INTERLABORATORY COMPARISON IN THE SOUTH EASTERN PART OF RA VI USING CALIBRATION KIT

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1. Introduction

The subject of this report is the evaluation of the interlaboratory comparison in the field of measurement of temperature, relative humidity and pressure. Interlaboratory comparison (ILC) serves as a tool for comparison of measurement results carried out by accredited or non-accredited calibration laboratories in the relevant field of measurement. ILC represents very effective means to demonstrate technical competence of the participant and also serves as a technical base for accreditation. Furthermore, it is the most important element for monitoring of quality of measurement results as required by ISO/IEC 17025 standard for laboratories in paragraph 5.9 [1]. An extensive participation of accredited laboratories in interlaboratory comparisons/proficiency testing is required by ILAC G13:2007 [2].

2. Participants

ILC was organized by the Regional Instrument Center (RIC) of Environmental Agency of the Republic of Slovenia. RIC has invited National Hydrometeorological Services (NHMS) in the South Eastern part of Europe (RA IV) to take part in the ILC: Austria, Croatia, Hungary, Bosnia and Herzegovina, Serbia, Montenegro, Macedonia, Albania, Greece, Turkey, Romania, Bulgaria and Moldova. Additionally, Laboratory of Metrology and Quality (LMQ - holder of a Slovenian national standard for temperature and relative humidity) was also invited to participate in calibration and data analysis. The LMQ is accredited as a provider of proficiency testing/interlaboratory comparisons by the Dutch Accreditation Council (RvA) under the number R-014. At current stage of the ILC a loop of four NHMSs calibration laboratory was concluded involving following laboratories: Meteorological and Hydrological Institute of Croatia, Hungarian Meteorological Service, Environmental Agency of the Republic of Slovenia and Slovenian Laboratory of Metrology and Quality. Other NHMSs calibration laboratories did not join the ILC due to different reasons.

2.1 Coding of the participants

Each laboratory has assigned a code number under which all the laboratory results is represented. The numerical series from 100 to 110 is used for coding. These code numbers do not correspond to the alphabetical order of participating laboratories or to their measurement order. In such way confidentiality of the results is assured.

3. Comparison schedule

The measurements of the ILC were carried out in the time period from July 2007 up to July 2008 with 4 participating NHMS laboratories: Croatia, Hungary and two Slovenian laboratories (RIC Ljubljana and Laboratory for Metrology and Quality – LMQ). These measurements have been performed in steps. The RIC as a pilot laboratory made a set of four calibrations of instruments: initial calibration, calibration after each step and final calibration. In such way stability of the units under tests could be assessed. This part is very important, if we want to independently evaluate results of every particular laboratory.

4. Subject of the ILC

In all three fields of ILC (pressure, relative humidity and temperature), typical meteorological ranges, where majority of the measurements and calibrations are made, has been chosen.

4.1 Temperature

The subject of the ILC is the calibration of the self-displaying digital thermometer (Pt1000) and two Pt100 (waterproof and non-waterproof). In case of these two Pt100, direct resistance measurements had to be made by some resistance bridge or mulitimeter. The calibration was to be made in the following measurement points using standard laboratory procedures:

-30	**-20**	**-10**	**0**	**10**	**20**	**30**	**40**	°C

As a permissible temperature medium temperature controlled liquid bath using standard medium or in case of one Pt100, which is used for air temperature measurements, calibration humidity generator, climatic chamber or salt solutions should have been used.

4.2 Relative humidity

The subject of the ILC is the calibration of the capacitive hygrometer Vaisala HMP45D. The calibration was to be made in the following measurement points at temperature 20°C using standard laboratory procedures:

10	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**	**95**	% r.h.

In case of salt solution calibration other calibration points can be used covering as wide measuring range as possible. As a permissible humidity medium humidity generators or humidity chambers should have been used.

