Establishment of a System for Uniformity and Stability Characterization of Liquid Calibration Bath Used Above Ambient Conditions: 50ºC to 75ºC Settings

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ARTICLE INFORMATION

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ABSTRACT

This paper aims to determine the characteristic of a Liquid Calibration Bath at above ambient temperatures using a validated method and procedure for measuring stability and uniformity. Existing method and procedure of the Metals Industry Research and Development Center were adopted and the measurements were carried out using Standard Platinum Resistance Thermometer (SPRT- Hart) and Platinum Resistance Thermometer (PRT - Isotech) which are coupled to a Digital Multimeter. Several measurements were taken at different time intervals taken on a number of identified locations in a systematic measurement pattern that covered the entire working space. Results of measurements showed that the stability of calibration bath at each temperature setting are more stable at lower temperatures and tends to increase as the temperature reaches 70ºC. The liquid bath was more uniform at lower temperatures. A full-scale research is recommended to pursue characterization of calibration bath using other types of liquid such as Ethyl Alcohol and Silicon Oil.

Introduction

Instrument calibration is one aspect in measurement where the calibration laboratories in the Philippines is continually improving to cope with the demand of continually advancing technology worldwide. This paper contributes to improving a metrological component in thermometer calibration in the Philippines that utilizes the direct proportionality of electrical resistance against temperature through resistance-temperature detector such as Platinum Resistance Thermometer. The competitiveness in the calibration of thermometers in the Philippines is largely dependent on what equipment our local laboratories have, which in most cases, the accuracies are less than what calibration laboratories abroad offer. Temperature calibration facilities abroad have different set-up and cannot be exactly duplicated by most of the local calibration laboratories in the Philippines. Among the problems arising from this situation is the scarcity of documented materials and research papers that provide detailed methods and procedures in conducting the stability and uniformity tests on calibration enclosures (or calibration bath). The limited availability of verified methods and procedures for determining the characteristics of calibration baths by many of our local calibration laboratories prevented them to evaluate further their best measurement capability and improve their accuracies. The evaluation of best measurement capability is substantial in every calibration laboratory nationwide that seek accreditation and formal recognition for competence in testing and calibration (PNS ISO/IEC 17025, 2017).

This study aligns to the concept of traceability in measurement wherein measuring instruments are calibrated using higher accuracy standards which are traceable to National Laboratories of each country, see Figure 1 below (excerpted from Estacio, 2011). Characterizing the behavior of uniformity and stability of calibration bath helps increase accuracy in calibration which improves uncertainty of measurement.

The Commonwealth Scientific and Industrial Research Organisation (CSIRO), Australia conducted characterization of liquid calibration enclosure (Connolly, 1994) where the results of stability ranged from 0.01°C to 0.05°C using Platinum Resistance Thermometers. The results of CSIRO maybe duplicated by other calibration laboratories if the laboratory environmental conditions and set-up of the same instrument were followed, however, the case may not be true in actual scenario as every instruments have their own properties and the errors of these instruments are influenced by their unique design and methods of calibration. In Glass Thermometer calibration, for example, most calibration laboratories use variable-temperature type of enclosure since the working thermometers are calibrated

