SIM
SISTEMA INTERAMERICANO DE METROLOGÍA

SIM METROLOGY SCHOOL

MASS METROLOGY

SPEAKER

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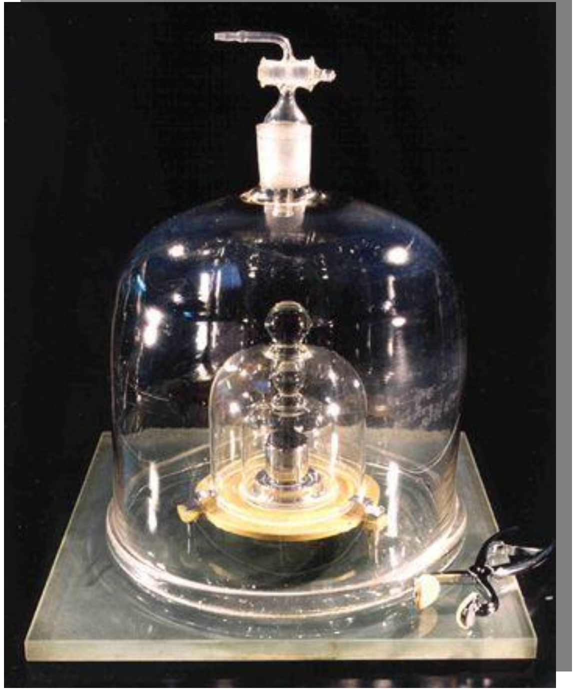
History of the kilogram definition

Changes in SI

Kilogram Definition:



3r CGPM, 1901 :

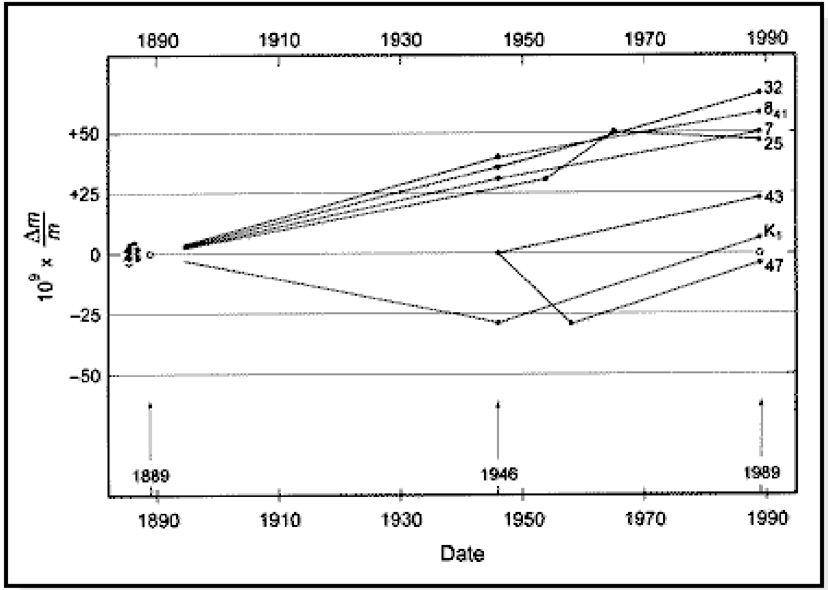
The kilogram is the unit of mass; it is equal to the mass of the international prototype of the kilogram

It is kept at the BIPM: IPK manufactured in 1879 and ratified in 1889; alloy of 90% Pt - 10% Ir .

It was the last SI base unit defined by a material artefact.

