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GUIDE TO CALIBRATION

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GUIDE TO CALIBRATION

This brochure was written within the framework of the OIML Development Council with the aim of guiding national organisations which intend to establish new or further develop existing facilities for calibration of working standards.

The suggestions or opinions expressed have no official standing and have received no official endorsement by the Organisation.

S.A. Thulin

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I - GENERAL CONSIDERATIONS

1. What do we mean by calibration ?

It is important first to clarify what we mean by calibration. Originally the word meant the procedure of sorting out by their size (in French "calibre") objects such as cannon balls.

When visiting laboratories you may hear somebody saying that an instrument needs calibration meaning in fact that it needs adjustment (many times preceded by repair !). The confusion between adjustment and calibration is very frequent as many electronic instruments and in particular those used for physico-chemical analysis frequently require both zero and span adjustment before use. In less frequent situations the use of the term calibration may simply refer to a check of tolerances in a similar way as the first mentioned "historical" meaning of calibration.

For the purpose of this guide we will for calibration generally use the definition given in the International Vocabulary of basic and general terms in metrology (VIM), i.e. "The set of operations which establish under specified conditions the relationship between values indicated by a measuring instrument or a measuring system, or values represented by a material measure, and the corresponding known values of a measurand".

By "known values" are usually meant the (conventionally) true values attributed to the standards and equipment used as references in the calibration.

According to this definition a calibration will generally result in a report or certificate stating the conventionally true values.

Most measuring instruments or measures will usually not be calibrated but adjusted by its manufacturer or by maintenance services to within specified tolerances which subsequently can be checked by procedures usually referred to as verification and which may result in a statement by the verifying body sometimes called verification certificate.

In the following we will however only deal with instruments or measures which are considered to be used as standards and as thus subjected to calibration in the strict meaning of the mentioned Vocabulary and the issue of a calibration certificate.

Finally we shall however not exclude the occasional use in common language of the word calibration such as when saying that "the instrument's calibration has been affected by rough handling", meaning that the indicated values are affected.

2. Objects to be calibrated - active and passive standards - redundancy

It may be useful at least for the discussion below to distinguish between 'active' and 'passive' devices or instruments. By 'active' devices we refer here to those which contain some form of electronic, electromechanical, pneumatic or hydraulic amplification, transformation or data processing. A 'passive' device is for instance a weight or a resistor.

Active devices like electronic balances, digital electronic voltmeters, etc. are nowadays most commonly used for calibration purposes because of convenience, high resolution, recording facilities etc. Though many of such instruments exhibit good long-term stability they frequently require at least a rapid check of one or several calibration points by use of passive devices.

Generally active devices may exhibit more erratic behaviour with time than truly passive devices and the same holds as regards their transportation.

Active standard devices are therefore most suitable for use as working standards in immediate connection to a passive calibration facility operated directly by the user.

Devices to be sent to national or other specialized calibration laboratories should preferably only be of the passive type. In the case of equipment comprising sensors such as load cells which are associated with electronic measuring and indicating devices, the sensors should preferably be calibrated separately and the measuring and indicating device provided with means for on-site calibration by use of a separate electrical circuitry.

There are unfortunately cases where the manufacturers of high-accuracy calibration equipment has not foreseen such separate calibration facilities by the user. In this connection standard laboratories should also be warned against procurement of equipment whether passive or active, which does not easily allow internal calibration of digits (for instance by containing only steps of 1 to 9 in each digit instead of 1 to 10).

Summarizing the above one may recommend that the transfer of the magnitude of a unit of measurement from one laboratory to another, especially when they are far away, should be done wherever possible by passive devices, or if this is not possible by use of sufficient redundancy.

Any passive standard likely to show an erratic behaviour due to handling and transport should also be subjected to redundancy by constituting a group of at least three units, so that if one of these units indicates a very different value from the mean when compared to the certified values it can if necessary be excluded from the group.

3. Uni-directional transfer

For the purpose of the discussion we would like to use the term uni-directional transfer for a procedure whereby the calibrated object or standard is not likely to be recalibrated and thus not returned to a standards laboratory for subsequent calibration.

On international level this kind of transfer is very common for practical and administrative reasons. Some types of standards, when not submitted to rough handling or wear, are in fact unlikely to change their metrological characteristics outside the required or expected limits of accuracy. Another example is reference materials such as hardness blocks and similar though sometimes interesting results about their evolution, or the evolution of the standard measuring equipment, can be obtained by return of such materials after an appropriate time to the original calibrating laboratory.

A common example of uni-directional calibration transfer is when a new measurement standard is of relatively low cost compared to transport and calibration fees, in which case it may prove easier to procure a new standard and have it calibrated in the country of manufacture instead of sending a reference device for recalibration at a national laboratory abroad.

If uni-directional calibration transfer is largely used it is of particular importance to provide for satisfactory procedures and means for the preservation of the national reference so as to avoid jumps in values or other inconsistancies resulting from the fact that the standards involved in successive transfers are different both as regards material and certification.

The exclusive use of uni-directional calibration is not advised when the transferred standard is likely to be influenced by the transport. Should the uni-directional procedure however be necessary, it must then be combined with sufficient redundancy requiring more units to be calibrated. A typical example is photometric standard lamps which furthermore preferably should be hand-carried, see below.

4. Bi-directional transfer

In a similar manner as the term used in the previous section we would like to call bi-directional transfer the procedure whereby a standard is sent to an official laboratory abroad for calibration and then returned to the proprietory laboratory.

To make best use of this procedure which usually is carried out for a main standard, such a standard kilogram, a standard resistor or a group of Weston cells, it is necessary to proceed as follows :

- control and if necessary trim the local comparison equipment (such as balances, bridges, voltage comparators, etc.)
- compare the travelling standards before departure with suitable local standards which shall not be otherwise used in the absence of the travelling standards
- compare the travelling standards upon their return with the local standards under exactly the same conditions as at the departure.
  For some types of standards like for instance Weston cells this latter comparison may have to be repeated after a further interval of one month, when all cells have duly stabilized.

The comparisons on local level of the travelling standard before departure and on return usually permit to check the influence of the transport.
If any significant differences are found between these two comparisons it may be appropriate to contact the laboratory which made the calibration abroad to find out if any adjustments have been made as for instance of thermostat temperature in the case of a Weston cell enclosure or if the travelling standard has been subject to any special treatment, such as cleaning in the case of a mass standard.

5. Transport

Many standards are likely to suffer during transportation from vibration, shock or variations in temperature or humidity. Time is also a factor which cannot be neglected. Though air transport is most frequently used it happens that equipment is blocked in customs warehouses for a long time, sometimes under very unfavourable conditions of climatic variations provoking condensation and consequent corrosion. Rough handling, frequent in warehouses may be of even more nuisance than the transport itself.

The general rule for delicate standards is to have them accompanied by a person who can carry them as hand-luggage. This is of course expensive in particular as regards bi-directional transfers but is absolutely necessary in the following typical cases :

- Weston cells (in thermostated enclosure with battery supply)
- Photometric lamps
- Travelling electronic standard clocks with battery supply.

Some other instruments sensitive to shocks or breakage are for instance :

- Standard platinum resistance thermometers (with freely suspended wire)
- High accuracy mercury-in-glass thermometers
- Hydrometers of highest resolution.

A number of other instruments can usually be adequately packed but may preferably be sent as accompanied luggage so as to keep the total transport time under control, avoid sources of corrosion and enable more rapid handling through customs. Such items are for instance the following standards :

- Weights
- Gauge blocks
- Divided rulers
- Resistors
- Capacitors
- Pyrometer lamps
  etc.

Whether a standard is hand-carried or not it must generally be packed in a suitable transport box which is clearly marked with its content, serial numbers and the name and address of the proprietory laboratory. If the transport box takes the form of a packing which can mistakenly be disposed of at the calibrating laboratory it shall in addition be clearly marked with big letters REUSABLE CONTAINER DO NOT DESTROY or similar.

6. When to calibrate ?

To the question "When is calibration or verification necessary ?" the laconic answer "always" would be the safest. It applies in particular to modern "active" instrumentation, which must, more and more, be provided with easy facilities for the checking of the calibration either automatically or by intervention of the user.

There has been extensive studies made on calibration intervals for commonly used instruments which are not provided with such calibration check facilities.

These studies aimed mainly at trying to establish methods for fixing calibration intervals. Some of these methods have been briefly mentioned in OIML International Document D 10 "Guidelines for the determination of recalibration intervals of measuring equipment used in testing laboratories". This Document was elaborated within the framework of a collaboration with ILAC.

An attempt was also made by ILAC to establish through an enquiry typical calibration intervals for specific types of instruments. The results to this enquiry were, however, confusing and did generally not enable to lay down any definite intervals for specific instruments. The enquiry covered in fact a very great variety of instruments of common use as well as a few standards. The replies from the testing laboratories were consequently incomplete as common instruments frequently have to be verified at the user's laboratory (or close to it) and only the standards are sent to a special testing laboratory.

Taking into account this distinction between calibration of instruments for direct use and that of standards we will in the following generally discuss the problems associated with standards of high accuracy used as national primary or secondary references. *

A measurement standard which is adopted or considered as the national reference should be preserved under suitable conditions within the central metrology laboratory of the country and should normally only be used for comparisons of secondary standards or travelling standards. There are however a few cases where there is no harm and even convenient to use directly a national standard for more general calibration purposes (e.g. a protected divided calibrated scale incorporated in a length measuring machine, or a stationary time and frequency standard).

Most national standards will be subjected to an initial calibration, usually at the national laboratory of the country of manufacture or in a few cases at BIPM.

The necessity for recalibration is determined by

a) accuracy required on national level taking into account existing calibration equipment

b) possible changes of the value of the standard with time under local atmospheric conditions

c) possible or suspected changes of the value of the standard due to its use.

It is likely that the value of standards changes more or less with time

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* Standards used by local verification offices are covered in the International Document No .. "Calibration and certification of verification of verification devices" drafted by SP 23-Sr 4.

either as a regular drift or in a more erratic manner. However, these changes may frequently be so small that they have no practical effect on the country's measuring system, point (a).

If this system is so highly developed that it requires the use of the full accuracy which can be provided by the national reference it is necessary to take into consideration changes according to point (b). Gradual changes with time can normally not be revealed but by recording the evolution of the standard at each calibration over a number of years. It can, however, be suspected that some types of passive standards change more in the first years after manufacture than later and that consequently a recalibration already after 1 or 2 years is suitable to find out or simply to confirm their stability within certain limits.

The storage temperature of such standards usually has a noticeable effect on drift. Some materials are more subject to drift than others in particular some alloys like brass and invar as well as various materials used in electrical standard resistors. Special types of steel used for gauge blocks or for making standard dynamometers or load cells usually require special heat treatment followed by a long stabilization period. The amount of drift may thus depend on the manufacturer's supply of suitably stabilized materials.

The last point (c) is however the most annoying especially with instruments such as dynamometers and load cells which may have been subjected to slight over-loads without visible effects. The means for detecting changes due to overload or bad handling consist in providing sufficient redundancy by the procurement of additional standards used only for control purposes, so-called witness standards (from French "étalons témoins").

A good reason for undertaking a recalibration of national standards may also be that there is a special opportunity offered by a foreign national laboratory or BIPM.

Summarizing the above it will be seen that the answer to the question "When is calibration of the national standards necessary ?" has to be based on pragmatic considerations. We will include some advice concerning this question in the later discussion when treating separately each quantity or component of the national measuring system.

7. Hierarchy schemes - Traceability - National Measurement System

Between the practical application of a measuring device and the physical definition of the unit of measurement used in the device there can be a multitude of steps which are technically, geographically and timewise separated. The link from the instrument to the definition of the unit is sometimes represented by a hierarchy scheme. Such schemes are useful but they are in most cases incomplete as they only connect various forms of standards of different accuracy but disregard, for reasons of easy presentation, a great many of the practical problems involved, such as scaling up and scaling down operations of the basic unit, particular characteristics of comparison equipment, time and distance. To this enumeration one may sometimes add factors such as administrative difficulties including finance, customs, etc.

Hierarchy schemes are therefore only reliable when considered on purely local level in situations where all these extraneous factors can be kept under control.

A developing country or a small-sized country which cannot afford to build-up a comprehensive measurement system without making use of extensive "calibration abroad" has frequently difficulties in establishing and maintaining the efficiency of a complete hierarchy scheme.

In a similar way the use of the word 'traceability' does in practice not have the full force of an 'unbroken chain of comparisons' as it is somewhat idealistically expressed in the International Vocabulary of Metrology. This chain is in fact as mentioned many times broken by distance, time, administrative difficulties, different laboratory practice and staff, etc. Much work is presently devoted in the world by standards bodies and analyst associations to harmonize procedures and instruments in order to improve measurement accreditation schemes. This, however, is outside the scope of this brochure.

In the absence of very strict "game rules" one must unfortunately consider that the term traceability in practice only means that there is some undefined relation between the indications of an instrument or a measure and the corresponding local, national or international measurement standard or physical definition. We must admit, however, that "traceable to" is a convenient term to use in common language when designating the source, standard or institute to which measurements are linked even without saying when and how. "Fully traceable to" would be stronger and could practically mean that there is within given limits of uncertainty no bias error of a measurement with respect to the stated source or laboratory.

The important matter for a small or a developing country is to know how it can establish a reliable measurement system based on the fundamental definitions of the units of measurement. This involves several practical considerations of technico-scientific as well as administrative nature whereby the factors time and money cannot be neglected.

The approach to this question should be pragmatic by considering each measurement problem or unit of measurement separately in the light of the most recent technology and experience rather than from a purely theoretical point of view. The local needs and prevailing conditions should form so to speak the terminal station of such a system to which there may lead as many tracks as there are units of measurement required.

The general rule for establishing such a system would be, at least for a small country, to build it up by starting at this terminal station and not at the other end of the far-away track leading to the physical definition. The reverse, more attractive from the philosophical point of view may however apply to countries having considerable funds and potential for scientific research.

In the following chapters we will use the term "national measurement system" to designate an institutional, technical and administrative structure embedding the practical aspects of hierarchy schemes for each quantity (or unit) as well as the associated traceability.