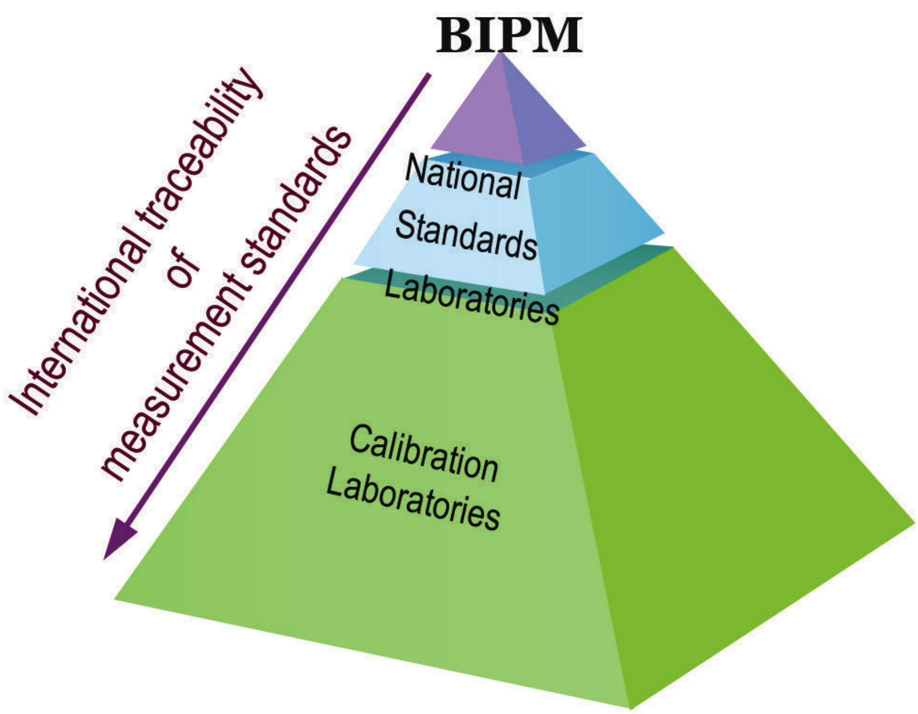

Figure 1. Traceability of Measurements Standards through Laboratories

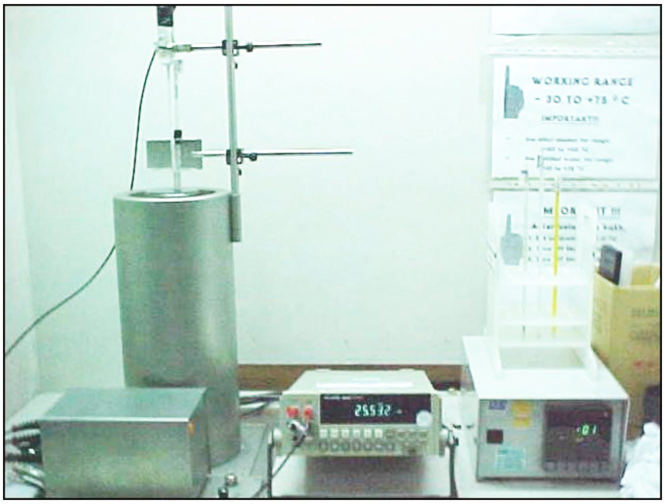

Figure 2. Calibration Set-up for Comparison Method of Thermometer

by comparison method (Estacio, 2011), see Figure 2. In this method, working thermometers are calibrated by comparison with another thermometer which has already been calibrated against another calibrated thermometer of higher accuracy in the chain of calibrations that is ultimately traceable back to a primary standard thermometer. The values obtained by CSIRO may still vary from the results of other laboratories depending on the location and instruments used.

Similar study from the works of CSIRO was conducted by Estacio in 2011 to address the lack of formal studies in characterizing calibration baths in the country, that covered temperature range from -30°C to +30°C using Ethyl Alcohol as the liquid medium. The instruments used were Platinum Resistance Thermometers, digital multimeters and liquid stirred calibration bath, where the results are comparable to the values obtained by CSIRO, however, its application is limited to low temperature ranges only.

As variable factors influence the performance of instruments at different environmental conditions and the limited range of characterization of the calibration bath covered by Estacio, this paper pushed for the continuation of characterization at above ambient conditions. Earlier investigation initially targeted up to 90^{0}C, however, due to increasing temperature variations as the temperature settings are increased, it was observed that bubbles developed in the calibration bath, thus, the test points were only fixed at a maximum of 75^{0}C. This paper covers test temperature settings from 50^{0}C to 75^{0}C and utilized only a single type of liquid all throughout the measurement proper and at an environmental condition of at least 23±3°C and 55±15% Relative Humidity (%RH). Distilled water was used in the calibration bath and measurements were taken on a number of identified locations in a systematic measurement pattern that covers the entire working space of the Liquid Calibration Bath. With these parameters the gaps of the previous studies will be addressed.

