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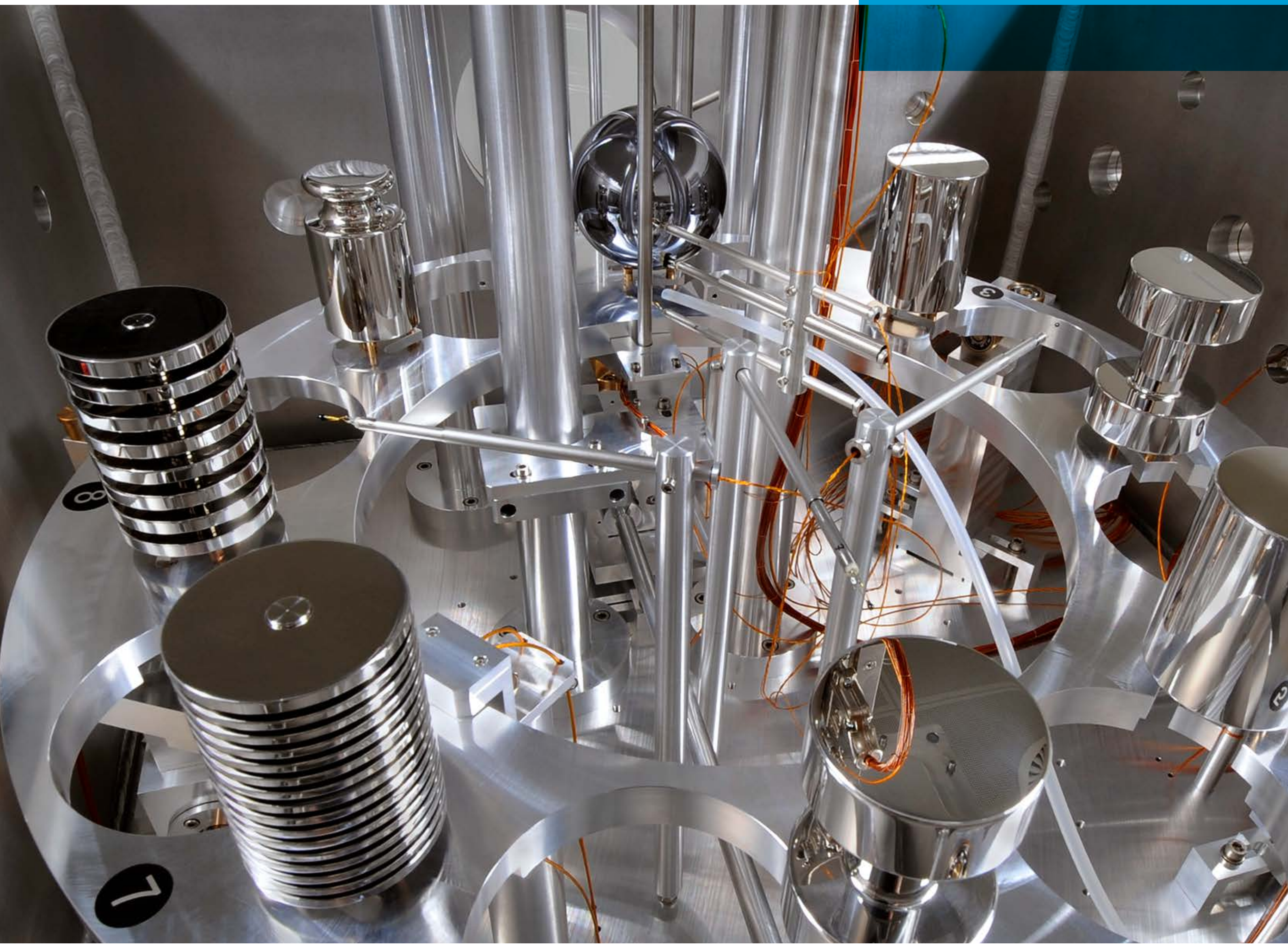

The Road to a Mass Laboratory

Design, training and equipment

Martin Firlus

german
cooperation
DEUTSCHE ZUSAMMENARBEIT

On behalf of

Federal Ministry
for Economic Cooperation
and Development

On behalf of the German federal government, the Physikalisch-Technische Bundesanstalt (PTB) promotes the improvement of framework conditions for economic activity, thereby supporting the establishment of metrology.

