A MASS COMPARISON AROUND THE WORLD

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ABSTRACT

A group of nine calibration laboratories performed an intercomparison of Conventional Mass in 2004. The comparison consisted of conventional mass for four weight pieces, namely 100 g, 10 g, 100 mg and 2 mg. All participating laboratories are accredited for this measurement. However, they are distributed around the world on different continents, they are accredited under different accreditation bodies and are traceable to different national standards.

This is special, as usually calibration laboratory intercomparisons are performed in one country only or among laboratories that are accredited by one accreditation body. In this intercomparison the only common point of traceability is the international kilogram prototype and the only common point of the quality assurance system of the participants it is their accreditation according to ISO 17025. The paper deals with the results of the intercomparison and hints at problems and findings that can be drawn from the evaluation of the results.

1. INTRODUCTION

Inter-Laboratory Comparisons (ILC) for calibration laboratories are a valuable means for evaluating the quality of a lab’s calibration results. Calibration laboratories usually welcome such ILC’s as they offer a unique chance to verify their results together with their uncertainty calculation. This strengthens the confidence among the national calibration services, the confidence of the lab in its own measurements and the confidence of the customers in the services offered by the lab. ILC’s among calibration labs are usually run nation wide within one accreditation body or as official EA-Intercomparisons. This leads to the following problems:

- Some countries have a large number of labs accredited for the same tasks (e.g. Germany, Conventional Mass). This makes the ILC’s long, complicated and expensive, which then means that they are conducted not very often.
- Some countries have only a small number of labs (e.g. Switzerland: only one). This makes an ILC un-feasible and senseless. (So here they are not conducted at all.)
- All such intercomparisons are usually traceable to one national standard only. Thus, they can make no statement about the international inter-correspondence of the respective unit.
- Official EA-Intercomparisons require considerable administrative work (thus they are not conducted very often).

These facts made the 9 laboratories within the METTLER TOLEDO company decide in November 2003 to perform their own intercomparison. This intercomparison should

- Cover all mass labs of the company around the world.
- Become a regular event to be repeated e.g. every 2 years.

The document [1] was used as a guideline where applicable.

2. THE LABORATORIES

The participating laboratories were in detail (alphabetical order of column 1):

Table 1: Participating Laboratories

Lab	Country	Accredited by	Traceable to	Uncertainty level
MT-ES	Spain	ENAC (Spain)	CEM (Spain)	“E2”-Lab
MT-F	France	COFRAC (France)	French National Standard	“F2”-Lab
MT-I	Italy	SIT	Italian National Standard Lab	“E2”-Lab
MT-JP	Japan	JCSS (Japan)	NMIJ (Japan)	“E2”-Lab
MTLabTec	Switzerland	SAS (Switzerland)	metas (Switzerland)	“E1“-Lab (RL)
MT-MX	Mexico	ema (Mexico)	CENAM (Mexico)	“F1”-Lab
MT-NA	U.S.A.	A2LA (U.S.A.)	NIST (U.S.A.)	“E2”-Lab
MT-NL	Netherlands	RvA (Netherlands)	NMI (Netherlands)	“E2”-Lab
MT-TH	Thailand	TISI (Thailand)	metas (Switzerland) (through RL!)	“E2”-Lab

As the Laboratory “MTLabTec” in Switzerland had the smallest accredited uncertainty, this lab was given the function of the reference lab (RL).

3. TASK AND TEST PIECES

The task for the intercomparison was the determination of the *Conventional Mass* or the *Conventional Mass error* of the weight Δm_c with respect to its nominal value of four weight pieces. The nominal values of the weights were chosen so that they fit into the scope of accreditation of all labs of the company. Eventually, one weight each of 2 mg, 100 mg, 10 g and 100 g were chosen. The milligram weights are made of wires and the larger weights are cylindrical knob weights, both types being made of stainless steel with a density of about 8000 kgm^{-3}. The technical specifications make them suitable for E1 class (which has the highest requirements regarding surface quality, magnetic properties, density etc.). The weights are standard METTLER TOLEDO products.

In order to prove stability of the weights, the pieces were calibrated in the reference laboratory, then stored for one month and then calibrated again. The weight values were thus proved stable (under consideration of uncertainties) (see graphs in Figures 1 through 4, first two measurements).