Significantly, the study can provide support to the local facilities to perform at par with international laboratories providing similar calibration services. This project further provides awareness on the importance of calibration in this field of measurement.

Purposes of the Research

The general objective of this study is to determine the characteristic of a Liquid Calibration Bath at above ambient temperatures using a validated method and procedure for measuring stability and uniformity. The specific objectives are (1) conduct stability test by determining the maximum temperature variation at a given temperature set point and time, (2) conduct uniformity test by determining the maximum difference in temperature between specified positions in the Liquid Calibration Bath, and (3) establish an initial curve fitting model describing the behavior of stability and uniformity of Liquid Calibration Bath.

Methodology

The methods of measurement adapted the procedure from available and existing method of the Metals Industry Research and Development Center (MIRDC). The measurements were conducted in the Temperature Calibration Laboratory of MIRDC following its existing ambient condition. The instruments used during test are shown in Table 1. This method, however, limited its scope on the use of stirred-liquid and overflowing calibration bath. The calibration chamber of different stirred-liquid bath can accommodate various temperatures ranging from -50^{0}C to $+600^{0}$C depending on the type of liquid used.

The measurements were carried out using Standard Platinum Resistance Thermometer (SPRT- Hart) and Platinum Resistance Thermometer (PRT - Isotech) which are coupled to digital multimeters. Both SPRT and PRT were immersed at the center position for the stability test while the temperature test points were fixed at 50°C, 55°C, 60°C, 65°C, 70°C and 75°C. The whole set up was warmed up for 30 minutes before setting the first temperature set point. After setting the first temperature set point, the set-up is stabilized further for 2 to 3 hours before initial measurements were taken, then, for every 5-minute interval, five temperature readings were taken every after a minute. The entire measuring process for stability took 30 minutes to complete one test point. Figure 3 shows the actual measurement set up for stability wherein both Hart SPRT and Isotech PRT were located at the center of the calibration bath and submerged at 200 mm depth.

Table 1.

List of Equipment Used.

Equipment Description	Code No.	Brand/Model
Calibration Enclosure	T049	ASL CB15-45e Calibration Liquid Bath
SPRT1 (Reference, Fixed)	T038	Hart 5699 SPRT
SPRT2 (Test, Movable)	T033	Isotech 670SH PRT

Indicator Used	Code No.	Indicator Setting
Agilent 34401A DMM	E050	4-wires, 100W
Fluke 8842A DMM	E004	4-wires, 100W

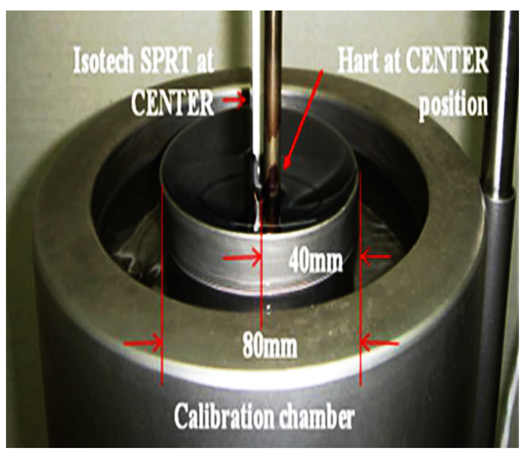

Figure 3. Actual set-up of and SPRT Hart PRT at Center Position

Uniformity test, was done at different immersion lengths of 75mm, 150mm, 200mm, 250mm with the same temperature test points fixed at 50^0C, 55^0C, 60^0C, 65^0C, 70^0C and 75^0C. The Hart SPRT was used to establish the reference point by immersing it at 200mm depth while being held fixed at the center of the calibration bath. The PRT (Isotech) was used to measure the temperature at five different positions including the center. Figures 4(a) and 4(b) illustrate the different immersion depths and measurement locations, respectively. Careful attention to instrument preparation, wirings and connections during set up was made to ensure that the liquid bath is functioning properly and filled with distilled water up to a level such that when it is switched on, distilled water will overflow from the inner tube or calibration chamber. The entire measuring set up was allowed to warm-up for 30 minutes prior to setting of test point. Right after warming and setting up, the first set test point, 50^0C, was set on the calibration bath.

