# **Fundamentals of Mass Determination**

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> 1. Auflage 2012. Buch. x, 114 S. Hardcover ISBN 978 3 642 11936 1 Format (B x L): 15,5 x 23,5 cm

<u>Weitere Fachgebiete > Chemie, Biowissenschaften, Agrarwissenschaften ></u> <u>Analytische Chemie > Massenspektrometrie</u>

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Figure 2.1: Kilogram prototype number 52 (stored under two glass covers)

# 2. Mass determination

# 2.1 Dissemination of the unit of mass

In 1883, from three Pt-Ir prototypes designated as KI, KII, and KIII, the prototype KIII was chosen by the CIPM as the international prototype of the kilogram and designated with the Gothic letter K. At the first General Conference on Weights and Measures in the year 1889, 30 of the first 42 available kiloaram prototypes were distributed by draw among the member states and the BIPM. Two additional prototypes (KI and No. 1) were presented to the BIPM for safekeeping as reference standards (témoins) in conjunction with the IKP. The remaining prototypes were retained for subsequent allocation and stored at the BIPM. The BIPM received two additional prototypes as working standards, France received five prototypes, several states two prototypes each, and the remaining states one prototype each [4]. In the years 1929 to 1993, 40 more prototypes were produced: most of these were distributed to additional member states of the Metre Convention as national prototypes [16]. For example, Switzerland has the prototype number 38; the national mass standard acquired in 1954 by the Federal Republic of Germany is prototype number 52 (Figure 2.1).

According to a recalibration performed at the BIPM in 2010, the mass of kilogram prototype number 52 was determined to be  $m_{52} = 1.000\ 000\ 292$  kg with a standard measurement uncertainty of  $u(m_{52}) = 6 \times 10^{-9}$  kg. Since the definition and realisation of the unit of mass is tied to a specific object, the unit of mass cannot be disseminated with higher accuracy than that permitted by mass comparisons with the international prototype. This explains the hierarchical structure of mass standards, which guarantees dissemination of the unit of mass with the highest possible level of accuracy.

# 2.1.1 Hierarchy of mass standards

The international prototype of the kilogram is at the top of the hierarchy for the dissemination of the unit of mass. The national prototypes are linked to the BIPM working standards, which are, in turn, linked to the international kilogram prototype (Figure 2.2). Therefore, the international kilogram prototype only had to be used in 1889, 1939, 1946, and most recently 1989/92 as a reference, and, thus, it is protected to a large extent against wear and possible damage. The unit is further disseminated by the respective national metrology institute, e.g. PTB, using stainless steel secondary standards (density 8000 kg/m<sup>3</sup>). From a metrological point of view, linking these secondary standards to the national prototype is the most difficult step, since the required transition from a density of 21500 ka/m<sup>3</sup> (Pt-Ir) to 8000 ka/m<sup>3</sup> (steel) causes a larger uncertainty of the air buoyancy correction than the uncertainty of weighing and other influencing augntities. The reference standards of verification authorities for legal metrology and the reference standards of calibration laboratories and other institutions are then linked to the PTB secondary standards. The prototypes, secondary standards and reference standards are standards of the highest accuracy. Handling always bears the risk of unexpected mass changes (e.g. due to wear, contamination) and possible damage. Therefore, the selected recalibration intervals should be as long as possible. but yet short enough so that significant changes in mass are recognised. The right-hand column of Figure 2.2 shows examples of the time between two recalibrations. Working and control standards are easier to replace. The interval at which they need to be verified or recalibrated depends on the conditions and frequency of use.

#### Figure 2.2:

Hierarchy of mass standards, using the Federal Republic of Germany as an example (Pt-Ir: platinum-iridium alloy, BIPM: Bureau Internationale des Poids et Mesures [International Bureau of Weights and Measures], CIPM: Comité International des Poids et Mesures [International Committee for Weights and Measures, Sèvres, F], PTB: Physikalisch-Technische Bundesanstalt)



## 2.1.2 Mass scale

In order to determine the mass of arbitrary objects, multiples and submultiples of the mass unit must be realised in the form of mass standards and be linked to 1 kg standards. This is done by representing the nominal values in each decade, using a combination of standards. In legal metrology, the following nominal values shall be used for weight sequences in a set of weights:  $1 \times 10^{n}$  kg,  $2 \times 10^{n}$  kg and  $5 \times 10^{n}$  kg,  $n \in \{..., -2, -1, 0, 1, 2, ...\}$  [17]. At least four standards are required per decade. The sequence 1, 2, 2, 5 is commonly used. In addition, the duplicate use of each nominal value, i.e. the use of six standards per decade with the values 1, 1, 2, 2, 5, 5 allows every nominal value in the mass scale to be covered twice [18]. Taking as an example the decade from 100 g to 1 kg, the first link-up weighing with a known mass  $m_{1kg}$  results in the equation

$$m_{1\rm kg} - m'_{1\rm kg} = x(1),$$
 (2.1)

where  $m_{1kg}$  is the mass of the standard with a nominal value of 1 kg (No. 1),

- $m'_{1kg}$  is the mass of the standard with a nominal value of 1 kg (No. 2),
- x(1) equals the mass difference as the result of the initial weighing.

Further equations, such as

$$m_{1\mathrm{kg}} - (m_{500\mathrm{g}} + m'_{500\mathrm{g}}) = \mathrm{x}(2),$$
 (2.2)

$$m_{500g} - m'_{500g} = x(4), \tag{2.3}$$

and the use of additional standards allow as many or more mass comparisons to be conducted as the number of standards of unknown mass. Thus, each decade and finally each set of mass standards can be derived from a single standard with a known mass [7, 19].

ę	0							
Weighing	1 kg	1 kg	500 g	500 g	200 g	200 g	100 g	100 g
x(1)	+	-					/	
x(2)	+		-	-				
x(3)		+	-	-				
<i>x</i> (4)			+	-				
<i>x</i> (5)			+		/-	-	-	
<i>x</i> (6)				+	-	-		-
<i>x</i> (7)					+	-		
<i>x</i> (8)					+		-	-
<i>x</i> (9)						+	-	-
x(10)							+	-
Decade 10 g to 10	00 g 📈							
Weighing	100 g	100 g	50 g	50 g	20 g	20 g	10 g	10 g
x(1)	+	-						
:								
r(10)							+	_
м(10)							1	
Decade 1 g to 10 g								
8	g							
Weighing	g 10 g	10 g	5 g	5 g	2 g	2 g	1 g	1 g
Weighing	g 10 g	10 g	5 g	5 g	2 g	2 g	1 g	1 g
Weighing Weighing	g 10 g 10 mg	10 g 10 mg	5 g 5 mg	5 g 5 mg	2 g 2 mg	2 g 2 mg	1 g 1 mg	1 g 1 mg
Weighing Weighing	g 10 g 10 mg +	10 g 10 mg	5 g 5 mg	5 g 5 mg	2 g 2 mg	2 g 2 mg	1 g 1 mg	1 g 1 mg
Weighing Weighing x(1) :	g 10 g 10 mg +	10 g 10 mg -	5 g 5 mg	5 g 5 mg	2 g 2 mg	2 g 2 mg	1 g 1 mg	1 g 1 mg
Weighing Weighing x(1) $\vdots$ x(10)	g 10 g 10 mg +	10 g 10 mg -	5 g 5 mg	5 g 5 mg	2 g 2 mg	2 g 2 mg	1 g 1 mg	1 g 1 mg
Weighing Weighing x(1) : x(10)	g 10 g 10 mg +	10 g 10 mg -	5 g 5 mg	5 g 5 mg	2 g 2 mg	2 g 2 mg	1 g 1 mg +	1 g 1 mg

# Decade 100 g to 1 kg

and for larger nominal values

# Decade 1 kg to 10 kg

Weighing	10 kg	10 kg	5 kg	5 kg	2 kg	2 kg	1 kg	1 kg
x(1)	+	-						
: x(10)							+	-

## Figure 2.3:

Example of derivation of a mass scale from a weighing scheme with seven unknown standards and ten weighings per decade [18]

Various weighing schemes can be used depending on the requirements and the specified weight sequence in a set of weights. Figure 2.3 illustrates an example with seven unknown standards (with the weight sequence 1, 1, 2, 2, 5, 5, 10) and ten weighings per decade. The first line shows that during the initial weighing, the known 1 kg standard (symbol "+") is compared to the unknown 1 ka standard (symbol "-"). The weighing result of this comparison is x(1). The over-determined equation system with ten equations and seven unknowns allows the unknown mass values of the individual standards to be calculated with the aid of a least-sauares adjustment (Appendix A.2). One of the 100 a standards that were determined in the first decade is the starting point for the comparisons in the following decade for the range from 10 g to 100 g, etc. This allows all of the following decades, e.g. down to 1 mg, as well as decades for nominal values greater than 1 kg, to be derived successively. Starting from the respective national kilogram prototype, national metrology institutes normally use several sets of mass standards to derive the mass scale across several decades (e.g. in the range of 1 mg to 5000 kg in the case of PTB).