A time-dependent correction factor for the drift of the weight values with time has been used sometimes in other mass intercomparisons ([2]). However, a time-dependent function is not always representative, because “wear” of a weight is generally a function of “use” and not of “time”. Additionally “wear” over the duration of the ILC is likely to be a step function, which cannot be modeled by a continuous function. Furthermore, it turned out that the data of the object did not show any significant signs of “wear” during the course of the intercomparison. Therefore it was decided not to introduce any correction function, as the weight values of the chosen weight pieces were stable.

4. UNCERTAINTIES

An important task for mass calibration laboratories is to determine if a weight is within the tolerance limits of the accuracy classes of [3] named E1, E2, F1, etc.. [3] also states that for this confirmation, the 95%-uncertainty should be not larger than one third of the width of the tolerance band. That is why mass calibration laboratories are often classified according to their ability to confirm that the weight of a mass piece is within the limits of a certain class (“E1-laboratory”, “F1-laboratory”, …).
For this reason mass labs are frequently accredited only to a best measurement capability

(BMC) which corresponds to the required third part of the tolerance span. This BMC is confirmed by accreditation when the lab can prove that it is for sure and always better than this limit. There is no need for the lab to have their “real” best capability accredited. Thus the “real” uncertainty of calibrations is sometimes not even calculated, and never stated, because it is smaller than the accredited uncertainty. This leads to the following problems:

- The picture of intercomparisons is incomplete, because the stated uncertainties are larger than they really are.
- The user of the weight – if he is using the exact value of the weight together with the associated uncertainty – uses an uncertainty figure that is too large.

However, solutions are scarce. An individual calculation of uncertainty of a weight piece would require a software solution or a sophisticated spreadsheet due to the fact that a large number of uncertainty sources can be identified. Mass laboratories (including even some national laboratories) commonly try to avoid such efforts and state “safe guess” values based on one third of the tolerance span in their calibration certificates.

5. SHIPMENT AND “LOOPS”

For logistical reasons, the intercomparison was arranged in three loops according to continents:

- Americas-Loop: Mexico, USA
- Asia-Loop: Thailand, Japan
- Europe-Loop: Spain, France, Italy and Netherlands

The weight pieces were examined and measured at the reference laboratory in Switzerland before and after each loop.

The weights were packed in individual weight boxes. A special re-usable padded cardboard box had been built with individual cavities for each weight box to ensure safe shipment. The intercomparison started in January and was completed in October 2004.

6. COMMUNICATION OF RESULTS AND CERTIFICATES

The laboratories sent their usual calibration reports to the reference laboratory immediately after finishing the measurements. A special form (as suggested in [1]) was not regarded as necessary, since the essential values can easily be identified in the certificate even in a foreign language. The values were immediately checked upon arrival in the RL and En-values calculated with respect to the previous reference lab values. This was done very quickly in order to be able to interrupt the loop in the case that suspicion had arisen about the proper quality of one of the test pieces. Certificates were to be issued in English if this was a common certificate language for the laboratory.

7. COMMUNICATION

The exchange of communication was done very effectively using an Internet forum (community). Participants of the intercomparison could log on to this password-protected Internet area and download instructions, time schedules and other important information. The forum was administered by the reference laboratory. The language for any communication was English.

8. PROBLEMS ENCOUNTERED

After receiving the weights back from the first loop, the reference laboratory noticed that the 100 g weight was slightly heavier than at the beginning of the test. The difference was in the order of magnitude of the uncertainty of the RL, but no reason (e.g. fingerprint, dirt) could be visually detected. The RL then decided to clean the weight gently with a dry microfibre cloth. After the cleaning, the initial value was confirmed. Also after the second loop, the same weight came in to the RL slightly heavier. Again, the same effect could be observed after cleaning.

The last lab of the third and final loop reported a very high value for the 100 g piece leading to an E_n-value of 0.95. The reference lab observed dust and fibers from the lining of the weight box in the "neck" of the weight. The dust was blown and wiped away and the value of the 100 g weight piece was confirmed. It turned out that the last lab had not cleaned the weight at all before their measurements although the prescribed procedure asked for a "careful dry cleaning". The weight was sent back to this lab for another measurement. Finally, an E_n-value of 0.32 was obtained after proper cleaning.

Laboratory #2 reported uncertainty values that were calculated by software. It turned out that some of these uncertainties were

a) even smaller than those of the RL and
b) corresponding to class E1-uncertainties. However, it is commonly assumed [3], that E1-uncertainties can only be achieved if the volume of the weight pieces are known by measurement and thus their (systematic) buoyancy effect in ambient air can be calculated instead of contributing to the uncertainty. Unfortunately, the uncertainty budget could not be re-produced or re-investigated.

