SMQ-ILC 2020-1





To participants

Report on an interlaboratory comparison (ILC) on the calibration of 4 laboratory balances



Balance 1: Sartorius MCA 10.6S Max 10,1g d = 0,000001g



Balance 2: Mettler Toledo XPR 205DRq Max 81/220g d = 0,00001/0,0001g



Balance 3: Ohaus PA 4102C Max 4100g d = 0,01g

Author

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Balance 4: Sartorius PMA 35001 Max 35kg d = 0,1g

2020-04-02

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Purpose and implementation of the comparison

This interlaboratory comparison serves as a tool to verify results from the measurement carried out by calibration laboratories. It is an effective method to demonstrate technical capacity of the participant and serves as a technical base for accreditation as required by ISO/IEC 17025:2017 (SS-EN ISO/IEC 17025:2018) as specified in point 7.7.2.

This inter-comparison was supported by the companies Sartorius International, Mettler Toledo Nordic, VWR International and Tillquist Sweden which provided the balances for the time of the exercise.

The weighing instruments no 1 and 2 are the most advanced micro and analytical balances on the market and they were installed by international technicians in order to provide the best possible conditions for the comparison measurements. This was also a specific challenge as many of the technicians before this inter-comparison had no calibration experience with those balances. A further benefit was the access to a generous location with suitable weighing tables for each balance in separate rooms which allowed a tight measurement scheme and efficient calibration work. This support is thankfully acknowledged.

Participating laboratories:

- VWR International AB, Sweden
- Instrument and Calibration Sweden AB, ICS, Sweden
- TILLQUIST(an ADDvise Group Company, Sweden
- Mettler Toledo AB, Norway
- Mettler Toledo AB, Sweden
- Mettler Toledo A/S, Denmark
- Karolinska Universitetssjukhuset Huddinge, Sweden
- DEKRA Quality Management AB, Sweden
- Analysvågservice AB, Sweden
- Flintab AB, Sweden
- Lahti Precision OY, Finland
- Eurofins Expert Services Oy, Finland
- Avantor Services, Netherlands
- MyCal AB, Sweden
- OptiCal I Linköping AB, Sweden
- JH Analys och Vågteknik AB, Sweden
- JB Scales AB, Sweden
- Vågkonsult AB, Sweden
- Pilot Laboratory—Swedish Metrology and Quality AB, Sweden

Some of the participants performed more than one calibration on the balances using different methods. A majority performed a calibration on all four balances, others only on one or two. During the exercise all together 68 calibrations were performed during 7 working days.

All participants have an accreditation by SWEDAC, DANAK, FINAS and RVA Netherlands

Planned calibration scheme:

Date	Participant
2020-02-14	SMQ, Pilot laboratory
2020-02-17	5 participants
2020-02-18	6 participants
2020-02-19	6 participants
2020-02-20	3 participants
2020-02-21	4 participants
2020-02-24	2 participants and pilot laboratory

The measuring scheme for the comparison

Principles on the calibration in general

Prior to each calibration by a participant the pilot laboratory accomplished an adjustment on each instrument and a stability check after each individual calibration. This adjustment was performed with the build-in weights or with external weights. The main purpose for doing so was to achieve as equal conditions as possible for all participants. In this way varying air density conditions over the experiment period could be largely eliminated. Further it was checked that no significant change had occurred before the next participant could start its calibration.

Weighing conditions during the measurement period

The balances used in this inter-comparison were a loan from four suppliers. They were installed in four separate rooms close to each other provided by VWR International AB in Stockholm for the whole time. This arrangement guaranteed that this exercise could be handled efficiently and enable the participants to work independently and undisturbed by each other.

Calibration instructions

The laboratories were allowed to use maximum 1,5 hours for each balance calibration. In the call they were advised to use their own calibration procedures with focus on the following points which were important for the inter-comparison outcome. They were not allowed to perform any type of adjustment on the balances themselves. This task was reserved for the pilot laboratory. In this way the laboratories were able to apply the uncertainty calculations presented in EURAMET Calibration Guide No. 18 Version 4.0 (11/2015), especially the scenario for an adjustment immediately prior to the calibration. Using own procedures also meant it was up to the laboratories which measurement points they would select, as long as the following three obligatory ones were included. Even the number of repetitions was free to choose. The laboratories further were encouraged to use the actually calculated uncertainty values even if those would differ from the CMC values in their accreditation.

