University *of Ljubljana*
Faculty *of Electrical Engineering*

PT protocol

INSTRUCTION FOR THE PARTICIPANTS IN THE PROFICIENCY TESTING SCHEME

Title: Intercomparison with SPRTs – IPA PT 1 - T

Date:
11.03.2010

Item:
Two Fluke HartScientific 25 Ω standard platinum resistance (SPRT) thermometers (serial numbers 1559 and 0374)

Coordinator:
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Laboratory of Metrology and Quality (MIRS/FE-LMK), RvA Reg: R-014
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Reference laboratory:
MIRS/UL-FE/LMK, SA Reg.: LK-002 (http://www.sa.gov.si/teksti-1/slo/katalog.htm)
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TABLE OF CONTENT

1 Introduction

The purpose of the proficiency test is to compare the results of the participating laboratories during calibration of a SPRT at fixed points in the range from the triple point of argon (-189,3442 °C) to the freezing point of zinc (419,527 °C) and at the same time to support the Calibration and Measurement Capabilities (CMC) entries of the participating laboratories. The IPA PT 1-T is using the same technical protocol used in the EURAMET comparison P. 552.

The circulating items are two Fluke HartScientific 25 Ω standard platinum resistance (SPRT) thermometers, one metal sheathed, type 5699, serial number 0374 and one quartz sheathed, type 5681, serial number 1559. The proficiency test includes six fixed points of ITS-90 in the range between -190 °C and 420 °C. The diameter of the metal sheath probe is Ø 5,6 mm and of the quartz sheath is Ø 7 mm. Both probes should be immersed in the fixed point at least 180 mm.

It is recommended that the participants use their standard procedure for the realization of ITS-90 and follow instructions from this protocol during the temperature calibration and if possible avoid making extra time-consuming measurements. The proficiency test is carried out in accordance with ILAC-G13:2007 and ISO Guide 17043, 2010.

1.1 Coordinator/Reference laboratory

University of Ljubljana, Faculty of Electrical Engineering
Laboratory of Metrology and Quality (MIRS/FE-LMK),
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1.2 Participants

There are six participants to this intercomparison. Contact details are as listed alphabetically:

Albania

Participating laboratory:	GDMC-General Directorate of Metrology and Calibration (AL)
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Measuring instructions for participants of IPA PT 1-T intercomparison

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Turkey
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1.3 Time schedule and deadlines

The deadlines for the calibrations appear in Table 1. Depending on the number of fixed point cells and measurements that have to be taken, all participating laboratories have from two weeks to seven weeks for calibration including transport to the next laboratory. The transport must be planned for each of the participating laboratories, so that the subsequent laboratory receives the equipment no later than on Monday in the first week, in which the calibration is planned to be carried out:

RS: 6. April 2010; TR: 1. June 2010; HR: 5. July 2010; MK: 2. August 2010; AL: 16. August 2010; SI: 30. August 2010

If a participant anticipates difficulties in keeping the deadlines, the coordinator must be contacted immediately. In such a case the other participants will be contacted as soon as possible and be informed about eventual changes.

Deadline for reporting the result is 2 weeks after the equipment has left the laboratory. It is important that the deadline is met since the results are being analyzed continuously by the reference laboratory. If there are any problems or doubt regarding the results of the participant laboratory, the laboratory will be contacted immediately. Any suspicion that the equipment is defect or drifted, will lead to return of the equipment to the reference laboratory, which then will make an extra check and take an appropriate action.

If deadlines are respected, the participants will receive the report of the proficiency test before 10. November 2010.

The first measurement in the laboratory RS is expected to start in 15th week of this year (6.April .2010).

