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First Interlaboratory Comparison on Calibration of Temperature-Controlled Enclosures in Turkey

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Abstract The number of accredited laboratories in the field of calibration of temperature-controlled enclosures has been increasing in Turkey. One of the main criteria demonstrating the competence of a calibration laboratory is successful participation in interlaboratory comparisons. Therefore, TUBITAK UME Temperature Laboratory organized the first interlaboratory comparison on "Calibration of Temperature-Controlled Enclosures" in Turkey as a pilot laboratory between January and November, 2013. Forty accredited laboratories which provide routine calibration services to the industry in this field participated in the comparison. The standards used during the comparison was a climatic chamber for the measurements at −40 °C, −20 °C, 40 °C and 100 °C and an oven for the measurements at 200 °C. The protocol of the comparison was prepared considering guide EURAMET cg-20 and BS EN/IEC standards 600068-3-5 and 600068-3-11. During the comparison measurements, each participant had the liberty to choose the most convenient calibration points in terms of their accreditation scope among the values mentioned above and carried out on-site measurements at UME. The details and the results of this comparison are given in the paper. Determination of the statistical consistency of the results with the uncertainties given by the participants can be assessed by the method of E_n value assessment for each laboratory. E_n values for all measurement results based on the results of pilot and participating laboratories were calculated.

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1 Introduction

One of the main criteria demonstrating the competence of a calibration laboratory is the successful participation in interlaboratory comparisons. Accredited calibration laboratories should obey the rules of ISO/IEC 17025 for their recognition by the customer and increase the quality of their calibration services [1]. National accreditation system controls the accredited laboratories in Turkey, and TURKAK is the authorized organization for the accreditation system. The number of accredited laboratories with scope covering calibration services in the field of calibration of temperature-controlled enclosures has been increasing in Turkey. TUBITAK UME Temperature Laboratory organized the first interlaboratory comparison on calibration of temperature-controlled enclosures in Turkey during January and November, 2013.

A total of 40 accredited laboratory or candidate laboratories for accreditation participated in the comparison which was piloted by TUBİTAK UME Temperature Laboratory. The instruments used during the comparison were a climatic chamber for the measurements at −40 °C, −20 °C, 40 °C and 100 °C and an oven for the measurements at 200 °C. The protocol of the comparison was prepared by considering EURAMET cg-20 [2] and of BS EN/IEC standards 60068-3-5 [3] and 60068-3-11 [4]. During the comparison measurements, each participant laboratory had the liberty to choose the most convenient calibration points in terms of their accreditation scope among the values mentioned above and carried out on-site measurements at UME.

2 Organization of Comparison

This interlaboratory comparison was organized by TUBİTAK UME to provide and demonstrate technical proficiency and quality of calibration results of accredited laboratories as a requirement of ISO/IEC 17025 item 5.9.

Participant information and comparison results were only shared with the participating laboratories and TURKAK because of privacy principles.

The technical protocol of comparison was prepared by TUBITAK UME and published at the website of TUBİTAK UME. Finally, the protocol was sent to all participant laboratories to be approved before the comparison begins.

Participating laboratories used their own reference standards (read-out device and probes). Before comparison begins, the traceability of standards used in the comparison were ensured and declared by filling out the required form.

The instrument to be calibrated during the comparison were a climatic chamber for the measurements at −40 °C, −20 °C, 40 °C and 100 °C and an oven for the measurements at 200 °C. The climatic chamber and oven used in the comparison are listed in Table 1.

Table 1 The temperature-controlled enclosures calibrated in the comparison

Instrument	Manufacturer	Serial number	Measurement values	Dimensions (cm)
Oven	Carbolite	20-603452	200 °C	46 × 65 × 41
Climatic chamber	Weiss	59226017690010	−40 °C, −20 °C, 40 °C and 100 °C	100 × 100 × 100

3 Measurements and Results

3.1 Measurements

According to the technical protocol, the initial and final measurements at each temperature comparison point were performed by the pilot laboratory, TUBITAK UME. The difference between the results was evaluated as the drift of the oven/climatic chamber (U_{drift}) during the comparison and included in the uncertainty budget and also in E_n calculations.

Each laboratory carried out on-site measurements at TUBITAK UME using their own reference standards according to the technical protocol. TUBITAK UME assigned the working volume of the temperature enclosure (climatic chamber and oven) for the comparison per of BS EN/IEC 60068-3-5 defining the working space as the part of chamber in which the specified conditions can be maintained within the specified tolerances. This is the part of the internal volume which is designed to guarantee temperature uniformity. According to BS EN/IEC 60068-3-5, temperature-measuring sensors were located in each corner and in the center of the working space (9 sensors). The measuring system was arranged in such a way that the temperature distribution of the unloaded test chamber would not be affected. Therefore, temperature-measuring sensors of all participating laboratories were located in this arrangement. All measurements of the comparison were performed in an unloaded working space.