Balance	1	2	3	4
Capacity,	Max 10,1g;	Max 220 g;	Max 4,1 kg;	Max 35 kg;
resolution	d=0,001mg	d=0,01/0,1mg	d=10mg	d=0,1g
Point 1	1 mg	500 mg	200 g	1 kg
Point 2	500 mg	5 g	1 kg	5 kg
Point 3	10 g	200 g	4 kg	20 kg

Compulsory measurement points

The laboratories were asked to hand over original calibration data in pre-defined forms either on paper or in digital form by e-mail before leaving the site. The final calibration certificate then should be sent to the organizer within one week, which most of them also managed to deliver.

Planning and instruction details

The laboratories were asked to hand over original calibration data in pre-defined forms either on paper or in digital form by e-mail before leaving the site. The final calibration certificate then should be sent to the organizer within one week, which most of them also managed to deliver. The evaluator used the principles of the ISO/IEC 17043:2010 in the reporting.

The participants should deliver calibration certificates which at least stated the measured values together with a belonging uncertainty for the points stated above. Several of them delivered data on further calibration points including e.g. eccentricity and repeatability checks as they do in a normal case.

It was further agreed that a participant not having the capability to calibrate the whole range of the instruments to perform a limited calibration. And it was possible to provide additional information or supplementary documentation eventually needed to understand the results.

Administrative information

Address to send weights in advance:	Dates and place:	Address to send the reports:
VWR International AB	February 17-24.	Swedish Metrology and Quality AB
ATT: SMQ	Adress Fagerstagatan 18A, vån. 2	Håkan Källgren
Domnarvsgatan 2B, Port 11	SPÅNGA	Dragspelsgatan 21
SE-163 53 SPÅNGA	Contact phone +46705774931	SE-504 72 Borås, Sweden
Sweden	(Håkan Källgren)	e-mail: <u>hakan.kallgren@smquality.se</u>

Summary of the timeline planning in the call:

- One week after the calibration/measurement send the calibration certificate to the evaluator of the intercomparing report.
- A draft report will be sent to the participants 2 weeks after receiving the last report
- Comments on the draft report within 1 week
- Final report will be finalized within 2 weeks after receiving comments from all participants

Analysis of the calibration results

The main information compared is the "error of indication" (EoI) at all measurement points. This error is simply the difference between the documented balance indication and the calibrated value of the used weights given by the participants. This information was mostly already provided in the calibration certificates. In some cases when the weights were not explicitly stated it was evaluated as the documented balance indication minus the nominal weight value by the organizer.

The quality of each individual measurement result is reviewed using the E_n – criteria. For each measurement point it is the distance of respective laboratory result to the corresponding reference value normalised with respect to the uncertainty in determine this difference.

$$E_n = \frac{x_i - x_{ref}}{\sqrt{U_i^2 + U_{ref}^2}}$$

x_i: Single measurement result (error of indication); index i counts the various participants.

x_{ref}: Provided reference value.

Ui: The estimated expanded uncertainty (k=2) stated by each laboratory for each calibration point.

U_{ref}: The estimated expanded uncertainty (k=2) of the reference value for the same calibration point.

Inter-comparison reference value

In every comparison the reference value is a crucial fact. ISO 13528 suggest various situations. In this exercise a pilot laboratory SMQ performed a first calibration at all measurement points before the start, termed pilot series P1. This was then repeated once more after the last participant had finished his work and is termed pilot series P2.

For the comparison the reference value x_{ref} as a function (balance, calibration point) was simply calculated as the average between the two corresponding pilot results.

$$x_{ref} = Eol(Ref) = \frac{1}{2}(Eol(P1) + Eol(P2)) = \frac{1}{2}(x_{P1} + x_{P2})$$

The pilot calibration results P1 and P2 were based on ten repeated measurements in every point. The main purpose for this choice was to take care of possible balance drift over the week of the experiment and to include the balance stability into the uncertainty of the reference value. As can be seen in the diagrams some drift could be found. It was, however, mostly in the range of the pilot-uncertainty or below, often within the resolution of the respective balance.

x_{P1}: The declared error of balance indication for respective weighing point in the pilots first calibration series before the inter-comparison exercise.

x_{P2}: The same information from the second calibration series after the inter-comparison exercise.