Table 1: Time schedule

Measuring instructions for participants of IPA PT 1-T intercomparison

2010

	weeks	March				April				May				June				July				August				September				October				November
	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
MIRS/UL-FE/LMK	X	X	X	X	X	X																															
RS							X	X	X	X	X	X	X	X																							
TR															X	X	X	X	X																	
HR																				X	X	X	X													
MK																								X	X											
AL																										X	X									
MIRS/FE-LMK																												R	R	R	R	X	X	X	X	X	X

R - reserved for any unexpected delay

1.4 Transportation of the equipment

As soon as the equipment is delivered/sent, the coordinator shall be informed (e.g. by e-mail). The equipment is then unpacked, and an inspection carried out. If the equipment has any visible damage due to transportation, this must be reported to the coordinator before the calibration begins.

The equipment is hand carried (personal transport) to the next laboratory.

The participating laboratory covers expenses of transportation to the next laboratory.

2 Description of the equipment

2.1 General

Four Standard Platinum Resistance Thermometer (SPRT) are supplied as the circulated instruments by reference laboratory. Two of them are sent around. The second pair is kept for safety at the reference laboratory. The SPRTs used for this comparison will be selected by the reference due to their very good stability.

The laboratory receives the following equipment:

- SPRT Fluke Hartscientific
  - Type 5699
  - Serial number 0374
  - Metal sheath
  - Ø 5,6 mm
  - Sensor length 5 cm
  - Measurement current < 2 mA
- SPRT Fluke Hartscientific
  - Type 5681
  - Serial number 1559
  - Quartz sheath
  - Ø 7 mm

Measuring instructions for participants of IPA PT 1-T intercomparison

- Sensor length 5 cm
- Measurement current < 2 mA

In a case any of the above-mentioned equipment is missing at the receipt, the coordinator must be contacted.

2.2 Environmental conditions

Calibration is carried out at an ambient temperature of nominal 23 °C. The ambient temperature shall be reported.

2.3 Handling

2.3.1 Packing and unpacking

Procedure for unpacking is as follows:

1. Inspect the transportation boxes for damage. If the boxes are damaged, the coordinator shall be contacted before continuing.
2. Unpack the equipment and check that all equipment mentioned in the section "Description of equipment" is present.
3. If any equipment is missing, the coordinator shall be contacted.
4. Inspect the equipment. If any of the equipment shows visible signs of damage, the coordinator shall be contacted.

The packing procedure is as follows:

1. Before packing, slowly cool down thermometers to room temperature and clean them with pure alcohol.
2. Place two SPRTs in their transportation box.
3. Check that all equipment mentioned in the section "Description of equipment" is packed before the equipment is transported to the next participant

2.3.2 Mounting

1. The SPRTs are cleaned before use with pure alcohol.
2. Thermometers are carefully placed in the calibration media (annealing furnace, or fixed point cell).
3. The SPRTs are connected to resistance bridge using 4 wire connection.

2.3.3 Precautions

- SPRTs are very sensitive device to vibration and mechanical shock.
- When not in use, it should be stored in a safe place in the provided transport boxes.
- Don't use current higher than 2 mA
- Check that the thermometers are completely clean and dry before placing them in the calibration media.
- Ensure that the thermometers are cooled down and cleaned with pure alcohol before placing them in the transportation box.

Refer to the manual (Chapter 5 Care and Handling Guidelines) or contact the coordinator in a case of doubt about the above-mentioned precautions.

3 PT protocol

The IPA PT 1-T is using the same technical protocol used in the EURAMET comparison P. 552
The MIRS/UL-FE/LMK will perform measurements at the beginning of the loop and at the end.
For the purpose of checking the performance of the equipment (e.g. operation) during the progress of the loop, first the measurements as described in section Inspection/Start-up are carried out. Hereafter the actual temperature calibration at fixed points is carried out.
It is recommended that the participants use their standard procedure for the realization of the ITS-90 and instructions from this protocol during temperature calibration and avoid making extra time-consuming measurements, if possible. For accredited laboratories it will be advantageous to apply the accredited procedures in preparation for later use of the report in relation with documentation to the accreditation body.
Details about the applied procedure can be stated in the report form.

3.1 Inspection/Start-up

1. Measure both thermometers at the triple point of water (TPW).
2. Correct for hydrostatic head and self heating.
3. Note the resistance for 0 mA current of both probes
4. Report the values from step 3 to the coordinator and wait before starting with further measurements.