# 2.2 Mass standards and weights

Language differentiates between "mass standards" and "weights". While there are no special regulations for mass standards regarding material, shape, surface characteristics, etc., there are international directives and recommendations that apply to weights as well as national regulations that establish error limits, materials, shapes, etc. [17, 20–22].

In practice, mass standards and weights are rarely used for weighing. In general, the user utilises (verified) weighing instruments which (are used in official or business transactions and) do not require mass standards (weights) for weighing. Instruments that are used in legal metrology are adjusted and verified with weights at intervals of one to four years. Comprehensive procedures (type approval, regular verification of the mass standards and weights used for adjustment and verification by verification offices and national metrology institutes) are in place to ensure that a verified instrument "measures correctly" even without weights. Therefore, mass standards and weights are primarily used to adjust and check weighing instruments and for precision mass determinations with relative uncertainties of <  $10^{-5}$  (see Section 2.3).

#### 2.2.1 Conventional mass and maximum permissible errors

The conventional mass  $m_c$  of a weighed object with the mass m and the density  $\rho$  at a reference temperature of 20 °C corresponds to the mass of a standard with a density  $\rho_c = 8000 \text{ kg/m}^3$ , which it balances in air with a reference density of  $\rho_0 = 1.2 \text{ kg/m}^3$ . Therefore, the conventional mass is a function of the mass m and the density  $\rho$  (see equation 1.10) [15, 23]

$$m_{\rm c} = m \frac{1 - \rho_0 / \rho}{1 - \rho_0 / \rho_c} = m \frac{1 - (1.2 \, \text{kg m}^{-3} / \rho)}{0.99985} \quad . \tag{2.4}$$

The conventional mass was introduced in order to reduce mass comparisons to a simple weighing process. With the introduction of standard conditions for the density of the weighed object ( $\rho_c = 8000 \text{ kg/m}^3$ ) and the density of the air ( $\rho_0 = 1.2 \text{ kg/m}^3$ ), reference conditions were defined for the adjustment of the instrument. The different weight forces of weighed objects with the same mass but different densities become comparable. If the density  $\rho$  of the weighed object deviates from the conventional density  $\rho_{c_{r}}$  an instrument indicates the conventional mass when weighing under standard conditions in air ( $\rho_a = \rho_0$ ). This weighing value corresponds to the same force action exerted on the instrument by a comparative mass  $m_{\rm c}$  with density  $\rho_{\rm c}$  at an air density of  $\rho_a = \rho_0$ . Since the conventional mass corresponds to the value of a comparative mass, the unit of the conventional mass is the kg. The mass m of a weighed object can be calculated from the conventional mass  $m_{\rm c}$  using equation (2.4). The relative deviation of the mass from the conventional mass is

$$\frac{m - m_{\rm c}}{m} = 1 - \frac{1 - \rho_0 / \rho}{1 - \rho_0 / \rho_{\rm c}} = \frac{\rho_0}{m} (V - V_{\rm c}) \quad , \tag{2.5}$$

with the "conventional volume"  $V_c = m_c/\rho_c$ . The term  $\rho_0(V-V_c)$  corresponds precisely to the mass of the air with the density  $\rho_0$  contained in the volume difference  $(V-V_c)$ . For a weighed object with a density of  $\rho > 1000 \text{ kg/m}^3$ , the relative deviation of mass from the conventional mass is less than 0.1 % (see Figure 2.4).





Relative deviation of the mass from the conventional mass  $(m-m_c)/m$ as a function of the density of the weighed object  $\rho$  (equation 2.5)

In legal metrology, weights are assigned to accuracy classes with defined error limits, otherwise called "maximum permissible errors" (mpe), according to international regulations (OIML R 111, Table 2.1). The specified nominal value of a weight is not the mass, but the conventional mass. The error limits also refer to the conventional mass. The accuracy class with the smallest mpe is class E<sub>1</sub>. The mpe for subsequent classes with the designations E<sub>2</sub>, F<sub>1</sub>, F<sub>2</sub>, M<sub>1</sub>, M<sub>2</sub> and M<sub>3</sub> (with M<sub>1-2</sub> and M<sub>2-3</sub> as interim classes [17]) increase by a factor of approximately  $\sqrt{10}$ , respectively. For each weight, the expanded measurement uncertainty U(k=2) of the conventional mass must be less than or equal to one third of the specified margin of error  $\delta m$ .

$$U \le \delta m/3 \tag{2.6}$$

The expanded measurement uncertainty U is part of the mpe, i.e. the conventional mass  $m_c$  of a weight may not deviate from the nominal value  $m_0$  by more than the difference between the specified margin of error  $\delta m$  and the expanded measurement uncertainty U.

$$m_0 - (\delta m - U) \le m_c \le m_0 + (\delta m - U)$$
 (2.7)

In order to meet the uncertainty requirements when calibrating the conventional mass of weights of a given accuracy class, weights of a higher class (usually the next-higher) are used; for example, standards of class  $E_2$  are used for weights of class  $F_1$ .

	Maximum permissible errors $\delta m$ in mg								
Nominal value	Class E1	Class E2	Class F1	Class F2	Class M1	Class M <sub>1-2</sub>	Class M2	Class M <sub>2-3</sub>	Class M3
5 000 kg			25 000	80 000	250 000	500 000	800 000	1 600 000	2 500 000
2 000 kg			10 000	30 000	100 000	200 000	300 000	600 000	1 000 000
1 000 kg		1 600	5 000	16 000	50 000	100 000	160 000	300 000	500 000
500 kg		800	2 500	8 000	25 000	50 000	80 000	160 000	250 000
200 kg		300	1 000	3 000	10 000	20 000	30 000	60 000	100 000
100 kg		160	500	1 600	5 000	10 000	16 000	30 000	50 000
50 kg	25	80	250	800	2 500	5 000	8 000	16 000	25 000
20 kg	10	30	100	300	1 000		3 000		10 000
10 kg	5.0	16	50	160	500		1 600		5 000
5 kg	2.5	8.0	25	80	250		800		2 500
2 kg	1.0	3.0	10	30	100		300		1 000
1 kg	0.5	1.6	5.0	16	50		160		500
500 g	0.25	0.8	2.5	8.0	25		80		250
200 g	0.10	0.3	1.0	3.0	10		30		100
100 g	0.05	0.16	0.5	1.6	5.0		16		50
50 g	0.03	0.10	0.3	1.0	3.0		10		30
20 g	0.025	0.08	0.25	0.8	2.5		8.0		25
10 g	0.020	0.06	0.20	0.6	2.0		6.0		20
5 g	0.016	0.05	0.16	0.5	1.6		5.0		16
2 g	0.012	0.04	0.12	0.4	1.2		4.0		12
1 g	0.010	0.03	0.10	0.3	1.0		3.0		10
500 mg	0.008	0.025	0.08	0.25	0.8		2.5		
200 mg	0.006	0.020	0.06	0.20	0.6		2.0		
100 mg	0.005	0.016	0.05	0.16	0.5		1.6		
50 mg	0.004	0.012	0.04	0.12	0.4				
20 mg	0.003	0.010	0.03	0.10	0.3				
10 mg	0.003	0.008	0.025	0.08	0.25				
5 mg	0.003	0.006	0.020	0.06	0.20				
2 mg	0.003	0.006	0.020	0.06	0.20				
1 mg	0.003	0.006	0.020	0.06	0.20				

# 2.2.2 Requirements

The requirements for weights refer to their physical and metrological characteristics. In order to ensure measurement trueness and stability that correspond to the respective accuracy requirements, the shape, dimensions, material, surface characteristics, density, magnetic properties, nominal values, and error limits of weights have been established in standards, directives, and ordinances [17, 20–22].

