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# Automated Controlled Stirred Liquid Baths System for Calibrating Contact Thermometers in the Temperature Range from -80°C to 420°C

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**ABSTRACT**

National Institute of Standards (NIS) is the national metrology institute in Egypt. It consists of more than twenty laboratories working continuously to realize the international standard units (SI units). Thermal metrology laboratory (ThML) is one NIS leading laboratory that has twice responsibilities, one for realizing the thermodynamic temperature and the other for introducing calibration solutions to all organizations in Egypt such as contact, non-contact thermometry, hygrometry, thermal analysis and viscosity. In this study, a newly developed automated firmware was used for controlling and regulating the temperature of stirred liquid baths and thermometry bridge system (SLBTBS) simultaneously and determined precisely the starting time of the measurements according to smart algorisms using compatible parameters of (LabVIEW®) tools. The protocol of measurements was dependent on an automatic investigation of the best stability period whereby the setting point lied in the specific temperature ranges related to hysteresis limits. Characterization tests and assessment for optimum conditions of the SLBTBS were done. The newly designed controller gave temperature stability better than 0.02K in the low-temperature range with better-linked uncertainty.

## 1. Introduction

The main mission of Thermal Metrology Laboratory (ThML) as a leading laboratory of the National Institute of Standards (NIS) in Egypt in the fields of thermometry, humidity and viscosity is conscientious of the development, improvement and maintenance of the national standards. Furthermore, ThML introduces services of testing and calibration to various organizations, factories, companies and scientific divisions such as medical, industrial agriculture, pharmaceutical and others that reflect the vital role of NIS in the quality infrastructure in Egypt.

The word ''Metrology'' is the measurement science. Measurements of any physical property like temperature should be in a good stabilization system. Measurements of mediums have great interest from the researcher, scientists, equipment developers and manufacturers. In the

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metrology field at National Metrology Institute, the main target accuracy and precision must not exceed a few mK.

The contribution of results of accuracy and precision in temperature measurements meant there were hard work done by the calibrators and temperature calibration facilities at ThML. The sensors and probes which measure the temperature need regular calibration. The calibration of temperature sensors is important for ensuring their good operation within the required accuracy limitation.

## 2. Methodology

A state of the art software was developed for a new controller and data acquisition firmware for all ThML stirred liquid baths to calibrate automatically all kinds of contact thermometers such as thermocouples, digital thermometers, bimetal and liquid in glass thermometers.

The firmware control, regulate, operate and measure the temperature of SLBTBS precisely using the strong coding, protocol and subsequence string under Laboratory Virtual Instrument for Engineering Workbench (LabVIEW®) software with similar mechanisms to those reported in Figure 1 [4,5,7].

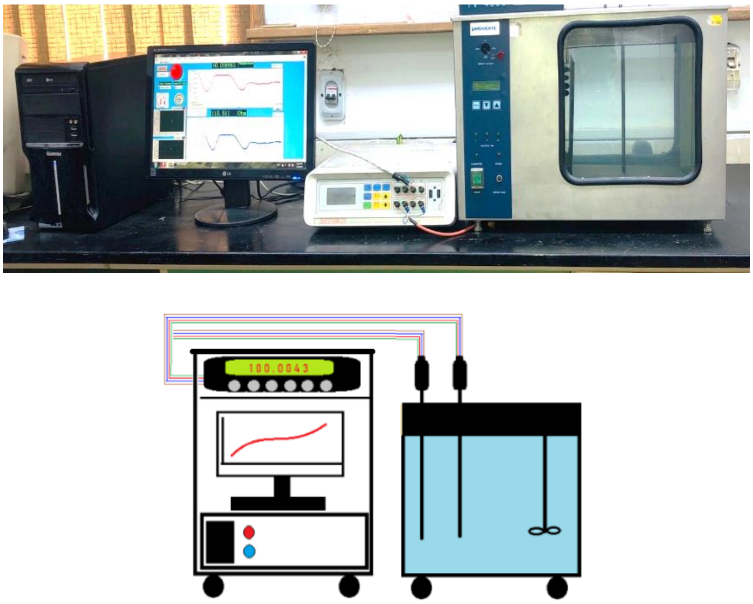


**Fig. 1.** SLBTBS

The novelty of this study is that for the first time there is a built-in unified system at NIS for calibrating all kinds of contact thermometers without any overlapped from the observer only when mounting and dismounting the unit under test equipment. The operator or technician adjusts the calibrating setting point and the limits of stability (*SP* ± α) where α is the error gain due to the response time at different setpoints, the firmware will follow up the algorithm instructions to control and monitor the setting point, stability, homogeneity, type A uncertainty and finally acquire the data after a ringing alarm that the bath under stability within the limits of stability values.