Uncertainty of inter-comparison reference value

Usually one would expect that the uncertainty of the average from two measurement series should be less than the uncertainty in each one. However, the two measurements cannot be regarded as the same measurement. Furthermore, a possible balance drift adds uncertainty to the reference value valid over the whole exercise. Therefore, the reference uncertainty was determined as:

$$U_{ref} = max(U_{P1}, U_{P2}) + \frac{1}{2}abs(x_{P1} - x_{P2})$$

In plain words the larger of the pilot uncertainties is linearly combined with half of the experienced drift. This means that during the week the reference value most probably moved in between the two respective pilot values and with falling probability could have moved up to the 95 % coverage of each of these two pilot values. Although this construct is not contained in any of the standards it seems to be the most reasonable approach.

U_{ref}: The expanded uncertainty of the reference value for each respective calibration point

U_{P1,2j}: The uncertainty of the pilot result 1 before or 2 after the inter-comparison for each calibration point. The larger of them is linearly combined with half of the balance drift between start and

end (absolute value). This guaranties that the reference value with its uncertainty includes balance drift and overlaps both results x_{P1} and x_{P2} with $|E_n| < 1$.

An absolute value of E_n of less than 1 is often used as a criterion for an acceptable measurement quality, see

ISO/IEC 17043:2010, B.4.1.1.

Traceability for the pilot values P1 and P2 at each point

The traceability for the pilot laboratory SMQ is established by calibration of its weights at RISE (the NMI in Sweden) and an accredited laboratory in France, ZWIEBELfor a 10 kg weight.

The reference weights used during calibration by the pilot laboratory are of class E1 and E2.

Analysis of uncertainty in the pilot values

The stated expanded uncertainty for the pilot values following Euramet guide 18 version 4.0 are calculated according to:

$$U_{Pilot} = 2 \cdot \sqrt{u_{ref}^2 + u_{stab}^2 + u_s^2 + u_{zero}^2 + u_{read}^2 + u_{ab}^2 + u_{ecc}^2 + u_{conv}^2}$$

Where the following contributions on a standard uncertainty level are related to the pilot (or reference) laboratory:

 u_{ref} = uncertainty of the reference weights, from the calibration certificate

 u_{stab} = uncertainty of the stability of reference weights

 u_s = the standard deviation of the mean value at each calibration point

 u_{zero} = uncertainty with respect to zero setting the balance in question

 $u_{read} = uncertainty based on the balance resolution (display)$

- u_{ab} = uncertainty caused by air buoyancy
- u_{ecc} = uncertainty due to balance eccentricity
- $u_{conv} = possible convection due to temperature differences between weight and balance$

The magnetic susceptibility on the reference weights was controlled and is not interfering with the weighing instrument.

Uncertainty of the reference weights Euramet guide 18 version 4.0 formula 7.1.2-2

Taken from the certificate and divided by the stated k-factor. In this case 2.

Stability of the reference weights Euramet guide 18 version 4.0 formula 7.1.2-11

The mean drift of the reference weights estimated from different calibrations

Standard deviation of the mean value of the calibration Euramet guide 18 version 4.0 formula 7.1.1-6

7(22)

Standard deviation divided by square root of the number of measurements

Uncertainty based on the accuracy of zero setting

The zero setting is within the range of ¹/₄ of the reading resolution. The resolution divided by 4 and rectangular distribution is used. Euramet guide 18 version 4.0 formula 7.1.1-2c

Uncertainty based on resolution of the display

Resolution on the balance in each calibration point. Rectangular distribution is used.

Euramet guide 18 version 4.0 formula 7.1.1-3a

Uncertainty based on air buoyancy

Uncertainty factor based on air buoyancy difference as described in guide EURAMET/cg-18-7.1.2-5a

Uncertainty based on eccentricity

A proportional figure from possible eccentricity influence divided by square root of 6 EURAMET/cg-18-7.1.1.4

Measuring results on calibration in comparison for four balances

The following tables and diagrams present the error of indication along with the stated measurement uncertainty for each calibration point. The data for the four balances is marked in different colours for the three respective calibration points. Each table also specifies the two pilot results and the calculated reference value and its uncertainty. These two values are used to calculate the various En-values for each participant. In the diagrams all results from all participants including the pilot data are presented graphically as symbols together with respective reported uncertainty. In each diagram the reference value is indicated as a broken line and the belonging uncertainty is given as a coloured band.