4.3 Pressure

The subject of the ILC is the calibration of the digital barometer Vaisala PTB220 ACA2A3A1AB. The calibration was to be made in the following measurement points using standard laboratory procedures:

80	**85**	**90**	**95**	**100**	**105**	**110**	kPa

As a permissible pressure medium, clean air or nitrogen without contamination by oil or dust particles should have been used. For reaching pressures lower than the atmospheric pressure only oil-free vacuum pump or pump equipped with a device for minimization of oil vapour emissions should have been used

5. Test items

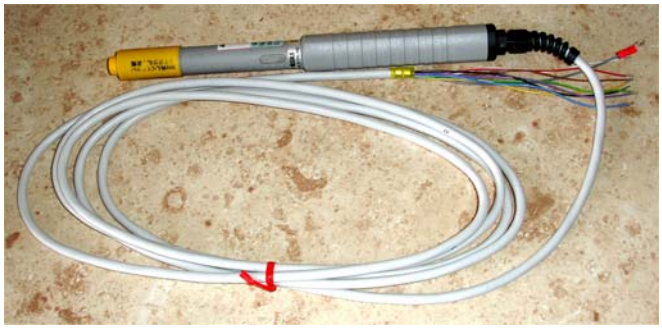
The measuring instruments were chosen from the instrument base of WMO/EARS. All of the instruments had very well-known metrological history prior to the ILC. Further more, prior to the calibration, test measurements were done in order to assess stability of the instruments. From the measurements of the laboratories, it has been concluded that all the instruments were stable enough and their short-term stability didn't influence final results of intercomparison.

5.1 Test items specification

Measuring quantity:	Relative humidity	Air Pressure
Measuring instrument:	Capacitive hygrometer	Barometer
Manufacturer:	Vaisala	Vaisala
Type:	HMP 45D	PTB220 ACA2A3A1AB
Serial number:	V3850021	A4610018
Measuring range:	(0.8 ÷ 100) %RH	(50 ÷ 110) kPa
Output:	Voltage (0..1V)	Digital display
Accuracy:	1 %RH	30 Pa

Measuring quantity:	Temperature	Temperature	Temperature
Measuring instrument:	Pt100	Pt100	Self displaying thermometer
Manufacturer:	Vaisala	Vaisala	PATTON
Type:	HMP 45D	DTS12G	GTH175/Pt – Pt1000
Serial number:	V3850021	V38505	KIT5
Measuring range:	(-40 ÷ 60)°C	(-80 ÷ 80)°C	(-199.9 ÷ 199.9) °C
Output:	Resistance	Resistance	Digital display
Accuracy:	0.2°C at 20°C	0.08°C at 0°C	-

Barometer	Temperature – Vaisala – DTS12G
	

Temperature – Vaisala – HMP45D	Temperature – PATTON – self-displaying thermometer
	

6. Environment

Ambient temperature:	(20 ± 3) °C
Relative humidity:	(30 ÷ 60) %

Barometer stabilization (warm-up) before measurements should have been made according to the common laboratory practice, but at least for 2 hours in the laboratory environment.

7. Measurement method

As to the measurement method, direct pressure/humidity/temperature comparison should have been used with reference standard of known traceability. It was recommended that the participants use their standard procedure during the calibration and if possible avoid making extra time-consuming measurements, as described in the ILC protocol.

8. Criteria for evaluation

The evaluation of the measurement results was made on the basis of the normalized error E_n. The normalized error E_n is given by the mathematic formula:

$$E_n = \frac{X_{lab} - X_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}} \qquad (1)$$

where:
X_{lab} - value measured by the participating laboratory,
X_{ref} - reference value,
U_{lab} - expanded (k=2) uncertainty of the value measured by the participating laboratory,
U_{ref} - expanded (k=2) uncertainty of the reference value.
If $|E_n| \leq 1$, the measurement result is assessed as satisfactory**,** if $|E_n| > 1$, the measurement result is assessed as unsatisfactory.

9. Measurement results

Since it is almost impossible to have calibration medium at the exact temperature, Pt100 (T,R) pairs of data were supplied by the participants at temperature values near to, but not exactly equal to the specified nominal temperatures. The participant data were therefore corrected back to the nominal temperatures in order to be compared. This was done using

standard coefficients from standard IEC 751 [4], to each of the participants data, and using the slope of this curve to correct the data back to the agreed nominal temperatures.

