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# Multi-Entrance Fixed Points as an Alternative to the Equalizing Block

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**Abstract** The uncertainty of comparison calibrations is often limited by temperature gradients and the stability of the isothermal zone. Traditionally, an equalizing block inserted into a bath or furnace is used to provide an isothermal zone during calibration. In this paper, the design and performance of a multi-entrance fixed point (MEFP) filled with mercury is described, with the goal to replace an equalizing block with the phase transition of mercury during melting/freezing. During this investigation, three thermometers were calibrated against a standard in the same bath at −39 °C, using both the standard equalizing block and MEFP. The measurement results clearly indicate that use of MEFP decreases the uncertainty contributions from temperature gradients and the stability of the bath by an order of magnitude compared to the standard equalizing block.

**Keywords** Comparison calibration · Equalizing block · Multi-entrance fixed point

## 1 Introduction

Dissemination of the International Temperature Scale (ITS-90) to lower-grade standards in Croatia [1] is, as elsewhere, achieved through comparison calibration. Some of the main uncertainty components in the uncertainty budget of the calibrated thermometer are the radial and axial gradients in the isothermal zone.

Comparison calibration in an isothermal block when low uncertainties are required is time consuming as it takes a long period of time for the system to achieve equilibrium. When several thermometers are to be calibrated using a high-precision bridge with

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a balancing time of approximately 20 s, the stability of the bath/furnace comes into play, as a longer time is needed for calibration. Then, the temperature of the isothermal zone may shift significantly between reading the standard thermometer and the devices under test. Generally, comparison calibration with low uncertainties requires either a bath/furnace with an advanced control system or a furnace with a heat pipe, with both solutions being expensive.

In order to investigate the possibility of economically improving our comparison calibration capability using available equipment, the principle of the phase transition used in standard temperature fixed points was adopted for comparison calibration. The phase transition in such a calibration is not intended to be the temperature standard itself, but only provides spatial thermal homogeneity and temporal stability. The multi-entrance fixed point (MEFP) with four entrance tubes, allowing simultaneous calibration of three thermometers against the standard thermometer, was designed in the Laboratory for Process Measurements. This concept allows the use of modest purity materials to fill the cell, since a standard thermometer is used for the calibration. For the same reason, the difference in pressure from nominal during the phase transition is unimportant. A bath/furnace with a poor controller setting can be used because the temperature arrest provided by the change of phase will provide ample temperature stability and uniformity.

## 2 Design and Fabrication of the MEFP

The starting point for the design of the MEFP was a standard single re-entrant well fixed point, as used for realization of the ITS-90 [2]. As the MEFP has to accommodate several re-entrant wells for the thermometers to be calibrated, a cell of larger diameter was adopted. The MEFP was designed from stainless steel, since a workshop with experience in stainless steel welding was available. Also, the handling of the larger amount of mercury is more assuring in a metal rather than a borosilicate glass container. The cell was manufactured from Type 304 stainless steel with four re-entrant wells, with an inner diameter of 8 mm. The re-entrant wells are welded together 30 mm from the bottom of the cell, and protrude from the cell in a radial pattern, in order to assure space for the thermometer heads (Fig. 1). The length of the tubes is 350 mm, and the distance between them at their top is 35 mm. This design assures that the thermometer sensors at the bottom of the re-entrant wells are surrounded by the largest amount of mercury, preventing formation of a ‘bridge’ during the phase transition. The cell itself has an outer diameter of 63 mm, allowing for 20 mm of mercury from the cell’s inner surface to the thermometer well. The length of the cell is 250 mm. Three small plates with two 5 mm holes were welded at the top of the cell to hold and position the cell in the bath, and the filling tube of 5 mm i.d. was welded in place with a needle valve at the top.

Prior to welding, all cell parts that come into contact with mercury were polished and were degreased with detergent. As the temperature of the phase transition in the cell is to be measured with a calibrated SPRT, no special care was taken to remove all residual traces from the cell that might contaminate the mercury and affect the temperature of the transition.