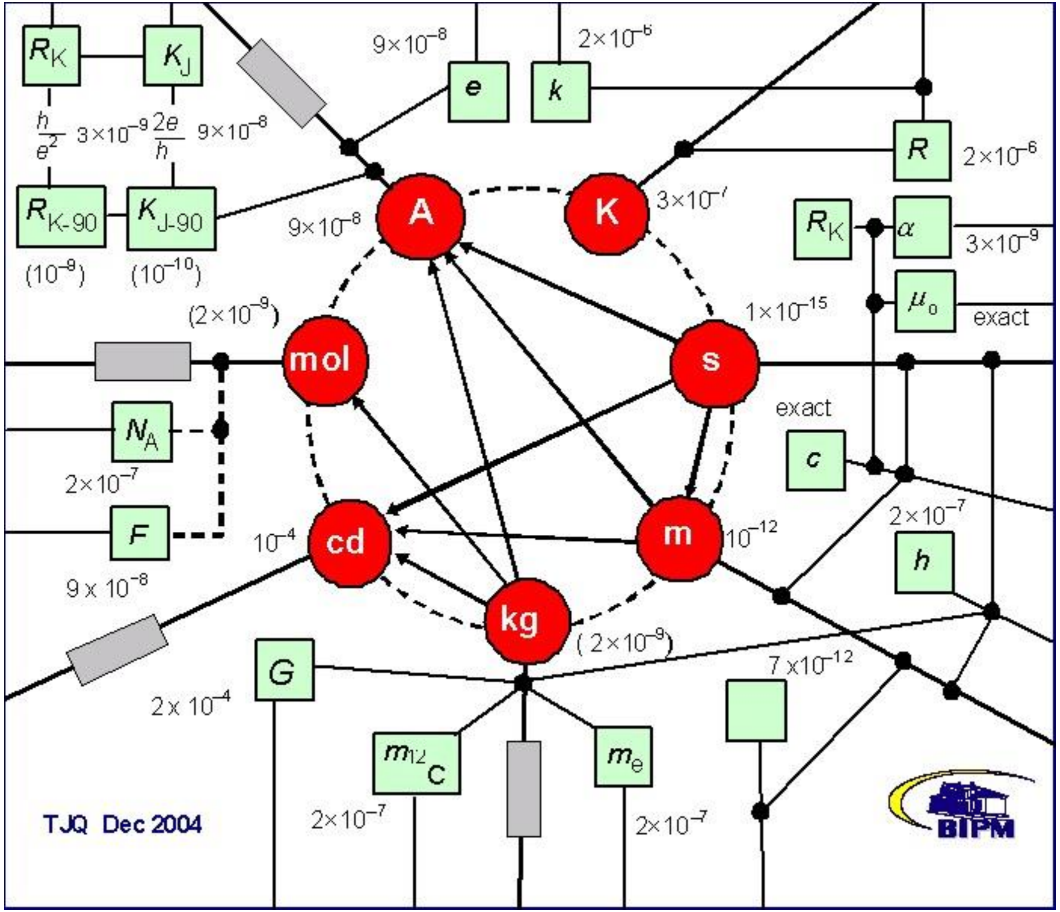
Drift of prototypes

- **Drift of 50 x 10^{-9} (50 mg) in the mass of prototypes in about 100 years (0.5 µg / year).**



NEW SI

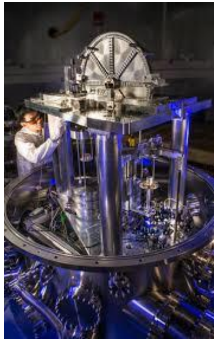

SI International System of Units
base units
defining constants
cd
mol
K
A
kg
m
s


R_K
K_J
h/e²
3×10⁻⁹
2e/h
9×10⁻⁸
R_K-90
K_J-90
(10⁻⁹)
(10⁻¹⁰)
e
k
2×10⁻⁶
R
A
K
3×10⁻⁷
R_K
α
3×10⁻⁹
μ₀
exact
(2×10⁻⁹)
mol
s
1×10⁻¹⁵
N_A
2×10⁻⁷
exact
c
F
9 x 10⁻⁸
10⁻⁴
cd
m
10⁻¹²
2×10⁻⁷
h
kg
(2×10⁻⁹)
G
7 x10⁻¹²
2 x 10⁻⁴
m₁₂C
m_e
2×10⁻⁷
2×10⁻⁷
TJQ Dec 2004
BIPM

kilogram

The kilogram, symbol kg, is the SI unit of mass.

It is defined by taking the fixed numerical value of the Planck constant h to be 6.626 070 15 × 10–34 when expressed in the unit J s, which is equal to kg m2 s–1 , where the meter and the second are defined in terms of c and ΔvCs



kilogram dissemination in SIM

- Once *h* the value of the Planck constant is fixed, the International Prototype of the kilogram no longer define the unit of mass
- Other techniques – specifically the XRCD Si spheres and the Kibble (Watt) balances – are the way to realize the unit of mass from its definition
- We can have realizations at any required mass value, not just at 1 kg
- SIM – through NIST and NRC – has two operational Kibble balances that have sufficiently low uncertainty at the level of 1 kg masses

kilogram dissemination in SIM

- On those NMIs that do not have a primary realization traceability *still rests on the calibration* of the 1 kg national standard that is at the top of each national mass scale.
- Existing dissemination schemes from a primary national standard – including PtIr National Prototypes or stainless steel OIML Class E1 weights – can still be used without modification
- NRC and NIST have discussed with SIM the idea of *disseminating primary calibrations* from Kibble balances to all of the SIM member states

CONVENTIONAL MASS

Forces involved in weighing

Forces involved in weighing

- Gravity.

$$F= M\,g$$

M- instrument mass.

g- gravitiy acceleration.