### 2.1 Equipment

ThML contains mainly five different liquid baths which are different in temperature ranges as shown in Table 1. Each one is connected to class A Fluke Pt100 from model 5626, with stability ± 0.003 °C and accuracy ± 0.006 °C from –80 °C to 0 °C, ± 0.012 °C from 0 °C to 420 °C and ± 0.015 °C to ± 0.022 °C from 420 °C to 660°C respectively.

**Table 1**
Set of equipment used in this system

| Equipment | Manufacture | Model | Temperature Range |
|---|---|---|---|
| Thermometry Bride | Isotech | TTI | -200 up to 700 $^{O}$C |
| Alcohol Bath | Fluke | 7381 | -80 up to 25 $^{O}$C |
| Alcohol Bath | aHetrofrig | 2100 | -20 up to 25 $^{O}$C |
| Water bath | Petrotest | TV4000 | 25 up to 95 $^{O}$C |
| Oil bath | Hart Scientific | 6054 | 100 up to 220 $^{O}$C |
| Salt bath | Hart Scientific | 6055 | 200 up to 420 $^{O}$C |
| 6 Class A Pt-100 | Fluke | 5626 | -200 up to 660 $^{O}$C |

Thermometer bridge model TTI has two channels A and B. Pt-100's is connected to a selector switch that has mainly four ports for four baths connected to channel A and the fifth bath connected directly to channel B of the bridge.

### 2.2 Interface Protocol

The steps for controlling and flowchart algorithm of the interface are presented in Figure 2. One of the most popular, familiar, easier and strongest programs that provide a good graphical user interface, equipment-PC communication, data acquisition and data analysis is LabVIEW [7-9]. Figure 3 and 4 show two main subVI (Virtual Instrument) governed under the main Graphical User Interface (GUI). The first is responsible for moderate stabilization and regulation of the bath's temperature as a mechanism protocol reported in [4-8]. The second is related to the microcontroller processor of TTI Itself as shown in Figure 4. It initiates by sending a string code defined by ASCII language to the processor of the TTI to demand a series of information.

The queued return back information from the processor responses is the immediate temperature in ohm, resolution of the indicator, relaxation time between each iteration loop and the working channel input that does the measurements. The encryption binary string transfer between the processor and PC is converted to a known decimal value [9-12].

Table 2 shows the firmware protocol that is applied between the TTI processor and PC by ASCII language [11]. The communication starts with RS232 configuration through VISA. For instrument control using VISA, it should be applying termination characters string as a slave code and separated by carriage return of line feed under the firmware that is considered as a master code.

**Table 2**
Firmware protocol codes (**TTI Configuration)**

| Code | Slave string | Meaning |
|---|---|---|
| INPUT A | Carriage return | Select the input channel of TTI leads A or B (Figure 4) |
| INPUT B | | |
| Wait 100 ms | ---------------- | Waiting time for relaxing equals 10 milliseconds |
| HR | Carriage return | Ask the bridge to measure high resolution (Figure 4) |
| Wait 100 ms | ---------------- | Waiting time for relaxing equals 10 milliseconds |
| MEASURE | Carriage return | Ask the bridge to respond with a momently reading value of Pt-100 in Ohm |
| Wait for 1 s | ---------------- | Waiting time for relaxing equals 1000 milliseconds |
| Select Pt100 codes: | ---------------- | The temperature in Ohm is going to be converted into °C through sub vi built in the program (Figure 4) |
| 012 | | |
| 013 | | |
| 015 | | |
| 022 | | |
| Wait for 10s | ----------------- | Waiting time for relaxing equals 10 seconds due to the complexity of the CVD inverse function |

