SMALL FIXED-POINT CELLS FOR USE IN DRY WELL BLOCK CALIBRATORS

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ABSTRACT

As part of a research project for the Combined Calibration Group (CCG) of the U.S. Armed Forces, three rugged fixed-point cells were developed for use in dry well block calibrators (DWBCs). The small fixed-point cells of the water triple point (0.01 °C), the Ga melting point (29.7646 °C), and the In melting point (156.5985 °C) are intended to provide either calibration temperature points for industrial platinum resistance thermometers (IPRTs) or single-point calibration checks on IPRTs for the CCG mobile calibration facilities. While the ITS-90 indium fixed point is defined as a freezing point, realization of the three fixed-point cells in the melting mode simplifies the realization process, yet still provides the required expanded uncertainty (k=2) of less than 10 m°C. These three fixed-point cells were tested in two DWBCs using standard platinum resistance thermometers (SPRTs) by measuring realization plateaus and by direct comparisons with the PRT Laboratory reference cells. Adequacy of immersion of the thermometers in the cells during realization of the fixed points in the DWBCs was checked and the results were used to quantify the immersion uncertainty. This paper presents the realization methods, behavior of the fixed-point cells in the DWBCs and the uncertainty assigned to each of cells.

1. INTRODUCTION

The use of fixed-point cells, defined on the International Temperature Scale of 1990 (ITS-90) is usually reserved for the calibration of standard platinum resistance thermometers (SPRTs) [1]. A research project, funded by Combined Calibration Group (CCG) of the U.S. Armed Forces, was undertaken to study the effectiveness of using small rugged versions of three ITS-90 fixed-point cells for use in commercially-available dry well block calibrators (DWBCs) to calibrate industrial platinum resistance thermometers (IPRTs). The three fixed-point cells used in this study are the triple-point of water [(TPW), 0.01 °C], the gallium melting point [(Ga MP), 29.7646 °C], and the indium freezing or melting point [(In FP/MP), 156.5985 °C]. The use of these fixed-point cells with a DWBC both minimizes the amount of calibration equipment needed to calibrate IPRTs and extends the calibration interval of the reference thermometers that are used in the CCG mobile calibration facilities.

The original design specifications for the small fixed-point cells were created by the Thermometry Group at the National Institute of Standards and Technology (NIST) and subsequently made available to two fixed-point cell manufacturing companies to bid on the fabrication order. The designs of the fixed-point cells are similar to that of the six Standard Reference Material® (SRM®) small fixed-point cells (SRMs 1968 to 1973), but with a more rugged outer enclosure for industrial use [2]. The commercial fabrication of the fixed-point cells facilitates the commercial dissemination of the ITS-90 to industry. To eliminate the need for additional equipment, the fixed-point cells are designed to slip fit in the standard 2.54 cm inner diameter (i.d.) chuck hole of a DWBC, so that the calibrator itself becomes the thermal enclosure used to realize the fixed-point. The application of the cells in the melting mode simplifies the realization of the three fixed points, while still meeting the CCG requirement of an expanded uncertainty (k=2) of 0.01 °C.

This paper describes the realization methods, the thermal characteristics during realization, and the uncertainty assigned to each of the fixed-point cells when used in a DWBC.

2. DESIGN OF SMALL FIXED-POINT CELLS

Two basic designs are used, one for the Ga and In fixed-point cells and one for the TPW cell. These small fixed-point cells of rugged design were fabricated with no fragile components and an outer diameter (o.d.) to allow a close fit in the 2.54 cm i.d. chuck hole of the DWBC. The 0.59 cm i.d. of the thermometer well of the fixed-point cells was chosen to provide a slip fit for good thermal contact with IPRTs and metal-sheathed SPRTs used in the CCG mobile calibration facilities.

The Ga and In fixed-point cells use a polytetrafluoroethylene (PTFE) inner crucible, well and cap to contain the metal sample and maintain purity. The metal sample purity is stated to be ≥ 99.9999 wt. % pure by the manufacturer of the fixed-point cells. The o.d. of the stainless steel outer shell is 2.4 cm and 12.7 cm in length. Approximately 133 g and 164 g of Ga and In are contained in the PTFE crucibles, respectively. The immersion depth (inside bottom of the stainless steel thermometer well to liquid metal surface) during realization is 7.5 cm. The electro-polished stainless steel outer enclosure is welded shut for an internal pressure of 77.5 kPa of Ar for In and 84.1 kPa of Ar for Ga during realization of the respective fixed points. Additionally, the rugged outer shell of stainless steel minimizes the possibility of contamination of the metal sample and accidental breakage of the fixed-point cell during use in an industrial environment.