- Air buoyancy.

$$F = - m_a\, g = - V\, \rho_a\, g$$

Archimedes´ Principle.

V – volume of weight.

$$V = m/ \rho m$$

m- weight density.

ρa- air density while weighing.

The air density is calculated according to the revised formula of CIPM-2007.

IOP PUBLISHING METROLOGIA

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Revised formula for the density of moist air (CIPM-2007)

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Abstract

Measurements of air density determined gravimetrically and by using the CIPM-81/91 formula, an equation of state, have a relative deviation of 6.4×10^{-5}.

This difference is consistent with a new determination of the mole fraction of argon x_{Ar} carried out in 2002 by the Korea Research Institute of Standards and Science (KRISS) and with recently published results from the LNE. The CIPM equation is based on the molar mass of dry

Magnetic forces involved in weighing

$$F_z = \mu_0 \iiint_V (M + \chi H) \frac{\partial H}{\partial z} \mathrm{d}V$$

- Vertical component of the magnetic interaction between the weight and balance
- H y M are vectors ; z is the vertical cartesian coordinate.

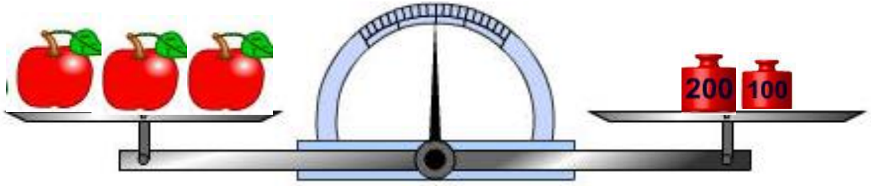
Conventional Mass Definition

The conventional mass of a body is equal to the mass of a standard weight that balances this body under "conventional" conditions, namely, ambient temperature = 20 °C, air density and standard weight density 8000 kg/m³.

Recomendación OIML R 111, p6.

$$mc = \left(\frac{1 - \frac{\rho_0}{\rho}}{1 - \frac{\rho_o}{\rho_c}} \right) m$$

- m_c: conventional mass.
- m: true mass.
- ρ_o: specific reference air mass 1,2 kg/m³.
- ρ_c: specific reference mass 8000 kg/m³.



WEIGHTS METROLOGICAL REQUIREMENTS

OIML R 111-2004

Weights Classification

According to OIML, weights could be classified as:

- Class E1: To ensure traceability between national mass standards (Mass value derivate from 1 kg standard). Calibration certicate must be available.

- Class E2: weights to calibrate F1 class weights and Type 1 balances, special accuracy.

Weights Classification

- Class F1: Weights to calibrate F2 class weights and class II balances.
- Class F2: Weights to calibrate M1 y M2 class weights and class II balances.
- Class M1: Weights to calibrate M2 class weights and class III balances.
- Class M2: Weights to calibrate M3 class weights and class III balances.
- Class M3: Weights to calibrate class III and IV balances.
- Class M1-2 Y M2-3: Weights from 50 to 5 000 kg to calibrate class III balances.

Maximum permissible errors Uncertainty

- MAXIMUM PERMISSIBLE ERRORS: Values are given in Table 1. The values, indicates the máximum diference between conventional mass and nominal mass of the weights.

- CALIBRATION UNCERTAINTY: For each weight, the expanded uncertainty, U, for k = 2, of the conventional mass, shall be less than or equal to one-third of the maximum permissible error in Table 1.

- $U \leq 1/3\ \delta m$

Table 1 Maximum permissible errors for weights (± δm in mg)

Nominal value*	Class E_1	Class E_2	Class F_1	Class F_2	Class M_1	Class M_{1-2}	Class M_2	Class M_{2-3}	Class M_3
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				

Shape - Construction

SHAPE:

- Must ensure the stability of the weight within the range of uncertainty declared for class.