The calibration bath was then monitored for its stabilization time. Various models of calibration bath have different stabilization time from ambient temperature to the "set point" temperature. The bath used in this study, however, followed the specification from the manufacturer's manual. After the stabilization time, the bath was further stabilized for five more minutes before the first reading of digital multimeter was recorded. Several measurements were then taken at fixed time intervals. The detailed step by step instruction on the data gathering procedure were followed as outlined in Estacio (2011, p.44-50).

The calibration bath stabilities and uniformities were calculated by computing the ranges of the maximum difference with the minimum difference measured at each set temperature points. The computed ranges were analyzed by curved fitting technique using polynomial regression. The analysis for uniformity, however, was made by grouping the data in the horizontal and vertical positions.

Results and Discussion

Stability Test for 50ºC to 75ºC.

The results of measurement show that the stability of calibration bath at each temperature setting tends to increase as the temperature reaches 75ºC. With reference to Figures 5(a) and (b), the calibration bath stabilities were calculated by taking the

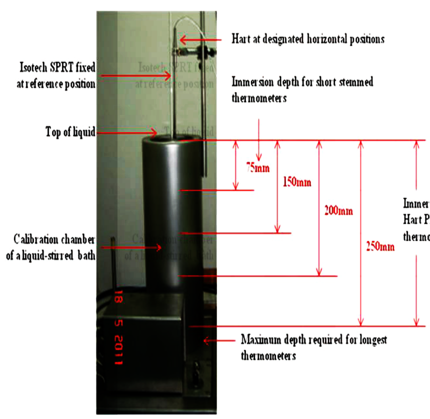

Figure 4(a). Different immersion Depth of the Isotech SPRT.

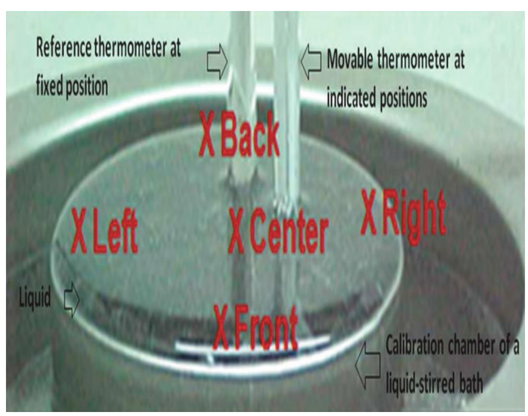

Figure 4(b). Different Locations of the Isotech PRT while the Hart SPRT is maintained at the center of the calibration bath

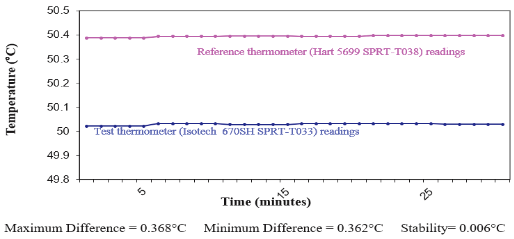

Figure 5 (a). Plot of Temperatures Measured by Hart-SPRT and Isotech PRT at 50ºC.

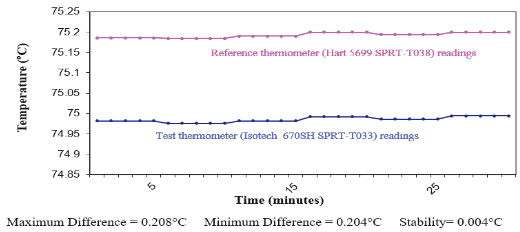

Figure 5(b). Plot of Temperatures Measured by Hart-SPRT and Isotech PRT at 75ºC.