3.2 Measuring protocol

After receiving approval from the reference laboratory to proceed with the PT, the participating laboratory can begin with measurements:

Measuring instructions for participants of IPA PT 1-T intercomparison

1. If laboratory has fixed points in the range including a freezing point of zinc, the SPRT annealing procedure is performed:

- Carefully insert the SPRT into a annealing furnace at 470 °C.
- Anneal the SPRT for two hours at 470 °C
- Carefully remove the SPRT from the furnace directly to the room environment.
- Re-determine the value of R_{TPW}

  If the resistance at TPW increases after annealing, contact reference laboratory for further instructions

  If the decrease in the TPW resistance of the SPRT after annealing is equivalent to 0,5 mK or larger proceed to a second SPRT annealing procedure. Re-determine the value of R_{TPW}.

  If the decrease in the calculated TPW resistance of the SPRT after second annealing is larger than 0,2 mK contact reference laboratory for further instructions

  If the decrease is less then 0,5 mK progress with measurements at fixed points.

2. If laboratory only has fixed points bellow a freezing point of zinc, no annealing is performed !!!

Calibrate the SPRT at all of the fixed points in the range of comparison, i.e., measurements at
TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg, TPW, Ar and TPW, in that order. If one or several fixed points are not available then the host laboratory may perform the comparison over a limited range. Existing techniques as used by the participating laboratory must be used.
In order to not increase the uncertainty on the comparison of the results the R_T values given by the different participants must approximately correspond to the same percentage of metal in liquid phase. This percentage is not easy to determine. So it is better to use the concept of percentage of time of the total duration of the plateau. It is asked that the R_T values correspond to the percentage of time given in the table below.

Fixed point	Type	% of time passed since the starting of the plateau
Ar	Triple	20 to 40 %
Hg	Triple	60 to 80 %
Ga	Melting	70 to 80 %
In	Freezing	20 to 30 %
Sn	Freezing	20 to 30 %
Zn	Freezing	20 to 30 %

Preferably, the measurements must correspond to the percentage given in the above table. But it is possible to use the measurements obtained in other conditions. In this case, two complementary information must be given in the report:

1- the approximate percentage of time at the moment of the measurement
2- an estimate of the potential difference between the values corresponding to the recommended percentage of time and to the percentage of time used by the participating laboratory

For each metal fixed point the $W=R_T/R_{TPW}$ is calculated. R_{TPW} is the TPW resistance measured immediately after the measurement of R_T. All the measurements at the fixed points should have been corrected for self-heating, hydrostatic head and, if any, the pressure effect.
At least 3 different phase transitions (3 freezing for Zn, Sn, In, 3 melting for Ga, 3 triple points for Hg and Ar should be performed). All three measurements for each fixed point are reported in the Excel spreadsheet including the calculated mean.

3.3 Reporting of results

The results are reported electronically in the forwarded Excel spreadsheet files (IPA2008PT1_T_CALIBRATIONDATA.xls, IPA2008PT1_T_Instrumentationdetails.xls, IPA2008PT1_T_Uncertaintyanalysis.xls). The green fields of the spreadsheets should be filled in, if possible.
In the report form, the participants are also asked to fill in details about the applied method, uncertainty sources, equipment and traceability, if this information does not appear from an issued calibration certificate.
The participants should send to reference laboratory the following information as well.

- For each fixed point cell that was used in the comparison, determine (using the circulating SPRT) and plot the change of phase transition temperature, dT, versus immersion, dh. On the same graph, plot the theoretical dT/dh curve using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text.
- Examples of Freezing curves in In, Sn and Zn cells, melting curve in Ga cell and triple–point curve in Hg and Ar

3.3.1 Uncertainties

Participants are requested to use the attached spreadsheet 'IPA2008PT1_T_Uncertaintyanalysis.xls' to calculate and report their estimated uncertainties for the determination of resistance ratios obtained from the SPRT at the fixed points that were used in this comparison. Calculations of uncertainties should follow the guidelines set out in the ISO Guide (1993) to the Expression of Uncertainty in Measurement.