CONSTRUCTION:

- **Class E1 and E2:** Shall be solid and shall have no cavity open to atmosphere. To weights E2 greater than 50 kg, the cavtiy shall be sealable and shall not exceed 1/1000 of the total volumen of the weight.
- **Class F:** May consiste in one or more pieces manufactured from the same material.
- **Class M:** May have cavity or not, weights greater than 100 g always present the cavity.

Material

- E class : Similar or better than austenitic stainless Steel.
- Clase F: Similar or better than brass.
- Clase M:
    - Weights less than 1 g: material with corrosión and oxidation resistance and hardness.
    - Weights M1 less than 5 kg, and M2 y M3 less tan 100 g are made by similar or better than brass.
    - The others are made by grey cast iron

Magnetic properties

- The PERMANENT MAGNETIZATION may not exceed máximum values permitted for each weight class.
- The magnetic susceptibility of a weight may not exceed maximum values permitted for their own class.

Density- Surface conditions

DENSITY:

The density of the material used for weights is specified shall be such that a deviation of 10 % from the specified air density (1.2 kg m–3) does not produce an error exceeding one-quarter of the absolute value of the maximum permissible error.

SURFACE CONDITIONS:

- Shall be such that any alteration of the mass ot the weights is negligible with respecto to the máximum permissible error.
- Considerations:
  - Base and corner: Smooth
  - Edges: roundes.
  - Surface porosity, roughness.

Adjustment - Marking

AJUSTMENT:

- Class E: by abrasion.
- Class F: by abrasión or refilling the adjusting cavities with the same material used to fabrished the weight.
- Class M:
  - From 1 mg to 1 g : by abrasion or cutting.
  - More than 1 g: filling the adjusting cavity with lead

MARKING:

Allowed for all classes except class E (*).

CALIBRATION OF WEIGHTS

OIML R 111-2004

Calibration Sequence

- Preliminary assessment tests should be performed in the following order (if applicable):

  a) Document review and visual inspection according to a checklist;
  b) Cleaning of the weight;
  c) Roughness surface verification;
  d) Magnetic properties measurements
  e) Volume-Density measurement;
  f) After density measurement, weight Cleansing must be repeated if liquid used for cleaning is different from water
  g) Thermal stabilization
  h) Calibration.

b-Cleaning

It is important to clean weights (only if necessary) before any measurements are made because the cleaning process may change the mass of the weight.

Cleaning should not remove any significant amounts of weight material.

Weights should be handled and stored in such a way that they stay clean.

Before calibration, dust and any foreign particles shall be removed. Care must be taken not to change the surface properties of the weight (i.e. by scratching the weight).

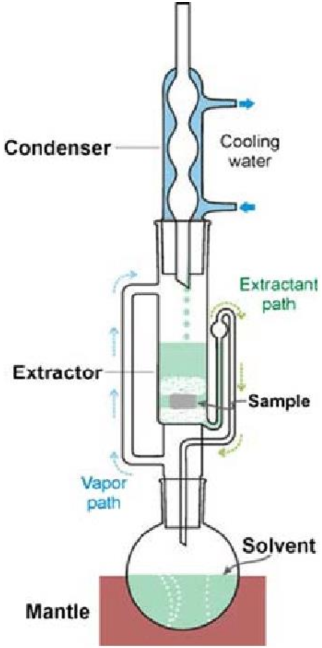
b-Cleaning

- Weights should be cleaned before use, with a soft, and clean brush.
- Do not apply other cleaning method, unless absolutely necessary. All weights have some type of gases on its surface to some degree remains constant in equilibrium with the environment.
- Deep cleaning changes the surface chemistry disturbing the balance achieved. In this case, weights with internal cavities should normally not be immersed in the solvent to avoid the possibility that the fluid will penetrate the opening
- After cleaning, weight mass seems to be lower and then in a lapse of time, the surface reached the equilibrium again.

b-Cleaning

- Be sure, that a deeper cleaning is needed.
- Cleaning not only invalidate the current calibration certificate, also affects the continuity of history calibration of the weight which will cause a negative impact on the budget of calibration uncertainty.
- Before cleaning, call the customer to agree the cleaning , warning the consequences
- Give an advice to client about improvement in the conditions of transport and storage of weights.