 Table 2.1: Maximum permissible

 errors ( $\pm \delta m$  in mg) for the conventional mass of weights according to the international recommendation

 OIML R 111 [17]

## 2.2.2.1 Shape

Weights must have a simple geometric shape without sharp edges or corners. In order to avoid deposits such as dust on the surface, they must not have any pronounced depressions. A high degree of stability, easy handling, and a favourable relationship between surface area and volume is ensured for mass standards and weights with a nominal value between 1 g and 20 kg by a cylindrical shape (Figure 2.5a) with a height-diameter ratio between 3/4 to 5/4. The block form with a fixed handle that does not protrude is also in widespread use for the range from 5 kg to 50 kg (Figure 2.5b). Weights with nominal values  $\geq$  50 kg are constructed so that, depending on the application, corresponding aids such as lifting and transportation equipment can be used safely and the weights can be stored securely. Weights with nominal values < 1 g are shaped as polygonal plates or wires so they are easier to handle and to differentiate. The shape of weights that are not inscribed with their nominal value must correspond to Table 2.2.

Nominal value	Polygonal plates		Wires	
5, 50, 500 mg	Pentagon	Pentagon		5 segments
2, 20, 200 mg	Square	Square	or	2 segments
1, 10, 100 mg	Triangle	Triangle		1 segment

Table 2.2:

Shape of weights with nominal values  $\leq$  1 g [17]







#### according to OIML R111 [17]. a cylindrical weights, b block-shaped weights

# 2.2.2.2 Material and surface properties

Weights must be made of a material that is highly resistant to corrosion caused by chemically and physically active substances in the atmosphere such as ozone, ammonia, oxygen, carbon dioxide, and water vapour. The material must have characteristics that ensure that changes in the mass of the weight that occur during normal use compared to the margin of error for the corresponding accuracy class can be disregarded. The surface of a weight must be smooth. Table 2.3 lists the maximum values for surface roughness according to the requirements specified in the OIML recommendation R 111. For weights with nominal values over 50 kg, twice the limit values in Table 2.3 apply.

Class	E1	E2	F1	F <sub>2</sub>
R <sub>z</sub> / μm	0.5	1	2	5
$R_{ m a}$ / $\mu { m m}$	0.1	0.2	0.4	1

The influence of the magnetic properties of a weight can be disregarded if the susceptibility and permanent magnetisation do not exceed the limit values specified in Table 2.4 and Table 2.5. The limit values were calculated so that, for commonly assumed maximum values of the magnetic flux density ( $B_z = 110 \ \mu\text{T}, \ \partial B_z/\partial z = -34 \ \mu\text{T/cm}$ , see [24]), the weighing results are not falsified by more than 10 % of the maximum permissible errors specified in Table 2.1.

#### Table 2.3:

Figure 2.5a-b:

Examples of the design of weights

Limit values for surface roughness [17]

ass de

Table 2.4: Limit values for magnetic

Table 2.5: polarisation [17]

Class			E1		E	2	F1		F <sub>2</sub>
<i>m</i> ≤ 1 g			0.25		0.9		10		-
$2 g \le m \le 10$	g		0.0	6	0.	18	0.7	7	4
20 g ≤ <i>m</i>			0.0	2	0.	07	0.2	2	0.8
Class	E1	E <sub>2</sub>	F1	F <sub>2</sub>	M <sub>1</sub>	M <sub>1-2</sub>	M <sub>2</sub>	M <sub>2-3</sub>	M <sub>3</sub>
Maximum polarisation $\mu_0 M / \mu T$	2.5	8	25	80	250	500	800	1600	2500

If the air density  $\rho$  deviates from the reference value  $\rho_0 = 1.2 \text{ kg/m}^3$ , this affects the determination of the conventional mass. In order to minimise this effect, limit values were established for the density of the weights [17]. The criterion is that the influence of a deviation in air density in a range of  $\pm 10$  % of the reference value is less than  $\frac{1}{4}$  of the maximum permissible errors specified in Table 2.1. Table 2.6 provides an overview of the resulting limit values for the individual accuracy classes.

In practice, the excellent material characteristics of austenitic steel with a density of 8000 kg/m<sup>3</sup> has proven itself well (e.g. steel X1NiCrMoCu25-20-5, material number 1.4539).

L										
Neminal			$ ho_{\min},  ho_{\max}$	(10 <sup>3</sup> kg m <sup>-3</sup> )						
Nominai	Accuracy class (no specifications for class $M_3$ )									
Vulue	E1	E <sub>2</sub>	F1	F <sub>2</sub>	M1	M <sub>1-2</sub>	M <sub>2</sub>	M <sub>2-3</sub>		
≥100 g	7.934 - 8.067	7.81 – 8.21	7.39 - 8.73	6.4 – 10.7	≥4.4	≥3.0	≥2.3	≥1.5		
50 g	7.92 - 8.08	7.74 – 8.28	7.27 – 8.89	6.0 - 12.0	≥4.0					
20 g	7.84 – 8.17	7.50 - 8.57	6.6 – 10.1	4.8 - 24.0	≥2.6					
10 g	7.74 – 8.28	7.27 – 8.89	6.0 - 12.0	≥4.0	≥2.0					
5 g	7.62 – 8.42	6.9 - 9.6	5.3 - 16.0	≥3.0						
2 g	7.27 – 8.89	6.0 - 12.0	≥4.0	≥2.0						
1 g	6.9 - 9.6	5.3 – 16.0	≥3.0							
500 mg	6.3 – 10.9	≥4.4	≥2.2							
200 mg	5.3 – 16.0	≥3.0								
100 mg	≥4.4									
50 mg	≥3.4									
20 mg	≥2.3									

Table 2.6: Lower and upper limit values for the density  $\rho_{\min}$ ,  $\rho_{\max}$  [17]

# 2.2.2.3 Handling and cleaning

Maximum accuracy mass standards and weights must be treated with extreme care. The weights are stored in dustproof boxes individually (usually from 1 kg and up) or as sets. They may only be handled with tweezers that have tips covered in plastic or another soft covering, weight forks made of wood, or with a clean, lint-free, non-greasing linen or leather cloth. Sets of weights are normally denominated so that each mass value can be represented by increments of the smallest weight in the set. According to OIML R 111 [17], the following increments are permitted:

$$(1, 1, 2, 5) \times 10^{n}$$
 kg  
 $(1, 1, 1, 2, 5) \times 10^{n}$  kg  
 $(1, 2, 2, 5) \times 10^{n}$  kg  
 $(1, 1, 2, 2, 5) \times 10^{n}$  kg

The exponent n is a positive or negative whole number, or zero.

For weights and weight sets of the classes  $E_1$  and  $E_2$ , a calibration certificate must always be issued for calibrations and tests performed, which are based on OIML R 111 [17]. For class  $E_2$ , such a certificate must include information about the conventional mass  $m_c$ , the expanded measurement uncertainty U, and the coverage factor k. In addition, certificates for weights of the class  $E_1$  must include information about the density or volume of each weight as well as a statement indicating if these values were measured or estimated.

Weights must be handled and stored in such a way that they remain clean. Before using the weights, minor dust deposits must be removed with bellows or a soft brush. Cleaning must not remove material from the surface or deteriorate the surface characteristics of the weight (e.g. scratches). Other contamination - such as finger prints caused by improper handling – can be removed by cleaning all or part of the weight in pure alcohol, distilled water, or alternatively a nondetrimental solvent. Hollow weights must not be immersed in the solvent, so that liquid does not enter through the opening. Depending on the degree of contamination, cleaning can cause changes in mass that cannot be disregarded (e.g. through changes to the sorption layers). In order to determine and document the effect of cleaning, mass determination before and after cleaning is recommended. The stabilisation periods specified in Table 2.7 must be observed after cleaning with alcohol or distilled water. Since cleaning with alcohol has a greater effect on the sorption layers, the stabilisation periods are longer than those after cleaning with distilled water.

Class	<b>E</b> 1	E2	F1	$F_2$ to $M_3$
After cleaning with alcohol	7-10 days	3–6 days	1-2 days	1 hour
After cleaning with distilled water	4-6 days	2-3 days	1 day	1 hour

 Table 2.7:

 Stabilisation periods after cleaning

 [17]

Weights have to stabilise to the environmental conditions at the measurement location before calibration. The temperature difference compared to the weighing chamber should be as small as possible, especially for weights of the E and F classes. The required period depends on the temperature difference between the weight and the environment at the beginning of the stabilisation process as well as the size and the margin of error for the weight. Table 2.8 provides an overview of the minimum periods. Up to a nominal value of 5 kg, a stabilisation period of 24 hours is recommended as a practical guideline.