9.1 Reference values and its uncertainties

After correction of the data next step was to calculate reference values and its uncertainties. Data from two laboratories, RIC and LMQ, have been used in order to calculate reference values and its uncertainties. In order not to unfairly bias the calculation of the reference value to either of reference laboratories, an weighted mean (weighted by their uncertainties) of values of calibrations these laboratories data points was used. Since any uncertainties in measurements made at the same laboratory are likely to be highly correlated, we have used the formula for correlated uncertainties [3]. Measurement uncertainties also incorporate the maximum instability of the measuring instrument being identified during the reference measurements at laboratories RIC and LMQ.

$$\hat{a}_1 = \frac{\sum_{i=1}^{n} \frac{y_i}{u^2(y_i)}}{\sum_{i=1}^{n} \frac{1}{u^2(y_i)}} \qquad (2)$$

$$u^2(\hat{a}_1) = \frac{1}{\sum_{i=1}^{n} \frac{1}{u^2(y_i)}} + \frac{Max(y_i - \hat{a}_1) - Min(y_i - \hat{a}_1)}{\sqrt{3}} \qquad (3)$$

Where:

$\hat{a}_1$ - intercomparison reference value,

y_i - measurement values of reference laboratories,

$u(y_i)$- expanded measurement uncertainty of measurement values,

$u(\hat{a}_1)$ - expanded uncertainty of reference value.

9.2 En ratio calculation

Temperature		-40°C	-30°C	-20°C	-10°C	0°C	10°C	20°C	30°C	40°C	50°C
Manufacturer: Patton Type: GTH175/Pt–Pt1000 Serial number: KIT5	Lab100		0.86	0.68	0.7	0.87	0.8	0.57	0.82	0.50	
	Lab101		-0.82	-0.27	-0.06	-0.02	0.24	0.41	0.48	0.86	
	Lab102*		-	-	-	-	-	-	-	-	
	Lab103		-0.52	0.07	0.14	0.02	0.32	0.07	0.48	0.16	
Manufacturer: Vaisala Type: DTS12G Serial number: V38505	Lab100	-0.03	0.01	-0.03	-0.01	-0.02	-0.03	0.04	0.02	0.06	0.06
	Lab101	0.12	0.03	0.03	0.07	0.10	0.10	0.07	0.00	-0.13	-0.31
	Lab102*	-	-	-	-	-	-	-	-	-	-
	Lab103	-	0.40	0.28	0.26	0.32	0.37	0.36	0.34	0.31	-
Manufacturer: Vaisala Type: HMP 45D Serial number: V3850021	Lab100			-0.33	-0.17	0.01	0.11	0.18	0.24	0.33	
	Lab101			0.07	0.05	-0.01	0.01	0.05	0.03	0.05	
	Lab102*			-	-	-	-	-	-	-	
	Lab103			-0.14	-0.1	-0.23	-0.32	-0.29	-0.52	-0.36	

* Laboratory Lab102 did not report any measurement uncertainty associated to the measurements.

Relative humidity		10%	20%	30%	40%	50%	60%	70%	80%	90%	95%
Manufacturer: Vaisala Type: HMP 45D Serial number: V3850021	Lab100	0.43	0.20	0.05	0.00	0.15	0.24	0.24	0.24	0.19	0.14
	Lab101	-0.41	-0.14	-0.10	-0.01	-0.15	-0.16	-0.20	-0.01	-0.22	0.07
	Lab102*	-	-	-	-	-	-	-	-	-	-
	Lab103	0.07	0.40	0.23	0.41	0.23	0.16	0.11	0.12	0.13	0.21

* Laboratory Lab102 did not report any measurement uncertainty associated to the measurements.