Balance 1: Satorius Max=10,1g Resolution(d) 0,001 mg

Calibration points: 1 mg; 500 mg and 10 g

Balance 1	Calibration poi	nt - 1 mg	
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	-0,0002	0,0015	0,28
1	0,001	0,0086	0,22
2	0,001	0,0056	0,32
3	0,002	0,0056	0,49
4	-0,0001	0,002	0,29
5	0	0,003	0,26
6	0	0,003	0,26
7	0,002	0,003	0,79
8	-0,001	0,004	-0,01
9	-0,001	0,003	-0,01
10	-0,001	0,003	-0,01
11	-0,001	0,003	-0,01
12	0	0,003	0,26
13	0	0,003	0,26
14	-0,001	0,003	-0,01
Pilot 2	-0,0017	0,0014	-0,29
Reference value	-0,00095	0,0022	

Table 1: Reported data from first calibration point; pilot-, reference data and En-values



Diagram 1: All reported results compared to the reference value and its uncertainty band

Comment: A shift of 0,0015 mg was observed between the two pilot results. This is slightly more than the resolution and of the same size as the measurement uncertainty. All results are totally acceptable.

Balance 1	Calibration point - 500 mg		
Participants	rticipants Error of indication		En-Value
	[mg]	[mg]	
Pilot 1	0,0014	0,0028	-0,04
1	0,001	0,036	-0,02
2	0	0,022	-0,07
3	-0,001	0,022	-0,11
4	0,0007	0,004	-0,17
5	0	0,008	-0,18
6	0,002	0,011	0,04
7	0,003	0,01	0,14
8	0,001	0,009	-0,06
9	0	0,009	-0,16
10	0	0,01	-0,15
11	0,002	0,011	0,04
12	0	0,011	-0,14
13	0,003	0,011	0,13
14	0,004	0,012	0,20
Pilot 2	0,0017	0,0027	0,04
Reference value	0,00155	0,0030	

Table 2: Reported data from second calibration point with pilot, reference data and En-values



Diagram 2: All reported results compared to the reference value and its uncertainty band

Comment: At the 500 mg point the stability over the whole exercise was excellent. The difference between pilot 1 and pilot 2 is 0,0002 mg, i.e. below the resolution of the balance. All results are comfortably within the uncertainty band of the reference value.

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Balance 1	Calibration point - 10 g		
Participants	Error of indication	or of indication Uncertainty	
	[mg]	[mg]	
Pilot 1	-0,0457	0,00956	0,02
1	-0,019	0,0977	0,27
2	-0,043	0,064	0,05
3	-0,043	0,064	0,05
4	-0,038	0,019	0,37
5	-0,043	0,021	0,13
6	-0,023	0,033	0,67
7	-0,036	0,025	0,37
8	-0,042	0,022	0,17
9	-0,039	0,025	0,26
10	-0,039	0,028	0,24
11	-0,022	0,03	0,76
12	-0,044	0,033	0,06
13	-0,041	0,025	0,19
14	-0,04	0,027	0,21
Pilot 2	-0,0463	0,0089	-0,02
Reference value	-0,046	0,0099	

Table 3: Reported data from second calibration point including pilot, reference and En-values



Diagram 3: All reported results compared to the reference value and its uncertainty band

Comment: At the maximum point the stability over one week was impressing with a shift of 0,00006 mg in the average of 10 repeated pilot weighings. All participants achieved very satisfying results. Three results show comparably large measurement uncertainty.

Balance 2: Mettler Toledo Max= 220 g; d= 0,01 mg and 0,1 mg

Compulsory calibration points: 500 mg, 5 g and 200 g

Balance 2	Calibration poi	nt - 500 mg	
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	0,001	0,008	-0,25
1	0,01	0,018	0,18
2	0,00	0,026	-0,18
3	-0,01	0,039	-0,36
4	0,00	0,026	-0,18
5	-0,01	0,03	-0,45
6	0,00	0,02	-0,10
7	0,01	0,02	0,17
8	0,01	0,03	0,13
9	0,00	0,02	-0,21
10	0,00	0,02	-0,21
11	0,00	0,01	-0,29
12	0,00	0,02	-0,21
13	0,00	0,04	-0,13
Pilot 2	0,010	0,012	0,22
Reference value	0,0055	0,017	

Table 4:	Reported EoI	-data from first	t calibration poin	nt and calculated]	En-values
				t and careatored	



Diagram 4. Distribution of 13 results around the calculated reference value with its measurement uncertainty band.

