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CALIBRATION OF SCIENTIFIC EQUIPMENT

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Measurements are based on standardized units and measuring instruments. Good measurement relies on the integrity of the measuring equipment used. Unfortunately, no matter how sophisticated measuring equipment's are, it degrades with time due to thermal, mechanical, electrical, and environmental effects. This degradation is called *drift*, and it is unavoidable. However, the effects of drift on the reliability of the measurements may be offset by a process known as *calibration*.

Accuracy and Precision

The accuracy of a measurement is the degree of its closeness to the *true or reference value*. The precision of a measurement is the degree of scatter of the measurement result, when the measurement is repeated a number of times under specified conditions (Fig. 1).

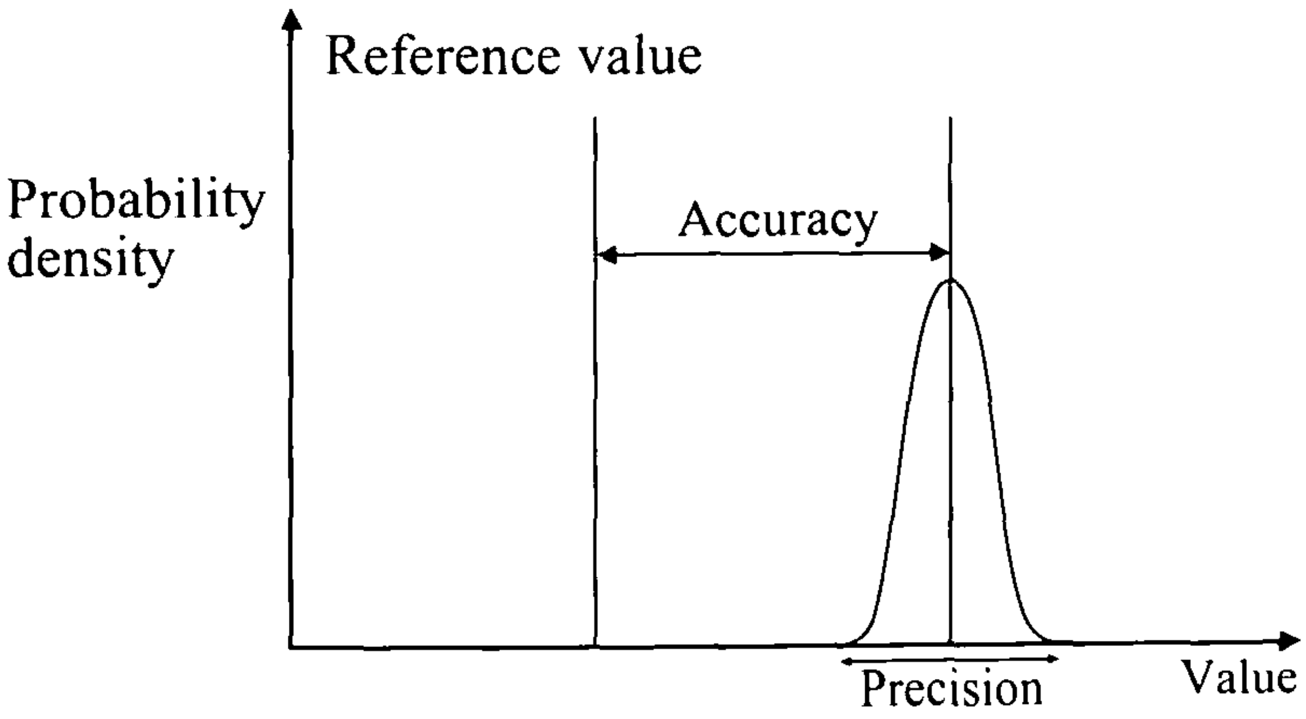

Figure 1. Accuracy and precision in measurements

Thus, a measurement system can be accurate but not precise, precise but not accurate, neither, or both. For example, if an experiment contains a systematic error, then increasing the sample size generally increases precision but does not improve accuracy. The result would be a consistent yet inaccurate string of results from the flawed experiment. Eliminating the systematic error improves accuracy but does not change precision

Standards and SI Units

In metrology, standards are set by regional or local authorities, often based on practical measures. The earliest examples of these standardized measures are **length, time**, and **weight**. These standards were established in order to facilitate day to day life and record human activity. Scientists made significant progress in metrology during the scientific revolution. The discovery of atoms, electricity, thermodynamics, and other fundamental scientific principles could be applied to standards of measurement, and many inventions made it easier to quantitatively or qualitatively assess physical properties, using the defined units of measurement established by science.