Other more elaborate methods may include ultrasonic baths or Soxhlet



Stabilization time after cleaning

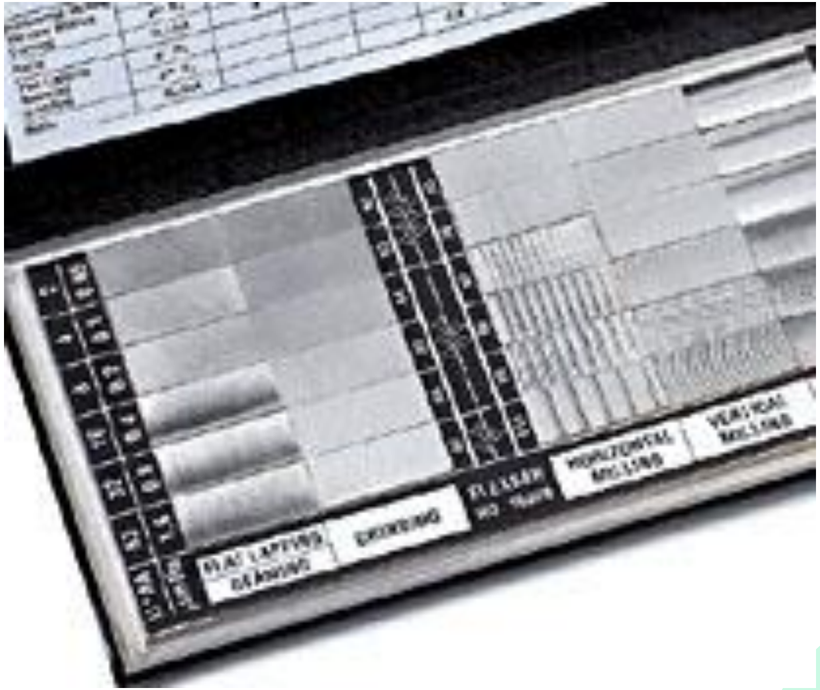
After weights are cleaned with solvents, they must be stabilized for the times given in Table B.1.

Table B.1 Stabilization time after cleaning

Weight class	E_1	E_2	F_1	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

c-Surface roughness

- The stability of the mass of a weight is highly dependent on the surface structure of the weight.
- A weight with a smooth surface is expected to be more stable than a weight with a rough surface so it is important to evaluate the roughness surface.
- The method using by roughness must be harmless to the weight.



e- Volume/Density and Magnetic properties determination

- Develop a procedure to determinate volume of weights, using a validated method y published by international recommendations or some method that is validated by a national laboratory.
- For laboratories that calibrates F2 weights or better accuracy, elaborate and implement a procedure to determinate magnetic properties of weights.

Why determinate the density?

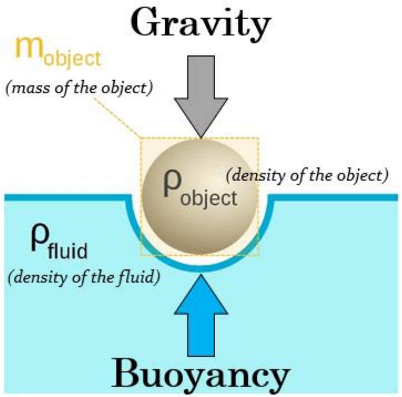
- If the air density, ρa, deviates from ρ = 1.2 kg m-3 more than ten percent (± 10%) and the density of the test weight, ρr, deviates from the reference density of the weight, ρt, the conventional mass should be corrected by the term C as follows:

$$m_{ct} = m_{cr}\left(1 + C\right) + \overline{\Delta m_c}$$

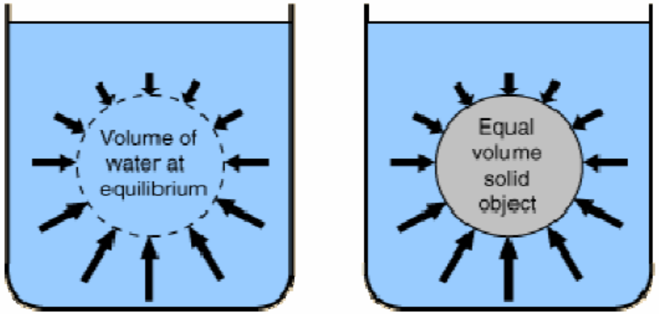
$$m_{ct} = m_{cr}\left(1 + \left(\rho_a - \rho_0\right)\left(\frac{1}{\rho_t} - \frac{1}{\rho_r}\right)\right) + \overline{\Delta m_c}$$

Density determination by hydrostatic weighing

When a body is submerged, it suffers an upwards force called buoyant force.