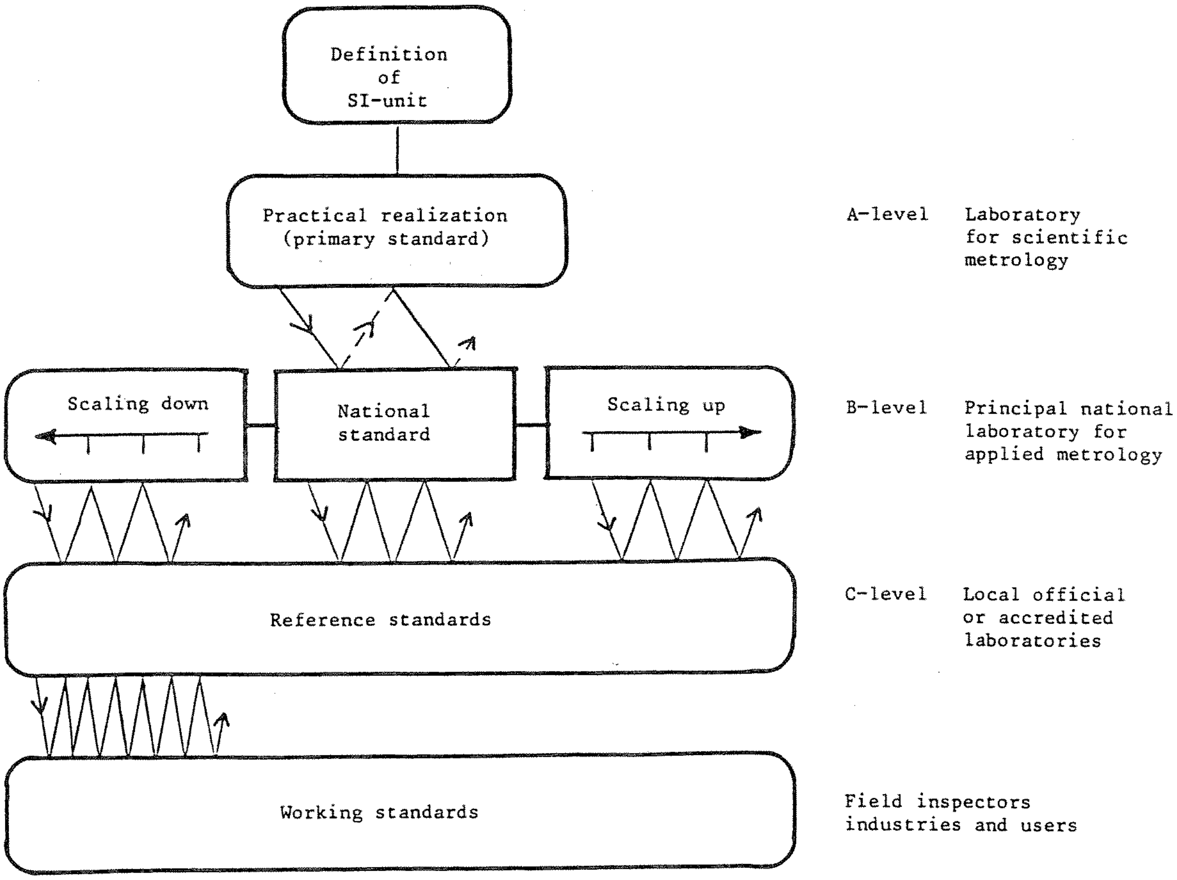

Fig.1 - Scheme of a national measurement system

For reasons of easy reference some reports on the organisation of metrology have used the following designations of metrology laboratories.

A-level - A scientific laboratory which has realized practically and maintains the relevant SI-unit in accordance with the resolutions of the General Conference of Weights and Measures (CGPM) and which calibrates other standards including national secondary standards in terms of this practical realization considered as the primary standard of the country.*

B-level - A central laboratory for applied metrology which generally maintains the country's secondary standards in the field of measurement concerned and which carries out the calibration of reference standards and reference instrumentation used by the other laboratories in the country.

C-level - Local laboratories capable of calibrating working standards used for direct verification in the field of common instruments. The C-level may include accredited industrial and user laboratories.

A diagram of the relation between these designations is shown in Fig.1. Note that in the diagram the qualifying term "primary standard" has been put between brackets as it usually has a relative meaning depending on countries. From the scientific point of view the primary standard is in fact that which under the best possible conditions can be directly realized using the definition of the corresponding unit of measurement. In a country which does not have a scientific metrology (A-level) laboratory the word "primary" may in common language for local purposes designate a national standard which from the international and scientific point of view would be considered as a secondary standard.

The administrative responsibility for the national units of measurement is frequently distributed over different laboratories according to main activities (mechanics, electricity, ionizing radiation etc.). A diagram like Fig.1 consequently applies individually to each unit or to a group of associated units.

Furthermore one finds that the A- and B-level laboratories are combined in several countries. In a small developing country there may be only one C-level laboratory which then is combined with the B-level facilities.

8. Calibration chains

Several highly industrialized countries have created so-called calibration services or calibration chains (from French : chaînes d'étalonnage) which, if A-level facilities exist, comprise schemes of A+B+C levels or, if this is not the case, only B+C level laboratories.

The aim of such chains is to ensure good traceability of manufactured instruments and other products or of measurements made for practical purposes.

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* For standards of mass, BIPM is according to this definition the only A-level laboratory though a number of countries have means for mass comparisons allowing practically the same repeatability as the main comparison balance of BIPM.

In such chains the critical links are frequently the C-level laboratories which usually have to be supervised through inspections by the official management of the chain. There is a quality promotion aspect behind the operation of these chains which may be of interest to developing and other countries when considering purchases of instruments and in particular of those which are not already subject to legal control in the country of importation. It may many times however not be sufficient to rely exclusively on the manufacturer's quality warranty even when it has been issued in the form of a certificate. Many instruments and in particular electronic ones may in fact change their characteristics during their shipment and their adjustment may change with time and climatic conditions. It is thus anyway necessary to provide for reliable local calibration (and adjustment) facilities in the country of importation covering a larger part of the practical activities than those usually covered by legal control.

A developing country must thus as a first step have a B-level laboratory with a minimum amount of integrated C-level facilities. This scheme will have to be progressively extended and reinforced as regards accuracy, sophistication and number of laboratory units in accordance with the practical needs of the country and available qualified staff and funds.

When a certain amount of these facilities are present it is necessary to consider schemes for their effective use to ensure traceability of measurements made in industries, analytical laboratories, hospitals etc.

One way of doing this would simply be to extend the amount of categories of instruments subject to legal control. This can usually be done for a certain number of items but will in many cases for the desired efficiency require a considerable amount of trained staff spending a great time and money on travels.

The alternative of creating a calibration chain copied on those existing in industrialized countries may seem more attractive especially from the financial point of view. There is however a risk that such a chain remains a paper exercise if the accredited laboratories, or the users of the working standards, do not have the appropriate training in metrology or simply do not dispose of sufficient time to correctly apply the calibration and adjustment operations.

For instruments and measurements which are not directly submitted to legal control it seems that a technically satisfying and economic compromise could still be found between the two described alternatives.

A country may for instance

- oblige or encourage, through lower taxes or customs duties, importers of instruments to maintain competent instrument repair and adjustment services which are subject to authorization and supervision by the B (+ C) level laboratory
- establish yearly calibration contracts between the B (+ C) level laboratory and users for individually designated instruments allowing planified calibration campaigns for the various categories and hence better use of staff and lower costs for both parties.

II - COMPONENTS OF A NATIONAL MEASUREMENT SYSTEM

We will below discuss the typical reference standards required in a National Measurement System and their links with the corresponding basic standards or definitions using facilities which may be built-up within a developing or small country with more or less calibration support from other countries.

Indications will in some cases be given as to how the scaling-up or scaling-down of the magnitude of the standards can take place on local level.

More detailed specifications of the standards and measuring equipment and their installation are given in the BIML brochures :

"Verification equipment for National Metrology Services" and
"Planning of metrology and testing laboratories".

A few fields of measurement not covered in the first brochure will for reasons of completeness be briefly summarized here regards the necessary verification equipment.

1. Length and dimensional measurements

1.1. Basic requirements

The national reference and associated facilities in the field of length measurements are mainly determined by the needs for calibration required by the local industries and possibly also for geodetic surveying.

The enforcement of regulations concerning trade does in fact usually only require reference standards of quite moderate accuracy and rather simple means for making the comparisons.

A small developing country which does not have a precision industry can thus start with procuring or constructing a simple comparator for visual comparison of measuring rules used in trade comprising a line standard divided in mm with a length of 1 to 5 m. Such a device with an accuracy of about ± 0.05 mm does not need recalibration as the stability of most metal alloys usually is sufficient provided of course that the rigidity of the construction is sufficient and not affected by its use.

An accuracy of line standards better than ± 0.05 mm is generally only required for the precision mechanical industry when using some form of measuring machine equipped with special optical projection or electronic read-out devices.

Industry does however make a more frequent use of end standards and many metrology services assisting industries may have to be equipped with reference sets of end standards and suitable comparison equipment.

1.2. National references for length measurements

It must be stressed that attention has to be paid to the fact that it is not easy to measure accurately line standards in terms of end standards or vice versa. The difficulty decreased when lasers and high-speed electronic interference fringe counting equipment became available and allowed the construction of length comparators operating on the principle of longitudinal displacement. Some of the national metrology laboratories are now equipped with such instruments.

The instrument installed at BIPM uses photo-electric microscopes and allows measurement of both end standards and line standards up to 1 m. The reference is a radiation from a He-Ne iodine stabilized laser which has been adopted by CGPM as a convenient practical means of realizing the new definition of the metre.

The majority of countries do however not have such facilities. A certain number of them use two different length references : one line standard (usually of H-section, length 500 or 1000 mm) and calibrated at BIPM and an interferometer together with a laser and other monochromatic sources for the measurement of end standards.

1.2.1. Precision line standards

It must be emphasized that high precision line standards of H-section (or of X-section when made from platinum-iridium alloy) cannot be used even for measurements of moderate accuracy unless a suitable measuring machine equipped with appropriate microscopes is available (the line width may in fact be as small as 5 µm). The use of such line standards for the verification of other measuring equipment used in industry is furthermore complicated by necessities for maintaining alignment, focussing and perpendicularity of the microscope that has to be installed on the machine to be tested.

Precision line standards made from special 56 % nickel steel may be very stable and show variations less than 0.5 µm per metre over a period of 25 years and do not need recalibration as long as the required accuracy is limited to approximately ± 1 µm.

This stability is usually also attained in several types of universal measuring machines of recent construction (Fig.2) which incorporate special material standards of length that cannot be sent to an official laboratory for calibration.

Such measuring machines can usually only be verified by external means such as a laser or, with much less accuracy, by use of additionally fitted microscopes and a precision line standard. They may have a digital display of the measured length and are usually convenient for measurements of various gauges and other mechanical parts. The accuracy of such measurements is generally limited to about ± 1 µm by the necessity for mechanical contact with the measured object which is detected by a special transducer. These machines can sometimes be fitted with additional external optical observation equipment (microscope of moderate resolution) so as to enable the verification of industrial and geodetic line standards to an accuracy of about ± 5 µm.

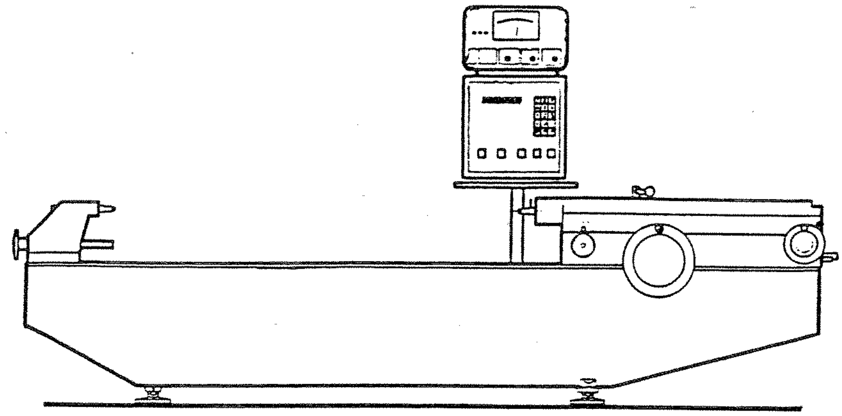

Fig.2 - Universal length measuring machine with incorporated line standard

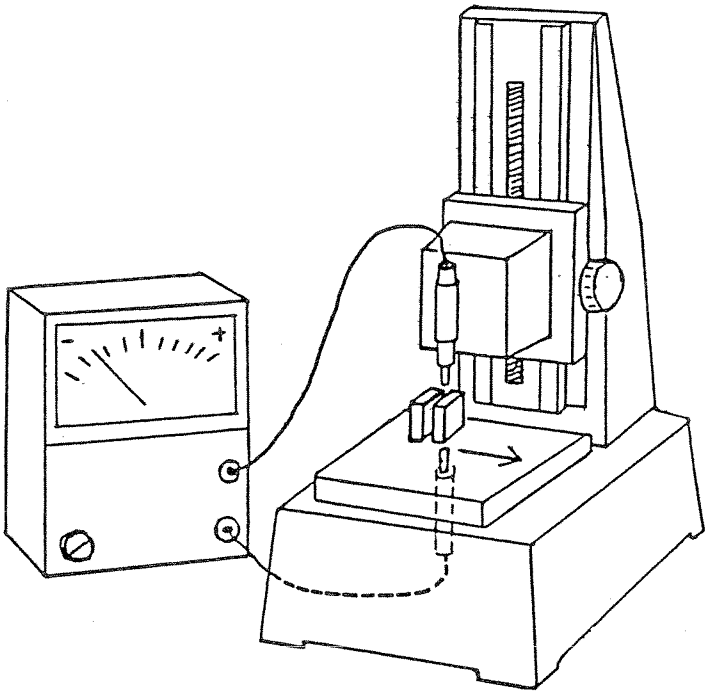

Fig.3 - Gauge block comparator using mechanical contact and two displacement transducers

1.2.2. End standards

Due to the small market most optical manufacturers have ceased to produce interferometers for gauge block measurements. A natural replacement would seem to be a fringe-counting apparatus adapted for the measurement of gauge blocks and using a stabilized laser, but commercialized versions are presently unknown.