**To next iteration**

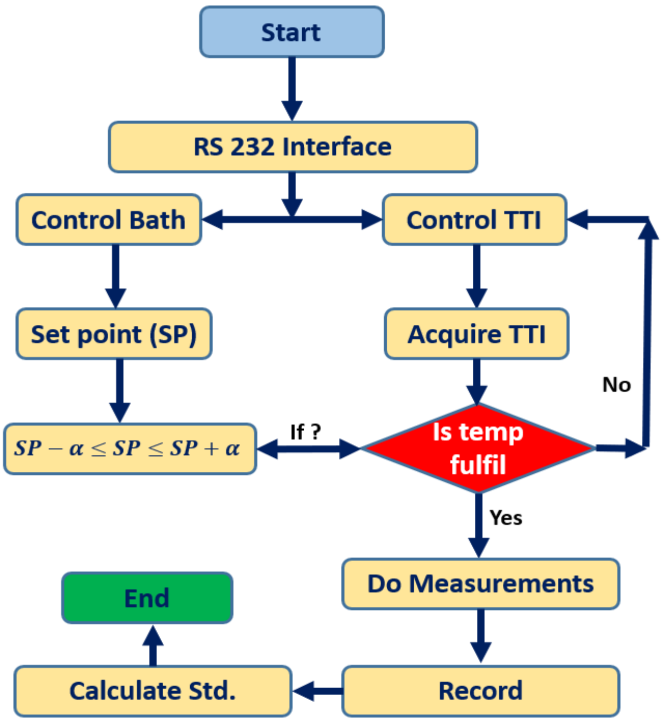


**Fig. 2.** Firmware flow chart protocol

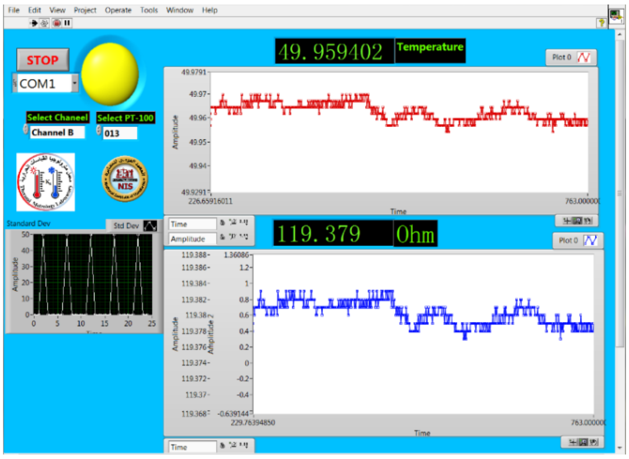


**Fig. 3**. A front interactive panel of the acquired data

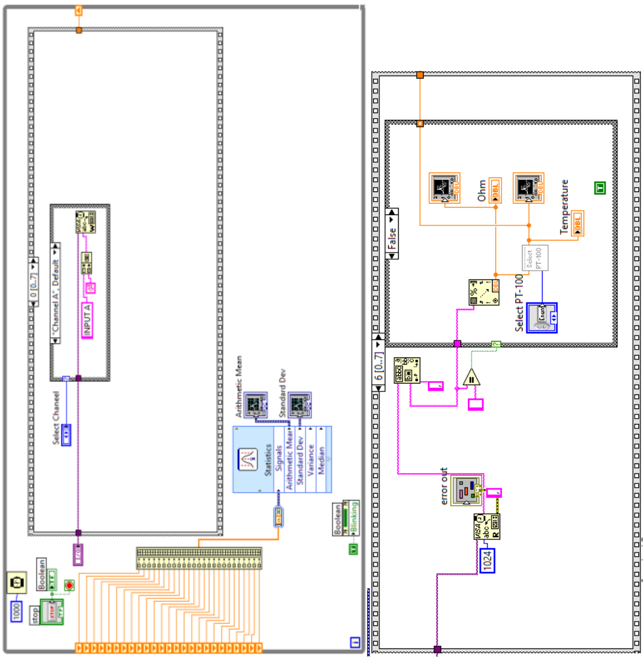


**Fig. 4**. First and last codes in the Stacked Sequence Structure

### *2.3 Callendar Van Dusen (CVD) Equation Conversion*

Temperature measurement is an indirect operation because there is no method to measure it directly. The measurement procedures measure certain physical property changes concerning the temperature. For an example, the measurement of temperature in platinum resistance thermometers based on the change in the resistance is referred as temperature changes. Measuring temperature using a resistance thermometer is considered the best and most accurate method. Platinum resistance thermometers are used for precise measurement of temperature by taking advantage of the linear change in resistance which concerns a temperature between −200 °C and 660 °C or higher with little higher uncertainty, and has a nominal value of 100 Ω.