The buoyant force experienced by a submerged object is equal to the weight of the liquid displaced by the object



Archimedes´Principle

$$F_b = m_F\, g$$

$$m_F = \rho_F\, V_o$$

$$F_b = \rho_F\, V_o\, g$$

$$V_o = \frac{F_b}{\rho_F\, g}$$

Table B.4 Methods for determining density.

Method	Description
A	Most accurate method. A hydrostatic technique comparing the test weight with a reference weight both in air and in a liquid of known density.
B	Fastest and most suitable method. Weighing the weight in water and verifying that the balance indication is within tabulated limit values, or calculating the density from the balance indication and the known actual mass of the test weight.
C	Separate determination of the mass and volume of the test weight. The volume is determined from the increase in the balance reading when the weight is suspended into a water bath placed on a balance pan.
D	This technique is suitable for weights > 1 kg. Weighing of a liquid-filled test container of well-defined volume capacity with and without the test weight inside.
E	This technique is appropriate for weights with cavities that must not be immersed in water. Calculating the volume from the dimensions of the weight.
F	Estimating the density based on the known composition of the alloy from which the weight is manufactured.

Table 5 - Minimum and Maximum limits for density

Nominal value	ρ_{min}, ρ_{max} (10^3 kg m^{-3})							
	Class of weight (for class M_3, no value is specified)							
	E_1	E_2	F_1	F_2	M_1	M_{1-2}	M_2	M_{2-3}
≥ 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							

D- Magnetic properties

- Modern scales using servo control systems.
- Some have motors for opening doors, moving weights, etc.
- There can be then electromagnetic forces between the magnetic fields in the balance and the weight placed on the pan that can lead to errors in the determinations.
- The amount of these forces depends on the magnetic properties of the weight.

Magnetization -M

- Parameter that specifies a magnetic state of material bodies such as weights, in the absence of an external magnetic field (most generally, magnetization is a vector whose magnitude and direction are not necessarily constant within the material).

- The magnetization of a body generates an inhomogeneous magnetic field in space and thus may produce magnetic forces on other materials.

Maximum polarization for weights

Table 3 Maximum polarization, $\mu_0 M$, (µT)

Weight class	E_1	E_2	F_1	F_2	M_1	M_{1-2}	M_2	M_{2-3}	M_3
Maximum polarization, $\mu_0 M$, (µT)	2.5	8	25	80	250	500	800	1 600	2 500

Magnetic susceptibility - χ

- Measure of the ability of a medium to modify a magnetic field.

  It is related to the magnetic permeability (μ) by the relation:

$$\mu / \mu_0 = 1 + \chi$$

- The quantity μ / μ_0 is referred to as the relative permeability, μ_r.

Limits of magnetic susceptibility

Table 4 Maximum susceptibility, χ

Weight class	E_1	E_2	F_1	F_2
$m \leq 1$ g	0.25	0.9	10	-
2 g $\leq m \leq$ 10 g	0.06	0.18	0.7	4
20 g $\leq m$	0.02	0.07	0.2	0.8

9.3 If the values of all local measurements of magnetization and susceptibility are less than these limits, then it may be assumed that the uncertainty components due to the magnetism of the weight are negligible. The maximum permanent magnetization and magnetic susceptibilities given in Tables 3 and 4 are such that, at magnetic fields and magnetic field gradients possibly present on balance pans, they produce a change of the conventional mass of less than 1/10 of the maximum permissible error of the test weight [8] [9].

Magnetic properties measurement

Two methods according to R111:

- B.6.5 The attracting method.
- B.6.4 BIPM Susceptometer.