The TPW cell is an electro-polished stainless steel crucible that is welded shut to provide a triple point during realization. Approximately 36 cm^3 of triple-distilled water is contained in the 2.4 cm o.d. and 12.7 cm length stainless steel crucible. The immersion depth (inside bottom of the stainless steel thermometer well to liquid water surface) during realization is 10 cm. It was decided that not using a PTFE inner crucible would improve the thermal contact between the thermometer and liquid-solid interface and that an electro-polished stainless steel crucible would not significantly leach impurities into the water over time. Using the first cryoscopic constant and Raoult's Law of Dilute Solutions [3], impurities in the water would need to reach an estimated concentration of about 100 µg/g to exceed the required expanded uncertainty (k=2) of 0.01 °C. The wall thickness of the thermometer well must be thin enough to provide good thermal contact, but thick enough to minimize the possibility of inwardly collapsing the thermometer well during the freezing of the water. As in metal fixed-point cells, the rugged stainless steel enclosure minimizes the possibility of contamination of the water sample and accidental breakage of the fixed-point cell during use in an industrial environment.

3. QUALIFICATION AND REALIZATION METHODS

3.1 Qualification of the small rugged fixed-point cells

The qualification of the small fixed-point cells performed in each DWBC included tracing three realization plateaus in a melting mode (and freezing mode for In), an immersion profile of an SPRT and IPRT in the fixed-point cells during realization, and three direct comparisons with the appropriate NIST reference cell. Using two DWBCs from different manufacturers yields information on the dependency of the realization temperature as a function of the DWBC. The melting and freezing curve plateaus are used to estimate the amount of impurities in the fixed-point sample. The duration of a realization plateau is a function of the DWBC set-point temperature. The effect of the DWBC set-point temperature on the plateau slope was determined by changing the DWBC set-point temperature for different plateau realizations. The immersion profiles of the SPRT and IPRT are used to estimate the amount of heat flux associated with the combined interaction of a DWBC, the small cell and thermometer during realization of the ITS-90 fixed-point temperature. The direct comparison measurements with the reference cell determine the relative hotness difference between the two cells. An additional test applied to the TPW cell is to test for a water hammer by shaking the TPW cell.

The two DWBCs cover a temperature range of at least –25 °C to 160 °C, thus allowing all three cells to be realized in each DWBC. The depth of the 2.54 cm chuck hole for both of the DWBCs is 14 cm with the heater zone covering the bottommost 12.7 cm. Using an SPRT the measured stability of these two DWBCs is no worse than ± 3 m°C at 0 °C, ± 2 m°C at 30 °C, and ± 10 m°C at 157 °C over ten hours. On the average, the vertical gradients (5 cm above the bottom and to the bottom of the thermometer well) of the two DWBCs were +0.015 °C, –0.015 °C, and –0.13 °C at 0 °C, 30 °C, and 157 °C, respectively. The digitally-displayed control set points of the DWBCs were incorrect by as much as 2 °C over the entire range.

The computer-controlled measurement system used to test the quality of the small fixed-point cells consisted of a 9½ digit ac resistance ratio bridge, thermostated (25 °C ±0.008 °C) ac/dc reference resistors, a scanner, two metal-sheathed SPRTs, and one metal-sheathed 100 Ω IPRT. Details of the NIST PRT Laboratory measurement system are found in Reference 4. The thermometer excitation currents used for the plateau measurements were 1 mA and for both the heat flux and comparison measurements were 1 mA and 1.41 mA. After each experimental measurement set on each fixed-point cell, the thermometer was measured in the reference TPW cell.

