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Characterization of Silicon Oil Calibration Bath Through Stability and Uniformity Tests Using Standard Platinum Resistance Thermometer and Precision Multimeter

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Abstract

Instrument calibration is a vital factor in ensuring that results of measurement are accurate up to an extent acceptable for a certain accuracy requirement. This paper presents the method of characterizing a Liquid Calibration Bath that uses Silicon Oil from 90ºC to 250ºC. Existing method and procedure that use Ethyl Alcohol and Water for low and ambient temperature ranges were followed. The measurements were carried out using two Standard Platinum Resistance Thermometers which are coupled to a digital multimeter. Several measurements were made at different positions and immersion lengths that covered the entire working space of the calibration bath. 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 200ºC. The temperature of the liquid calibration bath is more uniform at lower temperature setting as well. The temperature differences obtained were not significantly scattered since the curve-fitted equation describing the behavior of stability and uniformity do not deviate significantly from the actual measured values. This indicates that other calibration laboratories can also perform stability and uniformity tests unique to their set-up at an accuracy level consistent with industrial accuracy requirement.

Keywords: liquid calibration bath, stability, uniformity, characterization, measurement

I. INTRODUCTION

Various laboratories worldwide providing calibration of temperature measuring instruments have different set-up and most of the time cannot be exactly duplicated. Recent studies in characterizing liquid calibration baths through stability and uniformity evaluation are using two resistance thermocouple probes positioned at different locations in the calibration chamber as presented by [1-6]. This method was generally applied to various temperature ranges and type of liquids used, e.g. Ethyl Alcohol, Water and Silicon Oil. The method was specifically applied at -30°C to +30°C using ethyl alcohol to a liquid stirred overflowing calibration enclosure [8]. The works of [8] yielded values of temperature stability and uniformities comparable to Connolly and Horrigan to values ranging from -30°C to 30°C. Using the methodologies of these previous studies, a liquid stirred overflowing calibration bath was characterized at 50°C to 75°C using water as the medium [10] which yielded very close results to Connolly and Horrigan as well. The methods were then used to conduct characterization of a different calibration bath using Silicon Oil from 90°C to 150°C and yielded close results compared with previous studies [11]. Due to limited number of verified methods and procedures for determining the characteristics of calibration baths, many temperature calibration laboratories do not have a common basis in evaluating their best measurement capability in terms of the characteristic of their bath. Hence, the objective of this paper is to characterize a liquid stirred calibration bath using Silicon Oil by describing the behavior of stability and uniformity through a curved fitted mathematical equation.

Since Silicon Oil is used for high temperature applications, the previous works proved the suitability of the methodologies used for low temperature applications. This paper covered the characterization of liquid stirred overflowing bath up to the theoretical boiling point of Silicon Oil at 250°C. The calibration chamber of different stirred-liquid bath can accommodate various temperatures ranging from -50˚C to +600˚C depending on the type of liquid used [1,8]. Earlier test method initially was on low temperature range, however, due to increasing temperature variations as the temperature settings are increased, it was observed that there were changes in the behavior of the liquid used such as bubbles developed in the calibration bath, thus, the test points were only fixed at a maximum of 250°C.

This study limited the test temperature settings from 90˚C to 250˚C and utilized a single type of liquid only all throughout the measurement proper and at an environmental condition of at least 23±3°C and 55±15%RH. Silicone oil was used in the calibration bath. 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 [1,8].

The results of this study align 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 [7,8] as shown in Fig. 1. Characterizing the behavior of uniformity and stability of calibration bath helps increase accuracy in calibration which improved uncertainty of measurement.