There are however on the market a great number of comparators of gauge blocks using mechanical contact tips and equipped with inductive transducers though other means of sensing and amplification also exist (Fig. 3).

These types of comparator are quite easy to use and can be installed in laboratory rooms with less climatic requirements than for an interferometer but they require of course the use of a reference set of gauge blocks. Furthermore at least one second set of gauge blocks will have to be provided for current use so as to preserve the reference set from wear.

The primary reference set of gauge blocks has to be calibrated at an institute abroad having the necessary facilities. Previously it was usual to request such primary calibration only for some of the block sizes such as 10, 20, 25, 50, 75 and 100 mm. However, nowadays several of the national laboratories have automated equipment which allows calibration of most of the sizes at reasonable cost. If carefully preserved and not affected by corrosion and wear, recalibration of a primary set may only be required after 10 to 15 years and then be limited to a few of the biggest sizes to check for possible material changes.

Long end standards, in excess of 200 mm, usually require special measuring equipment. Reference standards of this kind are accepted for absolute calibration in terms of the definition of the meter by BIPM and a few national laboratories.

Long gauge blocks may also be compared by mechanical contact to other end standards in a universal measuring machine of the type shown in Fig. 2. The accuracy is then generally limited by alignment problems to about ± 2 µm (up to 1 m).

It should be stressed that calibration of length standards to accuracies better than 10 µm per metre requires careful temperature stabilization (the coefficient of expansion of steel is 11.10^{-6} per °C). A universal measuring machine must therefore be installed in specially climatized room (temperature gradients and variations less than 0.5 °C), see 1.3.

1.2.3. Measuring tapes

Temperature stability and gradients are also problems for accurate calibration of surveying tapes. These are normally supported on a bench made of stone or metal and compared to calibrated standard tapes. The great national metrology laboratories and geodetic institutes are provided with means for calibration of long lengths either by successive comparison to line standards of 4 m length or, more recently, using interferometry and laser sources.

The temperature stability requirements are however less stringent when two similar tapes are compared side by side as long as they have approximately the same coefficient of thermal expansion.

The requirements of standards for long length measurements in a small or developing country can therefore usually be satisfied by the use of a reference group of 3 calibrated standard steel tapes and a suitable bench of 20 (or 25) metres allowing the standards to be supported while submitting them to the force of stretching for which they have been calibrated (usually 50 N). Here again it may be appropriate to provide for a second set of tapes which is used for current calibrations.

The reference group of tapes should be recalibrated at 10 to 15 years intervals. As the cost of the tapes themselves usually is rather low one may use the method of uni-directional transfer by procurement of new tapes, however this method may lead to difficulties in interpretation in case the new standards show a significantly different result when compared locally to those of the old reference group.

If a line standard of the rigid type for one reason or another requires recalibration it is not necessary to redetermine the subdivisions but only the total length; the new positions of the subdivisions can in fact to be calculated by applying a simple rule of three. The same is not necessarily true for a tape measure which due to its flexible nature can have been subject to unequal stress in handling or rewinding. Recalibration for every meter may thus be appropriate.

The considerations above on suitable length standards are summarized in the Table 1.

Table 1 - LENGTH STANDARDS

Description	Nominal range	Adjustment accuracy	Procurement priority	Recalibration interval
Line standard in comparator for verification of length measures used in trade	1000 mm to 5000 mm	± 0.05 mm	1	uni-directional
Standard steel tapes for verification of measuring tapes	20 m	± (0.1 + 0.05 L) mm L in m	2	10 years
Set of calibrated gauge blocks	100 mm	OIML AA (ISO 00)	3	10 years
Gauge block comparator mechanical contact type	stand : 200 mm indicator : ± 1 to 10 µm	± 0.1 µm (resolution ± 0.01 µm)	3	-
Set of calibrated long gauge blocks 200 to 1000 mm *	1000 mm	OIML A (ISO 0)	4	10 years
Universal measuring machine with incorporated line standard *	1000 mm	± 1 µm	4	uni-directional

* requires special climatized laboratory.

1.3. Calibration temperatures

The standardized temperature for length measurements is 20 °C practically all over the world and certificates are normally established for this temperature only.

The temperature in practical use of a length measure may be sensibly different in particular in countries where the mean temperature usually is largely in excess of 20 °C. The International Organization for Standardization has provided for three values of standard temperature 20, 23 and 27 °C. The latter temperature may be representative for a number of subtropical countries but it is not practical, nor advisable, to consider this value for calibration rooms. The temperature of 23 °C is no doubt the best compromise for standards rooms even in hot countries and is nowadays largely used for electrical measurements.

Whenever a length measure is used at a different temperature than the reference temperature we must take into account its thermal expansion. It may thus be attractive to use for direct measurements in the field materials such as invar or fused silica which have negligeable thermal coefficients of expansion. This is still largely the case for determining the base lengths in geodetic surveying operations (triangulation) though electro-optical means nowadays tend to replace the classical methods. However these materials are hardly used for other practical measurements in particular in the precision industry. Invar is by the way not a very stable alloy for use as precision length standard.

The situation is slightly different in a length standards laboratory where the main activity is not to make direct practical measurements of length but to calibrate other length measures.

This goal can be reached by using standards which have a coefficient of thermal expansion which is as close as possible to that of the measures to be calibrated. The difference in temperature between the measure to be calibrated and that of the standard must however in all cases be small thus requiring good temperature uniformity and stability.

If this is realized, there will generally not be any problem in the choice of the exact value of the calibration temperature. This is the reason why a measuring machine should preferably have a line standard with the same coefficient of expansion as steel and reference gauge blocks to be made of steel rather than of other materials.

We can illustrate this discussion by considering for instance a measuring machine incorporating a line standard made from 56 % nickel steel and calibrated at 20 °C but which is used at a stable temperature of 23 °C. If the object measured, end measure or line standard, has the same coefficient of expansion there is no need for corrections. However if for instance the measure is made from stainless steel or brass the difference in thermal expansion coefficient will attain 5.10^{-6} per °C and in this case require a correction of 15 µm per metre.

2. Mass measurements

2.1. Basic requirements

In legal metrology the requirements for the mass standards are more exacting than for the other quantities.

In establishing the mass standards laboratory one must have in mind three distinct fields of application :

	Typical range
- heavy industrial weighing including weighbridges	50 kg to 50 t
- weighing instruments used for general trade	100 g to 50 kg
- weighing instruments used for pharmaceutical dispensing, chemical analysis and trade with precious metals	10 g to 5 kg

The first two categories concern instruments which belong to OIML class III with a typical value of n = Max/e = 2000 (maximum permissible error = e) and reaching exceptionally n = 5000 (maximum permissible error = 1.5 e). The relative uncertainty of the working standards used for verification should normally be at least 3 times smaller, i.e.

$$\frac{1}{3} . 1.5 . \frac{1}{5000} = 10^{-4}$$

It can be found that weights corresponding to OIML class M1 satisfy this requirement.

Weighing instruments used for pharmaceutical dispensing and chemical analysis belong to OIML class II with typical n-values of 20 000 but reaching sometimes 50 000 for modern analytical laboratory balances. The relative uncertainty of the working standards used for verification at full capacity of the latter must consequently be of the order of 10^{-5}. Usually OIML class F2 satisfies the requirements for in-service verification of such instruments. However examination of new analytical balances and similar current work in a central mass standards laboratory requires weights which are adjusted to OIML class F1 and have a relative uncertainty of 5.10^{-6} in the range of 100 g to 20 kg.

All sets of weights with denominations of 100 g and lower which are purchased for the verification of analytical balances should preferably be ordered as class F1 as these weights are not adjustable.They may in fact after some time in use become class F2.

2.2. The reference standards

Having defined in 2.1. the requirements for the working standards or standards used by inspector's we go a further step up to determine the accuracy of what we may call the reference standards which are used to calibrate the working standards and thus play a very important role.

In a small or a developing country there may only be one set of reference standards covering the full range of mass as required. In industrialized or geographically very extended countries there are usually several sets of reference standards located at subsidiary metrology laboratories all over the country. A simple scheme showing the interrelation or possible hierarchy of mass standards is shown in Fig. 4.

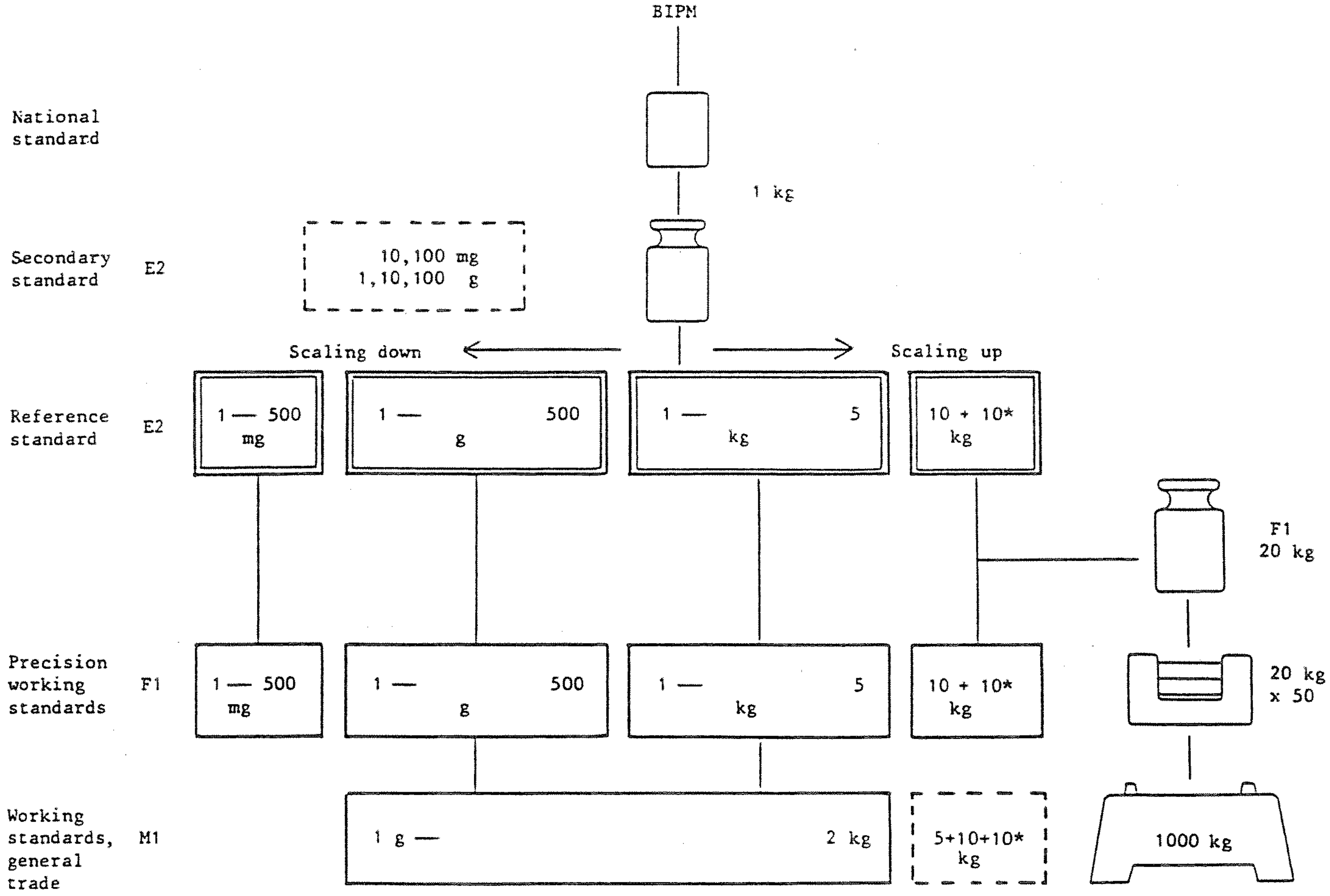

Fig.4 - A hierarchy scheme for mass standards

The reference set shall have adjustment tolerances which are at least three times lower than the best standards used for verification which are class F1. The adjustment of the reference set must therefore be that of class E2 as defined in OIML R 20. It is however necessary to determine the individual mass values of each weight with less uncertainty than their tolerance of adjustment.

The reference set or sets should therefore be calibrated at regular intervals to the national secondary standard using step-up and step-down procedures by group weighing as described in Appendix 1.

When reference sets are procured it is appropriate to obtain a full calibration from the national metrology institute in the country of manufacture. This initial calibration may in fact be used as a check of the correctness of the local calibration results. Typical uncertainties in the calibration of weights of class E2 and F1 are shown in Table 2.

Table 2 - Adjustment tolerances and typical uncertainties in calibration of precision weights

Nominal value	Class E_2		Class F_1	
	Tolerance	Uncertainty of measurement	Tolerance	Uncertainty of measurement
	mg	mg	mg	mg
50 kg	75	25	250	50
20 kg	30	10	100	20
10 kg	15	5	50	10
5 kg	7.5	2.5	25	5
2 kg	3.0	1.0	10	2
1 kg	1.5	0.5	5	1
500 g	0.75	0.25	2.5	0.5
200 g	0.30	0.10	1.0	0.2
100 g	0.15	0.05	0.5	0.1
50 g	0.10	0.03	0.30	0.05
20 g	0.080	0.02	0.25	0.02
10 g	0.060	0.01	0.20	0.01
5 g	0.050	0.01	0.15	0.01
2 g	0.040	0.005	0.12	0.01
1 g	0.030	0.005	0.10	0.01
500 mg	0.025	0.005	0.08	0.01
200 mg	0.020	0.003	0.06	0.01
100 mg	0.015	0.003	0.05	0.01
50 mg	0.012	0.003	0.04	0.005
20 mg	0.010	0.002	0.03	0.005
10 mg	0.008	0.002	0.025	0.005
5 mg	0.006	0.002	0.020	0.005
2 mg	0.006	0.002	0.020	0.005
1 mg	0.006	0.002	0.020	0.005

Note : The uncertainties of measurement are those stated by NPL, U.K. in the brochure NPL Measurement Services "Mass and Density".