The concept of establishing units of measurement was based on constants of nature, and thus making measurement units available "for all people, for all time". In the beginning a unit of length and mass were defined from the dimensions of the Earth, and a cube of water. The result was platinum standards for the meter and the kilogram established as the basis of the metric system in 1799 and further led to the establishment of the International System of Units or *Système International d'Unités*, abbreviated as SI units. This system has gained unique worldwide acceptance as definitions and standards of modern measurement units. The definitions and specifications of SI are globally accepted and recognized. The SI is maintained under the support of the Metre Convention and its institutions, the General Conference on Weights and Measures, CGPM, its executive branch the International Committee for Weights and Measures, CIPM, and its technical institution the International Bureau of Weights and Measures, BIPM.

As the authorities on SI, these organizations established and propagated the SI, with the objective to be of service to all. This includes introducing new units, such as the relatively new unit, the **mole**, to encompass metrology in chemistry. These units are then established and maintained through various agencies in each country so as to establish a hierarchy of measurement standards that can be traced back to the established standard unit, a concept known as *metrological traceability*. The definitions and base SI units published by the BIPM are given below and their symbols in Table 1.

Meter

The metre is the length of the path travelled by light in vacuum during a time interval of 1/299 792 458 of a second.

Table 1. The base SI units defined by the International Bureau of Weights and Measures (BIPM)

Quantity	Unit	Symbol
Length	meter	m
Mass	kilogram	kg
Time	second	s
Temperature	Kelvin	K
Electric current	Ampere	A
Luminous intensity	candela	cd
Amount of substance	mole	Mol

Kilogram

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

Second

The second is the duration of 9 192 631 770 periods of the radiation corresponding to the transition between the two hyperfine levels of the ground state of the caesium 133 atom.

Kelvin

The kelvin, unit of thermodynamic temperature, is the fraction 1/273.16 of the thermodynamic temperature of the triple point of water.

Ampere

The ampere is that constant current which, if maintained in two straight parallel conductors of infinite length, of negligible circular cross-section, and placed 1 m apart in vacuum, would produce between these conductors a force equal to 2×10^{-7} newton per metre of length.

Candela

The candela is the luminous intensity, in a given direction, of a source that emits monochromatic radiation of frequency 540 x 1012 hertz and that has a radiant intensity in that direction of 1/683 watt per steradian.

Mole

The mole is the amount of substance of a system which contains as many elementary entities as there are atoms in 0.012 kilogram of carbon 12.

Traceability

Traceability is defined as the *'property of a measurement result, whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty'.*

Traceability is the property of the result of a measurement. It is not achieved by following any one particular procedure or using special equipment. Merely having an instrument calibrated by an accredited laboratory is not enough to make the measurement results obtained by that instrument traceable to the realization of the appropriate SI unit or other stated references. The measurement system by which values are transferred must be clearly understood and be under control. Traceability gives the user confidence that the measurement results agree with recognized national/international standards within the stated uncertainty. It also ensures that the measurement results will be equivalent to those made using different instruments from different suppliers.

To support the claim of traceability, the provider of the measurement result or value of a standard must document the measurement process used to establish the claim and provide a description of the chain of comparisons that were used to establish a connection to a particular stated reference. All valid statements of traceability shall have the following common elements in them:

- A clearly defined particular quantity that has been measured.
- A complete description of the measurement system or working standard used to perform the measurement.
- A stated measurement result or value with a *documented uncertainty.*
- A complete specification of the stated reference at the time the measurement system or working standard was compared to it.
- An internal measurement assurance programme for establishing the status of the measurement system or working standards at all times pertinent to the claim of traceability.
- An internal measurement assurance programme for establishing the status of the stated reference at the time that the measurement system or working standard was compared to it.