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Introduction

On 16 November 2018 in Versailles, the National Metrology Institutes of the world resolved to fundamentally reform the International System of Units (SI).

The system of references we use to "measure the world" is well defined. For instance, we divide time into seconds, length into meters and mass into kilograms. The International System of Units (SI) has been approved by nearly 100 states and is thus a global success story. The SI is now being fundamentally revamped so that it will be able to face all scientific and technical challenges of the 21st century with ease.

This silicon sphere weighs exactly one kilogramm – down to the atom

To put it bluntly, the kilogram is outdated. It still is what it was at the end of the 19th century – namely, the mass of a particular metallic cylinder stored in a safe at the International Bureau of Weights and Measures (BIPM) near Paris. Each kilogram weight in the world is based on this prototype of the kilogram. And this is not all: numerous other units, such as the mole or the ampere, depend on the kilogram. If there is a problem with the kilogram, there is thus automatically a problem with these other units as well. The problems with the definition of the kilogram arise because the kilogram is realized as a material measure, i.e. as an object – and macroscopic objects are bound to undergo changes. The prototype of the kilogram and the national copies each member state of the Metre Convention has received are no exceptions to this rule. Today, if the assertion is made that no one knows how heavy a kilogram is with microgram accuracy, this may contradict the definition, but describes the problem quite adequately. These circumstances prompted metrologists to solve this problem.

Two experiments that are based on different principles have been conceived to make the kilogram's future stable. In one approach, the force of gravity that acts on a weight is compensated for by an electromagnetic force. This approach exploits several electrical quantum effects, so that these experiments, the so-called "watt balance experiments" (now known as Kibble balance experiments), provide a value of Planck's quantum of action h. Essential contributors to this experiment are located in Canada, the United States and England and now at PTB, too.

An alternative method to this (which is preferred by PTB) links a macroscopic mass with the mass of an atom. Counting an extremely large number of atoms can only be achieved if the atoms are located in a structure with a strong order, namely in a monocrystalline structure. This experiment (which is called the "Avogadro experiment" because it directly yields the Avogadro constant as a result) is based on a crystal sphere made of isotopically pure silicon which, as the base material, was concentrated in ten thousands of centrifuges. Despite the scientific competition prevailing between these two experiments, they will eventually have to find common ground.

Only if the results from both experiments are in agreement with each other will they pave the way for the new kilogram.

However, for this guide to establishing a mass laboratory, it does not matter whether mass is eventually traced to a silicon sphere or to a watt balance (Kibble balance).

Establishment of a Mass Laboratory

The accuracy or reliability of weighing results is closely connected with the place where mass comparators are installed, and also with the weights used, with the measuring room conditions and the staff qualification. The place of installation (measuring room) for mass comparators should be designed in such a way that disturbances of the environment (e.g. vibrations, climate) affect the measurement result only slightly.

1. Requirements to be met by a measuring room

1.1. The floor should allow a safe and vibrationless installation of weighing tables.

For this purpose, the area of installation of a weighing table must be separated from the remaining floor (joint). This prevents that impact sound influences the mass comparators, and thus the weighing result. The joint is closed again with suitable damping and sealing means.

The coating of the floor should prevent additional contamination by abrasion and guarantee optimal cleaning (PVC coating or plane flagstones).

1.2. The measuring room should have no windows (avoidance of solar irradiation) and only one access (avoidance of draught). It is recommended to paint the walls of the measuring room with a solvent-free, wiping-proof wall paint. Suitable wall coatings also improve the quality of the measuring room due to their insulating properties.

1.3. The kind and duration of the illumination of a measuring room depends on the air-conditioning system used. It should, however, be installed at a sufficient distance from the weighing table to minimize the influence of disturbing heat radiation on mass comparators and weights. Daily activating and deactivating of the lighting appliances influences the temperature of the measuring room and causes undesired temperature variations. This is why the lighting appliances should remain switched on continuously (24 h).