Pressure		800 hPa	850 hPa	900 hPa	950 hPa	1000 hPa	1050 hPa	1100 hPa
Manufacturer: Vaisala Type: PTB220 ACA2A3A1AB Serial number: A4610018 - **CELL 1**	Lab101	0.03	0.04	0.06	0.08	0.08	0.09	0.06
	Lab102*	-	-	-	-	-	-	-
	Lab103	0.05	0.06	0.12	0.04	0.26	0.22	0.22
Manufacturer: Vaisala Type: PTB220 ACA2A3A1AB Serial number: A4610018 - **CELL 2**	Lab101	0.07	0.09	0.07	0.03	0.05	0.04	0.03
	Lab102*	-	-	-	-	-	-	-
	Lab103	0.31	0.30	0.34	0.26	0.42	0.33	0.39
Manufacturer: Vaisala Type: PTB220 ACA2A3A1AB Serial number: A4610018 - **CELL 3**	Lab101	0.07	0.06	0.02	0.04	0.08	0.09	0.10
	Lab102*	-	-	-	-	-	-	-
	Lab103	0.28	0.22	0.23	0.22	0.37	0.34	0.39

* Laboratory Lab102 did not report any measurement uncertainty associated to the measurements.

10. Conclusion

From the results made by the participants as well as from the analysis of the results, it can be concluded that quality of the provided results is of high order. Even for the laboratory 102, which didn't provide with any uncertainty calculation, if we take typical uncertainties, which can be achieved with their equipment, their results are also very good, with all the values of E_n smaller than 1. In future it is expected that more laboratories take part in ILC.
The organization and evaluation of the ILC has been made in accordance with ILAC guide G13:2007 and ISO/IEC Guide 43-1:1997. These two documents represent current state of the art on the field of ILC.
Summary of the results is presented in the following table.

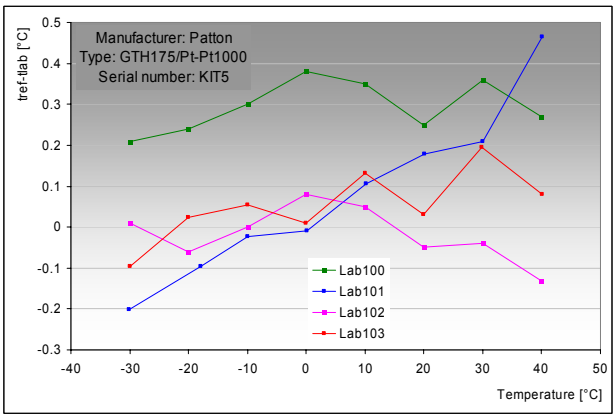
Code	Overall number of measurement performed	Number of satisfactory measurement results	Percentage of satisfactory measurement results in %
Lab 100	**35***	**35**	**100**
Lab 101	**56**	**56**	**100**
Lab 102	**56**	******	******
Lab 103	**55**	**55**	**100**

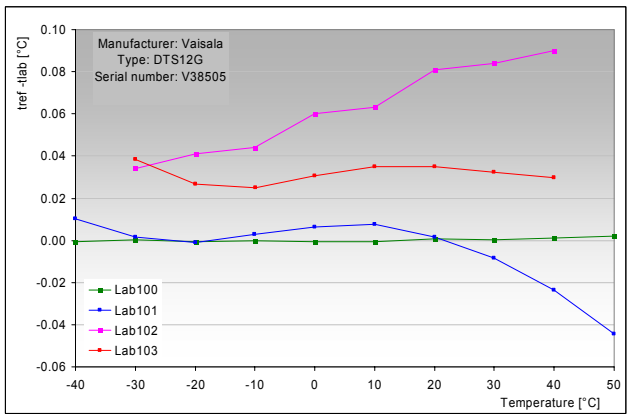
* Laboratory Lab100 did not participate in pressure part of the ILC.
** Laboratory Lab102 did not report any measurement uncertainty associated to the measurements.