Measuring instructions for participants of IPA PT 1-T intercomparison

For each uncertainty component, a standard uncertainty u_i and its associated degrees of freedom must be provided. The value of u_i should be given in terms of temperature. For type A evaluation, the number of degrees of freedom, is $n-1$ where n is the number of measurements. For type B evaluation, any input is assumed to have an infinite number of degrees of freedom. The combined uncertainty U, the effective degrees of freedom and, subsequently, the expanded uncertainty at 95% level of confidence are calculated as set out in the Guide.

To assist with the determination of measurement uncertainties, the following section explains the meanings of the uncertainty components given in 'IPA2008PT1_T_Uncertaintyanalysis.xls'.

W_t is determined according the following mathematical model obtained from the relationship.

$$W_t = \frac{(R_s + C_{Rs/3} + C_{Rs/4})*(X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(R_s)*(X_{0.01°C} + C_{X\,0.01/1} + C_{X\,0.01/2} + C_{X\,0.01/3} + C_{X\,0.01/4} + C_{X\,0.01/5} + C_{X\,0.01/6} + C_{X\,0.01/7})}$$

$$W_t = (1 + D_{Rs/3} + D_{Rs/4}) \cdot \frac{(X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(X_{0.01°C} + C_{X\,0.01/1} + C_{X\,0.01/2} + C_{X\,0.01/3} + C_{X\,0.01/4} + C_{X\,0.01/5} + C_{X\,0.01/6} + C_{X\,0.01/7})}$$

Where

Rs	reference resistor value at the time of TPW measurement
$D_{Rs/3}$	relative drift of the resistance of the reference between TPW and FP measurements $= C_{Rs/3} / R_s$
$D_{Rs/4}$	relative temperature variation of resistance of the reference between TPW and FP Measurements = $C_{Rs/4} / R_s$

Effects linked with triple point of water calibration:

$X_{0.01°C}$	reading on the bridge at the triple point of water

$C_{x0.01/1}$	water triple point reference including isotope variation
$C_{x0.01/2}$	Hydrostatic pressure correction
$C_{x0.01/3}$	Perturbing heat exchanges
$C_{x0.01/4}$	Self-heating correction
$C_{x0.01/5}$	Bridge linearity
$C_{x0.01/6}$	Ac/dc measurement correction
$C_{x0.01/7}$	SPRT internal insulation leakage correction

Effects linked with the considered fixed point calibration:

X_t	Reading on the bridge
$C_{Xt/1}$	Chemical impurities
$C_{Xt/2}$	Hydrostatic pressure correction
$C_{Xt/3}$	Perturbing heat exchanges
$C_{Xt/4}$	Self-heating correction
$C_{Xt/5}$	Bridge measurement correction, lack of linearity
$C_{Xt/6}$	Ac/dc measurement correction
$C_{Xt/7}$	Gas pressure correction

S_{Wt}	W_t scatter

Any participant can complete this table with additional component for taking in account specific experimental conditions. In particular, it could be necessary to include a component linked with SPRT internal insulation leakage correction at the Ga fixed point. On the other hand, if component is considered as negligible they have to be quoted as "negligible" and it value must be justified