Lab #1 reported uncertainties that were a little larger than one third of the respective tolerance band. Yet, the laboratory made a conformity statement based on this. It turned out that the respective national accreditation body had confirmed this procedure.

A typhoon storm delayed the progress of the intercomparison in Thailand, as due to weather conditions (which affect the buoyancy force of mass measurements in air) all measurements had to be stopped in the laboratory.

The holiday season delayed the progress of the intercomparison in the summer time. However, due to the short distances and some extra effort of the participants, the loop consisting of the four labs in Europe could recoup the lost time.

9. RESULTS

The Normalized Error values (E_n-values) for the final measurement results of the participating labs were evaluated with respect to the preceding RL-values according to the formula:

$$E_n = \frac{\Delta m_{c,l} - \Delta m_{c,RL}}{\sqrt{U_l^2 + U_{RL}^2}} \qquad (1)$$

where Δm_c are the measured Conventional Mass errors, U the 95-%-uncertainties (according to accredited BMC) and indices "*l*" for participating lab and "*RL*" for the reference laboratory.

The 32 final results of E_n are listed in Table 2 and the measurement results are displayed in graphical form with bars displaying the accredited uncertainty (k=2) in Figures 1 through 4. None of the results is larger than the critical value of 1.0.

Table 2: E_n-Values

Lab	2 mg	100 mg	10 g	100 g
1	0.54	0.48	-0.12	0.09

2	-0.22	-0.05	0.56	0.04
3	0.02	0.01	0.49	0.18
4	0.40	0.04	0.03	0.42
5	-0.13	-0.06	0.05	-0.40
6	0.09	-0.20	-0.25	0.33
7	-0.08	-0.24	0.24	0.66
8	-0.34	0.31	0.47	0.32

10. NON-CONFORMITIES

- Laboratory #2 and #7 reported uncertainties which were smaller than the accredited uncertainty without mentioning the reason for this.
- Laboratory #7 reported an uncertainty for one weight which was rounded to one digit so it became a little smaller than the accredited two-digit-uncertainty (“0.08” instead of “0.081”).
- Laboratory #2 and #8 issued a certificate without the title “calibration certificate”.
- Laboratory #1 issued a certificate with a conformity statement based on an uncertainty that was larger than one third of the tolerance span.

11. SUMMARY AND CONCLUSIONS

A laboratory intercomparison was run among eight labs and one reference lab in the METTLER TOLEDO company. The task was the calibration of the Conventional Mass of four weight pieces.
If weights were cleaned properly before calibration, none of the 32 E_n-Values was greater than 1
This is:

- Proof of the correct dissemination of the mass unit from the national laboratories which provide the calibration laboratories with traceability of their references (Table 1, column “traceable to”).
- Proof of the proper functioning of the accreditation system in each participating country.
- Justification and confirmation for the multilateral agreements (EA, APLAC, IAAC and ILAC) on recognition of certificates.
- Proof of the competence of the accredited METTLER TOLEDO labs.

REFERENCES

[1] European Accreditation Organisation: EA-2-03

[2] DKD: Final Report EA Interlaboratory Comparison Ma1 ‘Mass’, February 2001

[3] Organisation Inernational de Métrologie Légale: Recommendation R111, 1994

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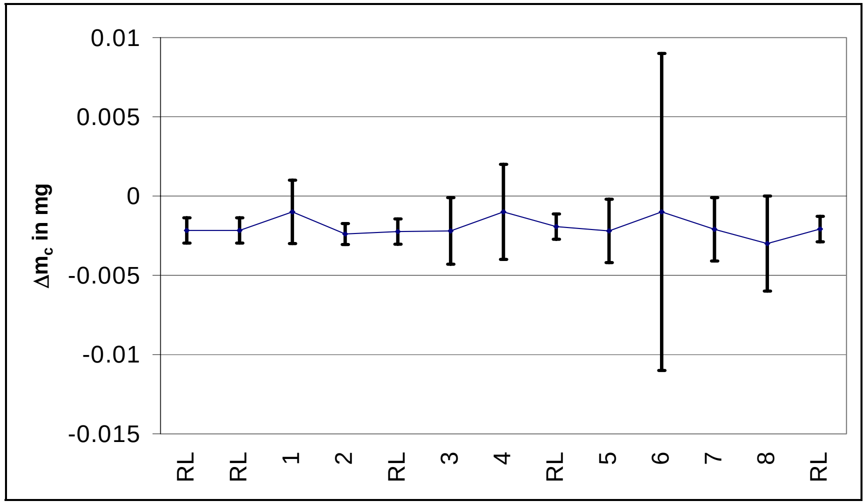