The material of the reference set should preferably be a non-magnetic stainless steel of identical composition and density as that of the secondary and primary 1 kg standards, see below.

It is important that the composition of the reference set of weights is such that it can easily be calibrated by group weighing methods. This requires duplication of some of the designations.

The common series with the multiples 1, 2, 2*, 5 requires in this case one auxiliary weight (from another box) of multiple 1. The same holds for the series 1, 1*, 2, 5 supplied by some manufacturers. The series 1, 1*, 2, 2*, 5 would be the most suitable for group weighing as it needs no additional weight. It also allows ratio arm tests on precision two-armed balances.

Another simple and very convenient solution as illustrated in Fig. 4 is to procure a box containing auxiliary class E2 weights of the following designations : 10 mg, 100 mg, 1 g, 10 g, 100 g. These weights can easily be shipped and calibrated at low cost by a foreign national laboratory and thus enable an convenient control of the local group weighings. The use of two complete reference sets is of course also a solution. Similar weights must always be suitably identified to avoid confusions.

The duplication of the 10 kg weights in the reference set is in any case preferable to a 20 kg weight since manufacturers encounter difficulties in obtaining the special non-magnetic stainless steel with the diameter required for the 20 kg denomination. Furthermore comparison balances with a capacity of 10 kg usually enable to reach a lower relative uncertainty than those of higher capacity.

2.3. The secondary 1 kg standard

The secondary 1 kg standard is used to calibrate the reference set by group weighing as described in Appendix 1. It is usually in the form of a cylinder with integral knob for easy handling using a chamois leather or a special plastic-lined fork tool. It should be made from non-magnetic stainless steel and adjusted to class E2 (see Note) * but its exact value should be determined by comparison to the primary national kilogram with an uncertainty less than ± 0.5 mg.

2.4. The primary national mass standard

The primary standard has usually the form of a massive cylinder without knob and thus requiring a special handling tool.

Taking into account the accuracy requirements for the reference standards and for the secondary 1 kg standard one may estimate that

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* Note : It should be stressed that the class E1 in OIML Recommendation No. 20 (1975 version) is unrealistic as it is extremely difficult to adjust for instance 1 kg standards to the accuracy required of ± 0.5 mg. Furthermore class E1 if realized would require the use of a material which has a density between 7930 and 8070 kg/m^3.

the uncertainty of the original calibration of the primary 1 kg mass standard should be of the order of 1.10^{-7} or 0.1 mg or less.

Furthermore variations with time in between calibrations should not exceed this value, which by no means must be confused with a tolerance in the adjustment of the nominal value. Primary 1 kg standards are adjusted by surface polishing or etching and deviations in adjusted mass from the nominal value of up to ± 1 mg are currently accepted, as in any case the exact value obtained from the fundamental mass comparison has to be used when calibrating secondary standards.

A developing country may be tempted to procure a primary mass standard made of platinum-10 % iridium alloy and similar to the 1 kg international prototype preserved at BIPM. This would most certainly be a mistake as the use of such a standard on local level would require a very stable environment and barometer and hygrometer measurements so as to enable the determination of the air density during all the comparisons with the secondary standards which are usually made from stainless steel. There is in fact a difference in air buoyancy of about 94 mg between the two kinds of standards. Even if the target for the local comparison is only to reach a global uncertainty of 0.1 mg, the air density would have to be determined to better than 0.1 %.

All comparisons of mass standards are made in air even at BIPM and the uncertainty in air density corrections presents actually a limit to the accuracy which can be reached in practical mass measurements around the standardized density level of 8000 kg/m^3. This limit is presently of the order of 0.03 mg and is higher than that imposed by the stability of platinum-iridium standards in between comparisons made at BIPM at about 20 year intervals.

Most stability problems of primary mass standards seem to be associated with surface effects and cleaning and it has so far not been brought to evidence that the standards made from austenitic (and non-magnetic) stainless steel, though of larger surface, have stabilities in mass value inferior to those made from platinum-iridium alloy.

The initial calibration of the primary national kilogramme should normally take place at BIPM which will state the exact value of the standard to an uncertainty which is usually of the order of ± 0.05 mg for stainless steel. The value of the BIPM certificate is valid for the true density of the material which shall be stated in the request for calibration. When this value is not known BIPM may on special request exceptionally determine the volume of the standard.

The recalibration period for standard kilogrammes forwarded to BIPM is about 20 years. In countries disposing of a well equipped mass laboratory, comprising a very sensitive 1 kg comparator, it may be advisable to dispose of one additional kilogramme of identical shape and material for use as witness or reserve standard in case of possible accidents and to enable to check by comparisons before and after recalibration that there has been no significant change in mass during transport, cleaning or other handling of primary standard. This witness standard shall normally not be used for calibration of other secondary standards. However, for many developing countries the 1 kg secondary standard described in 2.3. is sufficient.

2.5. Calibration of standards used for verification

In order to reduce the number of steps in the hierarchy of mass standards illustrated by Fig. 4 and make best use of calibration equipment and staff it is generally advisable in a developing country to make all comparisons of standard weights used for verification exclusively in the national standards laboratory.

Such comparisons can nowadays be highly automated using electronic balances associated with printers. (This equipment requires, however installation in a room with good temperature stability and without air draught).

In this way it will not be necessary to equip local verification offices with comparison balances except as required for direct comparison or adjustment of weights used in general trade and which are similar to class 0.

The central laboratory will thus have to calibrate at regular intervals, for instance every 2 years, all the sets of weights used by its verification services. These sets will as mentioned in 2.1 be of class F1 for the verification of precision weights and class II balances and of class M1 for the verification of weights used in general trade and of other weighing equipment which is usually of class III.

These calibrations are made by comparison, directly or indirectly, to the corresponding reference weights. It may thus be advisable to group the work so that a large number of verification weight sets are calibrated, or rather verified, at the same time in order to reduce the amount of manipulation of the reference standards.

The calibration and assignment of conventionally true values to the sets of weights requires that the influence of air buoyancy is taken into account as explained in the following section.

2.6. Air buoyancy corrections

By the adoption of a primary kilogramme standard of stainless steel accurate measurements of air density are not necessary in the national laboratory but will be the matter of BIPM.

If the secondary standards, as we have supposed, are also made from stainless steel the values obtained in the national comparisons will be scientifically correct as they express true mass.

However, when adjusting and verifying weighing equipment used for trade we must take into account the now world-wide accepted convention laid down by OIML RI 33 that the weighing shall be based on an assumed density of the weights of 8000 kg/m^3 and a mean density of the surrounding air of 1.2 kg/m^3. It must in this connection be remembered that weighing equipment which does not make use of weights such as spring balances and the majority of electronic balances have to be adjusted using weights; the problem as regards air buoyancy is thus identical to that of other kinds of weighing instruments. *

The values obtained from the local comparisons of the secondary standards shall for current calibrations of other sets of weights used in trade, thus be recomputed using the following formula :

$$m'_s = m_s \frac{\left(1 - \frac{1.2}{\rho_s}\right)}{\left(1 - \frac{1.2}{8000}\right)} \approx m_s \left(1 + \frac{1.2}{8000} - \frac{1.2}{\rho_s}\right)$$

where m_s' is the value in units of mass to be assigned to a secondary standard of mass m_s and true density ρ_s.

Once this operation has been accomplished it is not necessary to apply further density corrections for the weights used for verification.

In fact, if the density of the air when currently using such weights is about the same as during their original calibration (or adjustment) the effect of air buoyancy will be the same in both cases and the corrections cancel each other.

In practice the density of ambiant air does usually not differ by more than 10 %.

For a working standard weight of brass with $\rho = 8400\ \mathrm{kg/m^3}$ this would then mean a relative difference in upthrust of

$$\frac{\Delta m'}{m'} = 0.1 \cdot 1.2 \cdot \left(\frac{1}{8000} - \frac{1}{8400}\right) = 7.10^{-7}$$

When comparing this small hypothetical difference to the adjustment accuracy of class F1 weights which is at best 5.10^{-6} (for 100 g and above), we see that the effect of air buoyancy can be ignored as regards the calibration of the weights used for verification.

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* Independently of the problem of buoyancy it is important to note that significant differences in the value of the acceleration due to gravity may require adjustments of electronic balances on their site of use when n is higher than 1000.

3. Volume of liquids

3.1. The unit "litre" and the uncertainty of measurements

In the very beginning of the metric system it was planned that 1 kg should be the mass of 1 dm^3 of pure water at its maximum density, i.e. at about + 4 °C. Afterwards when the international kilogramme prototype had been created it was however found that this was not exactly realized and it was decided that name "litre" should designate the volume of 1 kg of water at + 4 °C. For a long time i.e. practically from 1901 to 1964 the exact relation was thus

$$1 \text{ litre}_{(1901-1964)} = 1.000\ 028\ dm^3.$$

However the 12th General Conference on Weights and Measures in 1964 decided to set

$$1 \text{ litre} = 1.000\ 000\ dm^3 \text{ exactly.}$$

The latter modification was of no practical consequence as most measurements of liquid volume are affected by wetting problems in the standard measures which at that time currently set the relative uncertainty to about 0.01 %. In recent years standard measures are manufactured in which the wetting effect is decreased by the use of a teflon coating enabling a lower uncertainty for capacities above 1 L.

Another limit to the accuracy which may be obtained in gravimetric measurements of liquid volume is usually set by the relative uncertainty of the density of pure water which is presently about 1.10^{-5}

3.2. Basic legal requirements

The legally prescribed limit of error for capacity measures used in ordinary trade as well as for liquid flow meters is generally ± 0.5 % for volumes of 2 L and more. For capacity measures in the range of 100 mL to 1 L the tolerances are usually even higher, typically ± 1 % (glass measuring cylinders) and up to ± 3 % (serving measures).

It is thus usually sufficient that working standard measures of 1 L and more which are used in the field have uncertainties less than ± 0.1 %.

3.3. Choice of reference standards

Working standards are frequently calibrated by transfer of water from automatic standard pipettes (or burettes) in the range of 50 mL to 10 L, or exceptionally up to 50 L, completed by overflow (weir) standard measures for higher capacities. A set of automatic pipettes requires a fixed installation and is recommended only if there is a reasonably great demand for calibration. They also require a supply of demineralized or distilled water but even in this case there may, unless the installation is in frequent use, be problems with cleaning and development of algae.

One-mark standard flasks of glass according to OIML R 4 (ISO 1042 grade A) are available from well-known laboratory suppliers and may constitute a substitute for the automatic pipettes in the range of

10 mL to 2 L and are easily transportable to other laboratories. They are however usually calibrated "contained" (symbol In) and therefore need to be checked by the gravimetric method if used for delivery (symbol Ex) into another measure whereby it is necessary to fix the drainage time (1 minute or 30 seconds). The difference between the contained (In) and the delivered (Ex) capacity for such flasks made from glass may typically be of the order of 0.1 % of the capacity for a 1 L flask increasing to 0.25 % for a 50 mL flask, when using water.

Transportable conical stainless steel measures with well defined fixed capacity (by application of a glass plate) may be used as reference standards in the range of 5 to 20 L.

In small countries it may not be necessary to make use of reference volume measures (local standards) for capacities up to 20 L but to calibrate all the working standards (at six months intervals) at the central laboratory directly by the gravimetric method as shown in Fig. 5. The central laboratory will however generally need a fixed reference standard of 100 L.

3.4. Gravimetric calibration of standard measures

Calibration by weighing with water (or other liquids) of volume standards up to 20 L (or more) is rendered much easier by use of modern direct reading and top loading balances. The method can therefore be applied not only to reference standards such as automatic pipettes but also directly to working standards and in particular to laboratory glassware.

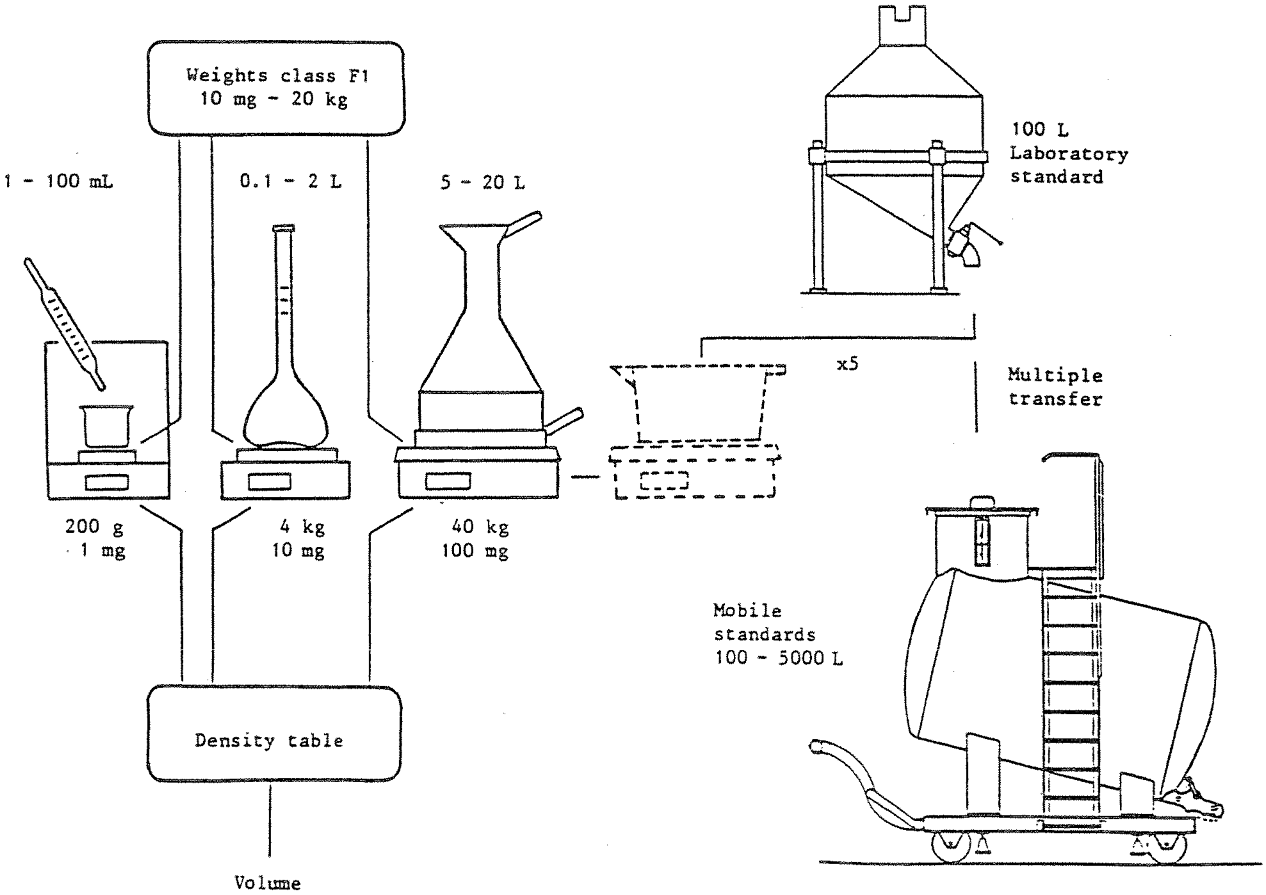

Fig.5 - Gravimetric calibration of liquid volume standards

The calibration must however take place in a draught-free thermally stable room and the balance must be calibrated initially using a set of class F1 weights. The temperature of the water used (and of the measure) shall be determined to about 0.1 °C.