1.4. The installation of mass comparators and thus the calibration of weights are performed under stable environmental conditions. To comply with this requirement in measuring rooms, an air-conditioning system is required.

The temperature and the relative air humidity must – with an existing ambient pressure – for example be regulated in accordance with the recommendations of OIML[1] R 111 (see 3.1.). In any case, it must be ensured that the environmental conditions meet the weighing specifications specified by the manufacturer. The temperature should, for example, lie between 18 °C and 27 °C. For the relative air humidity, values between 40 % and 60 % are usually recommended, as values below 40 % may lead to electrostatic charging and values above 60 % to corrosion.

1 OIML = Organisation Internationale de Métrologie Légale

2. Installation of mass comparators

2.1. Mass comparators should be installed on weighing tables which:

- transfer as few vibrations as possible (achievable by a high dead weight),
- do not bend (high solidity and material strength),
- are antimagnetic (no use of steel),
- are antistatic (no use of plastics or glass).

2.2. Installation of the mass comparators requires sufficiently large weighing tables. A weighing table should be used exclusively as working place for one mass comparator.

Weighing tables of stone, whose surfaces are polished, have proved their worth in mass laboratories.

Due to their dead weight, they reach a high stability, and the polished surfaces allow optimal cleaning.

3. Air density measuring instruments

3.1. Performance of a calibration requires the determination of the air density parameters (temperature, relative humidity and air pressure).

The international recommendation OIML R 111 (2004), Annex C, section C.2, Table C.1, contains recommended values for the individual accuracy classes.

It must be made sure that the uncertainties of the measuring instruments used – which determine the uncertainty of the air density – enter into the expanded uncertainty of a weighing result.

3.2. If calibrations are, for example, performed in accordance with the class E2 accuracy, the air density parameters could be determined with the following traceable measuring instruments (d = resolution):

- glass thermometer; d = 0.01 °C or
- electric resistance thermometer, d = 0.01 °C,
- electric capacitive humidity sensor, d = 1 % or
- Aßmann psychrometer, d = 0.1 °C,
- aneroid barometer, d = 0.5 mbar or
- electronic barometer, d = 0.1 mbar.

3.3. Data determination of the air density parameters should always be performed before a calibration.

OIML R 111 specifies temperature gradients which should not be exceeded.

Only if the temperature is determined directly on the measuring place (e.g. beside the mass comparator), a control of compliance with this recommendation during a calibration is possible. Rapid temperature changes must also be avoided by all means. For the determination of the relative humidity and air pressure, the values in the measuring room are, however, sufficient.

3.4. It should be possible to determine all air density data in the measuring room permanently in order to meet the conditions of OIML R 111 for temperature and relative air humidity. An electronic data acquisition allows the stored measurement values to be retrieved at any time and to be graphically represented if and when required. A simpler determination with mechanical drum writers is also possible and sufficient for the accuracy classes E2 to M3. Care must be taken that the changes (over 12 hours for the temperature and over 4 hours for the relative humidity) comply with the recommended values in Table C.1.

3.5 The local position of the mass laboratory (especially the altitude above sea level) is important for the determination of the air density.

The local air density must, for example, not deviate by more than 10 % from the conventionally determined reference air density (1.2 kg m^{-3}).

In the case of larger deviations, the conventional mass must be calculated from the mass.

4. Mass comparators

4.1. The selection of a mass comparator should meet the required metrological characteristics on the basis of the desired requirements. The following characteristics must be taken into account:

- Which nominal values shall be covered by the measurement range of the mass comparator?
- Weights of which accuracy classes shall be calibrated?
- Which resolution and maximum capacity are required?
- Is an automatic calibration with an incorporated calibration weight desired?
- reproducibility
- linearity
- simple handling and readability
- simple cleaning of the weighing room

4.2. If calibrations are, for example, to be performed from 1 mg to 10 kg and for the accuracy class E2, a distribution of the nominal values to four mass comparators has proved its worth in practical application. Mass comparators with the following specifications could be taken into account.