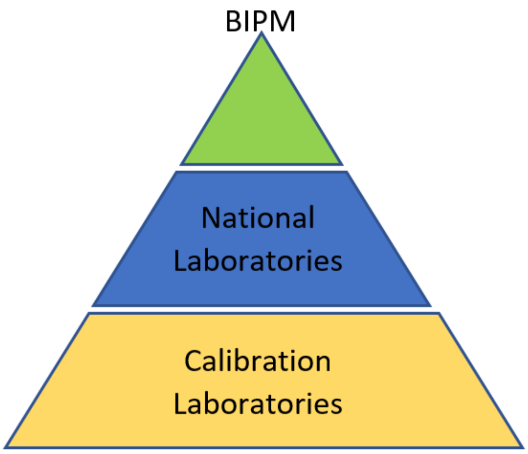

Fig. 1. Traceability of measurement standards of calibration laboratories

Most of the calibration laboratories use variable-temperature type of enclosure since the working thermometers are calibrated by comparison method [8], see Fig. 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 a chain of calibrations that should ultimately be traceable back to a primary standard thermometer [1, 9].

II. MATERIALS AND METHODS

The method of measurement adapted the procedure developed in the works of [8] and [10]. The characterization covers 90˚C to 250˚C, wherein the measurement results of [10,11] from 90˚C to 150˚C were augmented to the values obtained in this paper from 175˚C to 250˚C. The measurements were carried out using Standard Platinum Resistance Thermometer (SPRT- Hart) and Platinum

Fig. 2. Set-up for calibration of thermometers by comparison method using a calibration bath.

II.I Method for Evaluating Stability

Both SPRT and PRT were immersed at the center position for the stabilitys test at approximately 200mm immersion length from the surface of the liquid. The measurement method started by fixing the temperature, in this case, at 175˚C. After stabilizing the whole set up for 30 minutes, five measurement data were taken consecutively by taking the differences of the readings of the Hart SPRT and Isotech SPRT. The measurements were repeated every 5 minutes for 30 minutes, and thus, generating a total of thirty (30) measurement data. The Stability (S) is calculated by taking the difference of the maximum and minimum values out from 30 measurement data. This procedure is repeated for 200˚C, 225˚C, and 250˚C which yielded four (4) values of calculated stabilities at the different temperature settings. The new values of stabilities from 175˚C to 250˚C were augmented to the works of [10,11]. To describe the behavior of the stabilities obtained from 90˚C to 250˚C, a third-degree curve fit equation using polynomial regression was derived from the actual plot of the stabilities.

II.II Method for Evaluating Uniformity

Uniformity tests, on the other hand, were done at different immersion length of 75mm, 150mm, 200mm, 250mm while the temperature test points were fixed at 175˚C, 200˚C, 225˚C, and 250˚C. Uniform test setup is shown in Fig. 3. 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 Isotech SPRT was used to measure the temperature at different test points at Center, Front, Back, Left, and Right as illustrated in Fig. 4.

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Careful attention during set up were made to ensure that the liquid bath is filled with silicone oil up to a level such that when it is switched on, silicone oil will overflow from the inner tube of calibration chamber [10]. The whole 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, at 175 ^{0}C, 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.

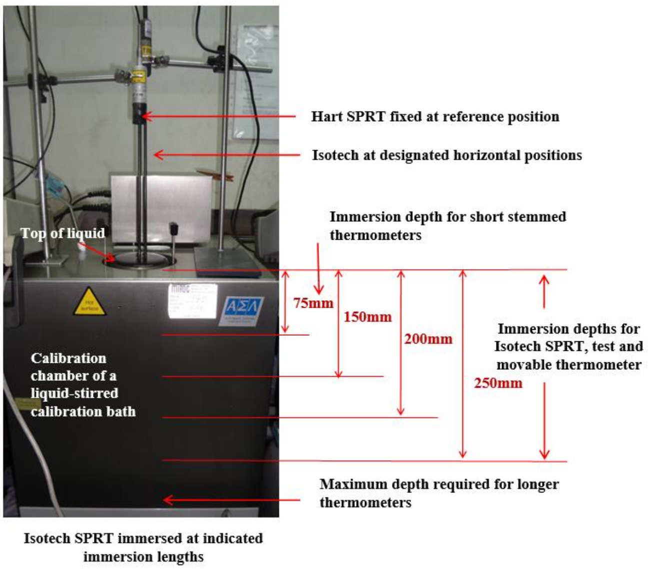

Fig. 3. Immersion length of the PRT for Horizontal and Vertical Uniformity Tests.

