 0.25-ohm HTSPRTs and two 1590 Super-Thermometers. Each HTSPRT was connected to a Super-Thermometer to allow the data to be recorded simultaneously. One HTSPRT was used to monitor the apparatus' temperature stability

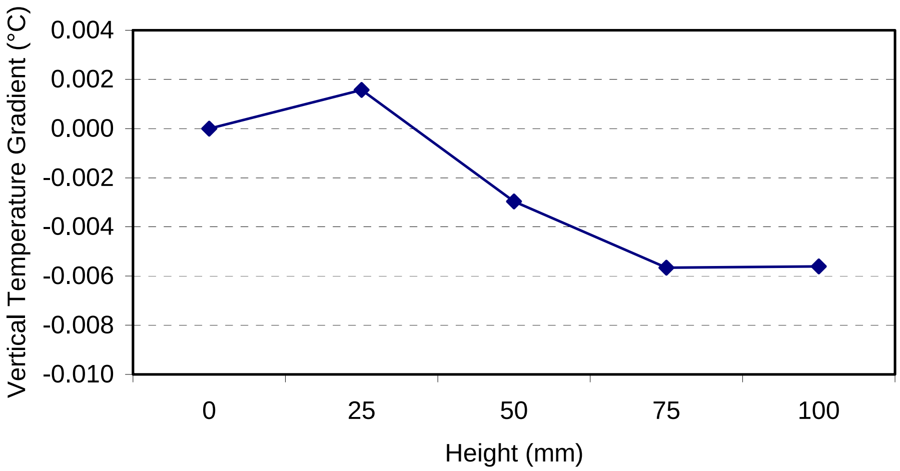

Fig. 6 Temperature vertical gradient at 500°C

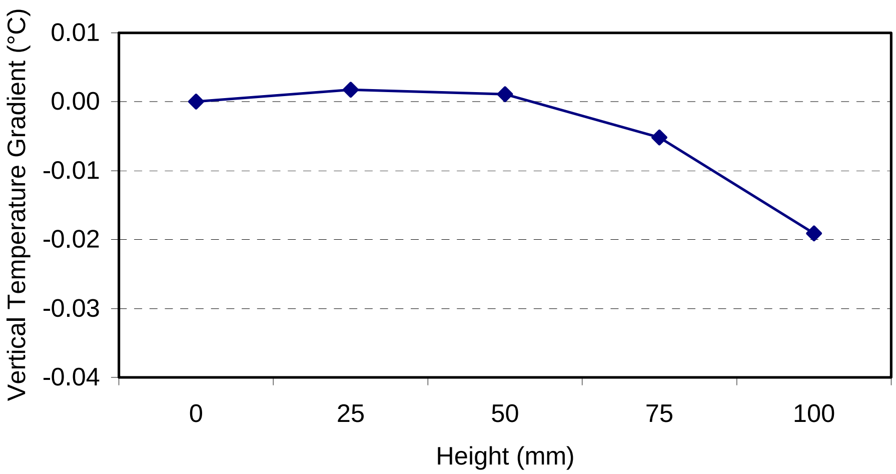

Fig. 7 Temperature vertical gradient at 850°C

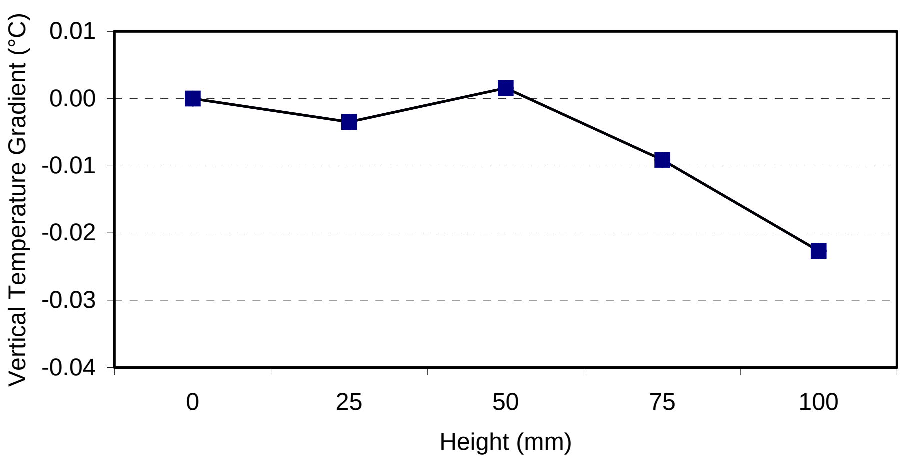

Fig. 8 Temperature vertical gradient at 1000°C

while the other HTSPRT was moved up 100 mm from the bottom of the thermometer well and then back to the bottom. The HTSPRT was moved in 25 mm increments and measurements taken at each depth. After every move and before each measurement, a four-minute stabilization period was allowed. The vertical temperature gradients, corrected for temperature stability, are shown in Fig. 6, Fig. 7, and Fig. 8. The results indicated the 100 mm vertical uniformity to be better than 8 mK at 500°C, 20 mK at 850°C, and 25 mK at 1000°C. At 50 mm from the well bottom, the vertical temperature gradients were within 5 mK over the full temperature range. This is very important because most elements are shorter than 50 mm.

Conclusions

This comparison-calibration apparatus may be used to calibrate and test precision thermometers and thermocouples in the range from 500°C to 1000°C. The temperature difference between the thermometer wells is less than 1 mK at 500°C, 2 mK at 850°C, and 3 mK at 1000°C. The vertical temperature gradient up to 50 mm from the bottom of the well is less than 5 mK over the entire temperature range. When the probes under test have elements of different lengths, the temperature uniformity is better than 6 mK. The apparatus has excellent horizontal and vertical temperature uniformity, and is well suited for comparison calibration of precision resistance thermometers and thermocouples.

Testing will continue. Several HTSPRTs and gold-platinum thermocouples with fixed-point calibration data will be calibrated using the comparison method in this apparatus. The comparison calibration results will be compared with the fixed-point calibration data.

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