Max. capacity	Indication	Nominal value
5 g	0.0001 mg	1 mg to 5 g
100 g	0.001 mg	10 g to 100 g
1 kg	0.01 mg	100 g to 1 kg
10 kg	0.1 mg	1 kg to 10 kg

4.3. On the basis of the accuracy class, a mass comparator is to be selected in such a way that its uncertainty component is balanced in proportion to the overall uncertainty of the weighing result. The most important uncertainty component of a mass comparator is calculated from its standard deviation, (s).
The specification of the manufacturer can be selected as a first approximation for the value of a standard deviation. It must be taken into account, however, that this indication is decisive for the smallest nominal value. It should, therefore, not exceed an amount of 30 % of the combined standard uncertainty u_c (k = 1).

Example:

1 mg E2, $U_{(k=2)}$ = 0.002 mg
u_c = U/2 = 0.002 mg/2 = 0.001 mg
s = 0.3 × 0.001 mg ≤ 0.0003 mg

5. Weights

5.1. A fundamental prerequisite for the comparability and comprehensibility of measuring results is their reproducibility. It must be made sure that the indications of different mass comparators are identical. The weights required for this purpose must have been traced back to the international kilogram prototype on the basis of comparison measurements with national standards.

In OIML R 111, the weights are classified into accuracy classes. The requirements regarding the single accuracy classes (e.g. material properties, surface properties etc.) are described in detail.

5.2. Weights are used for the calibration of weighing instruments or for the calibration of weights of a lower accuracy class. The accuracy class is a class designation for weights which meet specific metrological requirements to achieve that the mass values are within specified limits. Weights of the accuracy class E1 can, for example, be used for the calibration of weights of the accuracy class E2.

5.3. As to the weight sets required for a calibration laboratory, the individual nominal values should in each case be available twice. This guarantees that calibrations can also be carried out when a weight has been damaged or when a weight set has been sent away for recalibration.

5.4. Weight sets which are used for the calibration of other weight sets should be kept under glass jars (e.g. protection against contamination by dust) in lockable laboratory cabinets.

6. Personnel

6.1. A laboratory should have managerial and technical staff whose education and qualification meet the requirements of a calibration laboratory.

Studies of natural science or engineering science (e.g. mechanical engineering, physics) are prerequisites for the management of a calibration laboratory.

6.2. Basic and advanced training at other national institutes or accredited calibration laboratories improve the qualification for the application of international standards and recommendations (e.g. EA[1], OIML).

6.3. Thorough training of the operating staff is a prerequisite for a correct measuring process and influences decisively the quality of the measurement result. Basic and advanced training should take place at regular intervals in the respective calibration laboratory. Additional training specific to the existing measuring equipment enhances and stabilizes the practical and theoretical knowledge.

6.4. If other calibration laboratories are available in a country, basic and advanced training of the collaborators should be realized in seminars (performed by the management of the national calibration laboratory). This allows a continuous improvement of the requirements for the quality of a calibration laboratory to be achieved and ensured.

List of requirements to be taken into account:

- OIML R 111 (2004)
- ISO/IEC 17025:2005
- GUM[2]
- EA 4/07: Traceability of Measuring and Test Equipment to National Standards

1 EA = European Co-operation for Accreditation
2 GUM = Guide to the Expression of Uncertainty in Measurement

7. Calibration intervals of weights

As weights are – due to their frequent use – subject to large changes in the course of time, external influences (such as storage or handling) must be taken into account when assessing their calibration intervals. Thus, the long-term stability of weights is of decisive importance for laying down calibration intervals. Contrary to other measuring instruments (e.g. weighing instruments that are checked daily or maintained annually), no calibration intervals have been specified for weights.

7.1. Influence on weights

When weights are handled and stored under controlled environmental conditions, which have also been recommended by OIML R 111, this will increase their long-term stability and can lead to a clear prolongation of the calibration intervals (see the figure "Influences on a weight" below).