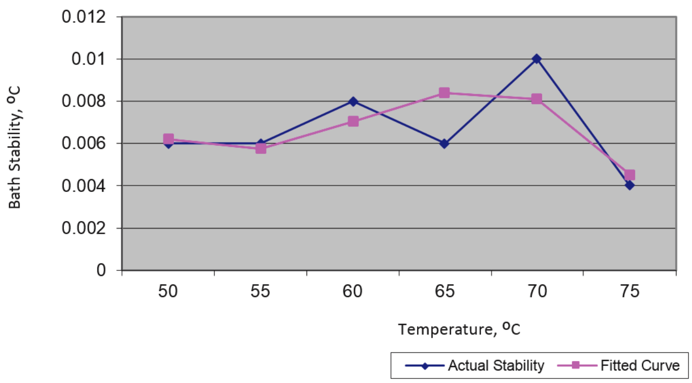

Figure 6. Comparison of Graph of the Actual Stability from the Curve Fitted Equation of S

range of the maximum difference with the Minimum Difference measured at each set temperature points, e.g. 50, 55°C, etc. The differences obtained were not significantly scattered since the standard deviation only ranged from 0.0016°C to 0.01°C. The graph shown in Figure 5(a) for 50°C corresponds to a standard deviation of 0.0025°C that exhibits a fairly straight graph. This also indicates that the measured values are way below the acceptable limit of 0.01°C to 0.05°C that was established in CSIRO (Connolly, 1994).

Temperatures measured at lower setting point, e.g. 60ºC, 65°C are more stable compared with higher settings, which were also consistent with the results of measurement conducted by Estacio (2011) for setting ranges below 30ºC. As the temperature set points approaches higher values, e.g. 70ºC and 75 ºC, it was observed that bubbles occasionally appeared in the bath.

Taking into consideration that the measured temperatures for stability analysis were taken at 200mm depth from the surface of the calibration bath, it is also considered consistent within the range of the results of uniformity test for both horizontal and vertical analyses presented in the succeeding discussions.

The waviness of the plot of temperatures in Figure 5(b) exhibited similarity in form. This result indicates that the response of both Hart-SPRT and Isotech PRT at higher temperature setting has not changed compared to its responses in lower temperature setting.

The computed stabilities for the temperature settings from 50ºC to 75ºC ranged from 0.004°C to 0.010°C and the plot of these Stabilities is shown in Figure 6. To describe the behavior of the stability of the liquid calibration bath, a curve fit

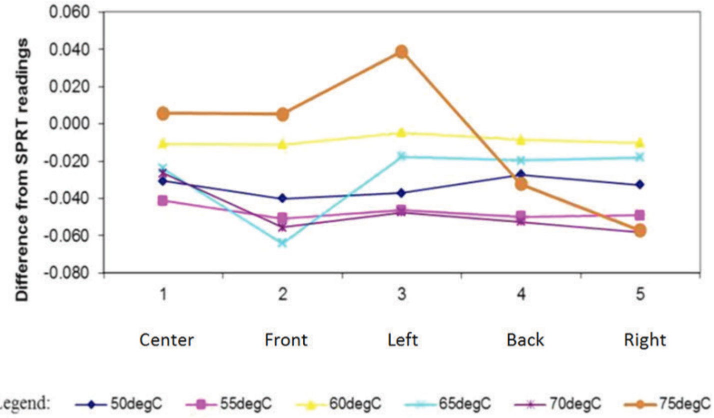

Figure 7. Graph of average of differences for Horizontal Uniformity

equation for S was derived using polynomial regression expressed as:

$S = 0.4714 – 0.0238t + 0.0004t^2 – 2.22x10^{-6}t^3$

Where:

S = Stability, ºC and t = temperature w/in 50ºC to 75ºC

The curve fitted equation for S is considered applicable within the range of 0.002°C. This range value estimates the accuracy of the curve fitted graph that describes the actual plot of the calibration bath stability. The derived curve fitted equation for S, also describes the behavior of the stability of the calibration bath within an 8-hour period as demonstrated during the actual measurements.