**Fig. 1** Design of the MEFP. Dimensions are in the text

The MEFP was filled with readily available mercury of moderate but untested purity. The Laboratory for Process Measurements, established some 50 years ago, maintains national standards for temperature and pressure. The pressure department [3] has a long tradition of precise measurements in both gauge pressure and barometric pressure, and over time an ample amount of leftover mercury from different U-tubes and barometers was collected. High-purity mercury is used for manometers, and it was assumed that it is sufficiently clean to fill the MEFP, even after being stored in glass bottles for a certain period of time. Such samples of mercury were used to fill the cell instead of being sent for recycling. For the filling process itself, a simple pouring method was used, whereby a large syringe with a 100 mm long needle was positioned inside the filling tube. The mass of the mercury poured into the cell was 6,250 g, which was enough to have a 150 mm column of mercury around the re-entrant well and 30 mm of mercury between the bottom of the cell and the wells. With the cell being 250 mm long, the upper 70 mm of the cell was left filled with air. Filling the cell completely with mercury would add 2,500 g of extra mass, but might also provide a suitable point for a bridge across the liquid/solid interface to form, as in this section the re-entrant tubes are closer to the cell's inner surface. Thus, the air gap at the top of the cell isolates that part of the re-entrant tubes from the cell walls and the bath, thermally anchoring the wells to the phase-transition mass at the same time.

## 3 Bath and Measurement Equipment

For the measurements, the same bath was used for the calibrations in the equalizing block and those in the MEFP. It is a commercially available parallel-tube bath, capable of covering the range from (−60 to 300) °C, depending on the working fluid used. The working volume is 350 mm × 90 mm. The fluid volume is approximately 8 l, and denatured ethyl alcohol was used for the calibration around −40 °C. An external immersion chiller was used for cooling. An equalizing block made of copper originally supplied with the bath was used. It has a 50 mm outer diameter, and a length of 150 mm. Four holes of 8 mm inner diameter and 120 mm in length are drilled into the block to accommodate thermometers. The depth of immersion was 350 mm, and the block was completely submerged in the alcohol to improve thermal contact in the thermometer wells.

The temperature measurements were performed with SPRTs, both metal- and quartz-sheathed, and the usual metal-sheathed Pt100s. No significant difference was observed that depended on the type of thermometer. The thermometer resistances were measured with an ASL F700B AC resistance bridge, which when used with a 25.5 Ω standard resistor gives a resolution of 0.25 mK. The bridge was connected to a 10-channel scanner, and all the data were collected via an IEEE-488 connection to a laptop computer. The average balance time for the bridge is (18–20) s, giving approximately 80 s for the full cycle with four thermometers. For ease of data interpretation, 30 s of balance time was selected throughout the test, making 2 min for the full cycle. The standard resistors were Wilkins-type (25.5 and 100) Ω, and were kept in their original thermostatted enclosures.

## 4 Measurement Results

The measurements in both the equalizing block and in the MEFP used the same bath, thermometers, and resistance bridge. The data were collected in the same manner to avoid biasing the results.

Results of the gradient and stability testing of the equalizing block are presented in Fig. 2. Four thermometers were placed in their respective holes, and foam insulation was applied to the thermometer parts protruding from the bath. After the bath stabilized at the set-point value, the scanning cycle was triggered. In each measurement cycle, all four thermometers were read after 30 s of bridge balance time. In total, 90 measurements were taken within a period of 180 min. To aid the visual representation of the thermal stability, the measured resistances of the three thermometers were offset in order to plot all readings on the same chart. To calculate the magnitude of the shift, mean values for all thermometers were calculated during a 30 min window (all thermometers were measured on the melting plateau). Then, for each thermometer, the difference between the mean of the standard thermometer and the mean of the respective thermometer was added.