The resistance–temperature characteristics of Pt-100 are usually expressed using Callendar Van Dusen (CVD) equation. Six Pt-100 were calibrated by comparison with the Standard Platinum Resistance Thermometer that is calibrated according to the requirements of ITS-90 in fixed points.
In the CVD Eq. (1), the resistance ratio $R(t)/R(t = 0\,°C)$ above 0 °C is expressed as R = $f\,(t)$.

$$w(t_{CVD}) = \frac{R(t)}{R(t=0\,)} \qquad (1)$$
$$R(t) = R_o\,(1 + At_h + Bt_h^2),\quad t \geq 0$$

Where, $t$ is the temperature expressed in °C, A and B are the polynomial coefficient and vary from one thermometer to another. The CVD equation is expressed by 2nd order polynomial as a function in $t$. The inverse function is $t = f^{-1}(R)$ as in Eq. (2);

$$t_h = -\frac{\sqrt{Ro\,(Ro\,A^2 - 4\,B\,Ro + 4\,B\,Rt)} + A\,Ro}{2\,B\,Ro} \qquad (2)$$

In the CVD Eq. (3), the resistance of the thermometer below 0 °C is expressed as

$$R(t) = R_o \left(1 + At_l + Bt_l^{2} + Ct_l^{3}(t_l - 100)\right), \quad t_l < 0 \tag{3}$$

Temperature can be determined using the following Eq. (4),

$$T_m = f^{-1}\left(\lim_{PV \to SP} R(T)\right) \tag{4}$$

Where, $T_m$ is the measuring temperature after the process variable (PV) temperature becomes close as possible to the set point temperature (SP). The inversion function within 1.8 m °C is as shown in Eq. (5) – (13).

$$t_l = \psi_2 + \psi_3 + 25 \tag{5}$$

$$\psi_2 = \frac{\sqrt{-9\,\psi_6^{\frac{2}{3}}\,\psi_5 - 12\,\psi_8\,\psi_5 - \left(\frac{B}{C} - 3750\right)^2 \psi_5 - 3\sqrt{6}\,\psi_9 \sqrt{2\left(\frac{B}{C} - 3750\right)^3 - 72\left(\frac{B}{C} - 3750\right)\psi_8 + 27\,\psi_9^{2} + 3\sqrt{3}\,\psi_7} - 12\left(\frac{B}{C} - 3750\right)\psi_6^{\frac{1}{3}}\,\psi_5}}{\psi_4} \tag{6}$$

$$\psi_3 = \frac{\psi_5}{6\,\psi_6^{1/6}} \tag{7}$$

$$\psi_4 = 6\,\psi_6^{1/6}\left(\left(\frac{B}{C} - 3750\right)^2 + \frac{300\,A}{C} + \frac{7500\,B}{C} + 9\,\psi_6^{\frac{2}{3}} - 6\left(\frac{B}{C} - 3750\right)\psi_6^{1/3} + \frac{12\,(Ro - Rt)}{C\,Ro} - 14062500\right)^{1/4} \tag{8}$$

$$\psi_5 = \sqrt{\left(\frac{B}{C} - 3750\right)^2 + \frac{300\,A}{C} + \frac{7500\,B}{C} + 9\,\psi_6^{\frac{2}{3}} - 6\left(\frac{B}{C} - 3750\right)\psi_6^{1/3} + \frac{12\,(Ro - Rt)}{C\,Ro} - 14062500} \tag{9}$$

$$\psi_6 = \frac{\left(\frac{B}{C} - 3750\right)^3}{27} - \frac{4\left(\frac{B}{C} - 3750\right)\psi_8}{3} + \frac{\psi_9^{2}}{2} + \frac{\sqrt{3}\,\psi_7}{18} \tag{10}$$

$$\psi_7 = \sqrt{27\,\psi_9^{4} - 256\,\psi_8^{3} - 16\left(\frac{B}{C} - 3750\right)^4 \psi_8 + 4\left(\frac{B}{C} - 3750\right)^3 \psi_9^{2} + 128\left(\frac{B}{C} - 3750\right)^2 \psi_8^{2} - 144\left(\frac{B}{C} - 3750\right)\psi_9^{2}\,\psi_8} \tag{11}$$