Figure 1: Result for the 2 mg piece

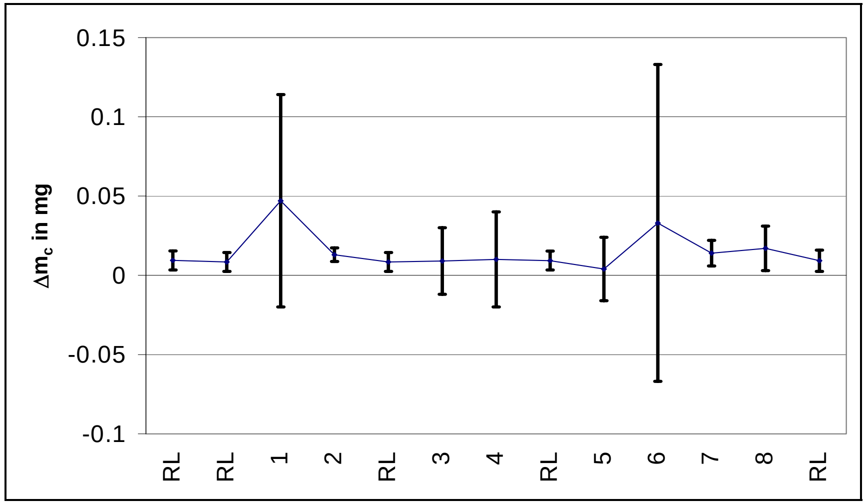

Figure 3: Result for the 10 g piece

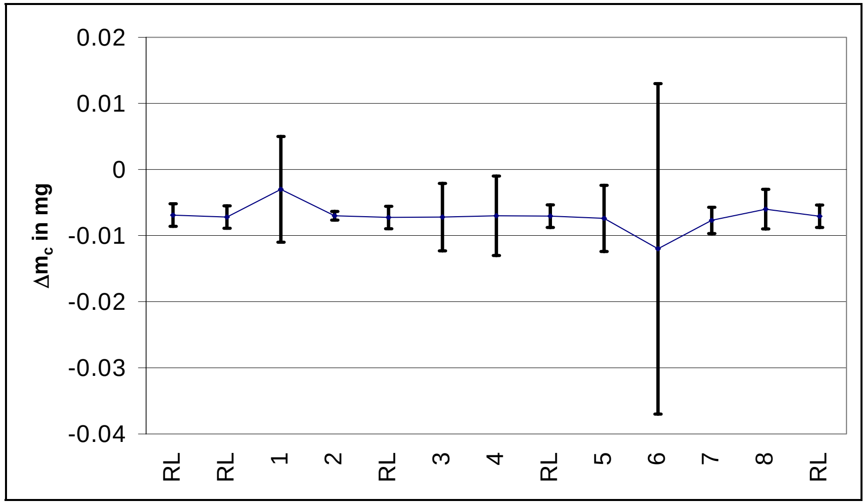

Figure2: Result for the 100 mg piece

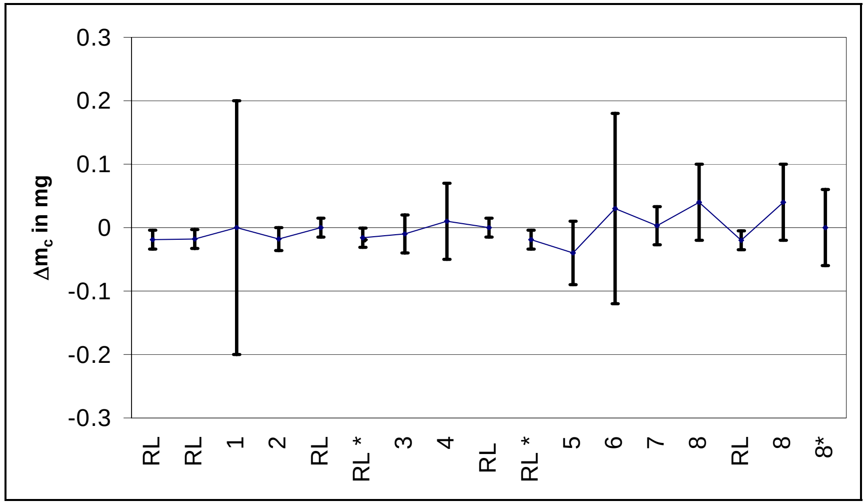

Figure 4: Result for the 100 g piece (RL* and 8* are after cleaning the weight piece)