As presented in Fig. 2, the readings of all the thermometers varied during the measurement period by approximately 15 mK due to instability of the bath. The

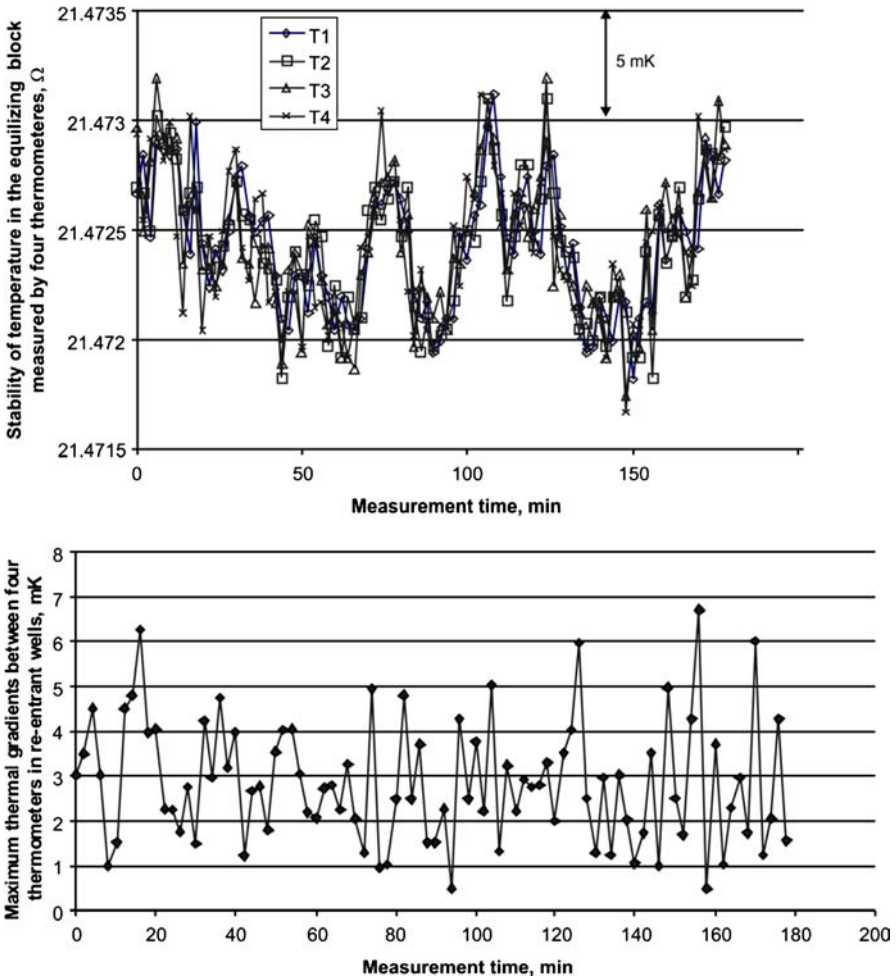


**Fig. 2** Gradients and stability measured in the equalizing block

maximum difference between the thermometer wells due to thermal gradients in that period was in the range from (1 to 6) mK.

The testing of the MEFP is presented in Figs. 3–5. The MEFP was submerged into the ethyl alcohol, and the same type of thermal insulation was applied to the thermometer stems protruding from the bath. The depth of immersion was approximately 350 mm, similar to that for the equalizing block. Ethyl alcohol was poured into the re-entrant wells to improve thermal contact. The MEFP was tested with two bath set-point temperatures for freezing and two bath set points for melting. In both cases, the procedure was the same as for the tests in the equalizing block. The thermometers were placed in their re-entrant wells and the scanning sequence started as the temperature of the bath approached the freezing point. No inner melt or freeze around the thermometer wells was initiated, leaving only the phase transition front