$$\psi_8 = \frac{25\,A}{C} + \frac{625\,B}{C} + \frac{Ro - Rt}{C\,Ro} - 1171875 \tag{12}$$

$$\psi_9 = \frac{A}{C} + \frac{50\,B}{C} - 125000 \tag{13}$$

## 3. Results and Discussion

For seven months, more than 120 runs for around 800 working hours in testing, improving techniques, trying different mechanisms and updating the calibration methods were done. Many data were recorded and acquired to find the values of prime situations and optimum conditions after a series of measurements and statistical analysis. Several assessment parameters were used to test the performance, quality and reliability of firmware, whether the examined parameters were temperature-stabled, whether the fast response to any set point changed and improved related uncertainty to build a complete image if that study make difference or not.

The stability of the temperature is the difference between the maximum and minimum during 100 cycles after controlling for the biggest 25 % of reading variations. The stability is expressed as plus or minus one-half of the recorded value and is determined by measuring the temperature in the bath's working volume in the middle. The greatest difference between the mean temperatures at any of these sites is the homogeneity, which is measured by taking temperature readings at the bath's center and corners.

Table 3 describes the measurements that were carried out at specific set points covering the whole range of study with a special basic statistics function that tests the validity of the system measurements. Figure 5 shows the overlapped temperature ranges covered by each medium. There are different liquid bathes as a thermostatic medium for hosting the thermometers with different ranges.

The normalized parameter $E_n$ was implemented to assess the performance of the process. It normalized the error in the result of two mediums in the overlapping range to expand uncertainty estimated using a coverage factor, $k = 2$. Hence $|E_n| \geq 1$ is interpreted as the result of 95 % confidence level (JCGM-100, 2008) as in Eq. (14) and (15) which is considered an unaccepted performance. Homogeneity of the liquid baths was determined using a set of pt-100 that are distributed in a uniform geometrical structure [13-15]. The stability and uniformity are summarized in Table 4.

$$(E_n)_i = \frac{x_{Medium\ 1} - x_{Medium\ 2}}{\sqrt{U^2(x_{Medium\ 1}) + U^2(x_{Medium\ 2})}}, \tag{14}$$

$$|E_n - number| = \begin{Bmatrix} < 1, & Successful\ Performance \\ Otherwise, & Unsuccesseful\ Performance \end{Bmatrix} \tag{15}$$

**Table 3**
Average selected values of measurements that were carried out

| Setting $^{o}$C | No. of runs | Operating-overlapped mediums | Median / $^{o}$C | Pt100 code | $E_n$ value, $\|E_n\|$ |
|---|---|---|---|---|---|
| -80.00 | 7 | Fluke-7381 | -80.002 | 013 | ----- |
| -50.00 | 6 | Fluke-7381 | -49.962 | 013 | ----- |
| -20.00 | 6<br>6 | Fluke-7381& aHetrofrig-2100 | -19.996<br>-19.982 | 013<br>012 | 0.023 |
| 0.00 | 6<br>8 | Fluke-7381& aHetrofrig-2100 | 0.0008<br>0.0025 | 013<br>012 | 0.028 |
| 20.00 | 6<br>6 | aHetrofrig-2100<br>Petrotest- TV4000 | 19.9909<br>20.0008 | 012<br>022 | 0.03 |
| 80.00 | 8<br>6 | Petrotest- TV4000<br>Hart scientific-6054 | 79.9961<br>79.9397 | 022<br>015 | 0.094 |
| 95.00 | 7<br>6 | Petrotest- TV4000<br>Hart scientific-6054 | 94.9998<br>94.9443 | 022<br>015 | 0.165 |
| 150.00 | 6 | Hart scientific-6054 | 149.9324 | 015 | ----- |
| 200.00 | 7<br>7 | Hart scientific-6054<br>Hart scientific-6055 | 200.0279<br>200.0301 | 015<br>013 | 0.264 |
| 220.00 | 6 | Hart scientific-6054<br>Hart scientific-6055 | 220.0246<br>220.0303 | 015<br>0.13 | 0.368 |
| 300.00 | 6 | Hart scientific-6055 | 300.0581 | 013 | ----- |
| 420.00 | 6 | Hart scientific-6055 | 420.0299 | 013 | ----- |