7.2. History of the mass change of a weight

To lay down a calibration interval, it is necessary to collect and evaluate all the information that is available about the time dependence of the mass change of a weight (or a weight set). The initial calibration and all the calibrations that follow should be carried out at constant time intervals.

The temporal drift behaviour of a weight can be evaluated with certainty only after the second recalibration has been carried out – i.e. when a total of three values is available.

7.3. E_n value

If the values of the individual calibrations (e.g. values from calibration certificates) are to be compared with each other, an objective evaluation of the time dependence of the mass change of a weight is necessary. Here, it is suitable to calculate, after each recalibration, the so-called E_n value. This E_n value is an important criterion to decide whether the recalibration interval can – or must – be maintained, prolonged or reduced.

$$|E_n| = \frac{m_{c,n} - m_{c,n-1}}{\sqrt{U_n^2 + U_{n-1}^2}} \le 1$$

$m_{c,n}$	conventional mass of the weight of the n^{th} calibration
$m_{c,n-1}$	conventional mass of the weight of the $(n-1)^{th}$ calibration
U_n, U_{n-1}	uncertainties (k = 2)

Influences on a weight:

Storage
(box, bell jar)

Climate
(temperature, humidity, air pressu

Operator
(care)

Handling
(auxiliary means)

Surface
(material, roughness)

Time
(calibration interval)

Example: 1 kg E1

Initial calibration m_0:	1 kg + 0.256 mg	U: 0.160 mg	
1st recalibration m_1:	1 kg + 0.302 mg	U: 0.160 mg	$\lvert E_n \rvert$ = 0.20
2nd recalibration m_2:	1 kg + 0.329 mg	U: 0.160 mg	$\lvert E_n \rvert$ = 0.12

7.4. Schedule of calibration intervals

As has turned out, the information required on the drift behaviour of a weight can be obtained within a relatively short time by limiting the calibration interval to – initially – two years. If, after a repeated recalibration, the absolute amount of the E_n value is clearly smaller than one (E_n < 0.6), the calibration interval can be prolonged. Here, the following schedule has proved its worth.

If weights – e.g. working standards – are used for the calibration of weighing instruments and are, thus, in constant use, a calibration interval of one year is required.

Calibration	**Calibration interval**		
	2 years	3 years	4 years
Initial calibration			
1st recalibration	x		
2nd recalibration	x		
3rd recalibration		x	
4th recalibration			x
...			
nth recalibration			x

7.5. Determination of the calibration intervals

If a prolongation of the calibration interval is desired, the complete documentation of all data (course of the calibrations and calculated E_n values) has to be submitted to the German Accreditation Body (DAkkS), for example. If the data submitted comply with the requirements ($E_n \leq 1$), the DAkkS can determine that the calibration interval is extended to the next higher step.

7.6. Evaluation

As weights underlie mass changes as a result of their use, E_n values larger than one are possible. If this is the case (E_n > 1), a reduction of the calibration interval is recommended. A critical analysis of the conditions of use of this weight has to be drawn up in accordance with the quality management (QM).

Practice has shown that very long recalibration intervals render the assessment of the drift behaviour of a weight more difficult, and that the mass will possibly be disseminated with a mass deviation which is too large. For example, in the case of a typical annual drift of 0.02 mg, the mass change of a 1 kg weight amounts, after four years, to up to 0.08 mg. For weight class E1, this already corresponds to the maximum permissible combined standard uncertainty uc = 0.08 mg. For that reason, recalibration intervals of not longer than four years should be aimed at.

Example: 1 kg E1

Date of the calibration	Calibration Certificate No.	Conventional mass	Uncertainty k = 2	$\lvert E_n \rvert$	Note
15 March 2005	PTB-04305	1 kg + 0.256 mg	0.160 mg	–	Initial calibration
26 March 2007	PTB-02107	1 kg + 0.302 mg	0.160 mg	0.20	1st recalibration
12 March 2009	DKD-K-	1 kg + 0.329 mg	0.160 mg	0.12	2nd recalibration

Notes

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