The user shall ensure that the calibration service provider fulfills the requirements stipulated above. An accredited laboratory conforming to ISO:IEC 17025 will ensure the traceability of the measurement results produced by the instrument.

The National Measurement Institute (NMI) provides the link to the definition of the unit. The hierarchy of traceability for a mass measurement and the uncertainty associated at each stage is illustrated in Fig. 2.

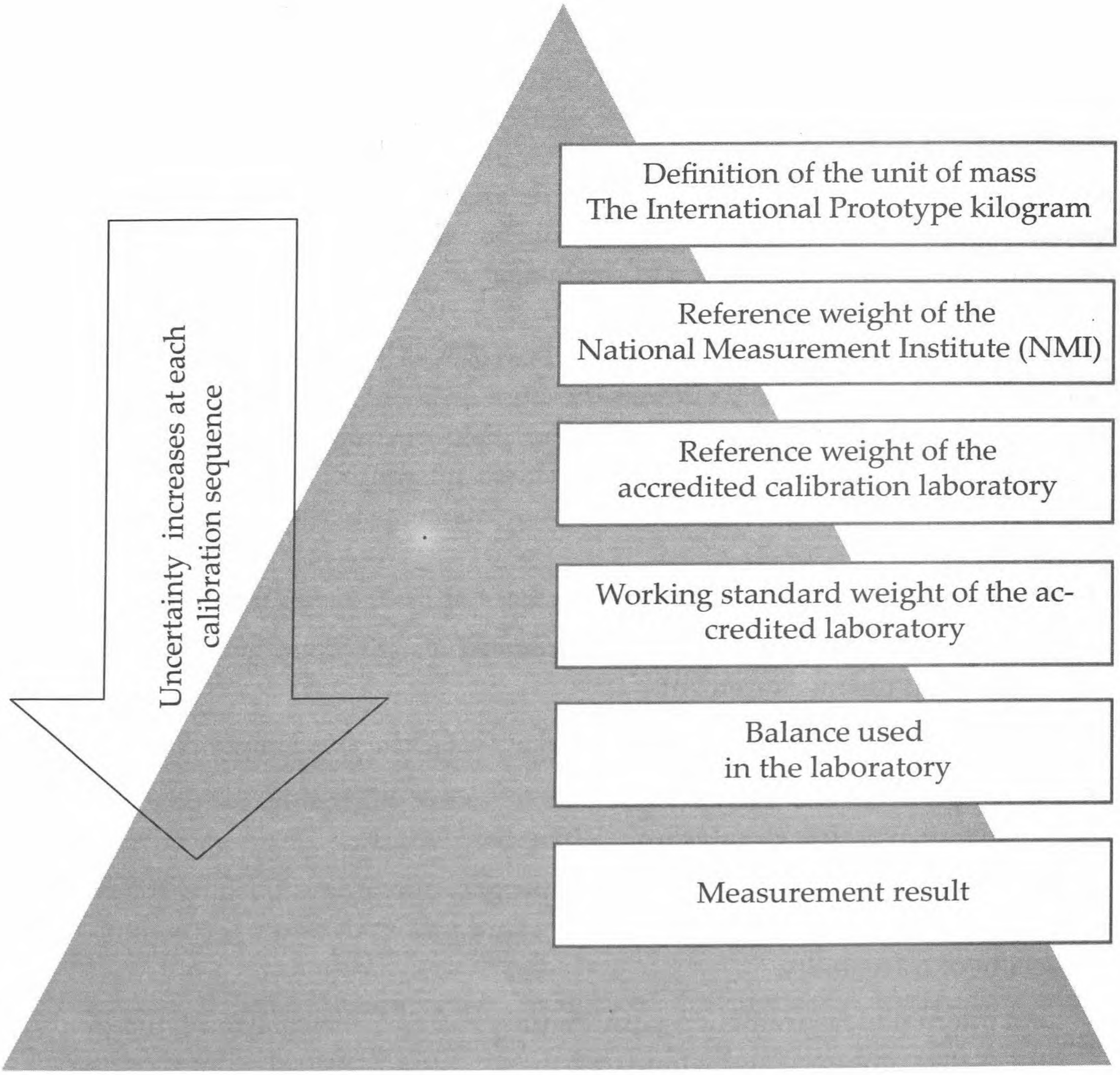

Figure 2. An example of traceability hierarchy of a simple mass measurement

Hierarchy of Measurement Standards

In metrology there are three different standards, namely primary, secondary and working standards forming a hierarchy. Primary standards have the highest metrological accuracy and their values are not compared with other standards of the same quantity. For example the Josephson junction is the primary standard for DC Voltage and the International Prototype kilogram maintained at the International Bureau of Weights and Measures (BIPM) is the primary standard for mass measurement. These standards are the highest level for mass and DC voltage measurements and are not referenced to any further standards.

Secondary standards are standards whose values are assigned by comparison with primary standards of the same quantity. Standard Platinum Reference Thermometer (SPRT), National Standard kilograms and solid state DC voltage are considered as secondary in metrology.

The Standards used routinely to calibrate or check material measures, measuring instruments or reference materials are categorised as working standards. The hierarchy of measurement standards is shown in Fig. 3.