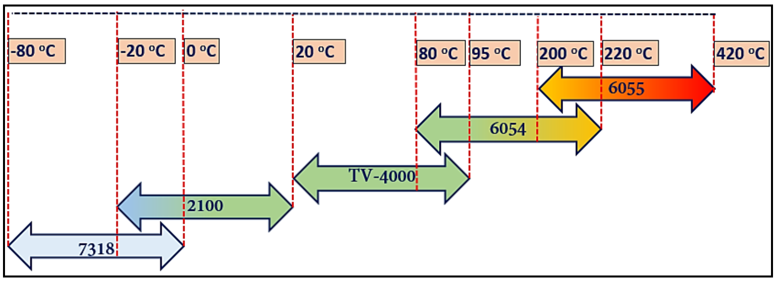


**Fig. 5**. Overlapped temperature ranges for the applied mediums

Figures 6 - 10 show the stability of temperature in minimum, maximum and overlapped checked temperatures for each bath. The stability for temperature was better than 0.04 °C, those values contributed to the uncertainty budget which shows some improvement and limitation for sources.

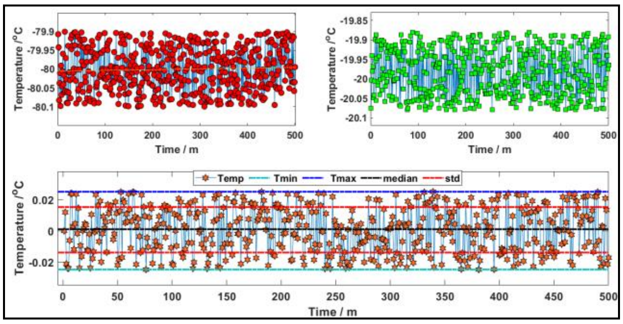


**Fig. 6**. Temperature stability at selected and overlapped set point of medium Fluke 7381

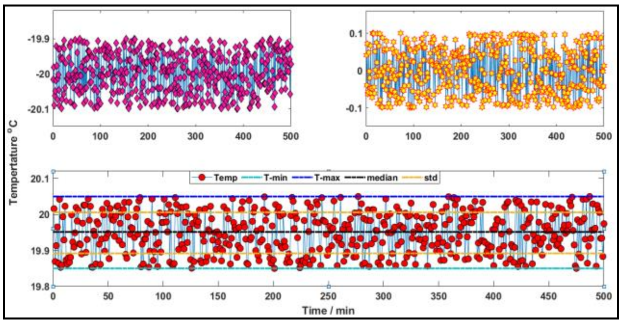


**Fig. 7**. Temperature stability at selected and overlapped set point of medium aHetrofrig-2100

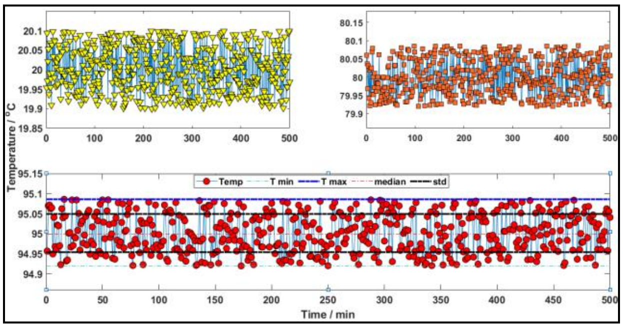


**Fig. 8**. Temperature stability at selected and overlapped set point of medium TV4000

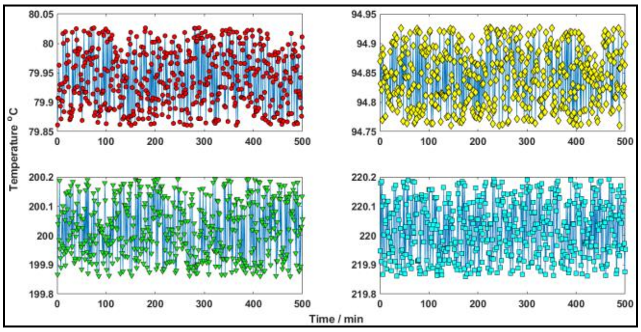


**Fig. 9**. Temperature stability at selected and overlapped set point of medium 6054

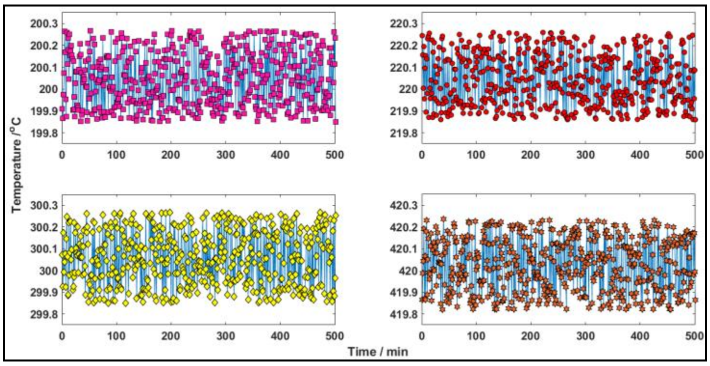


**Fig. 10**. Temperature stability at selected and overlapped set point of medium 6055

Tables 4 and 5 shows the uncertainty budget for sources that contribute to the measurements after characterization and the new firmware. Table 6 shows the published CMC scope in Egypt Accreditation Council (EGAC) [10], which is the official accreditation body in Egypt that shows the uncertainty for each calibration range.

**Table 4**
Uncertainty contributed values for each medium

| Bath | Stability / °C | Uniformity / °C |
|---|---|---|
| 7318 | 0.02 | 0.003 |
| 2100 | 0.015 | 0.005 |
| TV-4000 | 0.03 | 0.05 |
| 6054 | 0.02 | 0.03 |
| 6055 | 0.02 | 0.005 |

**Table 5**
Uncertainty budget for temperature