Comment: A shift of 0,009 mg between pilot 1 and 2 i.e. just below resolution. All results are within the uncertainty band of the reference value. All results are very well acceptable.

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Balance 2	Calibration p	point - 5 g	
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	-0,003	0,010	-0,30
1	0,02	0,025	0,52
2	0	0,045	-0,07
3	0,02	0,078	0,20
4	0,01	0,047	0,13
5	0,01	0,03	0,18
6	-0,01	0,03	-0,32
7	0	0,03	-0,10
8	0	0,03	-0,10
9	0,01	0,03	0,18
10	-0,01	0,03	-0,38
11	0,01	0,02	0,23
12	0,01	0,03	0,18
13	0	0,04	-0,08
Pilot 2	0,010	0,013	0,28
Reference value	0,0035	0,0195	

Table 5: Reported EoI-data from second calibration point and calculated En-values





Comment: The balance stability over one week from pilot 1 to pilot 2 is just above resolution (0,013mg). All results are completely acceptable within the uncertainty band of the reference value, which also is expressed by accepted En-values One result is given with comparably large measurement uncertainty.

Balance 2	Calibration po	pint - 200 g	
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	-0,11	0,12	0,13
1	-0,10	0,16	0,15
2	0,00	0,45	0,28
3	0,10	0,84	0,27
4	-0,10	0,49	0,07
5	0,10	0,2	0,91
6	-0,10	0,2	0,13
7	-0,10	0,4	0,08
8	-0,10	0,4	0,08
9	-0,10	0,3	0,10
10	0,00	0,2	0,52
11	0,00	0,2	0,52
12	-0,10	0,2	0,13
13	0,00	0,2	0,52
Pilot 2	-0,16	0,14	-0,12
Reference value	-0,134	0,162	

Tabell 6: Reported EoI-data from third calibration point with calculated En-values



Diagram 6: All reported results compared to the reference value and its uncertainty band

Comment: The balance drift from pilot 1 to pilot 2 amounts to 0,05 mg, however this corresponds to less than half the pilot uncertainty of 0,13 mg at average. Consequently participant 3 again has the largest uncertainty. Only result (5) is near the border of acceptance with its stated uncertainty. The rest is completely acceptable.

Balance 3: Ohaus PA 4102C; Max=4100g d=0,01g;

Calibration points: 200 g, 1 kg and 4 kg

Balance 3	Calibration poi	nt - 200 g	
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	0,7	8,1	0,03
1	0	15	-0,02
2	0	10	-0,03
3	0	10	-0,03
4	0	10	-0,03
5	0	20	-0,02
6	0	20	-0,02
7	0	13	-0,02
8	0	14	-0,02
9	-20	30	-0,65
10	10	30	0,31
11	0	20	-0,02
12	0	20	-0,02
13	10	40	0,24
14	-10	13	-0,66
15	-10	20	-0,48
16	0	20	-0,02
17	10	20	0,44
18	0	10	-0,03
19	0	20	-0,02
20	0	10	-0,03
Pilot 2	0,0	7,1	-0,03
Reference value	0 34	8.5	

Table 7: EoI results from 20 participants- all En-values clearly within ±1



Diagram 7: All 20 reported results compared to the pilot data and reference value and its uncertainty band

Comment: The possible drift of 0,7 mg is 1/10 of the pilot uncertainty – all participants overlap the reference value directly, which results in many low En-values.

Balance 3	Calibration point - 1 kg		
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	-6,2	8,8	-0,26
1	-10	17	-0,37
2	0	10	0,14
3	0	10	0,14
4	0	36	0,06
5	-10	20	-0,33
6	0	20	0,09
7	0	15	0,11
8	-10	29	-0,25
9	-20	30	-0,55
10	0	20	0,09
11	0	20	0,09
12	10	20	0,51
13	10	40	0,29
14	-10	14	-0,41
15	0	20	0,09
16	0	20	0,09
17	-21	20	-0,79
18	10	10	0,75
19	-10	20	-0,33
20	0	10	0,14
Pilot 2	1,8	8,0	0,27
Reference value	-2,2	13	

Tabell 8: All 20 results show accepted En-values below ±1



Diagram 8: All reported results compared to the reference value and its uncertainty band

Comment: The stated balance drift is 8 mg, i.e. just below the resolution 10 mg and equivalent to the uncertainty of the pilot measurements. All results are well acceptable.