The calibration can be done by weighing the standard empty and filled with demineralized water. An electronic top-loading balance with a capacity of 40 kg, a scale interval of 0.1 g, or if not available 1 g, and a sufficiently large loading platform will usually be suitable for gravimetric calibrations between 5 L and 20 L. Glassware may be calibrated using top-loading balances with lower capacities and a scale interval of 1 mg to 10 mg depending on the capacity.

The standard measures should be :
- weighed completely dry m_1
- filled with water, then fully emptied and drained for the specified drainage time or, if not specified, 30 seconds and then weighed again m_2
- filled with demineralized water, the temperature of which should measured, and then weighed m_3
- fully drained for the specified time or, if not specified, for 30 seconds and then weighed again m_4

The use of the values m_3 and m_1 will enable the calculation of the fully "contained" volume of the standard measure. However, if this measure is a brim or over-flow measure as is frequently the case, it will normally be used to transfer the liquid into a graduated working measure in which case the "delivered" volume applies. This can be obtained using in the calculations the weighing result m_3 from which the mean value $(m_2 + m_4)/2$ shall be subtracted.

The "delivered" volume can of course also be determined simply by weighing the delivered quantity collected by a non-calibrated container placed on the weigh pan of the balance. For reference measures made of metal or steel it may however be desirable to keep a record of the various calibrations through the years and the values of the "contained" volume will then provide better information about the influence of deformation whereas the "delivered" volume depends in addition on the cleanliness of the measure and more specifically on the nature of the liquid.

The computations must take into account the difference in buoyancy which results from weighing water in air using a balance which has been adjusted or calibrated with weights having a much higher density. If the calibration is not made at a temperature which is close to the reference temperature of the standard measure (usually 20 °C) it may also be necessary to apply a correction for the thermal expansion coefficient of the material of the measure.

The exact formula for calculating the volume at 20 °C temperature is thus

$$V_{20} = \frac{M}{\rho_W - \rho_A} \left(1 - \frac{\rho_A}{\rho_B} \right) \left(1 - \gamma \ (t - 20) \right)$$

where M is the mean difference in readings of the balance with and without water including corrections for balance errors

ρ_W is the density of water at the temperature t

ρ_A is the density of air

ρ_B is the density of the weights used to calibrate the balance

γ is the coefficient of cubical thermal expansion of the material of the measure.

The mass of the weights is adjusted on the basis of the conventional density, i.e. $\rho_B = 8000\ kg/m^3$. It will generally not be necessary to determine the true density of the air unless an uncertainty lower than ± 0.01 % is required. If the standardized conventional value of $\rho_A = 1.2\ kg/m^3$ is used the above formula may be written

$$V_{20} = \frac{999,85.M}{\rho_W - 1.2}\ [\ 1 - \gamma\ (t-20)]$$

where V_{20} is expressed in litres (L) and M in kg.

The values of ρ_W may be found in the table in Appendix 4.

For certain types of volumetric standards and in particular for delivery laboratory measures such as burettes and pipettes it is necessary to take into account the delivery time which should be specified and usually is rather long for calibration devices so as to reduce errors due to after-drainage. Details about the calibration of volumetric glassware can be found in the standard ISO 4787- Laboratory glassware - Methods for use and testing of capacity.

3.5. Calibration of standards above 20 L

Standard flasks made from steel or other metal will when certified at a foreign laboratory have to be recalibrated locally as they may have deformed with time or in transport. They are also subject to elastic deformation under the load of the liquid. The latter forms however part of the calibration as long as the liquids normally used do not vary in density.

In developing countries there may typically be a need for transportable working standards in the range of 100 to 1000 L or more which can be used for on-site verification in factories or at distribution centres for water or gasoline.

These working standards which require a relative accuracy of 1.10^{-3} or better can be calibrated by use of a 100 L reference standard having an accuracy of 1.10^{-4} and which should be available at the central laboratory. The direct gravimetric calibration of this reference standard would normally require a platform balance with a capacity of at least 200 kg and a scale interval and reproducibility of 10 g or better. Though such a balance may already be necessary for the calibration of heavy weights used for the verification of road weigh bridges, it may not prove convenient to adapt it for the calibration of the volume reference standards.

The method suggested in Fig. 5 is in this case more attractive. It uses successive weighing of smaller quantities of water, for instance about 20 L, on an electronic platform balance with a capacity of 40 kg, (or at least 30 kg) and a scale interval of preferably 0.1 g (or, if not available, 1 g).

It is convenient if the balance has taring facilities and is connected to a printer with a facility for adding and subtracting. It is of course necessary to avoid air draughts and any spillage of water in the operation. The collecting container may be equipped with a tap for easy and well defined drainage. Furthermore the balance must be calibrated at each volume determination by use of class F1 weights.

3.6. Calibration of flowmeters

Flowmeters for measurement of hydrocarbon liquids, water, milk etc are frequently used to determine a static volume and are therefore in many cases calibrated by use of volume measures and a start-stop method. Gravimetric methods using water are sometimes applied in specialized metrology laboratories. Larger quantities of liquids are measured by meters calibrated using fixed or mobile pipe provers. It should be emphasized that the indication of many types of meters are affected by the viscosity (and density) of the liquid. Calibrations of flowmeters must therefore preferably be done with the same or a similar liquid as that for which it is normally used. The liquid must furthermore not contain air or gas.

Verification of gasoline dispensers is usually done by 5 and 20 L working standards which can be easily handled and transported. A developing country is however also frequently faced with problems of verifying bulk-meters for measurements of volumes of more than 100 L. Meters used on tank trucks can in this case be verified to calibrated fixed volume standards installed at a test station in the fire-safe area of a hydrocarbon distribution centre.

The verification of meters installed at fixed locations or for aviation fuelling may require the use of a mobile unit which in its simplest form may consist of a trailer carrying a combination of a master meter and a volumetric standard measure.

Water meters are in most cases adjusted and sealed at the assembly or repair plant using a water meter test bench. Fixed reference standard volume measures from 100 to 1000 L can in these installations be connected to the test bench for calibrations. The legal metrology service will thus have to calibrate these measures using transportable working standard measures.

A water meter test station is sometimes installed in a legal metrology laboratory but it is preferable to carry out all the legal controls at the factory assembly plant or at the water distributors maintenance shop.

Occasional pattern approval according to OIML recommendation R 49 can when required probably also be undertaken at these workshops using mobile equipment calibrated at the central metrology laboratory.

3.7. Information on calibration of volume measuring instruments

The literature is extensive in this field. The basic methods are for instance described in the following book which also contains a bibliography :

Hayward, A.T.J. - Flowmeters, a basic guide and source-book for users, Macmillan Press, London 1975.

See also

- Defix A. - Mesurages des volumes de carburants et combustibles liquides, Publications de l'Institut français de pétrole, Editions Technip, Paris 1975
- Various papers in Techniques de l'Ingénieur - Mesure et Contrôle, volume R5, Paris
- Humphries J.W. - The calibration and verification of volumetric measures, Division of Applied Physics Technical Papers (1980) obtainable through the National Measurement Laboratory of Australia
- Papers published in the OIML Bulletin during 1987-1988 and which were presented at the OIML seminar on calibration of liquid volume measuring installations at Arles, France 1987.

4. Calibration of gas meters

The requirements for calibration of gas meters depend to a large extent on the management of the local gas distribution.

Domestic gas meters of the usual diaphragm type can simply be verified in the laboratory or on the site by another meter of higher quality used as working standard such as a standard drum-type gas meter (liquid-sealed test gas meter).

The reference working standard used by gas meter assembly and repair shops is usually a fixed air-liquid bell prover. These provers are initially calibrated in steps using successive transfer of air from a smaller bell-prover having a capacity of 200 to 500 dm^3 at maximum.

The latter can be calibrated by a special transfer standard comprising an immersion bottle of glass (capacity 50 L or less). The accuracy of a transfer standard of this type may reach ± 0.1 % but only if very careful attention is paid to temperature equilibrium problems during its calibration and use.

The necessity for procuring a transfer standard calibrated at a specially equipped official national laboratory or simply calibrated standard meters may be considered in cooperation with the gas distributors in the country concerned.

The testing of gas meters is described in detail in

- PTB Testing Instructions - Gas Volume Meters, 1982, and in
- Techniques de l'Ingénieur - Mesure et Contrôle vol R 5.

5. Density of liquids

Unless hydrometers are manufactured locally which may require suitable large scale certification equipment, hydrometer calibration is not likely to be very frequent except for cases where special legal verification schemes exist such as for alcoholometers. The latter may easily be compared in alcohol-water mixtures to standard density hydrometers using OIML alcoholometric tables.

5.1. Reference standards

The basic equipment may be composed of two sets of first grade laboratory standard hydrometers adjusted at 20 °C and delivered with calibration certificates from a recognized national laboratory.

Density range of the set	Range of each hydrometer	Subdivisions	Type
600 - 1000 kg/m^3	20 kg/m^3	0.2 kg/m^3	ISO 649 series L 20 Low surface tension
1000 - 2000 kg/m^3	"	"	ISO 649 series L 20 Medium surface tension

Recalibration of these hydrometers is not necessary but they must be carefully cleaned after use.

The comparison of other hydrometers or alcoholometers may take place using suitable liquids in a thermostated jacket of glass with flat high quality glass window connected to a refrigerated circulating thermostate with a stability of temperature setting 0.01 K, range - 10 to + 100 °C.

Details about the calibration and use are given in the ISO standard 649 - Density hydrometers for general purposes, part 1 Specification and part 2 Test methods and use.

Some practical hints :

- Hydrometers are usually fragile and break easily in transport, it is therefore recommended to procure at least two sets.Each hydrometer must be packed separately.
- The precision thermostat may be used also for other purposes.
- The equipment may conveniently be installed in the thermometer calibration laboratory.
- Some liquids used for calibration are very inflammable !!

5.2. Density determinations of liquids

Methods for the determination of the density of various liquids and semi-solid compounds are described in the BIML brochure :
Density Measurements - Guide for Inspectors, March 1987.

6. Force measurements

6.1. Basic requirements

A National Metrology Service may typically be required to make field calibrations of tensile strength testing machines in the range of 50 N to 600 kN (with an accuracy of 0.5 %) and of hydraulic presses (such as used for concrete tests) up to 2 MN with an accuracy of 2 % of the applied force.

Such calibrations are made by using dynamometers or strain gauge load cells with associated equipment. It should in this connection be borne in mind that the setting-up of dynamometers and load cells in tensile testing machines (when these cannot simply be tested in compression) will require the use of special fittings such as screw bars, ball joints etc and that some compression testing machines have quite reduced work space for accommodation of compression dynamometers which for capacities above 1 MN usually are bulky (and very heavy to handle). Whenever possible, it is therefore advised to make an inventory of the types of testing machines usually encountered in the country including their capacity and available work space including distance between jaws and platens. The dynamometers procured should only be used between about 20 % and 100 % of their nominal capacity, it is therefore important to adapt their capacity to be well in excess but not too much of the capacity of the machines to be calibrated.

In more special cases the service may also be asked to certify dynamometers or load cells in the central laboratory.

6.2. Calibration of working standards

The physical reference standards usually consist of loading machines with dead-weights of known mass used in conjonction with a determined value of the local acceleration due to gravity. Such machines are however, except for very low loads (e.g. up to 50 kN), extremely expensive and it is therefore advised whenever possible to take advantage of facilities existing in other national laboratories and simply procure suitable means for the transfer of the unit of force at the levels required.*

Transfer standards usually consist of compression dynamometers or strain-gauge load cells. The latter may be very accurate and reliable when employed with suitable bridges. However, if electronic direct read-out systems are used their reliability should be such that they can operate for at least two years without failure. Dynamometers with mechanical indication may for reasons of maintenance sometimes be preferred even though the resolution and convenience of use may be less.

The comparison of the dynamometers used in the field with the transfer standards may be done with moderate accuracy by substitution in a high-quality materials testing machine. Cascading of two compression

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* Weiler W., Sawla A. - Force standard machines of national institutes for metrology, report PTB-Me-22, PTB, Braunschweig, F.R. of Germany.

dynamometers in a press is usually not possible as alignment problems require the use of ball joints. Three identical dynamometers can anyway be compared to one of higher capacity in a good stability press according to the so-called pyramidal method.

An economical compromise consists of using an hydraulic comparator along the lines indicated in Fig. 6.

The most essential requirement on such a comparator is in addition to the minimum work-space that it shall be equipped with an hydraulic hand pump with very fine adjustment of the pressure and having very low leakage with time so as to allow more easily the simultaneous observations of the standard and the dynamometer tested.