Combined standard uncertainty on Wt

$$\sigma^2{}_{Wt} = \left(\frac{\delta Wt}{\delta D_{RS/3}}\right)^2 * \sigma^2{}_{D_{RS/3}} + \left(\frac{\delta Wt}{\delta D_{RS/4}}\right)^2 * \sigma^2{}_{D_{RS/4}}$$
$$+ \left(\frac{\delta Wt}{\delta X_{0.01°C}}\right)^2 * \sigma^2{}_{X_{0.01}} + \left(\frac{\delta Wt}{\delta C_{X\,0.01/1}}\right)^2 * \sigma^2{}_{C_{X\,0.01/1}} + ... + \left(\frac{\delta Wt}{\delta C_{X\,0.01/7}}\right)^2 * \sigma^2{}_{C_{X\,0.01/7}}$$
$$+ \left(\frac{\delta Wt}{\delta X_t}\right)^2 * \sigma^2{}_{X_t} + \left(\frac{\delta Wt}{\delta C_{Xt/1}}\right)^2 * \sigma^2{}_{C_{Xt/1}} + ... + \left(\frac{\delta Wt}{\delta C_{Xt/7}}\right)^2 * \sigma^2{}_{C_{Xt/7}}$$
$$+ 2 \cdot \rho_1 \cdot \left(\frac{\delta Wt}{\delta C_{X\,0.01/1}}\right) \cdot \left(\frac{\delta Wt}{\delta C_{Xt/1}}\right) \cdot \sigma_{C_{X\,0.01/1}} \cdot \sigma_{C_{Xt/1}}$$
$$+ .....$$
$$+ 2 \cdot \rho_6 \cdot \left(\frac{\delta Wt}{\delta C_{X\,0.01/6}}\right) \cdot \left(\frac{\delta Wt}{\delta C_{Xt/6}}\right) \cdot \sigma_{C_{X\,0.01/6}} \cdot \sigma_{C_{Xt/6}} + S_{W_t}$$

The values of $\rho_1, \rho_2, \rho_3, \rho_4, \rho_5, \rho_6$ will be taken as equal to Zero if the laboratory have not better information on these values. Taking these values as zero is justified because :
1) $\delta W_t / \delta C_{X0.001/i}$ is negative
2) The values of $\rho_1, \rho_2, \rho_3, \rho_4, \rho_5, \rho_6$ are positive.

Consequently to give a null value to these correlation coefficients leads to maximise the value of σ_{Wt}^2 .

$$\sigma^2{}_t = \left(\frac{\delta t}{\delta W_t}\right)^2 * \sigma^2{}_{W_t}$$

In sheet "IPA2008PT1_T_Uncertaintyanalysis.xls" the sensibility coefficient correspond to

$$\left(\frac{\delta t}{\delta W_t}\right) * \left(\frac{\delta W_t}{\delta i}\right)$$

for example the sensibility coefficient linked with the quantity $C_{Xt/1}$ is

$$\left(\frac{\delta t}{\delta W_t}\right) * \left(\frac{\delta W_t}{\delta C_{Xt/1}}\right)$$

Components explanation and proposal of evaluation

Quantity	Standard Uncertainty	Method	Evaluation
X_t	Repeatability of readings. No change during a short time	- Same SPRT - Same cell - Same freezing - Same day	Type A
$C_{Xt/1}$	Purity	- During the recent EUROMET Workshop Dr B.Fellmuth from PTB explained clearly that it was physically impossible to quote the uncertainty linked to the impurities in simply using the Raoult's Law. Therefore it is proposed to quote this uncertainty from the dispersion of a batch of cells. This batch can be the property of the laboratory or it is the set of cells which have been participated to previous comparison	
$C_{Xt/2}$	hydrostatic pressure correction	Estimated from the uncertainty of the sensible element position and the uncertainty of the free liquid level	Established by the Laboratory
$C_{Xt/3}$	Perturbing heat exchanges (between the sensor and the surrounding parts different in temperature from the liquid-solid phase change)	-Deviation from expected hydrostatic pressure correction obtained by changing immersion depth over 5 cm (length of the sensor) -Modification of the thermal exchange between thermometer and its environment	Established by the Laboratory Type B (Max value-Min value)/ 2√3

		-Use of different container design	
$C_{Xt/4}$	self-heating correction	Resolution of the bridge readings, uncertainty on the ratio between the two measuring currents Variation in self heating correction observed in an apparent similar environment	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{Xt/5}$	bridge linearity	Use of calibrated resistor for checking the bridge. Comparison between readings on different bridges. Checking the symmetry of the bridge (R1/R2 = 1/(R2/R1) ?)	Established by he Laboratory or btained in cientific literature
$C_{Xt/6}$	Difference between AC and DC measurements	Estimated by using DC and AC bridge	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{Xt/7}$	Gas pressure in the cell	Uncertainty on neutral gas pressure value during fixed point. 1 – open cells: uncertainty on line pressure measurement 2 - sealed cells: uncertainty on pressure measurement during sealing combined with temperature profile	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3