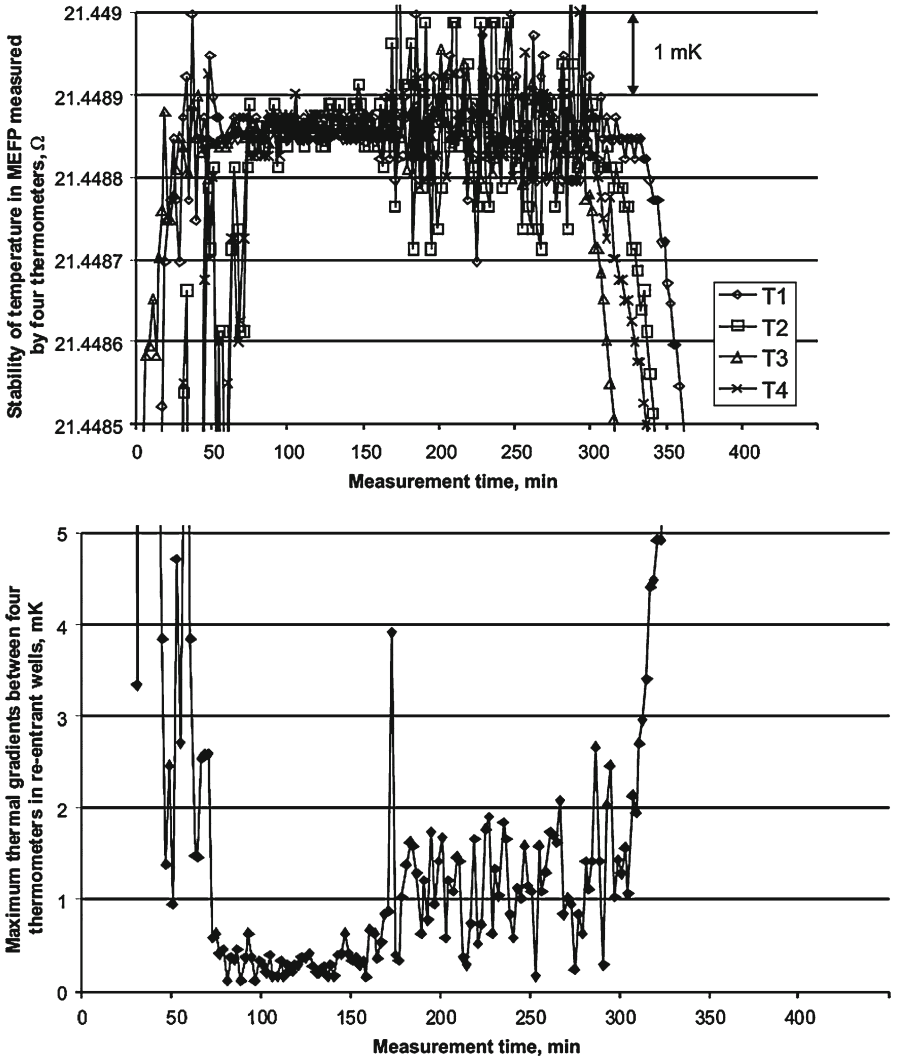


**Fig. 3** Gradients and stability measured in the MEFP during freezing, with the bath set point 0.4 °C lower than the freezing point

(both melting and freezing) to advance from the outer surface of the MEFP toward the thermometer wells. A balance time of 30 s before reading was assigned to each thermometer, making a total time of 2 min to scan four thermometers.

The purpose of the bath set points 0.4 °C lower than the freezing point and 0.4 °C above the melting point is to test the MEFP with the same temperature conditions as used for the standard mercury fixed point. In this thermal environment, the MEFP exhibits supercooling of 0.3 °C, and a certain time is required for the thermometers to stabilize after crystallization begins. The outcome of such a measurement is shown in Fig. 3. For the purpose of graphical representation, the values of the