Immersion profiles of a metal-sheathed SPRT in the three small rugged fixed-point cells during realization were performed to estimate heat flux uncertainty. The SPRT was measured on insertion into the thermometer well of the realized fixed-point. The estimated heat flux uncertainty for the interaction between the DWBCs, thermometer, and the realized fixed-point cells is 0.2 m°C, 0.2 m°C and 1.1 m°C, for the TPW, Ga MP, and In FP/MP cells, respectively.

3.2 TPW cell realization method

To realize the TPW in a melting mode, the cell is first placed in the DWBC, and the DWBC is then set at –5 °C to completely freeze the water sample over two hours. Second the DWBC is set at 0.1 °C to bring the cell to a temperature hotter than realization temperature to initiate the outer liquid-solid interface. Third, when the DWBC reaches the set point temperature of 0.1 °C, two copper rods are alternatively placed in the thermometer well for one minute each to induce the inner solid-liquid interface. Fourth, and finally, the block-chilled SPRT is placed in the thermometer well and thirty minutes are allowed to elapse for the thermometer and the TPW cell to thermally equilibrate prior to measurements. The outer liquid-solid interface, which acts as a buffer from temperature fluctuations of the DWBC, slowly melts inward during the realization. Plateau examples of the TPW cell in a melting mode realization for both DWBCs are given in Figure 1. The average melting range (0 % to 100 % liquid) for the TPW cell for six realizations is 1.0 m°C with a s.d. of 0.1 m°C.

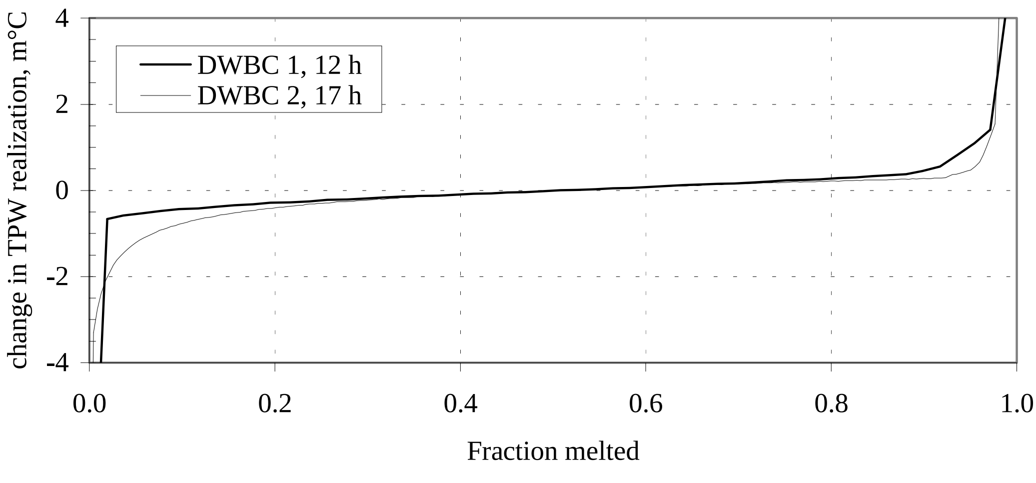

Figure 1. Plateau examples of the TPW cell realized in a melting mode in two DWBCs.

3.2 Ga MP cell realization method

To realize the Ga MP, the cell is first placed in the DWBC, and the DWBC is then set at –20 °C to completely freeze the Ga sample over two hours. Second the DWBC is set at 30 °C to bring the cell to a temperature hotter than realization temperature to initiate the outer liquid-solid interface. Because Ga MP cell is designed for industrial uses with an expanded uncertainty (k=2) requirement of 0.01 °C, no inner liquid-solid interface is induced. Third, and finally, when the DWBC reaches the set point temperature, thirty minutes are allowed to elapse for the thermometer and the Ga MP cell to thermally equilibrate prior to measurements. Plateau examples of the Ga MP cell in a melting mode for both DWBCs are given in Figure 2. The average melting range (0 % to 100 % liquid) for the Ga MP cell for six realizations is 0.8 m°C with a s.d. of 0.1 m°C.