The attracting method

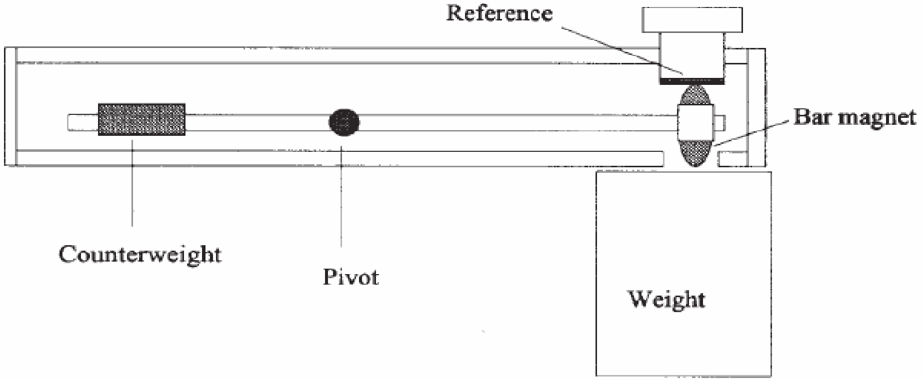
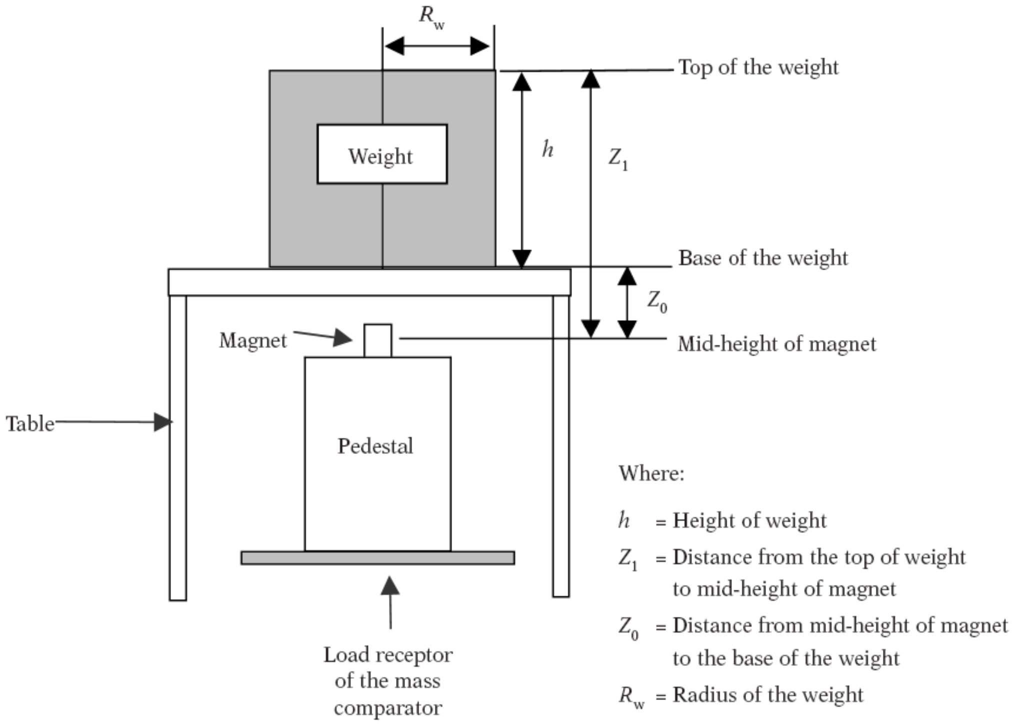


Figure 5. Design of a commercial permeability indicator.

Metrologia, **40** (2003) 339–355

Suceptometer Method.



g-Thermal stabilization

Prior to performing any calibration tests, the weights need to be acclimated to the ambient conditions of the laboratory. In particular, weights of classes E1, E2 and F1 should be close to the temperature in the weighing area.

The mandatory minimum times required for temperature stabilization (depending on weight size, weight class and on the difference between the initial temperature of the weights and the room temperature in the laboratory) are shown in Table B.2. As a practical guideline, a waiting time of 24 hours is recommended.

g-Thermal stabilization in hours

ΔT*	Nominal value	Class E_1	Class E_2	Class F_1	Class F_2
± 20 °C	1 000, 2 000, 5 000 kg	-	-	79	5
	100, 200, 500 kg	-	70	33	4
	10, 20, 50 kg	45	27	12	3
	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			0.5
± 5 °C	1 000, 2 000, 5 000 kg	-	-	1	1
	100, 200, 500 kg	-	40	2	1
	10, 20, 50 kg	36	18	4	1
	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			