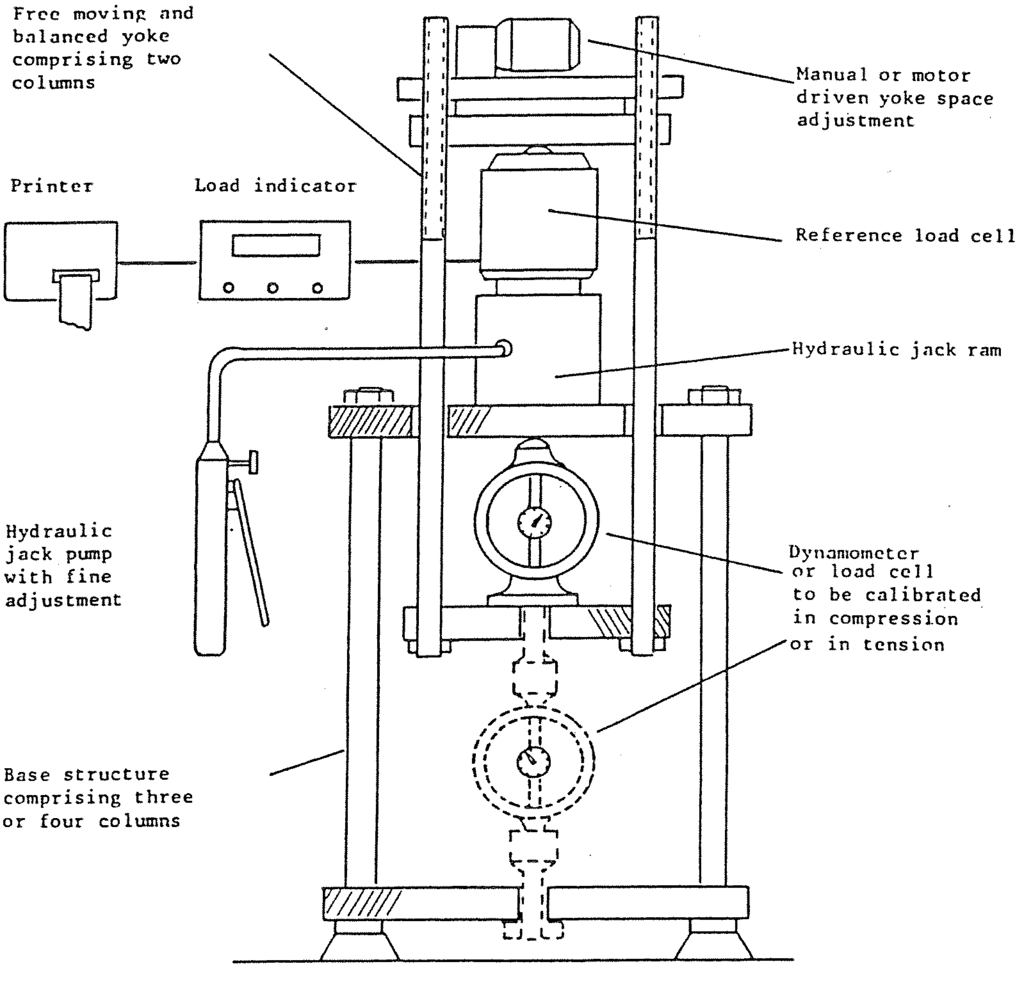

Fig. 6 - Principle of hydraulic dynamometer or load cell comparator

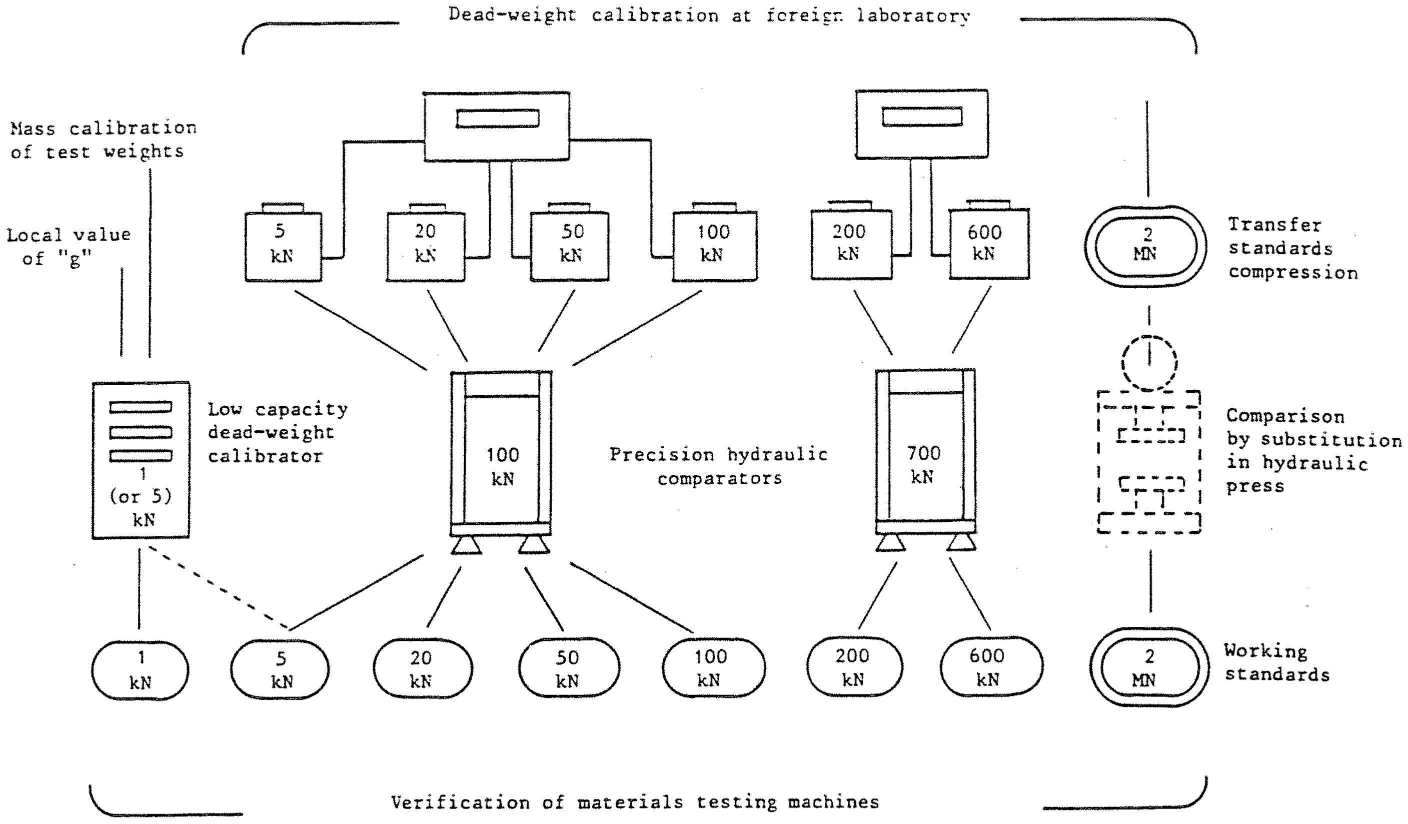

Fig. 7 - Simplified scheme for calibration of working standards for force measurement

In the synoptic diagramme in Fig. 7 we have indicated the use of two such comparators of moderate cost. For capacities above 600 kN it is considered that the field dynamometers are compared to the transfer standard by substitution in a suitable hydraulic press which does not need to be installed at the central metrology laboratory but should be selected for its good pressure adjustment facilities and good resolution of the readings of its force indicator.

6.3. Recalibration of the transfer standards

There is no doubt that force standards are presently most embarassing items as regards necessity for recalibration on account of their sensitivity to overloads, or other bad handling, and heavy weight as well as the need for verification of the accompaning electronic strain indicator in the case of load cells.

A typical recalibration period for working standards not suspected to have been overloaded is usually one year or less depending on their practical use in the field.

In a country where the amount of force calibration cannot economically justify the procurement (and housing) of standard dead-weight machines, it is necessary to make use of transfer standards. It is then preferable to make a standing arrangement with a foreign laboratory for regular recalibration of these transfer standards at intervals not exceeding two years.

The foreign laboratory equipped with dead-weight machines which is chosen for such an arrangement should preferably be located as closely as possible so as to reduce the cost of transport of this rather heavy equipment. Such recalibration schemes could typically be subject to regional cooperation arrangements.

The transfer standards should have repeatability errors less than 0.03 % and a warranted stability of calibration of 0.1 % for a period of 2 years. If hydraulic comparators similar to Fig. 6 cannot be procured or constructed it will also be necessary to procure a set of tension dynamometers or tension load cells to be used as transfer standards within the load range determined by the testing machine inventory.

The procurement of duplicate (or triplicate) series of reference standards identical to the transfer standards would be attractive from the point of view of ensuring internal control of their performance but this may prove to be too expensive.

It may however be advisable to procure at least two sets of dial indicator working standards whereby one set is normally preserved in the central laboratory for regular comparisons with the transfer standard.

6.4. Temperature influence on calibration of dynamometers

Dynamometers are generally calibrated for an ambient temperature of 20 °C.

If the temperature of use is comprised between 15 °C and 25 °C there is generally no need for temperature corrections. In other cases and for dynamometers made from low-alloyed steel the following correction formula may be applied

$$x_{20} = x_t \; [\; 1 - 0.00027\;(t - 20)\;]$$

where

x_{20} = calculated indication at 20 °C

x_t = measured indication (dial gauge difference with respect to the reading at zero force) at the temperature t

t = actual temperature of the dynamometer in °C.

The calculated indication x_{20} is then used with the calibration table of the dynamometer to find the true value of the force.

6.5. Calibration of materials testing machines

Though procedures for verification in the field are generally not treated in this brochure and may have to be adapted case by case we will below give some general indications in the case of materials testing machines.

It is first necessary to check that the machine operates properly and smothly and that in particular all mechanical parts associated with indicating or recording devices are not jammed, subject to excessive friction or other visible defects.

When the dynamometer which is used as working standard has been properly installed, load to maximum load several times before starting measurements. Make thereafter certain that the testing machine is set to zero and that the dial indicator of the dynamometer is set to the zero setting indicated in its certificate.

Select 4 to 5 points on the testing machine which should be calibrated, preferably corresponding to loads at which the working standard dynamometer has been calibrated.Increase slowly the load constantly in one direction. When the indication of the testing machine passes the choosen scale point, read the dial indicator of the dynamometer. On account of the instability of most hydraulic machines two observers will usually be necessary whereby one observes the machine indicator and announces to the other the crossing of the chosen scale points. At least 3 independant series of readings should be taken for each calibrated range of the machine. Between each series the working standard shall be repositioned and if possible rotated in a symmetric manner by 120 or 180 °.

So-called two-station universal testing machines need only to be calibrated in compression. One-station machines in which both tensile and compression tests are made in the same space, usually have electronic force measuring devices which have to be calibrated both in tensile and compression as their indication may not be symmetric.

The true force values are easily found by comparing the mean values of the readings with the dynamometer certificate using streight-forward interpolation. The maximum deviations between dial indicator readings for the same measuring point of the machine enable an estimation of the repeatability of the calibration (including the repeatability of the machine indications).

Testing machines (or dynamometers) which are used with both increasing and decreasing force (especially tensile testing machines) should be controlled for hysterisis (or backlash) in addition to the normal calibration with steadily increasing load. Hydraulic presses used for instance for testing building materials do not need to be tested for reversal errors.

6.6. Expression of errors of force measuring instruments

It is important to emphasize that errors of dynamometers, load cells or material testing machine indicators when expressed in % are relative errors which refer to the (conventionally) true value of the applied force and not to full scale as is the practice for electrical instruments (so-called fiducial errors).

To avoid confusions calibration certificates for tensile machines should as a rule be established by stating the (conventionally) true value in units of force (N or kN) for each measured point of the machine. The precision (repeatability) as well as the global uncertainty of the calibration can also be expressed in units of force.

7. Hardness measurements

Hardness testing machines can in principle be inspected as regards applied load and indenter measurements by using the equipment foreseen for force and dimensional metrology. However, it is much easier to do such checks using standardized hardness blocks, i.e. hardness blocks which have been calibrated in official or accredited laboratories using specially constructed machines with very carefully determined characteristics of the indenter. Such machines are however generally not needed unless standardized hardness blocks are to be specially manufactured in the country.

As a start it is thus suggested simply to procure sets of standardized hardness blocks and if required a few laboratory type hardness testing machines for Vickers and Rockwell B and C tests.

Calibration of hardness measuring machines by the metrology service is in most cases limited to those for steel and metals. We may thus suggest the procurement of the following blocks :

Standardized hardness blocks for verification of Brinell hardness testing machines under standardized load conditions for steel $F/D^2 = 30$ and in accordance with OIML R 9 and ISO 156 :

10 of hardness comprised between	100 and 200 HB
10 " " "	250 and 350 HB

Standardized hardness blocks for verification of Vickers type hardness testing machines for a load of 294.2 N (30 kgf) and in accordance with OIML R 10 and ISO 146 :

5 of hardness comprised between	100 and 200 HV30
5 " " "	250 and 350 HV30
5 " " "	600 and 750 HV30

Standardized hardness blocks for verification of Rockwell hardness testing machines for steel (Rockwell C), according to OIML R 12 and ISO 716 :

10 of hardness comprised between	20 and 30 HRC
10 " " "	35 and 55 HRC
10 " " "	59 and 65 HRC

Standardized hardness blocks for verification of Rockwell hardness testing machines for metals (Rockwell B), according to OIML R 11 and ISO 716 :

5 of hardness comprised between	40 and 60 HRB
5 " " "	80 and 100 HRB

All the blocks shall for each type be supplied by the same manufacturer.

Hardness blocks certified by national laboratories can generally be obtained through manufacturers of hardness testing machines.

The methods of verification of hardness testing machines are described in

OIML recommendations No. 37, 38 and 39. Consult also the following BIML brochures written by F. Petik :

- The metrology of hardness scales - Bibliography 1981
- Factors influencing hardness measurement, 1983
- Hardness blocks and indenters, 1984

8. Pressure measurements

8.1. Basic requirements

The current needs for verification of pressure measuring instruments cover mainly

- barometers used by meteorological and civil aviation services
- pressure gauges used for supervision of mechanical safety (boilers and other pressure vessels), tire pressure gauges
- pressure gauges used for the measurement of characteristics of materials (quality control) and in the oil industry.