The temperature-controlled enclosures were observed to stabilize within at least two hours after being adjusted for a new set point. The comparison measurements were taken at least for 30 min after stabilization reached. The time allocated for each cycle of measurements taken from nine sensors was approximately 1 min. At least 270 measurement data were collected and used for analysis. Temperature variation in space refers to the difference in maximum and minimum temperature in the working space in time after the temperature has stabilized.

Briefly, measurements of the comparison were taken by considering EURAMET cg-20 and BS EN/IEC standards 60068-3-5 and 60068-3-11 and technical protocol.

3.2 Results

After completing the measurements, each participant laboratory registered the equipment they used at the comparison, and measured temperature values with their uncertainties according to the technical protocol.

All participants have calculated uncertainty taking into consideration of the components defined in Table 2 according to BS EN/IEC standard 60068-3-11. The participant laboratories submitted the uncertainty for each measured temperature.

Table 2 Uncertainty budget of the measurement at 100 °C

Uncertainty source	Standard uncertainty (°C)
Reference instrument	
Calibration	0.010
Repeatability	0.006
Hysteresis	0.020
Temperature effect	0.006
Drift	0.014
Linearity	0.001
Resolution	0.003
Chamber	
Temperature Gradient	0.206
Temperature Fluctuations	0.009
Overall mean	0.002
Radiation effect	0.087
Drift of comparison	0.049
Expanded uncertainty (k = 2)	0.45

An example for the uncertainty budget at 100 °C measurements is given in Table 2. The table involves the uncertainty parameters with the corresponding contributions as obtained by the pilot laboratory. The uncertainty budget can be handled in two main groups; the components resulted by reference standards and by the temperature enclosure (climatic chamber and oven). The most important and effective uncertainty components are detailed below. All participants were required to assess these uncertainty parameters for each comparison point.

The temperature gradient could be derived from at least 30 min data. At each temperature measurement point, the mean and standard deviation of the mean of nine sensors have been calculated. The temperature variation in the working space appeared to be one of the most dominant uncertainty parameters.

Also, for each sensor, the average and standard deviation over the period of the test were calculated. The standard deviation was a measure of the fluctuations or temporal stability. The safest assumption was to take the largest value of standard deviation values.

According to DKD-R5-7 "Calibration of Climatic Chambers" and EURAMET cg-20 guides, the radiation effect makes a maximum contribution of 0.3 °C to the measurement uncertainty between the temperature range 0 °C and 50 °C. As stated in [5], the determination of the radiation effect can take place by measurement of the temperature in the center of the useful volume using a thermometer with low emissivity as well as a thermometer with high emissivity. The difference ascertained between the two thermometers is a measure of the radiation effect if wall temperature and air temperature are not identical. During the comparison, the radiation effect was evaluated and the difference between the indicated temperatures of low and high emissivity thermometers was found to be smaller than the value suggested by the literature. Nevertheless, it was decided to suggest to take the uncertainty arising from

Table 3 Comparison results

	$D_i = t_{Lab_i} - t_{ref}$/°C	Uncertainty /°C	E_n
(a) At −40 °C in climatic chamber			
L2	0.07	1.58	−0.04
L4	0.54	0.80	−0.54
L8	0.27	0.80	−0.27
L10	0.72	1.01	−0.61
L19	0.26	1.00	−0.22
L23	0.48	0.94	−0.43
L24	1.68	2.16	−0.75
L26	0.27	0.92	−0.24
L30	0.19	0.82	−0.18
L32	0.59	1.02	−0.50
L34	0.01	1.10	−0.01
L35	0.14	1.20	−0.10
(b) At −20 °C in climatic chamber			
L1	−0.08	1.28	0.06
L3	1.05	1.03	−0.94
L8	0.82	0.60	**−1.13**
L14	0.35	0.76	−0.40
L16	0.24	0.78	−0.27
L18	0.22	0.90	−0.22
L20	0.03	0.88	−0.03
L27	0.08	1.04	−0.07
L29	−0.04	0.94	0.04
L36	1.03	1.47	−0.67
L37	−0.22	1.20	0.18
(c) At 40 °C in climatic chamber			
L2	−0.78	0.79	0.92
L8	0.11	0.60	−0.17
L22	0.02	1.00	−0.02
L31	−0.33	1.06	0.30
L33	−0.02	0.70	0.02
L35	−0.73	0.92	0.75
(d) At 100 °C in climatic chamber			
L1	−0.89	1.26	−0.66
L2	−0.97	0.84	**−1.02**
L4	0.11	0.50	0.17
L5	−0.34	1.23	−0.26
L6	0.00	0.59	0.00
L7	0.13	0.70	0.16
L8	−0.38	0.89	−0.38