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Balance 3	Calibration point - 4 kg		
Participants	Error of indication	Uncertainty	En-Value
	[mg]	[mg]	
Pilot 1	-8,6	11	-0,53
1	-30	41	-0,75
2	-10	30	-0,41
3	20	10	0,49
4	30	120	0,19
5	0	20	-0,20
6	10	20	0,11
7	0	40	-0,13
8	-40	120	-0,38
9	-20	30	-0,67
10	-10	20	-0,50
11	-10	40	-0,34
12	40	30	0,85
13	40	40	0,71
14	-30	37	-0,80
15	-10	40	-0,34
16	-10	20	-0,50
17	-32	30	-0,97
18	30	20	0,72
19	-60	30	-1,68
20	10	10	0,13
Pilot 2	21,4	8,7	0,55
Reference value	6,4	26	

Table 9: The EoI results for calibration point 3 from 20 participants and belonging En-values.





Comment: A clear balance shift (3 times the pilot uncertainty) is observed. A clear spread is observed that is not reflected in the individual relative low uncertainties. It is assumed that the larger spread is caused by the handling of the two 2 kg weight pieces (eccentricity aspects). Only one result is definitively outside the En-border, but three others are near.

Balance 4: Sartorius Max=35 kg d=0,1g

Calibration points: 1, 5 and 20 kg

 Table 10: 21 plus 2 pilot results for first calibration point including uncertainties and En-values

Balance 4	Calibration point - 1 kg		
Participants	Error of indication	Uncertainty	En-Value
	[g]	[g]	
Pilot 1	-0,05	0,074	-0,17
1	0,0	0,1	0,22
2	0,0	0,1	0,22
3	0,0	0,2	0,14
4	0,0	0,1	0,22
5	0,0	0,08	0,24
6	0,0	0,2	0,14
7	0,0	0,1	0,22
8	0,1	0,1	0,95
9	0,0	0,2	0,14
10	0,0	0,2	0,14
11	0,0	0,6	0,05
12	0,0	0,2	0,14
13	0,0	0,1	0,22
14	0,0	0,2	0,14
15	0,0	0,1	0,22
16	0,0	0,1	0,22
17	0,0	0,2	0,14
18	0,0	0,1	0,22
19	0,0	10,0	0,00
20	0,0	0,11	0,21
21	0,0	0,08	0,24
Pilot 2	-0,01	0,068	0,17
Reference value	-0,03	0,094	



Diagram 10: All 23 reported results compared to the reference value and its uncertainty band

Comments: The detected balance drift amounts to1/3 of the resolution. Almost identical results throughout, probably due to the limited resolution and few repetitions. Only one result close to the Encriteria.

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Balance 4	Calibration point - 5 kg		
Participants	Error of indication	Uncertainty	En-Value
	[g]	[g]	
Pilot 1	-0,20	0,066	0,00
1	-0,2	0,10	0,00
2	-0,1	0,17	0,55
3	-0,2	0,20	0,00
4	-0,2	0,30	0,00
5	-0,2	0,10	0,00
6	-0,2	0,20	0,00
7	-0,2	0,10	-0,08
8	-0,2	0,10	0,00
9	-0,2	0,20	0,00
10	-0,2	0,20	0,00
11	-0,2	0,60	0,00
12	-0,2	0,20	0,00
13	-0,2	-0,13	0,00
14	-0,2	0,20	0,00
15	-0,2	0,10	0,00
16	-0,2	0,10	0,00
17	-0,2	0,20	0,00
18	-0,2	0,10	0,00
19	0,0	10,00	0,02
20	-0,2	0,12	0,00
21	-0,2	0,12	0,00
Pilot 2	-0,20	0,066	0,00
Reference value	-0,20	0,066	

Table 11: 23 results in comparison with reference value – all calculated En-values accepted



Diagram 11: 23 results only two deviating with one resp. two scale divisions apart. But the balance demonstrates a clear error. This balance was intentionally adjusted with a false weight, which gave this error.