Many pressure gauges used in industrial production processes are however usually not subject to verification.

The facilities for calibrations to be provided by national metrology services should be planned consequently. Details for moderately costful verification equipment are given in the BIML brochure : Verification Equipment for National Metrology Services.

8.2. Reference standards

The reference standards usually consist of a standard mercury manobarometer and a set of standard piston dead-weight pressure gauges also called pressure balances.

These reference standards should be delivered with calibration certificates from a recognized national laboratory.

They do however generally not need recalibration abroad but procedures should be established for on-site control of their performance.

These controls may for a mercury barometer simply consist in checking the vacuum, the cleanness of the mercury meniscus and of the glass tube followed when necessary by cleaning of the tube and replacement of the mercury. Facilities for filling the barometer must thus be available especially as it may not prove convenient to ensure a safe transportation of a filled barometer over long distances.

For piston manometers the verification may essentially consist first of an initial control that the mass of each weight (and of the piston if demountable) corresponds to the value indicated in the calibration certificate. These mass determinations should be repeated after a few years of use as well as replacement of the fluid used and cleaning of moving parts.

8.3. Influence of the acceleration due to gravity

Certificates for mercury barometers and for pressure balances will usually be based on a conventional value of the acceleration due to gravity sometimes called "standard gravity" the value of which is exactly $g_n = 9.806\ 65\ m/s^2$.

In order to use this barometer or pressure balance at a location where the acceleration due to gravity is g_L it will be necessary to multiply all the values in the certificate by the ratio g_L/g_n.

The acceleration due to gravity varies with latitude and altitude to an important extent and it is therefore necessary to know its value for the location of the barometer or pressure balance with an accuracy of at least 10^{-4} in relative value.

If it is not possible to undertake absolute measurements of the acceleration due to gravity in the country or to arrange for a liaison by gravimeter with foreign gravity stations the following formulae may be used to estimate the roughly local value (to within a few parts in 10^{-4}) :

$$g_L = 9.780\ 318\ (1+0.005\ 3024 \sin^2 \varphi - 0.000\ 0059 \sin^2 2\varphi) - 2.10^{-6}.H$$

where φ is the latitude and H the altitude in m with respect to mean sea level. (For more details see Thulin S.A. - The local value of g, Bulletin de l'OIML No.94, mars 1984, p.23-26).

8.4. Information about calibration of pressure measuring instruments

The precautions for handling and maintenance of mercury barometers and piston pressure balances as well as descriptions of their calibration and practical use including the necessary corrections are given in several available brochures, in particular :

- Measurement Services : Pressure and Vacuum by the National Physical Laboratory, U.K., 1984
- The Pressure Balance - A Practical Guide to its Use by S. Lewis and G.N. Peggs, National Physical Laboratory, U.K., 1979
- Liquid Manometers - PTB Testing Instructions, Physikalisch-Technische Bundesanstalt, Braunschweig 1980
  exists also in German and Spanish languages.

See also
- OIML draft (SP 11-Sr 7) Barometers
  and
- OIML draft (SP 11-Sr 3) Pressure Balances - General technical requirements and verification methods

9. Humidity measurements

9.1. Reference methods

The basic reference methods for calibrating humidity measuring instruments are mainly

A. The determination of the humidity of a material by weighing before and after drying (by heat or vacuum)

B. The production of humidity from known amounts of dry matter and fully absorbed water.

The former method is the primary standard method for all humidity measurements. It is also commonly used for checking hygrometers for materials such as grain, wood etc. The equipment then required consists of

- an analytical balance capacity 100 g (or more), scale interval 0.1 mg
- a drying oven with precision adjustment of temperature to ± 1 °C temperature range up to 150 °C
- flat containers with tight covers in glass or non-corroding metal
- dissicator (glass jar).

The accuracy of instruments measuring the humidity of solid materials depends to a large extent on the material itself and the operating procedure for their verification will therefore also have to be adapted consequently. See for instance OIML R 59 Moisture meters for cereal grains and oilseeds and OIML R 92 Wood moisture meters.

Air hygrometers of various types can in principle be calibrated by the method B using a humidity generator which produces a mixture of dry and water vapour saturated air at known volumes, temperature and pressure. Such an apparatus is, however, in most cases associated with a dew point hygrometer which is used as a transfer standard calibrated at a laboratory having gravimetric facilities according to method A.

9.2. Use of hygrostats for calibration of air hygrometers

As commercial versions of high precision humidity generators are scarce it is generally easier to make calibrations of air hygrometers using a salt hygrostat composed of either simply a small glass bell jar or dissicator or better a larger metal, glass or plastic air-tight cabinet provided with internal air circulation or air stirring and a free-standing tray containing water saturated solutions of certain salts.

A compilation of data for such salts has been made by Greenspan (see literature below). A selection of these data and descriptions of hygrostats are included in a draft by the OIML reporting secretariat SP 30-Sr 3 on a scale of relative humidity.

As examples the following currently available salts can be used :

	Relative humidity over the saturated solution at 20 °C
Potassium sulphate	97 %
Sodium chloride	75.5 %
Magnesium chloride	33 %
Lithium chloride	12 %

The accuracy attained depends to a large extent on the uniformity of temperature in the test cabinet. For the salts indicated above the uncertainty in the calibration of air hygrometers can usually be expected to remain within ± 1 %.

Salt hygrostats may be obtained commercially. Though such hygrostats are quite practical for the calibration of hair hygrometers they may not always be suitable for some hygrometers using chemical sensors (such as those employing lithium chloride layers).

If a climatic cabinet is procured for general testing purposes air hygrometers may also simply be checked to a good Assman type psychrometer (wet and dry bulb hygrometer) having certified thermometers or still better to a dew point mirror hygrometer. Several firms have in recent years put on the market electronic versions of such instruments which use Peltier effect (semi-conductor) cooling of the mirror and photoelectric or ultrasonic detection of the appearance of condensation or frost on the mirror. Such dew-point hygrometers are expensive but probably the most accurate reference instruments for direct measurement of the humidity of air. They should however preferably be certified by a national metrology laboratory having absolute calibration facilities.

9.3. Literature on air hygrometry

The following publications may be consulted for information concerning air hygrometry and calibration of hygrometers :

- Wexler A. and Brombacher W.G. - Methods of measuring humidity and testing hygrometers,
  NBS Circular 512 (1951) (also included in NBS Special Publication 300, Vol.8)
- Hickman M.J. - Measurement of humidity,
  NPL Notes on Applied Science No.4, HMS Stationary Office (1958)
- Young J.F. - Humidity control in the laboratory using salt solutions, a review
  Journal of Applied Chemistry , Vol.17, Sept.1967, p. 241-244
- Greenspan L. - Humidity fixed points of binary saturated aqueous solutions,
  Journal of Research of NBS, Vol. 81 A, No.1, Jan-Feb.1977, p.89-96
- ISO/R 483 - Plastics - Methods for maintaining constant relative humidity in small enclosures by means of aqueous solutions, 1966
- IEC Publication 260 - Test enclosures of non-injection type for constant relative humidity, 1968

10. Temperature measurements

10.1. Requirements

A legal metrology service may currently be requested to make verifications of temperature and calibrations of thermometers in the range of - 50 to + 250 °C. The needs of industry may also require calibration of thermocouples up to 1200 °C and less frequently the service may be requested to verify optical disappearing filament pyrometers. Medical thermometers are also frequently subject to legal control before sale.

It is necessary to stress that many temperature control instruments used in industry cannot be correctly calibrated in a laboratory unless all the conditions of their practical use are realized and it is therefore difficult to give a legal character to such calibrations.

The metrology service may however occasionally be requested to make temperature verifications on the site using its own instrumentation (liquid-in-glass or resistance thermometers, thermocouples or optical pyrometers).

10.2. Reference instrumentation

The International Temperature Scale of 1990 is based on the use of a certain number of fixed point temperatures (freezing points and triple points) which can be realized in the laboratory.

The following interpolation instruments are used between the listed fixed points of the ITS-1990 (see Note)

- platinum resistance thermometers between 13.81 K and 962 °C (the latter value is the freezing point of silver)
- radiation pyrometer according to Planck's law for temperatures over 962 °C.

The basic references in a developing country may typically consist of three long stem standard 25 ohm platinum resistance thermometers which can be used between - 200 °C and + 660 °C. (Lower and higher temperature ranges may be reached by special versions of platinum resistance thermometers). A set of three platinum to platinum/10 % rhodium thermocouples may constitute a secondary standard to cover the range of + 300 to 1200 °C.

There are automatic bridges for resistance thermometers which measure very accurately resistance ratios with respect to a standard resistor and are convenient to use. In a similar way a precision digital voltmeter may be used for measurements with thermocouples

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Note : The differences between the new scale ITS-90 and the previous scale IPTS-68 are as follows

at 20 °C	- 0.005 °C	and at 600 °C	- 0.1 °C
100	- 0.026	800	+ 0.3
200	- 0.04	1000	- 0.2

provided that it can be frequently calibrated using a passive device such as a potentiometer. A simplified equipment scheme is shown in Fig. 8.

10.3. Required fixed points

The need for fixed points depends much on the extent to which the laboratory desires to be independant of calibration services abroad. It should be emphasized that the fundamental fixed points generally require great care in their realization and maintenance.

A suitable ice point is in any case necessary for current use with the resistance thermometers, thermocouples and other thermometers. For a resistance thermometer the resistance at zero degree (ice resistance R_o) may drift with time without affecting the resistance ratios (R_t/R_o).

Calibration tables are therefore usually established as a function of a resistance ratio. A standard resistance thermometer used in connection with an ice point allows an accuracy as good as 0.01 °C to be reached within the range of - 50 to + 200 °C. Water triple point cells which define the + 0.01 °C point, allow accuracies better than 0.001 °C but they are more elaborate to prepare for use.

The accuracy which can be attained when using the reference resistance thermometer for calibrations will anyway be severely affected by the properties of the thermometer to be calibrated, therefore the accuracy of 0.01 °C at room temperature for the basic standard may for a metrology service constitute a realistic target.

A few national laboratories have developed secondary fixed points in the form of sealed cells which are commercially available and may be convenient to use for controlling the performance of resistance and other thermometers.

They can generally be directly used when placed in thermally controlled liquid baths or furnaces.

Among the materials used in these secondary points the following can be suggested as they cover the most frequently used range of measurement :

mercury (triple point)	- 38.83 °C	(Values expressed in ITS-90
gallium (melting point)	29.765 °C	to be confirmed by cell
indium (freezing point)	156.60 °C	supplying laboratory)

If such cells are available for calibration controls there may be no need for recalibration of the resistance thermometers at other laboratories.

10.4. Calibration of thermocouples

The thermocouples of platinum-platinum/10 % rhodium used as secondary standards may suffer at the highest temperatures if they are brought in contact with other metals or silica. They are generally mounted for use in twin bore tubes of pure aluminium oxide of length up to 500 mm.

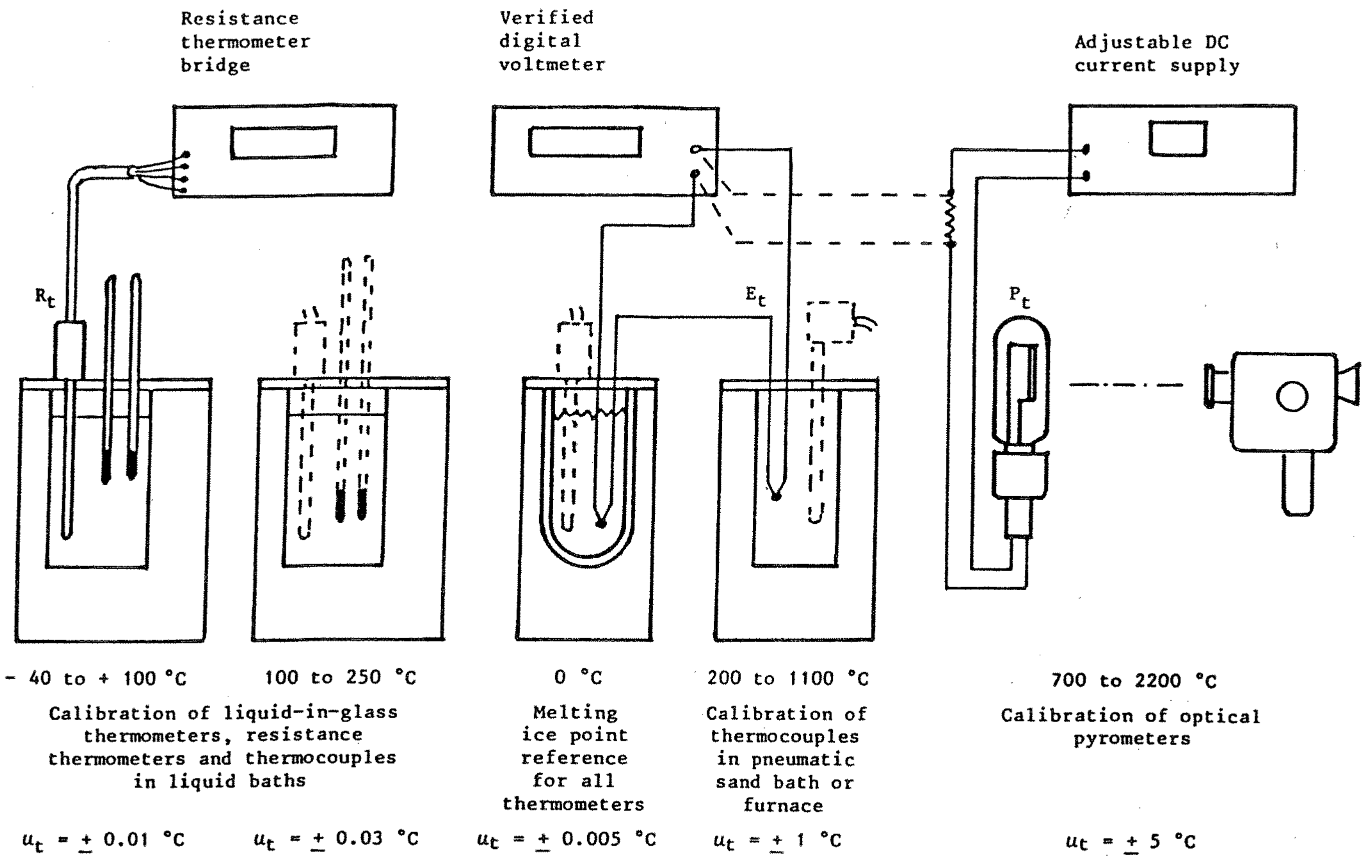

Fig. 8 - Scheme for calibration of commonly used temperature measuring devices.