| Symbol | Uncertainty source | Value Xi (±°C) | Divisor | Sensitivity coefficient, Ci | Uncertainty contribution, Ui (±°C) |
|---|---|---|---|---|---|
| $U_{st}$ | Standard uncertainity | 0.005 | 1 | 1 | 0.005 |
| $U_{dri}$ | Drift of Standard | 0.01 | $\sqrt{3}$ | 1 | 0.006 |
| $U_{st}$ | Brige uncertainity | 2.65E-03 | 1 | 1 | 2.65E-03 |
| $U_{ice}$ | ice point UUT (Pt-100) | 0 | $\sqrt{3}$ | 1 | 0 |
| $U_{res}$ | Resolution | 0.005 | $\sqrt{3}$ | 1 | 0.029 |
| $U_{uni}$ | Uniformity | 0.003 | $\sqrt{3}$ | 1 | 0.0017 |
| $U_{sta}$ | Stability | 0.01 | $\sqrt{3}$ | 1 | 0.058 |
| $U_{rep}$ | Repeatability(typeA) | 0.003 | 1 | 1 | 0.003 |
| | Combined Un. | | | | 0.0106 |
| | Expanded Un. | | | | 0.02 |

**Table 6**
CMC scope

| Measured quantity | Range (°C) | Calibration and measurement capabilities* | Brief description of measurement and equipment used |
|---|---|---|---|
| Calibration of liquid in glass thermometers, digital thermometers, In stirred liquid baths (Permanent) | -80 to < 20<br>> 20 to < 90<br>> 90 to < 200<br>> 200 to 450 | ± 0.03 °C<br>± 0.07 °C<br>± 0.05 °C<br>± 0.03 °C | SOP NO NIS-32-OP-7.2.1.1-03<br>Platinum resistance thermometer<br>code 015<br>code 013<br>code 012<br>code 022 |

Figure 11 shows the comparison between uncertainty values published before and those obtained from this study. Figure 12 shows the growth and increment of baths' stability against the temperature. We can determine the stability and uniformity of the medium at any set point in between the studied one using the third-order polynomial fitting, especially if the thermometer and mediums are fixed and non-interchangeable.