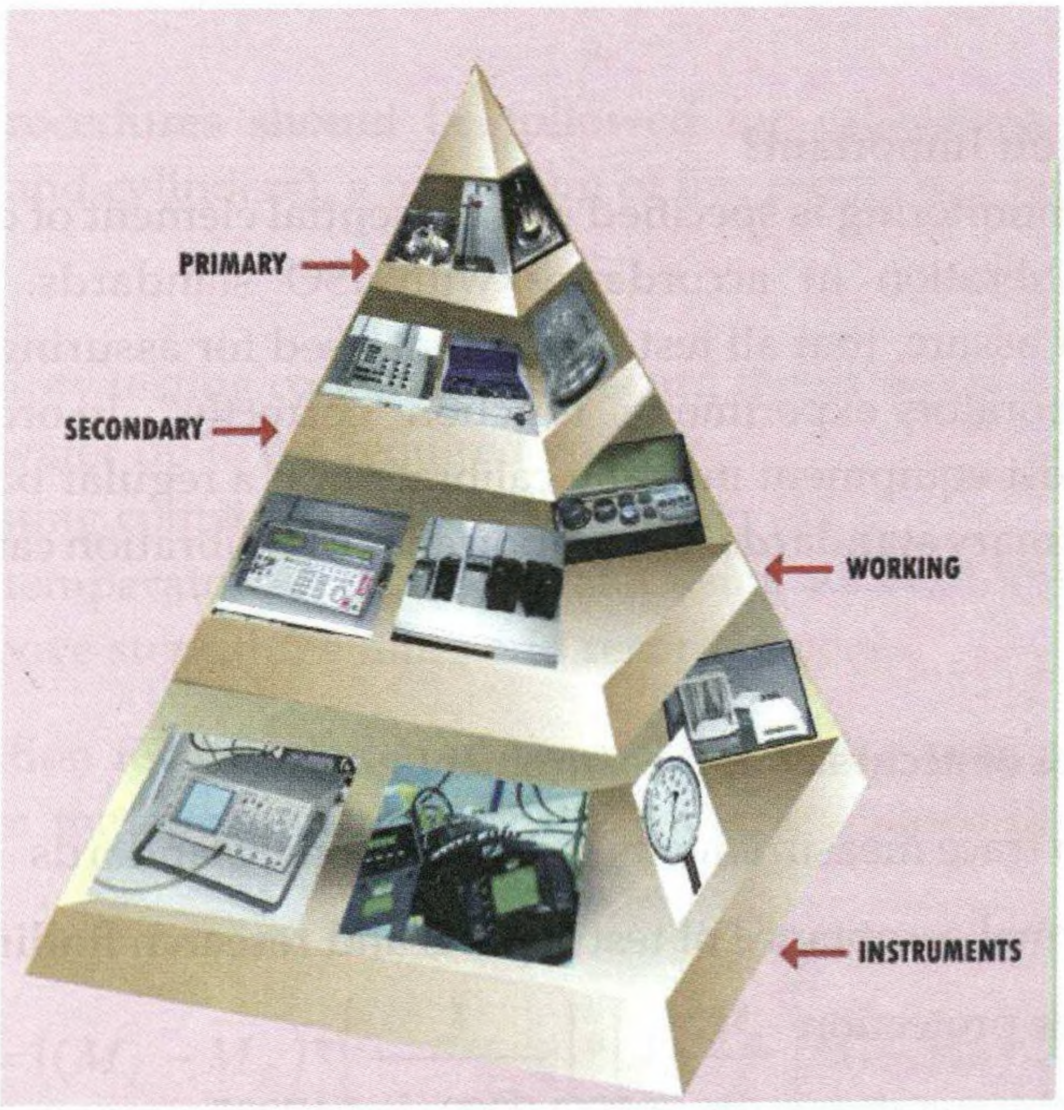

Figure 3. Hierarchy of measurement standards

The main reason for establishing a hierarchy of standards is to minimize the use and handling of the higher level standards and to preserve their values (Fig. 4).