Δ <b>T*</b>	Nominal Value	Class E1	Class E <sub>2</sub>	Class F1	Class F <sub>2</sub>
	1000, 2000, 5000 kg	-	93**	79	7
	100, 200, 500 kg	-	70	33	4
	10, 20, 50 kg	45	27	12	3
±20 °C	1, 2, 5 kg	18	12	6	2
	100, 200, 500 g	8	5	3	1
	10, 20, 50 g	2	2	1	1
	< 10 g	1	1	1	0.5
	1000, 2000, 5000 kg	-	51**	1	1
	100, 200, 500 kg	-	40	2	1
	10, 20, 50 kg	36	18	4	1
±5 °C	1, 2, 5 kg	15	8	3	1
	100, 200, 500 g	6	4	2	0.5
	10, 20, 50 g	2	1	1	0.5
	< 10 g	0.5	0.5	0.5	0.5
	1000, 2000, 5000 kg	-	16**	1	0.5
	100, 200, 500 kg	-	16	1	0.5
	10, 20, 50 kg	27	10	1	0.5
±2 0	1, 2, 5 kg	12	5	1	0.5
	100, 200, 500 g	5	3	1	0.5
	< 100 g	2	1	1	0.5
	1000, 2000, 5000 kg	-	-	-	-
	100, 200, 500 kg	-	1	0.5	0.5
.05.00	10, 20, 50 kg	11	1	0.5	0.5
±0.5 C	1, 2, 5 kg	7	1	0.5	0.5
	100, 200, 500 g	3	1	0.5	0.5
	< 100 g	1	0.5	0.5	0.5

#### Table 2.8:

Minimum stabilisation periods in hours for temperature equalisation between the weight and the weighing chamber [17]  $*\Delta T$  = Temperature difference between the weight and the weighing

chamber at the beginning of the stabilisation process

 $\ast\ast$  Value not specified in OIML R 111 (2004), only valid for 1000 kg

# 2.3 Physical weighing principles and methods

Weighing normally compares weight forces. According to equation (1.12), the weight force  $F_G$  of a body is the product of its mass *m* and gravitational acceleration *g*. Weighing is based on this relationship. Therefore, the mass of two bodies is equal if they exert the same weight force at the same gravitational acceleration (e.g. at the same location). Weight forces can only be compared directly in a vacuum. In air, the weight and buoyancy forces are overlaid vectorially. For a balance with equal arms in equilibrium (Figure 2.6), this overlay leads to the torque equation

$$l_{\rm L}(m_1 g_{\rm L} - V_1 \rho_{\rm aL} g_{\rm L}) = l_{\rm R}(m_2 g_{\rm R} - V_2 \rho_{\rm aR} g_{\rm R})$$
(2.8)

with the designations

- $m_1, m_2$  mass of the bodies (1 and 2),
- $V_1, V_2$  volume (capacities) of the bodies (1 and 2),
- $g_{\rm L}, g_{\rm R}$  local gravitational acceleration (left and right),

 $l_{\rm L}$ ,  $l_{\rm R}$  length of the effective lever arms (left and right),

- $\rho_1$ ,  $\rho_2$  density of the bodies (1 and 2),
- $\rho_{aL}$ ,  $\rho_{aR}$  air density during weighing (left and right).



Figure 2.6: Forces and torques on a balance with equal arms

Spatial variations of the gravitational acceleration in the vicinity of the balance can normally be disregarded. With  $g_{\rm L} = g_{\rm R}$ ,  $l_{\rm L} = l_{\rm R}$ ,  $\rho_{\rm aL} = \rho_{\rm aR} = \rho_{\rm a}$  and  $V_{1,2} = m_{1,2}/\rho_{1,2}$ , it follows that the mass  $m_2$  is

$$m_2 = m_1 \frac{1 - \rho_a / \rho_1}{1 - \rho_a / \rho_2} \quad . \tag{2.9}$$

In case of differences between the densities of the masses involved, the respective ratio between the air density and the solid density at the time of the comparison has a direct effect on the result of the mass determination. At a relative measurement uncertainty of up to  $10^{-3}$ , a buoyancy correction is generally not required; therefore, the weighing value read from the instrument can be considered as the direct mass determination result (see also Sections 2.2.1, 2.5.1 and Figure 2.4).

The following physical principles are used in order to determine mass or the conventional mass using weighing instruments:

- Full compensation of the weight force of the weighed object by applying weights or mass standards (mass comparison), e.g. using a mechanical balance with equal arms, mechanical balances with built-in weights and mechanical substitution beam balances
- Partial compensation of the weight force of the weighed object with dial weights or permanently installed counterweights and additional fine compensation, e.g. with electromagnetic (electrodynamic) force compensation in case of electromechanical dial weight balances, or with built-in counterweights and partial compensation of the weight force through electromagnetic force compensation in case of electronic comparator balances
- Full compensation of the weight force of the weighed object through counterforces that are not weight forces (force comparison), e.g. in case of inductive or capacitive load cells and electronic analytical balances with full electromagnetic force compensation, i.e. with a continuous measurement range between the minimum and maximum capacity

All of these principles are based on force comparison. However, if the compensating force is caused by a comparative mass (mass comparison), forces of the same origin are being compared. Therefore, changes of gravitational acceleration or air density have no effect or a significantly reduced effect.

The following weighing procedures are used to determine mass or the conventional mass:

- In proportional or simple weighing, the weighed object is applied to the load receptor (load pan) after the instrument is zeroed and the mass (the conventional mass) is read.
- Differential weighing, i.e. the mass comparison of the weighed object with a mass standard (reference standard), using the transposition method (Gaussian weighing) is only possible on balances with equal arms. Here the specimen (the weighed object) and the reference standard are exchanged on the weighing pans at least once, and the results of both weighings are averaged.

Differential weighing using the substitution method (Borda weighing) is possible on all types of instruments. Here the specimen (the weighed object) and the reference standard are compared on the same weighing pan in succession. With beam balances, the second weighing pan is loaded with a fixed auxiliary load (tare load).

For precision mass determinations with a relative uncertainty of  $< 10^{-5}$ , differential weighing is essential. Meanwhile, differential weighing using the substitution method has become the method of choice for modern comparator balances. It allows for simpler instrument designs and handling, resulting in shorter measurement times compared to the Gaussian method. In addition, the ability to automate the weighing process by using suitable weight-exchange mechanisms further increases measurement accuracy.

# 2.4 Scales and mass comparators

The first weighing instruments were simple balances. After the invention of the sliding weight scale, which is ascribed to the Romans, developments in the 19<sup>th</sup> century included mechanical weighbridges, crane scales, deflection scales, spring scales, and automatic weighing instruments. After the continued development of these mechanical weighing instruments up to the Second World War, the development of electromechanical instruments commenced; this led to a new variety of weighing instruments with various load cell principles [7].

## 2.4.1 Weighing instrument classifications

Analytical balances (laboratory balances) and comparator balances are used for high-accuracy mass determination. Analytical balances are instruments with a high resolution, where the scale interval *d* is usually less than or equal to the maximum capacity *Max* times  $10^{-5}$ . The maximum capacity is usually no more than 10 kg. Verifiable analytical balances are classified as weighing instruments of special accuracy (OIML accuracy class I) and weighing instruments of high accuracy (OIML accuracy class II) [12, 25]. Analytical balances are frequently divided into the classes of instruments listed in Table 2.9, depending on the scale interval and maximum capacity [7, 26–28].

The term "comparator balance" or "mass comparator" has become commonly accepted for instruments with an even higher resolution, e.g. with a number of scale intervals of  $n > 5 \times 10^7$  [26–28].

Designation	Common Maximum Capacity Max	Common Scale Interval d	Common Number of Scale Intervals n = Max/d
Precision balances	100 g 10 kg	1 mg 100 mg	104 105
Weighing instruments of special accuracy			
Macro balance	100 g 1 kg	100 µg	10 <sup>6</sup> 10 <sup>7</sup>
Semi-micro balance	25 g 100 g	10 µg	2.5×10 <sup>6</sup> 10 <sup>7</sup>
Micro balance	5 g 25 g	1μg	5×10 <sup>6</sup> 2.5×10 <sup>7</sup>
Ultra-micro balance	≤ 5 g	0.1 µg	≤ 5×10 <sup>7</sup>

#### Table 2.9:

Common classification of analytical balances for high-accuracy mass determination

## 2.4.2 Mechanical balances with equal arms

Up to the beginning of the 20<sup>th</sup> century, the mechanical balance with equal arms represented the preferred design for analytical and laboratory balances; it is still used by some national metrology institutes today because of its high degree of measurement accuracy. Meanwhile, it is of next to

no practical importance due to the disadvantages associated with its use (time-consuming measurement, complex operation, low comfort, sensitivity to vibrations and tilting) and the great amount of progress made with comparator balances with electromagnetic force compensation; therefore, we refer to additional literature here [7].