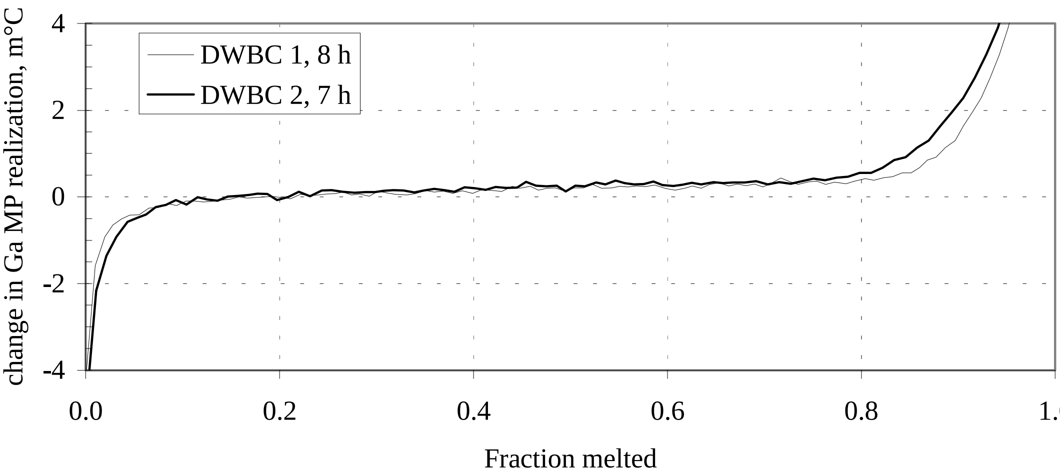

Figure 2. Plateau examples of the Ga MP cell realized in two DWBCs.

3.3 In FP and In MP cell realization methods

The In fixed-point cell may be realized either as a FP or as a MP. The In FP is the preferred realization method with the smallest uncertainty. The In MP, however, is easier to realize in an industrial environment.

To realize the In FP, the cell is first placed in the DWBC, and the DWBC is then set at 160 °C to completely melt the In sample over three hours. Second the thermometer is placed in the thermometer well and the DWBC is set at 150 °C to bring the In sample to a temperature lower than realization temperature to supercool the In sample and initiate the outer liquid-solid interface. Third, when the thermometer indicates recalesence, the DWBC is set at 156.1 °C and the thermometer is removed for one minute, reinserted for one minute, removed for one minute and then reinserted to induce the inner solid-liquid interface. Fourth, and finally, thirty minutes are allowed to elapse for the thermometer and the In cell to thermally equilibrate prior to measurements. The outer liquid-solid interface, which acts as a buffer from temperature fluctuations of the DWBC, slowly freezes inward during the realization. Plateau examples of the In cell in a freezing mode realization for both DWBCs is given in Figure 3. The average freezing range (0 % to 100 % liquid) for the In cell for six realizations is 0.1_4 m°C with a s.d. of 0.0_4 m°C

To realize the In MP, the ambient temperature cell is first placed in the DWBC, and the DWBC is then set at 155 °C to prevent a possible overshoot of the DWBC over the In MP temperature. Second the DWBC is set at 157.1 °C to bring the cell to a temperature hotter than realization temperature to initiate the outer liquid-solid interface. Because In cell in a melting mode is designed for industrial uses with an expanded uncertainty (k=2) requirement of 0.01 °C, no inner liquid-solid interface is induced. Third, and finally, when the DWBC reaches the set point temperature, thirty minutes are allowed to elapse for the thermometer and the In cell to thermally equilibrate prior to measurements. Plateau examples of the In cell in a melting mode realization for both DWBCs are given in Figure 3. The average melting range (0 % to 100 % liquid) for the In cell for six realizations is 0.0_8 m°C with a s.d. of 0.0_3 m°C.