Table 3 continued

	$D_i = t_{Lab_i} - t_{ref}$/°C	Uncertainty /°C	E_n
L9	0.22	1.02	0.20
L10	0.12	1.03	0.11
L11	0.32	1.10	0.27
L13	0.83	1.33	0.59
L14	−0.08	0.65	−0.10
L15	1.04	0.90	**1.04**
L17	−0.23	0.82	−0.24
L18	0.12	0.88	0.12
L19	−0.30	1.00	−0.27
L20	0.15	0.89	0.15
L21	−0.21	0.65	−0.26
L27	−0.20	1.11	−0.17
L36	−0.82	1.06	−0.71
L38	−0.07	0.93	−0.07
(e) At 200 °C in oven			
L2	0.30	1.26	0.18
L3	−2.05	4.34	−0.46
L8	−1.32	2.88	−0.43
L15	0.62	3.80	0.16
L16	−0.31	3.75	−0.08
L22	−1.20	2.83	−0.40
L23	−1.66	2.32	−0.65
L24	−0.48	4.16	−0.11
L26	−2.10	2.70	−0.72
L28	0.11	3.05	0.03
L29	−2.59	2.99	−0.81
L30	−1.08	2.00	−0.48
L31	−2.32	3.19	−0.69
L32	0.17	3.05	0.05
L33	−2.09	2.20	−0.85
L34	−1.30	3.20	−0.39
L37	−0.56	1.70	−0.28

the radiation effect in measurement uncertainty budget as 0.3 K for temperatures from −40 °C to 100 °C and 1 K from 100 °C up to 200 °C based on the fact that not all of the participating laboratories have the capability to assess radiation effect.

It was observed that the lowest uncertainties were given by TUBITAK UME. As a matter of fact, this is understandable since most of the participating laboratories were traceable to TUBITAK UME. Also the reference equipments (Pt-100 thermometers instead of thermocouples, high accuracy read-out device instead of industrial