## 2.4.3 Electromechanical dial weight balances

Modern, high-resolution weighing instruments are equipped with an electromagnetic force compensation. The highest resolutions of up to  $10^{-10}$  times the maximum capacity are achieved by electromechanical dial weight balances and electronic comparator balances with partial electromagnetic force compensation (the electrical weighing range is usually  $10^5 d$  to  $10^6 d$ ). Although electromechanical dial weight balances have been losing importance to electronic comparator balances since around 1990, they are still relatively common due to their ruggedness and very high accuracy in conjunction with a full weighing range. Figure 2.7 shows the basic structure of an electromagnetic dial weight balance.



The essential mechanical components are: The balance beam (1), the suspension (2), the main and secondary knife-edges (3a, 3b), the locking system and locking lever (4a, 4b), the dial weights (including the adjustment weight)

#### Figure 2.7:

Schematic illustration of a dial weight balance with electromagnetic force compensation and an automatic weight-exchange mechanism. 1 balance beam; 2 suspension; 3a main knife-edge, 3b secondary knife-edge; 4a, 4b, locking system, locking lever; 5a dial weights (including adjustment weight), 5b rotary switch for dial weights; 6 compensation system (electromagnetic force compensation) with: 6a electro-optical position sensor, 6b coil, 6c permanent magnet; 7 counterweight; 8a sensitivity adjustment, 8b zero point adjustment; 9 pan brake; 10a automatic turntable for weightexchange mechanism, 10b lifting and rotating mechanism, 10c gear motor; 11 levelling screws; 12 weighing table.

with the rotary switches (5a, 5b), the fixed counterweight (7), the sensitivity and zero point adjustment (8a, 8b), and the pan brake to dampen the suspension oscillations after loading (9). The illustration also shows an automatic weight-exchange mechanism which consists of a turntable to hold several weights (10a), a lifting and rotating mechanism (10b) and a motor (10c).

The essential components of the electromagnetic compensation system are: The electro-optical position sensor (6a) consisting of the light source (LED), the light gap and the differential photodiode; the coil (6b) and the permanent magnet in a pot-type system (6c). The position sensor acts as a displacement transducer and the coil in the magnet system serves as an actuator for a PID controller that helps keep the vertical position of the balance arm at rest. Weight force differences are measured as proportional current changes. The electrical weighing range has to be adjusted with one or more adjustment weights – usually installed in the instruments – so that the weight force differences are indicated in units of mass.

The relationship between a load m' and the instrument display  $m_W$  (in units of mass) is illustrated in Figure 2.8, based on the example of an electromechanical dial weight balance with 10 dial weight steps.



In this case the usable weighing range includes all loads between the minimum capacity ( $m'_{\min}$ ) and the maximum capacity ( $m'_{\max}$ ); the load is compensated by the dial weights combined with electromagnetic force compensation.

#### Figure 2.8:

Adjustment characteristic of an electromechanical dial weight balance. O-10 steps of the weight-dialing mechanism, m' load on the weighing pan (in units of mass), m'min minimum capacity, m'max maximum capacity, mw balance indication (in units of mass), mwmax balance indicotion upper limit,  $\delta_{mW}$  linearity error,  $\Delta m'_{j}$  electrical weighing range for the selected step j (here j = 4). The largest positive or negative deviation from the theoretical linear curve shape is referred to as the linearity error  $\delta m_W$ . In case of dial weight balances, it mainly depends on the adjustment of the dial weights as illustrated in Figure 2.8. Therefore, substitution weighings are performed without changing the dial weight step and with an approximately constant load on the instrument in order to prevent linearity errors during high-accuracy mass determination (see Section 2.3).

### 2.4.4 Electronic analytical and comparator balances

Due to their high level of operator comfort, combined with a high resolution of up to  $5 \times 10^7$  scale intervals, electronic analytical and comparator balances have prevailed over all other types of instruments where laboratory weighing technoloav is concerned. The high resolution can only be achieved with electromagnetic force compensation; it usually fully compensates for the weight force (entire measurement range from the minimum capacity to the maximum capacitv). Built-in counterweights are also used in case of extreme-Iv high-accuracy requirements; in this case, the weight force is only partially compensated (restricted weighing range near the maximum capacity). Figure 2.9 shows the schematic structure of a top-loading electronic comparator balance with a fixed counterweight. The load receptor with the weighing pan (1) is auided by two parallel auide pairs so that only vertical movements are possible. The weight force is transmitted to the lever (6) via a gimbal-mounted load receptor (2), the pan carrier (3), a coupling element (4), and a flexible bearing (usually a cross flex bearing) (5). A fixed counterweight (7) is located on the longer lever arm to mechanically compensate most of the effective weight force; some comparator balances also have several counterweights with different nominal values ("dial weights") which can be activated from the outside. The remaining weight force is electromagnetically compensated by a coil (9) that resides in the air gap of a permanent magnet system (8). As described above, the inductor current is controlled by an electro-optical position sensor (10-12) in relation to the load, so that the slit aperture (11) at the end of the longer lever arm remains in a defined rest position.

Mass determination



#### Figure 2.9:

Schematic illustration of a comparator balance with fixed counterweight and electromagnetic force comparison of part of the weight force.

- 1 weighing pan,
- 2 gimbal-mounted load receptor,
- 3 pan carrier,
- 4 coupling element,
- 5 flexible bearing,
- 6 lever,
- 7 counterweight (fixed),
- 8 permanent magnet system,
- 9 compensation coil,
- 10 photodiode,
- 11 slit aperture,
- 12 light source (LED),
- 13 pan lock,
- 14 tare disk e.g. *Max*/2 (can be installed in addition).

Modern electromagnetic force compensation load cells have a monolithic design (Figure 2.10). This manufacturing technique makes it possible to reduce the number of functional components. The number of fine mechanical assembly and adjustment steps is reduced and the reliability of the system is improved.

Commercially available comparator balances (also see Appendix A.3) can meet practically all metrology requirements. National metrology institutes and calibration laboratories can meet the requirements of the highest accuracy class E1 according to the international OIML recommendation R 111. Using the example of PTB, Figure 2.11 illustrates the uncertainty levels achieved for absolute mass determinations compared to error limits of the OML accuracy classes [17].



α

b



Figure 2.10:

Monolithic electromagnetic force compensation load cells produced with different manufacturing technologies.

a Monolithic load cell with mechanical elements produced by electric discharge machining (for greater visibility the model has been cut in the left one-third and the outer right part, Mettler Toledo);

**b** Monolithic load cell manufactured as a milled block system (Sartorius).



Expanded measurement uncertainties (k = 2) of PTB for mass determinations according to the BIPM CMC tables<sup>3</sup> (1) and the error limits for classes E<sub>1</sub> (2a) and F<sub>1</sub> (2b) according to OIML R 111 [17]

<sup>3</sup> Calibration and measurement capabilities (CMC) [29]

# 2.5 Influencing quantities

During weighing in air, the main correction is the air buoyancy correction. However, other influencing quantities and disturbance factors must be considered in order to achieve a relative uncertainty  $<1\times10^{-5}$  (Table 2.10). These quantities, which are usually linked to the environment, can affect the weighing instrument, the mass standards and the weighed object, respectively. The main influencing quantities and disturbance factors will be examined below, and measures to prevent or correct their undesirable effects will be discussed.