II.III Measurement Procedure for Uniformity Tests

Initial measurements were made at 175ºC set point. The reference Hart SPRT was held fixed at the center position with 200mm immersion length, While the Isotech SPRT was also placed side by side with Hart SPRT, however, its immersion length is fixed at 75mm. Refer to Fig. 4. After the initial stabilization of 30 minutes, five measurements were taken at every 1-minute interval, by taking the difference of the readings of Hart SPRT and Isotech SPRT. While the temperature is held fixed at 175ºC and immersion length of 75mm, the measurement is then repeated for the other positions following the sequence: Front-Left-Back-Right. The data gathered at 175ºC set point and 75mm immersion length were evaluated by calculating the Horizontal Uniformity (HU) and Vertical Uniformity (VU) using the following expressions:

$$HU_{(t,i)} = Max\{PA_{(C)}, PA_{(F)}, PA_{(L)}, PA_{(B)}, PA_{(R)}\} - Min\{PA_{(C)}, PA_{(F)}, PA_{(L)}, PA_{(B)}, PA_{(R)}\} \quad (1)$$

Where: HU(t,i) - Horizontal Uniformity at set temperature.

t – Temperature set point held constant for a particular measurement cycle.

i – Immersion Length, See Fig. 3.

PA – Position Average; the average of the difference of readings at various positions at a particular t setting.

$$VU_{(t,i)} = Max\{IA_{(75)}, IA_{(150)}, IA_{(200)}, IA_{(250)}\} - Min\{IA_{(75)}, IA_{(150)}, IA_{(200)}, IA_{(250)}\} \quad (2)$$

Where: VU(t,i) - Vertical Uniformity at set temperature.

t – Temperature set point held constant for a particular measurement cycle.

i – Immersion Length, See Fig. 3.

IA – Immersion Average; the average of the difference of readings at various immersion lengths at a particular t setting.

Fig. 4. Different positions of the Isotech SPRT for Horizontal and Vertical Uniformity Tests, excerpted from [8].

The whole cycle is repeated for 200ºC, 225ºC and 250ºC temperature settings and yielded four (4) sets of HUs and VUs. The instruments used for both stability and uniformity tests are listed in Table 1. A detailed step by step instruction on the data gathering procedure was followed as outlined in [8].

Table 1. List of equipment used

Equipment Description	Code No.	Model/Specifications
Calibration Enclosure	TO49	ASL CB15-45e Calibration Liquid Bath
SPRT1 (Reference, Fixed)	TO38	Hart 5699 SPRT
SPRT1 (Test, Movable)	TO33	Isotech 670SH PRT
Agilent 34401A DMM	E050	4-wires, 100W
Fluke 8842A DMM	E004	4-wires, 100W

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III. RESULTS AND DISCUSSION

III.I Result of Stability Test for 90⁰C to 250⁰C.

Results of measurement show that the stability of liquid calibration bath at each temperature setting tends to increase as the temperature reaches 250ºC. With reference to Fig. 5, the calibration bath stability was calculated by taking the range of the maximum temperature difference with the minimum temperature difference measured at each set temperature points, e.g. 175°C, 200°C, etc. The calculated stabilities ranged from 0.01°C to 0.09°C. The differences obtained were not significantly scattered since the standard deviation ranged only from 0.005 to 0.033. However, these indicate that the measured values up to 200°C are within the acceptable limit of 0.01 to 0.05 that was established by [1].