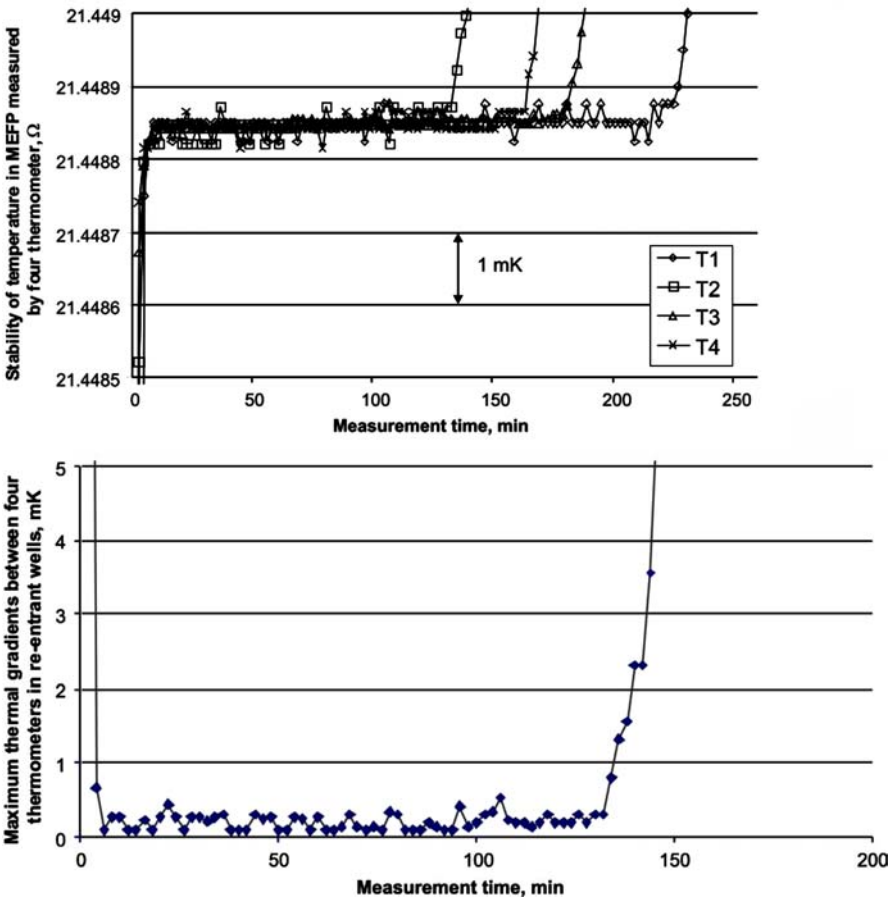


**Fig. 4** Gradients and stability measured in the MEFP during melting, with the bath set point 0.4°C higher than the melting point

thermometers are shifted, as described before. After stabilization, the thermometers in the MEFP showed stability better than 5 mK over 200 min, and better than 1 mK for a duration of 80 min. The difference between the re-entrant wells was smaller than 2 mK for 200 min and smaller than 0.5 mK for 80 min. During the melting of the MEFP with a temperature offset of 0.4 °C, better results were obtained, as presented in Fig. 4. The stability of thermometers was maintained for 120 min within 0.5 mK, while the maximum difference between the re-entrant wells was less than 0.5 mK for 120 min. During melting and freezing of the MEFP with a small temperature offset, the MEFP gives long but unstable plateaux due to the formation of a bridge across the liquid–solid interface. Depending on the starting point for nucleation, some thermometers would stabilize before the others. Also, for an average comparison calibration, there is no need for plateaux longer than required to read 10 measurements of all four thermometers. This is why the melting and freezing of the MEFP with larger temperature offsets was started, yielding shorter but more stable plateaux. When a bath set point 1.5 °C