Influencing quantity/ disturbance factor	Effect on	Effect
Air density	Instrument/weights	Air buoyancy on instrument components and weights (systematic errors)
Temperature variation over time	Instrument	Drift of instrument indication, sensitivity
Temperature gradients and differences	Instrument/weights	Systematic errors, convection, and an increase in standard deviation
Temperature ≠ 20°C	Weights	Volume change
Air pressure variation	Instrument	Drift and/or variation of the instrument indication
Humidity change	Instrument/weights	Drift of instrument indication, change of adsorption layers
Surface roughness and contamination	Weights	Adsorption layers, long-term stability
Position of centre of gravity, gravitational acceleration	Weights/force- compensated instrument	Systematic errors, change of instrument sensitivity/adjustment
Electrostatic charges	Instrument/dielectric weighing goods	Systematic errors, increased standard deviation or drift of the measurement values
Magnetic fields	Instrument/weights with too high susceptibility or magnetisation	Systematic errors, location-dependent measurement values
Vibration, tilting	Instrument	Increase in standard deviation, systematic errors
Eccentric load on the weighing pan	Instrument	Systematic errors

#### Table 2.10:

Influencing quantities and disturbance factors in high-accuracy mass determination

### 2.5.1 Air buoyancy correction

A mass comparison using substitution weighing in air for a standard with mass  $m_{\rm R}$  (density  $\rho_{\rm R}$ ) and the specimen with mass  $m_{\rm T}$  (density  $\rho_{\rm T}$ ) results in the weighing values

$$m_{\rm WR} = m_{\rm R} \, \frac{1 - \rho_{\rm a}/\rho_{\rm R}}{1 - \rho_{\rm o}/\rho_{\rm c}} \frac{1 - \rho_{\rm o}/\rho_{\rm J}}{1 - \rho_{\rm aJ}/\rho_{\rm J}} \,, \tag{2.10}$$

$$m_{\rm WT} = m_{\rm T} \frac{1 - \rho_{\rm a}/\rho_{\rm T}}{1 - \rho_{\rm 0}/\rho_{\rm c}} \frac{1 - \rho_{\rm 0}/\rho_{\rm J}}{1 - \rho_{\rm aJ}/\rho_{\rm J}} \quad .$$
(2.11)

Here  $\rho_{aJ}$  is the air density during adjustment of the weighing instrument and  $\rho_J$  is the density of the weight used to adjust the instrument. Equations (2.10) and (2.11) result in the weighing equation for mass determination in air

$$m_{\rm T} (1 - \rho_{\rm a}/\rho_{\rm T}) = m_{\rm R} (1 - \rho_{\rm a}/\rho_{\rm R}) + \Delta m'_{\rm W} , \qquad (2.12)$$

with

$$\Delta m'_{\rm W} = \Delta m_{\rm W} \left( 1 - \rho_0 / \rho_{\rm c} \right) \frac{1 - \rho_{\rm aJ} / \rho_{\rm J}}{1 - \rho_0 / \rho_{\rm J}} .$$
(2.13)

 $\Delta m'_{\rm W}$  refers to the corrected and  $\Delta m_{\rm W}$  to the uncorrected weighing difference, and  $\rho_{\rm a}$  is the air density during the weighing. In the prevalent situation where the air density during adjustment is approximately  $\rho_{\rm aJ} = \rho_0$ , the following applies:

$$\Delta m'_{\rm W} = \Delta m_{\rm W} \left( 1 - \rho_0 / \rho_c \right) = 0.99985 \, \Delta m_{\rm W} \quad . \tag{2.14}$$

This means that

$$\Delta m'_{\rm W} = \Delta m_{\rm W} \tag{2.15}$$

can be substituted as an approximation for sufficiently small weighing differences.

If the densities  $\rho_{\rm T}$  and  $\rho_{\rm R}$  are replaced with the volumes  $V_{\rm T}$  and  $V_{\rm R_r}$  then the weighing equation that corresponds to equation (2.12) is

$$m_{\rm T} = m_{\rm R} + \rho_{\rm a} \left( V_{\rm T} - V_{\rm R} \right) + \Delta m'_{\rm W} .$$
 (2.16)

The term  $\rho_a(V_T-V_R)$  denotes the air buoyancy correction. Depending on the accuracy requirements, calculating the air buoyancy correction requires a more or less accurate determination of air density (see Appendix A.1) and possibly of the volume of the standards (see Section 3). If auxiliary masses are used with the standard ( $m_{ZR}$ ,  $V_{ZR}$ ) and/or the specimen ( $m_{ZT}$ ,  $V_{ZT}$ ), the complete weighing equation is as follows:

$$m_{\rm T} = m_{\rm R} + m_{\rm ZR} - m_{\rm ZT} + \rho_{\rm a} \left( V_{\rm T} - V_{\rm R} + V_{\rm ZT} - V_{\rm ZR} \right) + \Delta m'_{\rm W} .$$
(2.17)

If the conventional mass  $m_{\rm cT}$  of the specimen is being determined instead of the mass  $m_{\rm T}$  (on an electromagnetically compensated analytical or comparator balance), the weighing equation

$$m_{\rm cT} = m_{\rm cR} (1+C) + \Delta m_{\rm W}''$$
 (2.18)

can be derived with the instrument indication

$$m_{\rm WR} = m_{\rm cR} \, \frac{1 - \rho_{\rm a}/\rho_{\rm R}}{1 - \rho_{\rm 0}/\rho} \, \frac{1 - \rho_{\rm 0}/\rho_{\rm J}}{1 - \rho_{\rm al}/\rho_{\rm J}} \tag{2.19}$$

and

$$m_{\rm WT} = m_{\rm cT} \, \frac{1 - \rho_{\rm a}/\rho_{\rm T}}{1 - \rho_{\rm o}/\rho} \frac{1 - \rho_{\rm o}/\rho_{\rm J}}{1 - \rho_{\rm aJ}/\rho_{\rm J}}.$$
(2.20)

Here

$$C = \frac{(\rho_{\rm R} - \rho_{\rm T})(\rho_{\rm a} - \rho_{\rm 0})}{(\rho_{\rm R} - \rho_{\rm 0})(\rho_{\rm T} - \rho_{\rm a})}$$
(2.21)

is the air buoyancy correction, and

$$\Delta m''_{\rm W} = \Delta m_{\rm W} \frac{1 - \rho_{\rm aJ}/\rho_{\rm J}}{1 - \rho_{\rm 0}/\rho_{\rm J}} \frac{1 - \rho_{\rm 0}/\rho_{\rm T}}{1 - \rho_{\rm a}/\rho_{\rm T}}$$
(2.22)

is the corrected weighing difference. In many cases, the air buoyancy correction *C* is so small compared to the uncertainty that needs to be achieved for the specimen that it can be disregarded. Under normal environmental conditions, the relative correction of the weighing difference  $\Delta m_{\rm W}$  according to equation (2.22) is usually also very small (generally <1.5×10<sup>-5</sup>), so that it is not required if the condition

$$\Delta m_{\rm W} < 30000 \, d$$
 (2.23)

is met for the weighing difference. For  $\rho_{a}$  =  $\rho_{a\text{J}}$  =  $\rho_{0}$  , the relation

$$m_{\rm cT} = m_{\rm cR} + \Delta m_{\rm W} \tag{2.24}$$

applies exactly. This once again illustrates the advantage of the conventional mass. If the instrument adjustment and the determination of the conventional mass are performed at an air density of  $\rho_0 = 1.2$  kg m<sup>-3</sup>, air buoyancy correction – in contrast to mass determination – is not required!

## 2.5.2 Thermal influences

If the temperature in the measurement room is not constant over time, the temperature changes cause dimensional changes to the mechanical weighing system as well as changes to the electrical and magnetic characteristics of an electromagnetic force compensation system. Therefore, the result is not only a drift or variation of the instrument indication but also a change in the sensitivity of the instrument. Temperature changes or variations over time can be reduced, e.g. by suitable air conditioning of the measurement room or by using a basement room located below around. Certain limit values for temperature changes in the laboratory can be derived, depending on the required measurement uncertainty (see Table 2.11). Temperature differences between the standard, the weight to be calibrated, and the instruments are especially critical, even for mass determinations with relative uncertainties of approximately  $1 \times 10^{-5}$ . If there is no thermal equilibrium, temperature gradients cause convection effects in the weighing chamber, which cannot only lead to an increased standard deviation but also especially to systematic errors in the weighing difference [30]. A sufficiently long waiting period (usually several hours) to allow the temperature of the specimen and the standard to adapt to the temperature in the weighing chamber can solve this problem (large weights can be left in the vicinity of the instrument under a common bell jar). In order to minimise temperature gradients in the weighing chamber, the exposure of weighing instruments and weights to direct thermal radiation (e.g. from solar radiation or radiators) should be avoided.