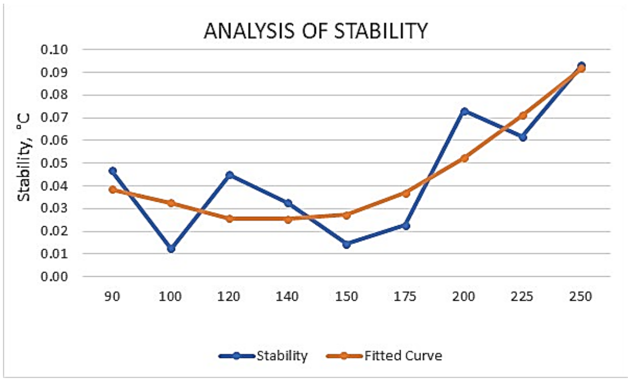

Fig. 5. Result of stability from 90°C to 250°C

Temperatures measured at lower setting point, e.g. 90ºC, 175°C, 0°C are more stable compared with higher settings which also comparable to the results of measurement conducted by [8] for setting ranges below 30ºC. Taking into consideration that the measured temperatures for stability analysis were taken at 200mm depth from the surface of the calibration bath, it is considered uniform within the range of the results of uniformity test for both horizontal and vertical analyses.

To describe the behavior of the stability of the liquid calibration bath, a curve fit equation for stability (S) was derived using polynomial regression expressed as:

$$S = 0.197 - 0.003t + 1.55x10^{-5}t^2 - 2.1x10^{-8}t^3 \quad (3)$$

Where:

S = Stability, ºC

t = temperature w/in 90ºC to 250ºC

III.II Results of Uniformity Test

The uniformity of temperature inside the Calibration Bath is largely affected by the movement of liquid inside. The circulating system tends to cause uneven temperature at various points in the bath [1]. Improving the rate of flow of liquid may increase the uniformity of a bath. However, it is not always attributed to design configuration since there are limitations on the amount of stirring that can be increased without causing overheating. Hence, the following part of this study is the results of evaluation of the uniformity of the calibration bath for horizontal and vertical directions.

III.II.I Uniformity test with respect to horizontal position, HU

Using the average of the values of the difference of readings with respect to horizontal position, i.e. average of center, average of front, average of left, average of back and average of right positions in the calibration bath, the HUs at different positions and immersion lengths were calculated using equation (1). The calculated HUs at 175°C to 250°C ranged from 0.027°C to 0.035°C. When the HU results of [10] for 90°C to 150°C ranging from 0.004°C to 0.012°C are augmented to the new values obtained at 175°C to 250°C, the resulting graph exhibited a sudden increase of about 0.025°C at the augmented points as shown in Fig. 6. This sudden increase can be attributed to varying environmental conditions at the time of data gathering, it can be noted that the data from 175°C to 250°C were obtained at different a period. However, the sudden increase can still be considered acceptable since the range of values fall within the expected accuracy of the calibration bath.

Fig. 6, showing the uniformity of the bath with respect to horizontal position indicates, increasing trend as the temperature setting approaches 250°C. The behavior of HUs as it approaches temperature settings near the boiling point of Silicon Oil tends to increase as compared with lower temperature settings. This behavior is also true with the result of stability measurement. At higher temperature settings, the Silicon Oil becomes less viscous and tends to generate bubbles which directly affect the uniformity of temperature at the different measurement points in the calibration bath.

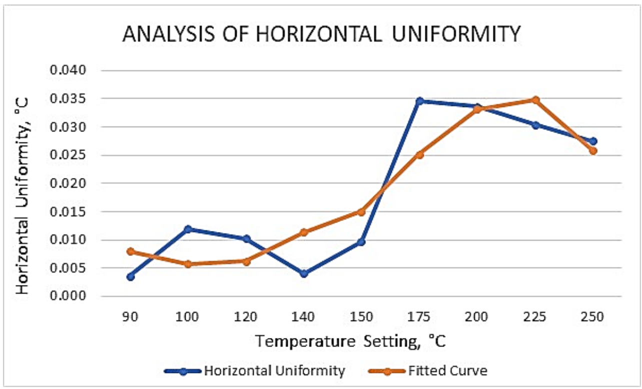

Fig. 6. Result of Horizontal Uniformity at 90°C to 250°C.

The behavior of HU is described in the equation below using curve fitting method applied in deriving the stability equation.

$$HU = 0.150 - 0.003t + 2.23x10^{-5}t^2 – 4.56^{-8}t^3 \quad (4)$$

Where:

HU = Horizontal Uniformity, ºC

t = temperature w/in 90ºC to 250ºC

The deviation of the curved fitted equation for HU from the actual value ranged from -0.007°C to +0.01°C. Which is considered acceptable up to the accuracy level of the calibration bath.