Uniformity Test

The uniformity of temperature inside the Calibration Bath is affected by the movement of the liquid inside. The circulating system tends to cause uneven temperature of the liquid at various points in bath. Improving the rate of flow of liquid may also improve the uniformity of a bath. However, it may not always be a practical solution to improve the uniformity of temperature by altering the design of the stirring system of the bath since there are limitations on the degree by which "stirring" can be increased or decreased without causing overheating of the bath stirring system. Hence, the evaluation of the uniformity of the calibration bath was done at horizontal and vertical positions. In the same way as stability is considered, the derived uniformity equation can be used in practical estimation of errors in uncertainty of measurement.

Uniformity Test with Respect to Horizontal Position.

The recorded measurement results are the differences of readings at each position, e.g. center(C), front(F), left(L), back(B), and right(R), relative to the center position as specified in Figure 4(b) and at different immersion depths. In plotting the graph of Uniformity for Horizontal position (HU), each point of the graph represents the average of the difference of readings of each same positions at different immersion depths. Shown in Figure 7 are the graphs of averages of C,F,L,B,and R differences with respect to various temperature settings.

Following the plot of 50degC from Figure 7, the Horizontal Uniformity is calculated by taking the difference of the

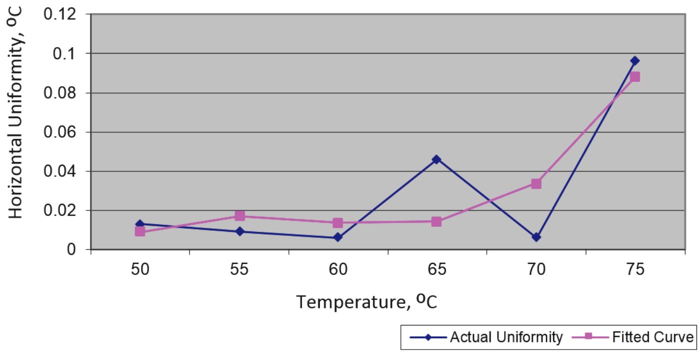

Figure 8. Comparison of graph of the actual Horizontal Uniformity from the curve fitted equation of HU.

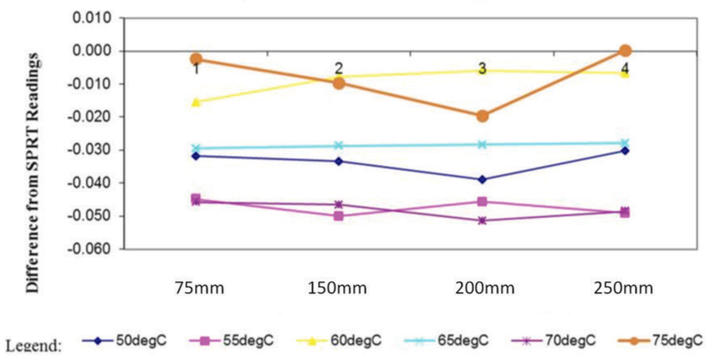

Figure 9. Graph of average of differences for Vertical Uniformity

maximum value minus the minimum value, in this case

$HU_{50°C}$ = (-0.027) – (-0.040) = +0.013°C

By applying the same concept of computation for other temperature settings, a plot of Horizontal Uniformity is generated in Figure 8 where a curve fitting equation for HU is derived using polynomial regression as shown below:

$$HU = -4.0783 + 0.2108t - 0.0036t^2 - 2.044x10^{-5}t^3$$

Where:

HU = Horizontal Uniformity, ºC

t = temperature w/in 50ºC to 75ºC

The graph of the fitted curve in Figure 8 shows that the horizontal uniformity reaches a value of about 0.05°C at a temperature of approximately 72°C which is comparable to the value of 0.05°C obtained by CSIRO (Connolly, 1994). The application of equation for the fitted curve, however, is limited up to 72°C only.