Measuring instructions for participants of IPA PT 1-T intercomparison

Quantity	Standard Uncertainty	Method	Evaluation
$X_{0.01°C}$	a) Repeatability of readings. No change during a short time	- Same SPRT - Same cell and same mantle realization - Same day	Type A
	b) Repeatability of temperature realised by cell	- Same SPRT (assumed stable) - Same cell - Different realisations of the mantle (1) - Different dates of measurement for take into account mantle ageing (2)	Reasonably large set of data: type A Small number of data: type B (1)PD rectangular and symmetrical: (Max value-Min value)/ 2√3 (2)PD rectangular and not symmetrical: (Max value-Min value)/ √3
	c) Short Repeatability of SPRT to be calibrated	- Same cell - Variation between TPW measurement before and after the considered fixed point	
$C_{X0.01/1}$	Purity and isotopic composition	Comparison between several cells from different sources in the same conditions. Use of the interlaboratory comparison data.	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{X0.01/2}$	Hydrostatic pressure correction	Estimated from the uncertainty on the distance between the platinum sensor and the free liquid level	Established by the Laboratory Type B (Max value-Min value)/ 2√3
$C_{X0.01/3}$	Perturbing heat exchanges (between the sensor and	-Deviation from expected hydrostatic pressure	Established by the Laboratory

	the surrounding parts different in temperature from the liquid-solid phase change)	correction obtained by changing immersion depth over 5 cm (length of the sensor) -Modification of the thermal exchange between thermometer and its environment -Use of different container design	Type B (Max value-Min value)/ 2√3
$C_{X0.01/4}$	self-heating correction	Resolution of the bridge readings, uncertainty on the ratio between the two measuring currents Variation in self heating correction observed in an apparent similar environment	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{X0.01/5}$	bridge linearity	Use of calibrated resistor for checking the bridge. Comparison between readings on different bridges. Checking the symmetry of the bridge (R1/R2 = 1/(R2/R1) ?)	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{X0.01/6}$	Difference between AC and DC measurements	Estimated by using DC and AC bridge	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{X0.01/7}$	SPRT internal Insulation leakage (if any)	Decrease in resistance over some hours in the triple point	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3

Quantity	Standard Uncertainty	Method	Evaluation
$D_{RS/3}$	Lack of stability of the reference resistance value	Negligible if measurement performed in a short time (within two successive days)	Established by he Laboratory Type B (Max value-Min alue)/ √3 (PD: rectangular o symmetrical) (Max value-Min value)/ 2√3 (PD: rectangular symmetrical)
$D_{RS/4}$	Change in value of the standard resistor with thermostat temperature	- uncertainty on calibrating temperature - uncertainty on temperature at time of use - uncertainty on temperature coefficient	Established by the Laboratory Type B (Max value-Min alue)/ 2√3
S_{Wt}	W_t scatter	- Same SPRT - Same cell - Different W values	Reasonably large set of data: type A Small number of data: type B PD rectangular and symmetrical: (Max value-Min value)/ 2√3 Different days

The laboratories which normally issue calibration certificates (e.g. the accredited laboratories), should send a standard certificate to the coordinator.
The results shall be sent to the coordinator no later than **2 weeks** after having finalized the calibration. Electronic reporting by e-mail is preferred.

Outline of statistical analysis

The assigned values are to be determined as the arithmetic mean of measurements made by participants. Any outliers are detected by Cochran's test and Grubbs' test (ISO 5725-2, 7.3.3 and 7.3.4).

Information to be returned to participants

The participants will receive summary of all measurements, assigned values and uncertainties of assigned values, and table of degree of equivalence, as required by BIPM MRA and presented in report of EURAMET 552, chapter 11.

4 Appendix A: Report form

Standard form for reporting of equipment information, results and uncertainties.

The form has been forwarded electronically.