III.II.II Uniformity test with respect to vertical position

From the same measurement data gathered, the average of temperature differences for each immersion length and temperature setting were computed and used as input to equation (2) to determine the different values of vertical uniformity at different temperature settings from 175°C to 250°C. The vertical uniformity obtained from 175°C to 250°C ranged from 0.023°C to 0.044°C. These values when augmented to the results of [10] from 90°C to 150°C yield increasing trend which is consistent with those patterns exhibited by stability and horizontal uniformity results. Fig. 7 shows the uniformity of the bath with respect to vertical position.

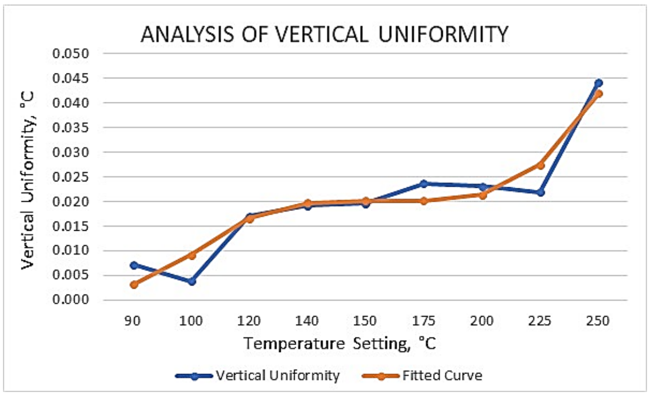

Fig. 7. Result of Vertical Uniformity at 90°C to 250°C

The behavior of VU is described in the equation (5) using curve fitting method.

$$VU = -0.159 + 0.003t - 1.95x10^{-5}t^2 + 3.89^{-8}t^3 \quad (5)$$

Where:

VU = Vertical Uniformity, ºC

t = temperature w/in 90ºC to 250ºC

The values generated by the curved fitted model for vertical uniformity deviates from the actual values of within -0.006°C to +0.003°C. This range fall within the accuracy of the calibration bath.

IV. CONCLUSION AND RECOMMENDATION

Based on the results of this study, local calibration laboratories can also perform the stability and uniformity tests on a calibration bath at an accuracy level consistent to the hierarchy of accuracy set by national laboratories, since the measured values obtained based on the procedure outlined were close to the values obtained in similar tests conducted in a national laboratory abroad and have very small standard deviations. After series of measurements done to establish the stability and uniformity of the calibration bath, as well as the analyses of gathered data, it is also concluded that the method and procedure applied in this study can establish the characterization of a temperature-controlled calibration bath by describing the behavior expressed in mathematical model. The derived mathematical models of S, HU and VU can be utilized in evaluating the uncertainties of measurement during calibration of various temperature measuring instruments.

This further concludes that the procedure for testing adapted in this study suits the local accuracy requirement in industrial application. The results also show the increasing temperature difference as the test temperature setting reaches 175^0C, where it was observed that bubbles started to develop in the calibration bath. Presence of bubbles affect the uniformity and stability of the calibration bath as this influence the homogeneity of the silicone oil.

A full-scale study is recommended to pursue further validation of the method and procedure through evaluation of uncertainty of measurements. The En-value test can also be performed to broaden the applicability of this characterization method. For other calibration laboratories considering the suitability of this method and procedure in the determination of the stability and uniformity of temperature-controlled calibration bath, the associated uncertainty of measurement unique to their test setup should take into account the effects of the following parameters: (1) environmental conditions of ambient temperature range of 20 to 26°C and relative humidity range of 40 to 55% R.H. , (2) the enclosure is a stirred-liquid and overflowing calibration liquid bath operating at 150°C to 250°C and liquid used is only silicone oil throughout the test and (3) test thermometers' accuracy of at least ± 0.05°C.

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