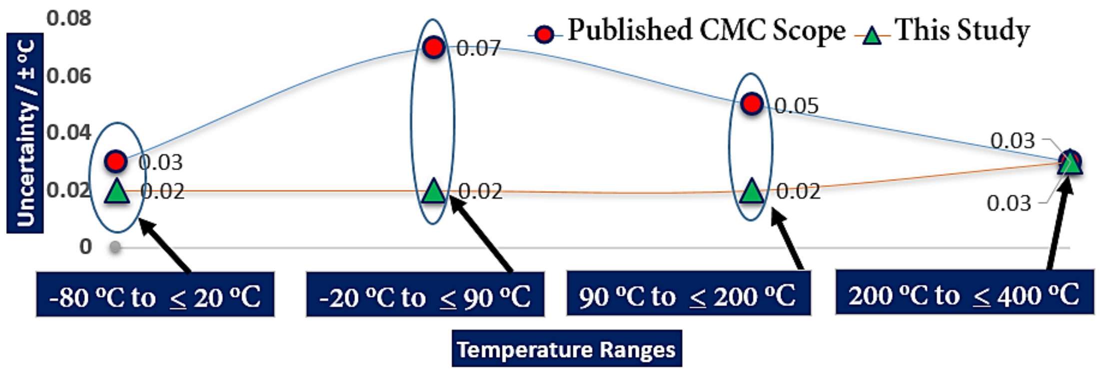


**Fig. 11.** Comparison between uncertainty values published before and those obtained from this study

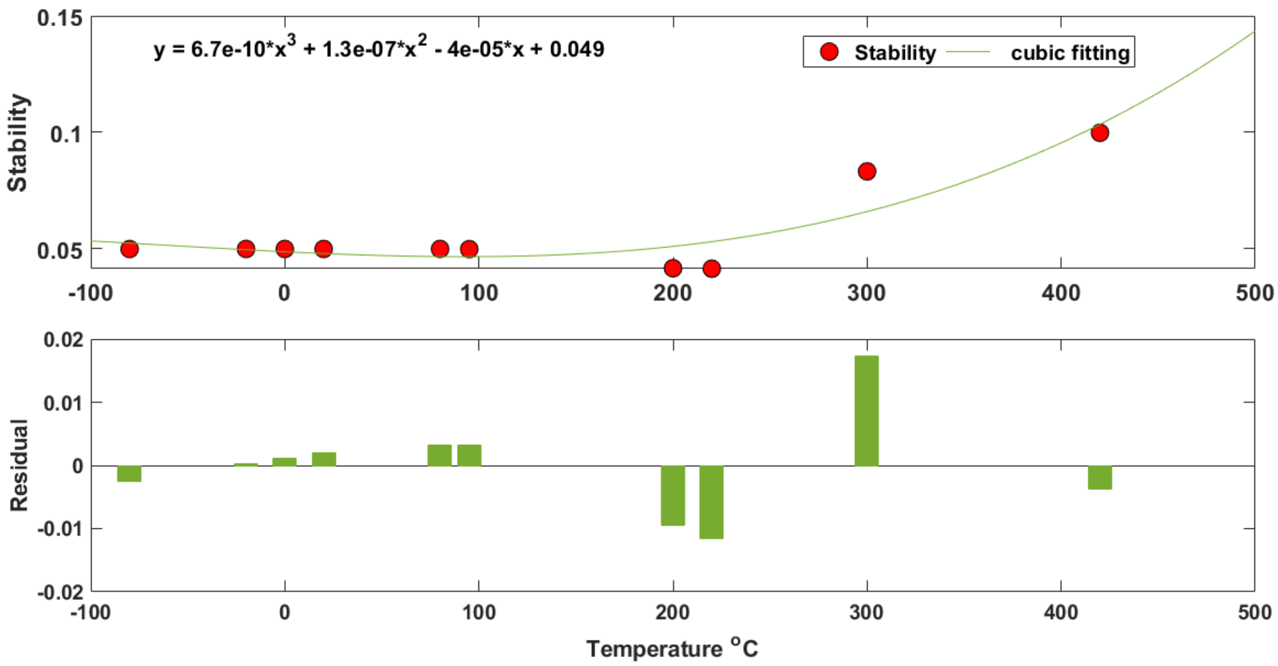


**Fig. 12**. The growth and increment of baths instability against the temperature

## 4. Conclusions

Performance examination, assessment and metrological characterization of the SLBTBS have been done. The statistical analysis of the acquired data and results show decreasing in the calibration time, operation cost and improvements in the combined uncertainty that is linked to the measurements. The modified system was introduced to routine calibration in the laboratory and fulfills the requirement of technical assessment from the Egyptian Accreditation Council (EGAC) as an official accreditation body in Egypt. The improvement of the linked uncertainty values will be published to replace the reported unified NIS scope.

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