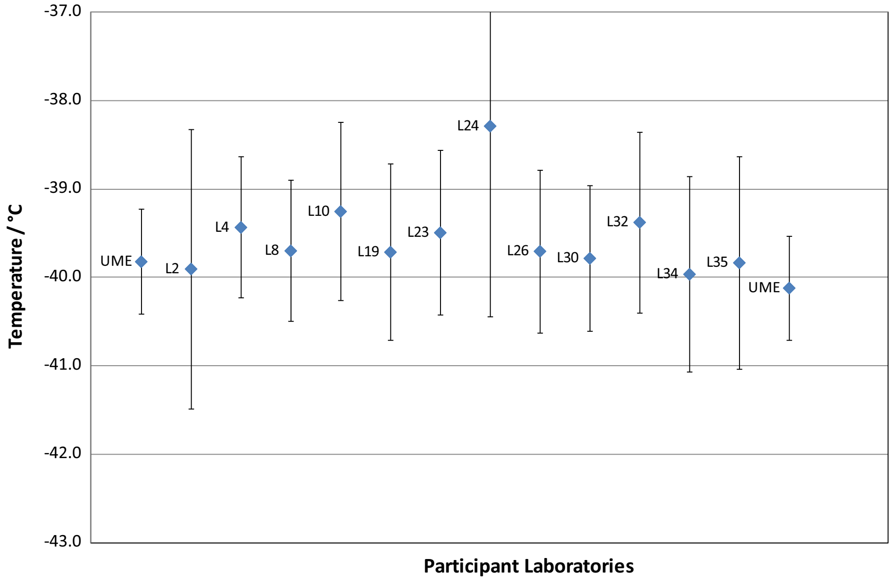

Fig. 1 Measurement results at −40 °C in climatic chamber

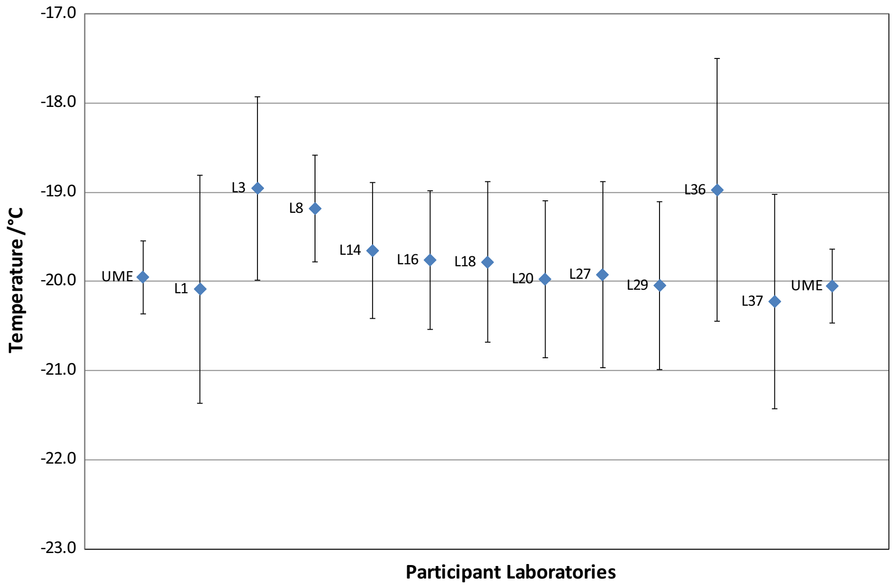

Fig. 2 Measurement results at −20 °C in climatic chamber

temperature data logger, etc) employed by pilot laboratory had lower measurement uncertainties.

During evaluation, TUBITAK UME values were regarded as reference values (t_{ref}) and the deviation of the participant laboratories' from the reference value was calcu-

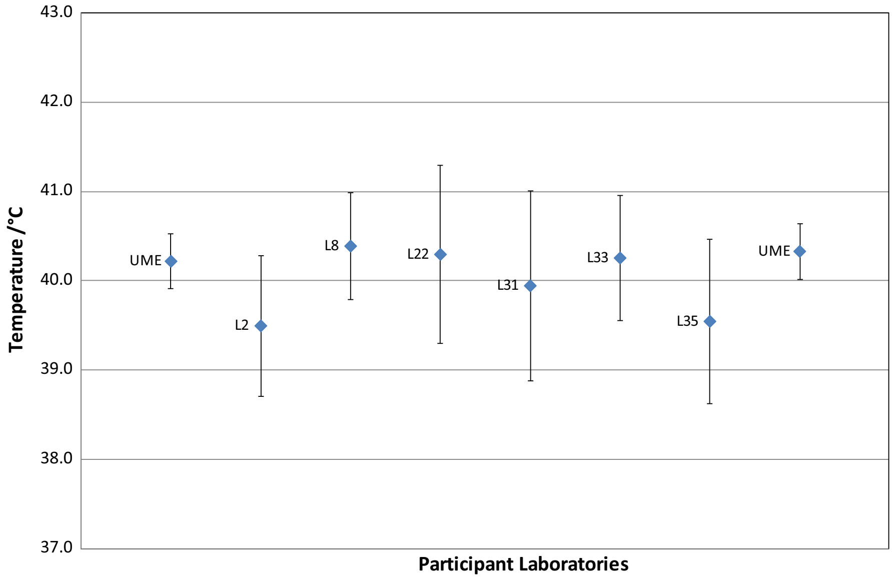

Fig. 3 Measurement results at 40 °C in climatic chamber

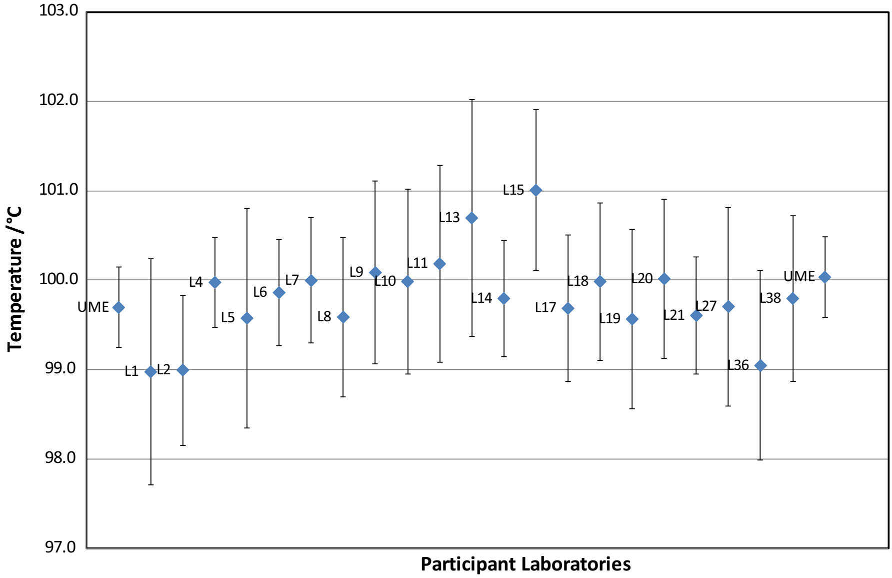

Fig. 4 Measurement results at 100 °C in climatic chamber

lated with the given uncertainties. The uncertainty associated with the reference value includes the drift over the period of comparison.