<table>
<tr><th>ΔT*</th><th>Nominal value</th><th>Class E_1</th><th>Class E_2</th><th>Class F_1</th><th>Class F_2</th></tr>
<tr><td rowspan="6">± 2 °C</td><td>1 000, 2 000, 5 000 kg</td><td>-</td><td>-</td><td>1</td><td>0.5</td></tr>
<tr><td>100, 200, 500 kg</td><td>-</td><td>16</td><td>1</td><td>0.5</td></tr>
<tr><td>10, 20, 50 kg</td><td>27</td><td>10</td><td>1</td><td>0.5</td></tr>
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<tr><td rowspan="6">± 0.5 °C</td><td>1 000, 2 000, 5 000 kg</td><td>-</td><td>-</td><td>-</td><td>-</td></tr>
<tr><td>100, 200, 500 kg</td><td>-</td><td>1</td><td>0.5</td><td>0.5</td></tr>
<tr><td>10, 20, 50 kg</td><td>11</td><td>1</td><td>0.5</td><td>0.5</td></tr>
<tr><td>1, 2, 5 kg</td><td>7</td><td>1</td><td>0.5</td><td>0.5</td></tr>
<tr><td>100, 200, 500 g</td><td>3</td><td>1</td><td>0.5</td><td>0.5</td></tr>
<tr><td>< 100 g</td><td>1</td><td colspan="3">0.5</td></tr>
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* ΔT = Initial difference between weight temperature and laboratory temperature.

h-Weights Calibration

MEASURAND

- The measurand is the conventional mass of the weight in calibration and expanded uncertainty associated with the measurement must be equal to or less than 1/3 of the maximum permissible error, EMT, of the weight to be calibrated.
- The conventional measuring unit mass is the kilogram, kg. multiples or submultiples of the unit may also be used, eg. gram, milligram, ton.

Typical measurement range

- 1 mg a 5 000 kg

Expected measurement uncertainty

- For each weight, the expanded uncertainty U for k = 2 of the conventional mass shall be less than or equal to 1/3 of the maximum permissible error specified in OIML R 111.

Calibration Procedure :

- It involves the comparison of two or more weights, normally an unknown and a standard, by placing each one in turn on the balance pan and noting the reading.
- The comparison must be consistent in determining the conventional mass, comparing the gravitational force exerted on the load receptor plate of an instrument for weighing:
- by the standard weight and the weight to be calibrated.
- There are diferent types of cycles to calibrate: ABBA, AB, AB1B2.....BnA, etc., where:
    - A: standard weight
    - B: weight to calibrate

Methods

1) The direct comparison method:

E_1 → E_2

2) The subdivision or multiplication method that applies for a set of weights.

E_1 → Set of Weights E_2

Subdivision example:

Reference weight	vs	5 + 2 + 2* + 1
Reference weight	vs	5 + 2 + 2* + 1*
5	vs	2 + 2* + 1
5	vs	2 + 2* + 1*
2 + 1	vs	2* + 1*
2 + 1	vs	2* + 1*
2 + 1*	vs	2* + 1
2 + 1*	vs	2* + 1
2	vs	1 + 1*
2	vs	1 + 1*
2*	vs	1 + 1*
2*	vs	1 + 1*

Standard weight: 1 kg

5: 500 g

2: 200 g

2*: 200 g (.)

1: 100 g

1*: 50 g + 20 g + 20 g (.)+10 g

Mass Standard:

- Must be at least one class higher accuracy than the weights to calibrate, ie, they must have a maximum permissible error (EMT) less than or equal to one third of the weight to be calibrated.
- Should have equal nominal values to the test weights.
- Calibration Certificate must be valid.
- It must be kept protected against deterioration or damage due to shock, vibration, etc

$E_1 \rightarrow E_2$

$E_2 \rightarrow F_1$

. . .

$M_2 \rightarrow M_3$

Weighing-Comparators - balances

Weighing instruments must have sufficient scope measurement according to the nominal value or the weights that will be calibrated.

Any of these conditions must be complied: the resolution (d) and standard deviation (s), in the range of interest:

- d ≤ EMT /10 , and s ≤ 2d
- $\sqrt{\frac{d^2}{12}+\frac{s^2}{n}} \leq \frac{EMT}{6}$ n = measurements number in repeatability test.

Environmental conditions

- Laboratories that calibrate weights E1, E2, F1 and F2 class, should have appropriate equipment to measure air temperature, atmospheric pressure and relative humidity.
- This is necessary to make the correction for air buoyancy for the determination of mass or conventional mass.
- Calibration certificates of these equipment should be valid.
- Laboratories that calibrate weights class M1 and lower only should know the reference environmental conditions.
- The weight calibration laboratories located at sea level, can avoid air buoyancy correction, but should demonstrate that this correction is neglectable in usual laboratory conditions.

Calibration, verification, adjustment.

- **CALIBRATION**: Determination of error of a weight and uncertainty by comparison with a standard using a specific methodology.
- **VERIFICATION**: Declaration by the weight calibration that belongs to the class being declared.
- **ADJUSTMENT**: Value changes of weight mass to carry the declared class.

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Thanks!

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