0	Specimen (calibration standard) T:		
0	$U_{\rm T}/m$ (100 g $\le m \le$ 50 kg)	1.5×10 <sup>-7</sup>	5×10-7
Ē	Class	Eı	E <sub>2</sub>
	R <sub>a</sub> / μm	0.1	0.2
	R <sub>z</sub> / μm	0.5	1.0
Φ	χ	0.02	0.07
	$\mu_0 M_z / \mu T$	2.5	8.0
S S	Reference standard R:		
S	$U_{\rm T}/m$ (100 g $\le m \le$ 50 kg)	≤5×10 <sup>-8</sup>	$\leq 1.5 \times 10^{-7}$
$\leq$	Class	"E <sub>0</sub> "4	E1
	R <sub>a</sub> / μm	< 0.1	0.1
	R <sub>z</sub> / μm	< 0.5	0.5
	χ	< 0.02	0.02
	$\mu_0 M_z / \mu T$	< 2.5	2.5
	Volume V:		
	$U_{\rm VT}/V_{\rm T} \ (m \ge 100 \ {\rm g})$	3×10-4	1×10-3
	Measurement room:		

Volume V:					
$U_{\rm VT}/V_{\rm T}$ ( $m \ge 100$ g)	3×10 <sup>-4</sup>	1×10 <sup>-3</sup>	3×10 <sup>-3</sup>	1×10 <sup>-2</sup>	3×10 <sup>-2</sup>
Measurement room:					
$U_{\rm p}$ / mbar	0.3	1.0	3.0	10	30
U <sub>t</sub> <sup>'</sup> / ℃	0.1	0.2	0.5	0.5	1.0
$U_{ m hr}$ / %	2	2	5	10	10
$dt_{1h} / °C$	± 0.3	± 0.7	± 1.5	± 2.0	± 3.0
$dt_{12h}$ / °C	± 0.5	± 1.0	± 2.0	± 3.5	± 5.0
$dh_{4h}$ / %	± 5	± 10	± 15	_	-

#### Table 2.11:

Requirements for measurement rooms and weights of a calibration laboratory for mass determination [17, 31].

#### m mass

 $R_{av}$   $R_z$  surface roughness (mean roughness value or average roughness depth) χ magnetic susceptibility  $\mu_0 M_z$  magnetic polarisation U expanded uncertainty  $(U = 2u_c, \text{ see Section 4}),$ p air pressure t temperature h<sub>r</sub> relative humidity  $dt_{1h}$ ,  $dt_{12h}$  maximum temperature change within one or twelve hours  $dh_{4h}$  maximum humidity change within four hours

The temperature dependence of the volume usually cannot be disregarded when the mass standard and the weight to be calibrated have different densities and/or different volume expansion coefficients. For example, this applies to mass comparisons between stainless steel standards and Pt-Ir prototypes. If the volume V of a weight determined using hydrostatic weighing is specified for the reference temperature  $t_{0}$ , the following applies to a temperature t that deviates from  $t_0$ :

 $1.5 \times 10^{-6}$ 

Fι

0.4

2.0

02

25

 $E_2$ 

0.2

1.0

0.07

8.0

 $\leq 5 \times 10^{-7}$ 

5×10-6

 $F_2$ 

1.0

5.0

0.8

80

F<sub>1</sub>

0.4

2.0

0.2

25

≤1.5×10<sup>-6</sup>

 $1.5 \times 10^{-5}$ 

M<sub>1</sub>

\_

250

 $F_2$ 

1.0

5.0

0.8

80

 $\leq 5 \times 10^{-6}$ 

$$V(t) = V(t_0) [1 + \alpha_v (t - t_0)], \qquad (2.25)$$

#### where:

 $\alpha_{\rm V}$  is the volume expansion coefficient

(for the temperature range  $t_0$  to t) and

is the reference temperature for volume determination to (normally,  $t_0 = 20$  °C).

The volume expansion coefficients of some materials commonly used in mass determination are listed in Table 2.12.

<sup>&</sup>lt;sup>4</sup> "E<sub>0</sub>" is not an official designation for an accuracy class of OIML R 111 [17]. Standards used to calibrate class E1 weights must have comparable or better metrological characteristics than the weight being calibrated. Thus, the designation "Eo" is a continuation of the system for error limits and uncertainties below the accuracy classes of OIML R 111.

Material	α <sub>V</sub> / K <sup>-1</sup>	Literature
Stainless steel (CrNi 18 8)	4.8×10 <sup>-5</sup>	[32]
Platinum-iridium (90 10)	2.6×10 <sup>-5</sup>	[33]
Brass (CuZn 62 38)	5.4×10 <sup>-5</sup>	[32]
Nickel silver (CuNiZn 62 15 22)	5.4×10 <sup>-5</sup>	[32]
Silicon	7.7×10 <sup>-6</sup>	[34]
Zerodur®	≤1.5×10 <sup>-7</sup>	[35]

Table 2.12:

Volume expansion coefficients  $\alpha_{\rm V}$ (t = 20 °C) of some materials commonly used in mass determination

# Example:

Temperatures:  $t_0 = 20 \text{ °C}, t = 21.5 \text{ °C}$ Mass standard (platinum-iridium):

$$\begin{split} m_{\rm R} &= 1 \text{ kg} \\ V_{\rm R}(t_0) &= 46.4300 \text{ cm}^3 \\ V_{\rm R}(t) &= 46.4318 \text{ cm}^3 \\ \Delta V_{\rm R} &= V_{\rm R}(t) - V_{\rm R}(t_0) = 0.0018 \text{ cm}^3 \end{split}$$

Specimen (stainless steel):

$$m_{\rm T} = 1 \text{ kg}$$
  

$$V_{\rm T}(t_0) = 124.3800 \text{ cm}^3$$
  

$$V_{\rm T}(t) = 124.3890 \text{ cm}^3$$
  

$$\Delta V_{\rm T} = V_{\rm T}(t) - V_{\rm T}(t_0) = 0.0090 \text{ cm}^3$$

Total change of volume difference:

 $\Delta V_{\rm T} - \Delta V_{\rm R} = 0.0072~{\rm cm^3}$  Change in air buoyancy:  $(\Delta V_{\rm T} - \Delta V_{\rm R}) \cdot \rho_{\rm a} = 8.6~{\rm \mu g}$ 

In this example, the temperature dependence of the volume plays a role that cannot be disregarded since so-called prototype balances, i.e. 1 kg mass comparators with a standard deviation of  $s \le 1 \mu g$ , are used for mass comparisons with Pt-Ir prototypes (see Appendix A.3). On the other hand, air buoyancy changes due to volume changes can always be disregarded for mass comparisons between weights made of the same material, e.g. stainless steel or brass, since both the expansion coefficients and the densities are very similar.

# 2.5.3 Air pressure, relative humidity, adsorption

Air pressure changes during a mass comparison can affect the weighing value, since the latter can only be pressure compensated for a certain density of the weights being calibrated. Many analytical and comparator balances that display the conventional mass are pressure compensated for the reference density  $\rho_c = 8000 \text{ kg m}^{-3}$ . If the material density deviates from this value, e.g. in the case of platinumiridium ( $\rho = 21500 \text{ kg m}^{-3}$ ), air pressure deviations can cause an increase in the standard deviation for the mass comparison. Therefore, for highest-precision weighing with Pt-Ir prototypes, the prototype balance is frequently installed in a pressure-tight enclosure (see Appendix A.3). High-accuracy mass determinations with analytical and comparator balances are usually carried out in a restricted humidity range between 40% and 60%. A relative humidity of less than 40 % may cause electrostatic charges and significant systematic errors. On the other hand, very high relative humidity ( $h_r > 60$  %) increases the risk of corrosion. Furthermore, humidity changes always affect the mass standard or the specimen due to sorption effects on the surfaces. While they are exposed to air, the surfaces of solids are always covered with adsorption layers consisting mainly of chemically and physically sorbed water [36, 37]. The chemisorbed layers have considerably larger binding energies than the physisorbed layers, so that the former are almost independent while the latter are dependent on relative humidity. For example, the surface coverage of polished stainless steel standards at a relative humidity of  $h_r = 0 \%$ is between  $\mu_{h=0} \leq 0.1 \,\mu \text{g cm}^{-2}$  (cleaned surfaces) and a maximum of approximately  $\mu_{h=0} = 0.8 \ \mu g \ cm^{-2}$  (uncleaned surfaces or surfaces which have not been cleaned for a long time) [38, 39]. Therefore, the mass of the chemisorbed layer can be estimated at approximately 15 µg to 120 µg for a 1 kg steel standard (surface  $A = 150 \text{ cm}^2$ ). The physisorbed adsorption layer on stainless steel standards changes depending on the surface condition, i.e. surface cleanliness and roughness, with the relative humidity according to the Brunauer-Emmett-Teller (BET) equation [38, 40]

$$\mu = \frac{m_{\rm A}}{A} = \mu_{h=0} + \frac{\mu_{\rm m} c_{\rm B} h}{(1-h) \left[1 + (c_{\rm B} - 1) \cdot h\right]} .$$
(2.26)