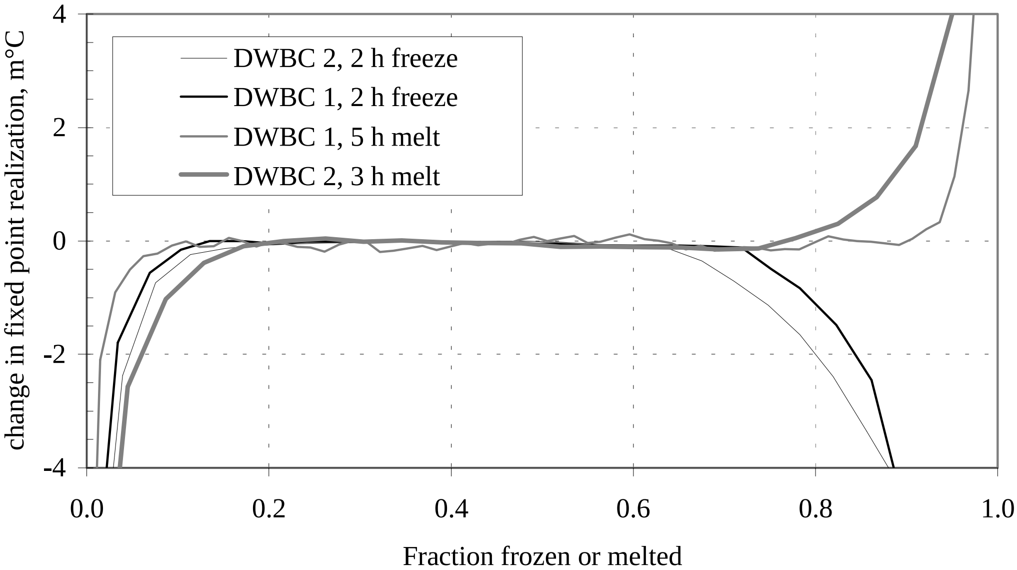

Figure 3. Plateau examples of the In FP/MP cell realized in a melting and freezing mode in two dry-well block calibrators.

4. UNCERTAINTIES

For the three small fixed-point cells, the uncertainty components used to estimate the expanded uncertainty (k=2) of realization when used in a DWBC are given in Table 1.

Table 1. Estimated uncertainties assigned to small fixed-point cells as realized in dry-well block calibrators.

Uncertainty Item	**TPW (0.01 °C)**	**Ga MP (29.7646 °C)**	**In FP/MP (156.5985 °C)**
	m°C	**m°C**	**m°C**
Type A			
Realization to Realization Reproducibility (n=18)	0.1	0.1	0.2
Type B			
Chemical Impurities	0.1	0.1	0.3
Heat Flux	0.2	0.2	1.1
Immersion Depth of Thermometer	0.0	0.0	0.0
Self-Heating	0.1	0.1	0.1
Gas Pressure	0.0	0.0	0.0
Comparison with Reference	0.1	0.1	0.5
Expanded Uncertainty (*k*=2)	0.6	0.6	2.5

The Type A value is calculated from the pooled standard deviations (of a single reading) of the realization to realization repeatability of a cell used in each DWBC, the realization to realization reproducibility of a cell used in both DWBCs, and of the comparison measurements. The Type B uncertainty contributions are chemical impurities, heat flux, exact immersion depth of the thermometer, thermometer self-heating, and gas pressure.

5. RESULTS AND CONCLUSIONS

The quantification process of the small rugged fixed-point cells shows that realization of the fixed points in a DWBC gives an expanded uncertainty of less than the required 10 m°C. The major contributor to error in realization is the heat flux uncertainty of realizing the fixed-point cell in a DWBC. Over a one-year period, there was no apparent change in the realized temperature of the fixed-point cells. There was no difference in the results from using the two different DWBCs. Based on the quantification of the three small fixed-point cells as realized in a DWBC, Table 2 gives the serial numbers, relative hotness to appropriate NIST reference cell, and the U (k=2) assigned to each cell.

With an uncertainty [U (k=2)] of 0.01 °C, these fixed-point cells are useful in calibrating IPRTs, validating the calibration status of thermometers and reducing the uncertainty assigned to the thermometers in an industrial environment. Applying this fixed-point cell technology for use in a DWBC to the other fixed points of the Hg TP, Sn MP and Zn FP/MP is a possibility.

Table 2. List of the serial numbers of three small rugged fixed-point cells, relative hotness of the three cells to appropriate NIST reference cell, and the U (k=2) assigned to each cell.

Fixed-Point Cell	cell X – NIST reference, m°C	U (k=2), m°C
TPW (s/n 31004)	–0.2	0.6
Ga MP (s/n 33001)	–0.2	0.6
In FP (s/n 34002)	–0.8	2.5

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* Commercial equipment identified in this paper does not imply recommendation or endorsement by NIST.

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