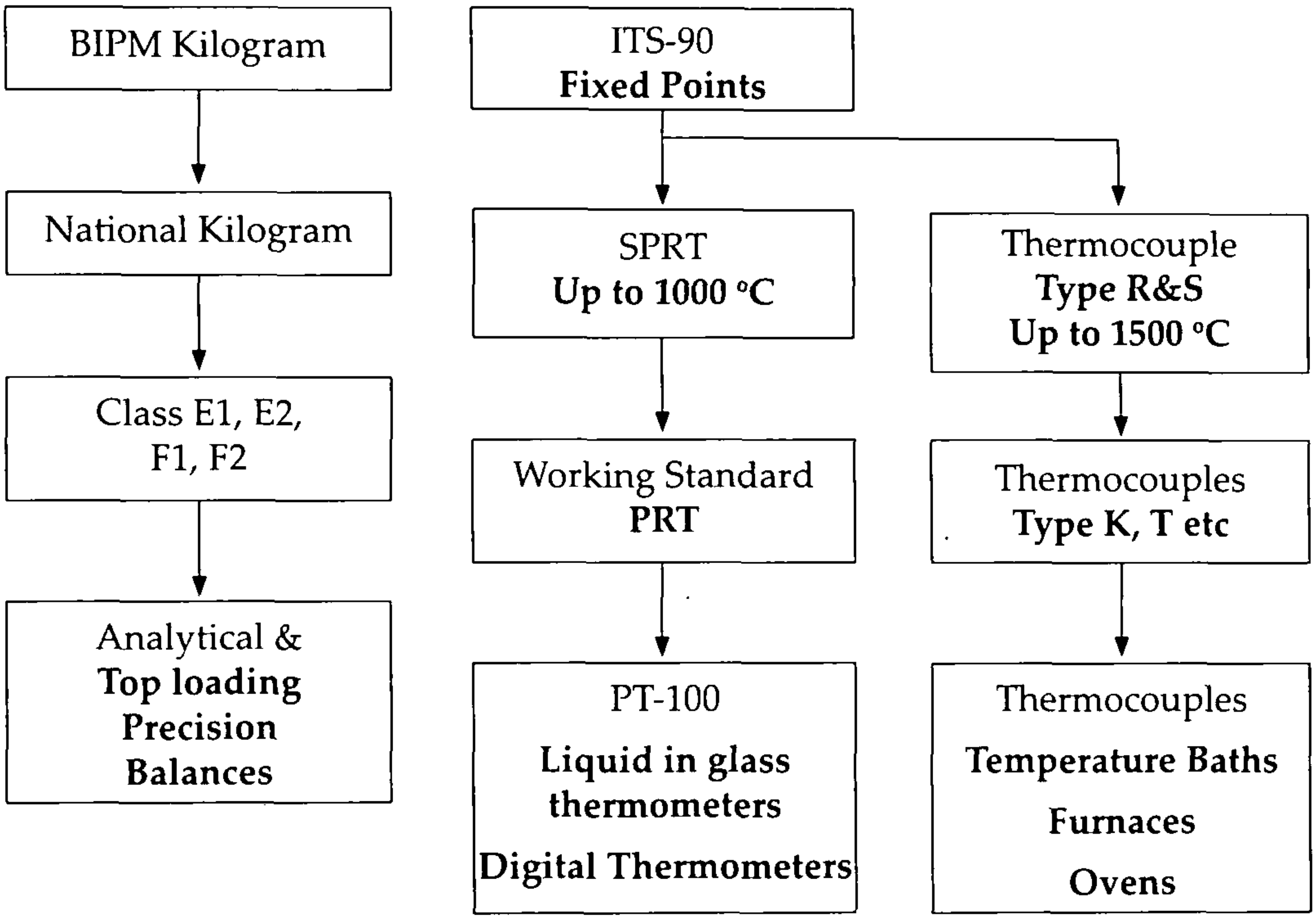

Figure 4. Hierarchy of mass and temperature measurements

Why is Calibration Important?

Test equipment monitoring is specified as an essential element of quality assurance for standard calibration in accordance with ISO standards. Test equipment monitoring must assure, that all test equipment used for assuring product quality measures and laboratory experiments, function "correctly". In order to ensure that this is the case, test equipment must be calibrated on a regular basis, and must be retraceable to **Primary standards.** The importance of calibration can be summarized as follows:

- Assurance of accuracy of measurements
- Ability to trace measurements to International standards
- International acceptance of test reports and research findings
- Consumer protection
- Meeting the requirements of ISO 9000 and 17025

Calibration of Volumetric Glassware by the Weighing method

The weighing method is the most common technique used for calibration of glassware such as volumetric flasks, burettes etc. The internal surface of volumetric vessels should be cleaned thoroughly before calibration (see Chapter 9 under Cleaning Glassware). Oil or grease deposits should be removed by a suitable solvent. The vessel should be nearly filled with an aqueous solution and shaken vigorously. It should then be repeatedly rinsed with distilled water, until all traces of the detergent are removed. The following conditions should meet the calibration of glassware:

- Prior to any calibration test, acclimatize the apparatus and materials to the ambient conditions of the laboratory by exposing them to the laboratory atmosphere.
- During the calibration, the maximum temperature variation in the laboratory should not exceed ± 0.5 °C per 2 hour.