The parameters of the so-called BET isotherms in equation (2.26) are:  $m_A$  mass of the adsorption layer, A surface of the weight,  $h = h_r/100$ ,  $h_r$  relative humidity in %,  $\mu_{h=0}$  surface coverage for h = 0,  $\mu_m$  change to surface coverage due to a monomolecular layer,  $c_B$  BET constant. For carefully polished stainless steel standards (average roughness depth  $R_z \le 0.1 \mu$ m), the following parameters were determined experimentally [38, 39]:

- Cleaned surfaces ( $\mu_h \le 0.1 \ \mu g \ cm^{-2}$ ):  $\mu_m = 0.0084 \ \mu a \ cm^{-2}$ 

$$\mu_{\rm m} = 0.0084 \ \mu g \ crc_{\rm p} = 8.9$$

- Uncleaned surfaces ( $\mu_{h=0} \ge 0.7 \ \mu g \ cm^{-2}$ ):  $\mu_m = 0.018 \ \mu g \ cm^{-2}$  $c_B = 11.2$ 



Figure 2.12:

Experimentally determined and interpolated BET adsorption isotherms for polished stainless steel surfaces (average roughness depth  $R_z \le 0.1 \,\mu$ m) [38, 39]:

a) For cleaned surfaces with a surface coverage of  $\mu_{h=0} \le 0.1 \ \mu \text{g cm}^2$ . b) For uncleaned surfaces with a surface coverage of  $\mu_{h=0} \ge 0.7 \ \mu \text{g cm}^2$ .

 $\mu \qquad \text{surface coverage} = \text{mass of} \\ \text{the adsorption layer per unit of} \\ \text{surface area,} \\ \end{array}$ 

 $h_{\rm r}$  relative humidity in %,

- $h = h_{\rm r}/100,$
- $\mu_{h=0}$  surface coverage for h = 0.

Figure 2.12 shows the curve shapes of the two BET isotherms. For example, the mass of a cleaned 1 kg stainless steel weight ( $A = 150 \text{ cm}^2$ ) increases by  $\Delta m_A = 2.9 \text{ µg}$  and the mass of an uncleaned 1 kg weight increases by  $\Delta m_A = 6.2 \text{ µg}$  when the relative humidity increases from 20 % to 70 %. This illustrates that such changes in mass caused by adsorption layers also need to be considered only in case of the highest-accuracy mass determination (weighing with prototype balances).

In addition to the cleanliness of the surface, roughness can also influence adsorption behaviour and, therefore, the long-term stability of weights. International upper limit values have been established for the average peak-to-valley height  $R_z$  and the average roughness value  $R_a$  (arithmetic mean of the deviations from the centre line of the roughness profile) for weights [17], which also apply to mass standards used in accredited calibration laboratories [31] (see Table 2.11).

#### 2.5.4 Centre of gravity position, gravitational acceleration

Differences in the vertical positions of the centres of gravity  $z_s$  between the standard and the specimen have an effect on very accurate mass determination and on hydrostatic weighing due to the vertical gradient of gravitational acceleration  $\partial g/\partial z$ . In measurement locations close to the surface of the earth, the relative gradient is normally approximately  $\partial g/(g \cdot \partial z) = -2.5 \dots -3.5 \times 10^{-7} \text{ m}^{-1}$ , depending on the local underground conditions and topography [41]. To correct precise mass determination, the approximation

$$\frac{\partial g}{g \, \partial z} = -3 \times 10^{-7} \, \mathrm{m}^{-1} \tag{2.27}$$

is generally sufficient. For example, a mass comparison of two 1 kg weights with a difference in the vertical positions of the centres of gravity of  $\Delta z_s = 20$  mm results in a correction of +6 µg for the weight with the higher centre of gravity. For hydrostatic weighing of a 10 kg weight, the correction amounts to -1.5 mg if the centre of gravity of the weight that is immersed in water is 50 cm below the centre of gravity of the reference standard in air. Changes in gravitational acceleration g that occur over time can also affect weighing results; for example, the daily and monthly relative fluctuations are up to  $\Delta g/g = \pm 1.5 \times 10^{-7}$ , while the maximum relative fluctuations per minute are  $\pm 0.8 \times 10^{-9}$  [41]. However, this only affects proportional weighing with g-dependent (force compensated) analytical balances (Sections 2.3 and 2.4). Since gravitational acceleration also depends on the geographic latitude and altitude, weighing instruments of special accuracy and weighing instruments of high accuracy are adjusted at their place of use (Appendix A.7 and A.8). On the other hand, changes in gravitational acceleration do not play a role in differential weighing.

#### 2.5.5 Electrostatic fields

Electrostatic forces between the weighed object and the weighing instruments and/or the environment can cause significant variations in the instrument indication as well as unknown systematic weighing errors. Whereas the electrical potential of metallic components of an instrument, especially the housing and the suspension, can be brought to the same potential (ground potential), the weighing of nonconductive (dielectric) objects frequently causes problems. The following procedures can be used to discharge or reduce the effect of electric charges:

- Increasing the relative humidity at the measurement location or installing the instruments in a practically closed chamber in which the relative humidity is artificially increased;
- Shielding electrostatic forces by inserting the non-conductive object in a metallic container with a known mass (Faraday cage); since only the conductive surface matters, a thin metallic foil is sufficient;
- Discharging the dielectric body using a suitable radioactive preparation (e.g. α-emitter) or ionised air, generated by a high-voltage discharge.

# 2.5.6 Magnetic fields

Magnetic fields outside and inside the weighing instruments (e.g. for instruments with electromagnetic force compensation) can also cause systematic weighing errors if the magnetic susceptibility of the weighed object is too high or if it is itself magnetised [17, 42].

Therefore, international limit values have been specified for the permanent magnetic polarisation  $\mu_0 M_z$  (magnetisation  $M_z$ ) and the magnetic susceptibility  $\chi$  of weights (see Table 2.4 and Table 2.5); exceeding these values is not recommended [17]. If the magnetic polarisation and susceptibility of a weight are smaller than the specified maximum values, it is assumed that the contribution of the measurement uncertainty resulting from the magnetic characteristics of the weight is small enough to be disregarded when calculating the combined uncertainty of the mass determination. The maximum values for polarisation and susceptibility have, in fact, been selected so that during mass determination of a weight, there is no deviation larger than  $1/_{10}$  of the maximum permissible error [17, 24].

The magnetic properties of a mass standard should be determined prior to calibration in order to ensure that their influence on the mass determination can be disregarded. A corresponding investigation for weights made of aluminium is not required since they are not magnetic and their susceptibility is clearly below 0.01. Methods to verify the magnetic properties of weights are described in the OIML recommendation R 111 [17].

# 2.5.7 Mechanical influences

Mechanical influences on weighing instruments can include vibration or tilting (inclination). The influence of vibration depends on the type of instrument and the direction of the vibration vector. For example, a mechanical balance with equal arms is not very sensitive to vertical movements, moderately sensitive to horizontal movements, and very sensitive to tilting.

On the other hand, all instruments that use other forces to compensate for all or part of the gravitational force – that is, all types of load cells as well as electromagnetically compensated instruments – are susceptible to vertical movements. The frequency range that affects weighing is between the reciprocal value of the time interval for one weighing cycle (approx.  $10^{-3}$  Hz) and the reciprocal value of the shortest averaging period for an indicated value (approx. 10 Hz). To reduce weighing errors due to mechanical influences, installing the instruments on a stable, solid weighing table that stands directly on the floor or is attached to a stable wall is recommended.

Systematic errors due to eccentric loads on the weighing pan occur particularly with top-loading instruments; therefore, special centring facilities are offered for comparator balances (e.g. the "LevelMatic<sup>®</sup>" or a gimbal-mounted load receptor) which largely eliminate errors caused by eccentric loading.