By using the assigned measurement and uncertainty values of the participants laboratories, the temperature difference (D_i) and the uncertainty (U_{D_i}) for each laboratory

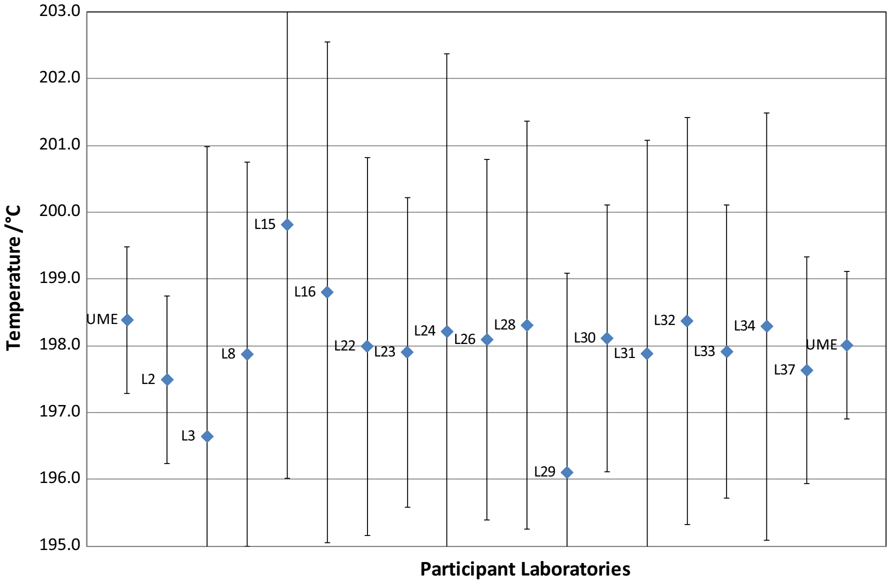

Fig. 5 Measurement results at 200 °C in oven

has been calculated according to the formula given in [6,7]

$$D_i = D_{t_{lab_i} - t_{ref}} = t_{Lab_i} - t_{ref} \tag{1}$$

$$U_{D_i} = \sqrt{(U_{Lab_i})^2 + (U_{ref})^2} \tag{2}$$

Also the E_n value has been calculated according to the formula given below:

$$E_n = \frac{t_{Lab_i} - t_{ref}}{\sqrt{(U_{Lab_i})^2 + (U_{ref})^2}} \tag{3}$$

The temperature differences obtained for each participant laboratory from the reference value with the associated uncertainty values and calculated *En* values are given in Table 3a, b, c, d and e.

The results with $|E_n| < 1$ are accepted as satisfactory, while the ones with $|E_n| \geq 1$ are accepted as unsatisfactory for a coverage factor of k = 2.

Also, Figs. 1, 2, 3, 4 and 5 show these results graphically with error bars corresponding to the expanded uncertainties of each participating laboratory.

4 Conclusion

First time in Turkey, comparison on "calibration of temperature-controlled enclosures" was organized with 40 participant laboratories including National Metrology Institute of TURKEY (TUBITAK UME) as pilot laboratory. The technical protocol of the comparison was prepared by the pilot laboratory and approved by participating laboratories. The pilot laboratory evaluated the comparison results and constituted the link between the laboratories. Finally, for each comparison temperature value, the E_n values of each participant laboratory have been determined.

When the comparison results are evaluated based on E_n values, it was seen that:

One participant at $-20\,^{\circ}\mathrm{C}$ and two other laboratories at $100\,^{\circ}\mathrm{C}$ measurements obtained E_n values greater than 1. The rest of the participants obtained E_n values smaller than 1. Even though most of the participant laboratories have demonstrated equivalent measurement results with *En* value being less than one; it is observed that in some cases this E_n value was obtained by declaration of very high uncertainty value—far away from supporting the accreditation scope—by the participant. The participant laboratories were recommended to focus on this issue and to make improvements in their measurement systems.

Finally, the comparison can be considered as successful since it enabled to clarify the status of the accredited laboratories in the field of the calibration of temperature-controlled enclosures within the scope of ISO/IEC 17025.

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