Comments: No balance drift found, low reference uncertainty. Generally excellent conformity with extremely low En-values, but quite some difference in uncertainty judgement. Participant 19 stated extremely large uncertainty.

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Balance 4	Calibration point - 20 kg		
Participants	Error of indication	Uncertainty	En-Value
	[g]	[g]	
Pilot 1	-0,58	0,093	-0,36
1	-0,8	0,1	-1,53
2	-0,6	0,59	-0,14
3	-0,6	0,3	-0,25
4	-0,5	1,2	0,01
5	-0,7	0,25	-0,63
6	-0,4	0,2	0,45
7	-0,6	0,2	-0,45
8	-0,7	0,1	-0,99
9	-0,6	0,4	-0,20
10	-0,6	0,4	-0,20
11	-0,7	0,7	-0,26
12	-0,7	0,2	-0,73
13	-0,5	-0,62	0,02
14	-0,7	0,2	-0,73
15	-0,7	0,2	-0,73
16	-0,5	0,2	0,06
17	-0,8	0,2	-1,12
18	-0,4	0,2	0,45
19	-1,0	10	-0,05
20	-0,7	0,26	-0,61
21	-0,7	0,38	-0,45
Pilot 2	-0,45	0,093	0,36
Reference value	-0,52	0,16	

Table 12: 23 EoI results for calibration point 3 including 2 pilot, reference data and En-values.





Comments: Here the handling of the two 10 kg weights probably cause the spread in connection with the balance eccentricity. All participants clearly state an increasing balance error, which is due to an intentional miss adjustment by the pilot before every calibration start. A clear spread between the participants in result and uncertainty statement can be seen. Two En-values are doubtless outside and one directly on the edge.

Final conclusion

In this inter comparison all the participants could demonstrate a convincing capacity to calibrate various balances which they partly did not have any former experience of. Only in a very limited number (3) of calibration points (total number 204) the En-criteria [-1<En<+1] was exceeded. A few En-values (4) were close to the En-limit, but the vast majority (197) was very satisfying with many really low values. This indicates that most stated uncertainties, even the relatively low ones, are justified. This is true in comparison to all other participants showing an excellent agreement, but especially in comparison to the pilot laboratory. Due to the possible balance drift and the definition of reference uncertainty, which eventually could be large, the participants could win some advantage for lower En-values compared to a single pilot uncertainty. However, this drift was mostly not significant and would only in four cases possibly influence the En- value to be on the correct side. It is remarkable that the advanced balances 1 and 2 only revealed totally acceptable results and it also interesting that the three failures to meet the En-critera occurred when two weight pieces were used together (2 kg one result, 10 kg two results). Some of the stated uncertainties were relatively large compared to other participants. Using the En-formula with a lower Ui could show if still a reasonable value would be achieved.

General comments (not a part of the inter-comparison)

The design of the calibration certificates was very different e.g. a calibration point was indicated with its nominal value. In other cases, the used load including the calibrated mass value of the used weight was specified

The result was indicated in one of the following ways in the heading of the column:

- Result (including all figures)
- Error
- Correction
- Deviation (without definition)

The uncertainty was normally rounded to the resolution of the balance. Sometimes it was given with more than 2 significant figures and the heading of the column was normally:

- Indicated as uncertainty
- Indicated as expanded uncertainty

The indicated result and uncertainty were sometimes given in the same unit (e.g. g and g) sometimes both differed by a factor of 1000 (e.g. g and mg)

After receiving the draft report one laboratory detected a mistake in using the software to calculate the uncertainty for balance 4. Instead of high 10 g for all three calibration points the correct values should have been 0,1, 0,2 and 0,6 g for respective points 1, 5 and 20 kg. With these corrections the corresponding En-values would change from 0,00 to 0,22 (1 kg); 0,02 to 0,95 (5 kg) and -0,05 to -0,75 (20 kg). This indicates acceptable uncertainty judgements. However, these corrections can not be introduced into the final report.

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References:

- ISO/IEC 17043:2010 Conformity assessment General requirements for proficiency testing
- ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories
- ISO 13528 Statistical methods for use in proficiency testing by interlaboratory comparison
- Evaluation of measurement data Guide to the expression of uncertainty in measurement GUM:2010
- EA-4/02 M:2013 Evaluation of Uncertainty of Measurement in Calibration