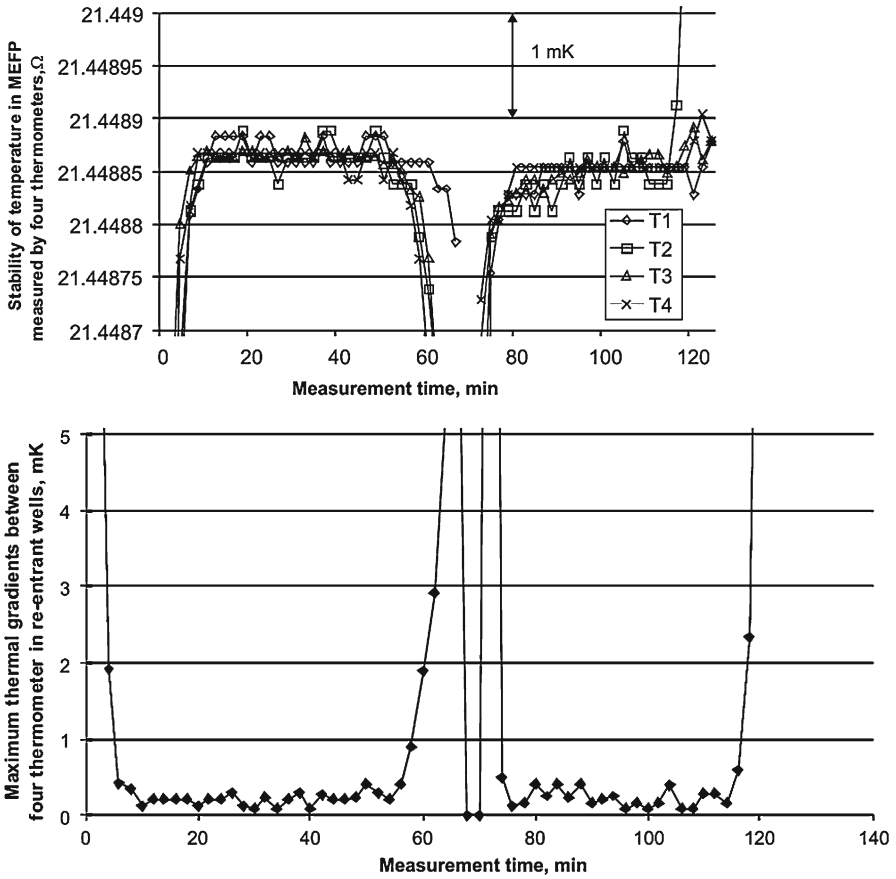


**Fig. 5** Gradients and stability measured in the MEFP during freezing and melting. The bath set-point temperature for freezing was 1.5°C lower than the freezing point, and the set-point temperature for melting was 1.5°C higher than the melting point.

lower than the freezing point and 1.5 °C higher than the melting point was applied, the four thermometers stayed on the plateau during melting and freezing for typically (35–40) min, as presented in Fig. 5. During this period, the difference between the re-entrant tubes would not exceed 0.5 mK, and the melt and freeze can be executed in a shorter period of time. This setup also better imitates the situation where a bath with larger gradients and poorer stability is used.

The MEFP was not evacuated prior to closing; thus, all phase transitions occur at temperature 3 mK higher than the mercury triple point, with a reproducibility of 0.3 mK. Measurements performed with the mercury inside the MEFP in the all-solid or all-liquid state were compared with the measurements in the equalizing block. The results were almost identical, with the temperature inside the MEFP clearly following the instability of the bath, while gradients were of the same magnitude as in the equalizing block, an order of magnitude larger than those measured during the phase transition.

## 5 Conclusions and Perspectives

During testing, the concept of the MEFP showed both strong and weak points. Uncertainties due to instability and gradients in the bath at −40°C were lowered by an order of magnitude, with the same equipment and with very small costs. Less time is required for the calibration, and the calibration of multiple thermometers can be automated. On the other hand, the weak points are that the phase transition in the MEFP, unlike the heat pipe, occurs at only one temperature, and unlike the fixed point, a calibrated standard is needed. The first issue can be addressed with the production of several MEFPs with careful selection of cell fillings to cover a certain temperature range. Low melting-point alloys used for prototyping in industry are already available at low cost, with melting points in the range from (57 to 300) °C, depending on the composition. Suitable materials must have a single melting point, as for example in the low-range alloys based on gallium ((11 and 29.7) °C), bismuth ((47, 60, 70, 92, 100, and 271) °C), indium ((125, 135, 150, and 157) °C), tin ((183, 199, and 232) °C), and lead ((292, 304, 315, and 327) °C). For higher temperatures, eutectic alloys based on zinc, gold, and aluminum ((356, 363, 382, 424, 525, and 577) °C) should be investigated.

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