- Set or read a meniscus against a reference line or scale.
- Make sure that the vessel or weighing bottle and the distilled water are at room temperature.

The following procedures should be followed for calibration. For graduated glassware (flasks and cylinders), a maximum of five points will be selected.

Procedure

- Weigh the empty testing container and record the value (M_E).
- Fill distilled water up to the required volume.
- Weigh the testing container with distilled water (M_L).
- Measure the temperature of distilled water using a thermometer.
- Repeat the above steps three times and record the data.

The general equation for calculation of the volume at the reference temperature of 20 °C, V_{20}, from the apparent mass of the water, contained or delivered, is as follows.

$$V_{20} = \left((M_L - M_E\right)*\left(\frac{1}{\rho_w - \rho_a}\right)*\left(1 - \frac{\rho_a}{\rho_b}\right)*\left(1 - \gamma(t - 20)\right))$$

where,

M_L	is the mass of the vessel with distilled water in grams
M_E	is the mass of the empty vessel in grams
ρ_a	is the density of air, in grams per milliliter
ρ_b	when weighing in air as though the density of the weight was 8.0 g/ml.
ρ_w	is the density of water at t °C, in grams per milliliter.
γ	is the coefficient of cubical thermal expansion of the material of which the item of glassware tested is made in reciprocal degrees Celsius,
t	is the temperature of the water used in testing in degrees Celsius.

where,

t_a temperature of air (°C)

$$\rho_a = \frac{0.00348444 * p - h * (0.00252 * t_a - 0.020582)}{(t_a + 273.15) * 1000}$$

h relative humidity (RH %)
p Pressure of air (Pa)

$$\rho_W = \frac{a + bt + ct^2 + dt^3 + et^4 + ft^5}{1000}$$

where,

$a = 999.7173$

$b = 0.09631$

$c = -0.01165$

$d = 0.000211712$

$e = -3.49072X10^{-6}$

$f = 2.62183X10^{-8}$