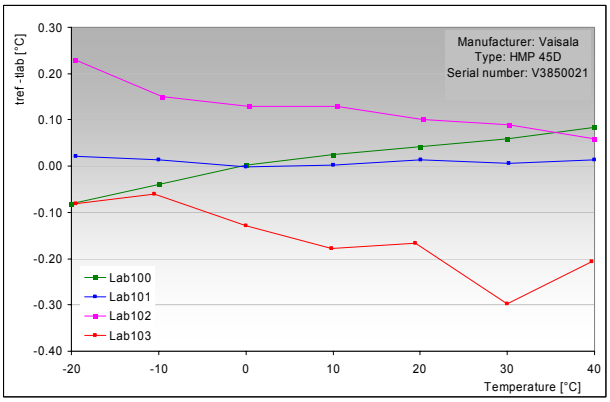
11. Reference

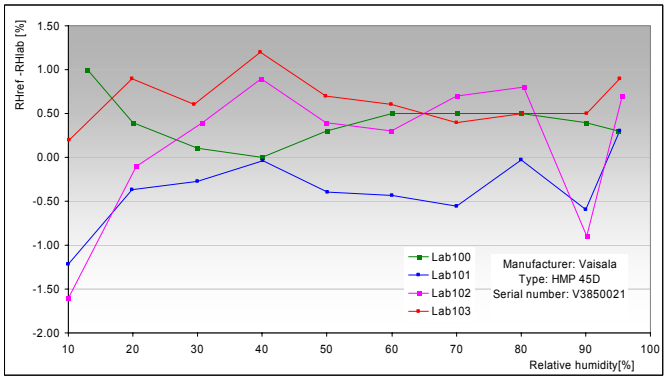
[1] ISO/IEC 17025: 2005: General requirements for the competence of testing and calibration laboratories
[2] ILAC Guide 13, 2007: Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes
[3] Nielsen Lars, Danish Institute of Fundamental Metrology, February 2000
[4] IEC 751:1995: Industrial platinum resistance thermometer sensors
[5] ISO/IEC Guide 43-1:1997: Proficiency testing by interlaboratory comparisons – Part 1

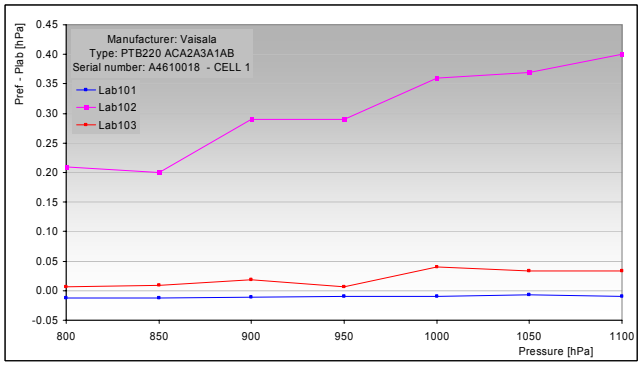
Appendix 1: Graphical presentation of measurement results

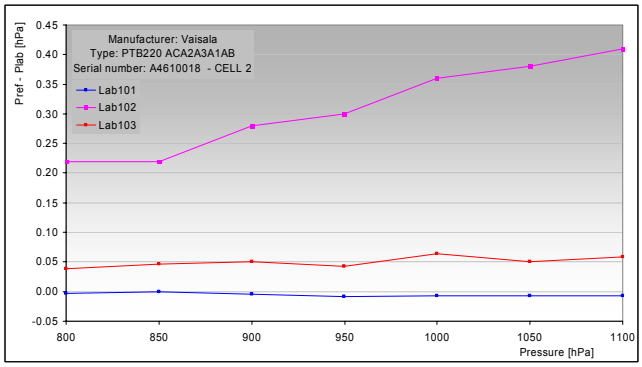


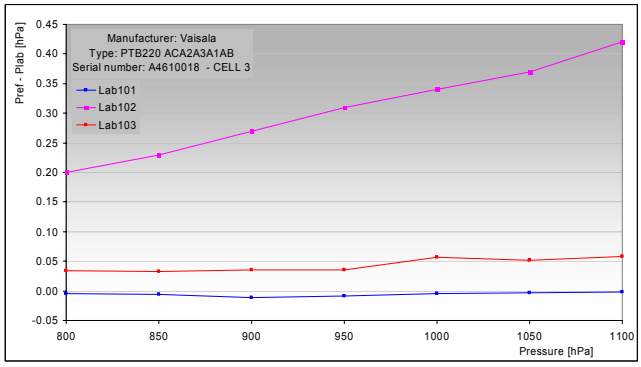












Appendix 2: Graphical presentation of En values