Uniformity Test with Respect to Vertical Position

The Uniformity for Vertical position (VU) was evaluated by getting the average of readings at each immersion depths, e.g. average of all differences taken at 75mm immersion depth at 50°C, etc.. By plotting the computed average of readings, a graph is generated as shown in Figure 9, wherein each point represents the average of differences at every immersion depths and

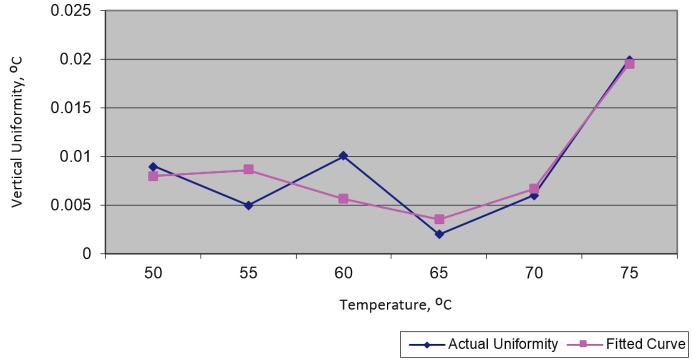

Figure 10. Comparison of graph of the actual Vertical Uniformity from the curve fitted equation of VU

temperature settings. The bath locations 1, 2, 3, and 4 correspond to the immersion depth of 75mm, 150mm, 200mm, and 250mm, respectively.

Following the plot of 50degC from Figure 9, the Vertical Uniformity is calculated by taking the difference of the maximum value minus the minimum value, in this case

$$VU_{50°C} = (-0.030) - (-0.039) = +0.009°C$$

By applying the same concept of computation for other temperature settings, a plot of Vertical Uniformity is generated in Figure 10 where a curve fitting equation for VU is derived using polynomial regression as shown below:

$$VU = -1.17371 + 0.0613t - 0.00105t^2 - 5.926x10^{-6}t^3$$

Where:

VU = Vertical Uniformity, ºC and t = temperature w/in 50ºC to 75ºC

Unlike the curved fitted equation for Horizontal Uniformity, the derived equation for the vertical uniformity can be applied up to 75°C since the graph in Figure 10 indicates a value of 0.02°C which is way below the value of 0.05°C obtained by CSIRO (Connolly, 1994).

Conclusion and Recommendations

In filling in the gap of our local facilities to perform at par with international counterpart laboratories in the field of temperature calibration, this study established a characterization method of a Liquid Calibration Bath at above ambient temperatures for local application by way of describing the behavior of stability, horizontal uniformity and vertical uniformity using curve fitting models.

Based on the results of analysis, the method and procedure used in this study can characterize a temperature-controlled calibration bath through the evaluation of stability and uniformity since the differences measured in various positions and temperature settings of the bath are comparable to the values observed by a national laboratory, in this case CSIRO, which also followed similar measurement method.

Furthermore, the procedure for testing adapted in this study suits the local accuracy requirement in industrial applications. The results also show that as the test temperature setting approaches 75^{0}C, bubbles started to develop in the calibration bath, and thus, influence the values of uniformity and stability of the calibration bath, which are evident in the increasing temperature differences of the SPRT and PRT.

The equations derived describing S, HU and VU can be used to estimate the temperature conditions at any given location inside the calibration bath. The accuracy of these equations can be further evaluated through uncertainty of measurement estimation. Once the uncertainty components of S, HU and VU are determined, their contribution to the overall uncertainty of measurement for temperature calibration will be clearly defined.

A full-scale research is recommended to pursue characterization of calibration bath using other liquid, e.g. Ethyl Alcohol and Silicon Oil. Also, validation of the method and procedure through evaluation of uncertainty of measurements and En-value tests can be performed for further in-depth analysis of this characterization method. In considering to adopt this method and procedure in the determination of stability and uniformity of a temperature-controlled calibration bath for local calibration laboratories, the test setup and working conditions are limited to the following parameters: (1) environmental conditions with respect to ambient temperature and relative humidity should range from 20°C to 26°C and 40% to 55% RH, respectively, (2) the enclosure is a stirred-liquid and overflowing liquid calibration bath operating at 50°C to 75°C, (3) the liquid used is only distilled water throughout the test, and (4) the accuracy of test and reference thermometers must be at least ± 0.05°C. Future directions in characterization of liquid calibration bath can cover higher temperature ranges up to 250°C.

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