Typical uncertainty u_t stated with respect to ITS.
Standards calibrated at an A-level laboratory :
R_t = platinum resistance thermometer
E_t = platinum-10% rhodium platinum thermocouple
P_t = tungsten ribbon lamp

For a developing country standard thermocouples may preferably be calibrated by unidirectional transfer i.e. they are not recalibrated but replaced by new thermocouples calibrated in a foreign laboratory at suitable reference points of the ITS.

Detailed specifications about the calibration equipment for thermometers operating by contact methods are given in the BIML brochure "Verification Equipment for Legal Metrology Services".

10.5. Radiation pyrometry

As the above-mentioned brochure does not include equipment for the verification of optical pyrometers it may be appropriate for reasons of completeness to deal briefly with possible requirements in this field.

Partial or total radiation pyrometers are extensively used in industry for process control. The calibration at a central laboratory of the output of such instruments as a function of temperature does most times not have much meaning as the true temperature will in practice greatly depend on the emissivity factor of the radiant surface and of reflexions from other thermal sources on that surface.

Disappearing filament optical pyrometers are less influenced by the emissivity factor and can generally be used for temperature measurements subject to corrections. They are much used for spot checks of material or furnace temperatures whenever approximate black-body conditions can be simulated through use of cavities and screens. For these reasons and with the main scope of maintaining reproducibility in high temperature measurements optical pyrometers are frequently calibrated by national metrology laboratories.

This calibration can take place by using a special furnace simulating a black-body in which case the temperature has to be measured and controlled by standard thermocouples. A special optical pyrometer such as used for comparison of black-body temperatures can also be used for interpolation purposes as provided by the ITS.

Disappearing filament optical pyrometers are however most frequently calibrated against tungsten ribbon lamps which are used as transfer standards. The fact that the tungsten ribbon in these lamps has an emissivity factor as low as 0.4 does not effect the transfer to the true temperature scale as long as the pyrometers to be calibrated all have an effective wavelength near 655 nm which is a standardized value for disappearing filament pyrometers and lamp calibrations.

The equipment for calibration of disappearing filament pyrometers is not very expensive and may simply consist of suitable calibrated tungsten ribbon lamps, a current regulated direct current supply and a current measuring equipment consisting of a shunt and a suitable digital voltmeter (Fig.8). As this equipment will generally not be in very frequent use the digital voltmeter may be the same as that used for thermocouple measurements. More detailed specifications are given below.

The tungsten ribbon lamps have tendency to change in calibrated values with time of use especially when run at high temperature. It is for this reason important that they are properly aged before calibration.

Calibrated ribbon lamps can generally be sent by air freight though carried as hand-luggage is preferable. Taking into account that their cost of procurement is in most cases low compared to the cost of calibration and transport they may instead of a recalibration be replaced after some years of use.

10.6. Suggested equipment specifications for verification of visual disappearing filament optical pyrometers

- Calibrated tungsten ribbon lamps and accessories for calibration of optical pyrometers consisting of

  3 Vacuum type for temperature range 700 to 1400 °C

  3 Gas-filled type for range 1300 to 2200 °C

  2 Special mounting ceramic sockets for the lamps

  The lamps shall be aged for 100 h at maximum operating temperature and furnished with calibration certificates with respect to current, established by an official national laboratory in terms of ITS.

- Direct current power supply stabilized in current to 0.01 %, maximum current 30 A (for a 1.5 mm wide filament), maximum voltage 50 V. Regulation by incorporated four-thermal precision shunt, short-circuit safety devices (when connected as voltage supply). Current (and voltage) controls to consist of at least two potentiometers, one preferably with multi-turn potentiometer so as to allow a current resolution of 1 mA.

- Two four-terminal shunts, accuracy ± 0.01 %, current ranges 10 A and 30 A. Output voltage preferably : 30 mV for 30 A and 100 mV for 10 A (so as to allow direct evaluation of current using a digital voltmeter connected to the potential leads).

- Digital voltmeter for general standards laboratory use and thermocouple measurements equipped with low-thermal binding posts and externally adjustable zero.
  5 full digits, highest resolution 1 µV (or better)
  Measurement ranges 0.1, 1, 10 and 100 V.
  Linearity and six months stability of calibration 0.005 % (or better). Adjustable easy calibration using external voltage standard (such as a Weston cell or potentiometer) without need of other specialized equipment.
  Preferably with four-terminal resistance measuring facility 10 MΩ to 0.01 Ω accuracy 0.01 % (for use on industrial resistance sensors). Insulated output for digital printer.

10.7. Bibliography on calibration of thermometers and radiation pyrometers

Some methods of test and calibration of thermometers and thermocouples are described in national standards (such as ASTM, BS, DIN, GOST, etc.) which it may be advisable to consult. The calibration of thermometers and in particular liquid-in-glass thermometers and thermocouples requires knowledge of their performance and shortcomings under various conditions. It is therefore advisable to consult the literature on these subjects out of which we give a few examples below :

BIPM - Supplementary information for ITS-90
BIML - Guide to practical temperature measurements

Rahlfs P., Blanke W. - Liquid-in-glass thermometers, PTB report (also available in German and Spanish)

Bliek L. - Principles of electric temperature measurement, PTB report E-13e, Braunschweig, July 1981

Barber C.R. - The calibration of thermometers, British Calibration Service Guidance Publication 5501

Coates P.B. - The design of a standards laboratory for thermometry NPL report QU 64, May 1982
National Physical Laboratory, Teddington, Middlesex, UK

Coates P.B. - Tungsten ribbon lamps for the calibration of disappearing filament pyrometers, NPL report QU 62, March 1981

NPL Measurement Services - Temperature, 1985

NBS Monograph 150 - Liquid-in-glass thermometry, 1976

NBS Circular 5906 - Methods of testing thermocouples and thermocouple materials, 1958

NBS Monograph 126 - Platinum resistance thermometry, 1973

NBS Monograph 41- - Theory and methods of optical pyrometry, 1962

The following publications are available from
CSIRO Division of Applied Physics
National Measurement Laboratory
P.O. Box 218
Lindfeld, N.S.W. 2070, Australia

- Techniques for the calibration of liquid-in-glass thermometers, by M. Beavis (1981)
- The melting point of ice as a reference temperature for mercury-in-glass thermometers, thermocouples and platinum resistance thermometers by M. Beavis (1981)
- Liquid-in-glass thermometers - care and use in measurement by M. Beavis (1982)

Consult also the following OIML International Recommendations :

R 7 Clinical thermometers (mercury-in-glass) (work is also undertaken by SP 12-Sr 7 on clinical electrical thermometers)

R 18 Optical pyrometers of the disappearing filament type

R 48 Tungsten ribbon lamps for calibration of optical pyrometers (work is also undertaken by SP 12-Sr 6 on total radiation pyrometers)

R 84 Resistance-thermometer sensors made of platinum, copper or nickel (for industrial and commercial use)

SP 12-Sr 9 (Draft) - Verification method for reference and ordinary PtRh10-Pt and PtRh13-Pt thermocouples

11. Electrical measurements

11.1. Basic requirements

In many countries only electrical energy meters and associated devices are subject to legal control.

In practice the local needs for calibration go frequently largely beyond this restricted field as many other testing laboratories, industries, universities and administrations require reliable national references for the various electrical units.

Digital electronic equipment of much higher resolution than previously is now commonly used which results in the necessity of having calibration equipment with increased accuracy.

If we leave aside the specific requirements for calibration within the high-frequency field which is usually taken care of by telecommunication administrations the necessities for calibration concern mainly :

- direct voltage and current
- resistance and capacitance
- alternating voltage current, power and energy at low frequency (mainly 50 or 60 Hz).

Specifications for calibration instrumentation for these quantities (with the exception of capacitance) are included in the BIML brochure "Verification equipment for national metrology services".

11.2. Selection of reference instrumentation

The schemes suggested in the brochure on equipment are intended to provide the national metrology service with a line of "passive" equipment which is reliable with time or which can easily be calibrated in terms of the electrical units maintained at the great national laboratories and at the BIPM.

At the same time the service may for convenience use a number of "active" electronic instruments to speed-up calibrations in particular if a large number of measurements have to be made.

A good practice is in fact to employ simultaneously at least two different types of instruments such as for instance a calibrated voltage source together with a precision digital voltmeter though strictly only one of these instruments would be needed. This form of duplication or reduncancy gives a good metrology assurance provided at least one of the instruments is checked regularily to the "passive" reference standards.

To counteract influence of equipment failures it is also advisable to procure duplicates of some of the electronic equipment. In this respect it is advisable to standardize upon certain types of electronic instruments (such as digital voltmeters) so as to enable easy interchanges in the laboratory or between laboratories.

The reference instrumentation should be chosen in such a way as to reduce as much as possible the necessity for calibration in foreign laboratories. This can be done by procuring whenever possible a type of equipment which only requires an initial calibration or verification i.e. uni-directional transfer. This is for instance the case for the winding ratios in capacitance bridges using ratio transformers and in the current comparators incorporated in certain patterns of resistance bridges and instrument transformer test sets.

Resistors have tendancy to change their values with time and therefore need recalibration especially if they have been accidentally overloaded. Direct current ratio devices such as voltage dividers (in the range of 1500 V to 1 V) and compensating potentiometers (for measurements of DC voltage below 2 V) should preferably have the main decades constituted by resistors of the same type of construction and material so as to ensure that the drift in the total resistance of the divider does not sensibly affect the ratios.

It should furthermore be ensured when making procurements of voltage dividers and potentiometers that they are so constructed as to facilitate calibration of the ratios by internal comparison of the various resistors or voltage steps (so-called auto-calibration).

11.3. The national standard for voltage

The reference for voltage measurements, and indirectly also for current measurements through use of four-terminal resistors, will in many countries be constituted by a group of standard Weston cells containing a saturated solution of $CdSO_4$ and maintained in a thermostated enclosure.

The temperature at which the cells are maintained in these enclosures is frequently 30 °C at which value the temperature coefficient is about 56 µV/°C. If it is desired to make use of the highest accuracy of the voltage standard it is thus important that the temperature inside the enclosure can be controlled to about ± 0.01 °C and furthermore that this temperature can be measured from time to time by an independant thermometer ensuring a reproducibility over several years of ± 0.02 °C or better. This thermometer will consequently have to be verified at regular intervals (for instance yearly).

A drift in the temperature control (or in the temperature measurement) can be a source of error greater than the instabilities of the standard cells and furthermore will not be detected by the usual intercomparisons of the cells contained in the same enclosure !

11.4. The transfer of the unit of voltage

The unit of voltage is basically obtained through scientific experiments referred to as absolute determinations involving measurement of forces. The various results of these determinations are discussed on international level within the Comité Consultatif d'Electricité and a mean value is established which is preserved through standards maintained at BIPM and at the great national laboratories. This mean value has several times been subject to changes in accordance with the scientific progress.* Whenever a voltage standard is calibrated at another laboratory than BIPM it should therefore be ensured that the certificate refers to the latest mean value maintained by BIPM and not to a national volt which for historical or other reasons may in some cases have a different value.

Several countries preserve the level of the volt at their national metrology laboratories by use of Josephson junctions for which the assigned value of the fundamental constant (2e/h) should be in line with the most recent decision of the Comité Consultatif d'Electricité and in principle based on the above-mentioned absolute determinations.

Other laboratories inside or outside these countries make use of travelling standards for the transfer of the voltage unit.

The travelling standards consist usually of a portable thermostated enclosure containing 4 saturated Weston cells. So-called electronic voltage standards are also available. Their principle of operation is frequently based on the use of specially selected semi-conducting Zener diodes placed in a thermostat or otherwise thermally compensated.

As the operating principles of these two main types of voltage transfer standards are very different it may at a distant location be advisable to make use of both types whereby the electronic standard may serve as a redundant control unit. In case of significant differences between the indication of the two types of standards, for instance in excess of 10 µV at the 1 V level as indicated by a high resolution digital voltmeter, it will then be necessary to arrange for a recalibration of one or both devices at a suitably equipped national laboratory.

The transportable thermostated enclosures have generally a stand-by battery for 24 h operation of the thermostat which as alreay mentioned is frequently set to + 30 °C. This temperature is rather low for transport through certain countries and the duration of travel may sometimes also be longer.

However even when the environment during transport is such that the thermostat cannot operate correctly, or is switched off, there will generally be no great harm to the in-use performance of the cells provided they are not subjected to very high (more than + 38 °C) or very low temperatures (less than + 10 °C). Far more important is the fact that the enclosure with the cells has to be transported upright and not submitted to shocks nor to vibration. It has thus to be hand-carried. Upon return in the laboratory the enclosure must be stabilized for a period of two weeks with its thermostat operating before making use of the values of the cells as indicated in the calibration certificate. After this period of stabilization in the laboratory the differences in voltage between the cells should be

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* Note : The representation of the volt which is supposed to be universally applied from 1 January 1990 is about 8.10^{-6} higher than the mean reference level established in 1972.

close to those calculated from the calibration certificate within limits of ± 2 to ± 3 μV at maximum.

An advantage with some of the electronic standards is that they can be sent by air-freight without need for being hand-carried provided of course they are not subject to excessively rough handling in loading and unloading of the shipments.

The procedure for comparing the standard cells is simple : If a sensitive null detector and suitable potentiometer is not available a digital voltmeter with a resolution of 1 μV or less may be used provided that in all cases care is taken as regards all contacts and binding posts which must be of copper or gold-plated but never nickel-plated because of the high thermal emfs which may develop. The cells are usually compared in opposition. Care should always be taken in the use of the cells that the external circuit has a sufficient resistance. Sometimes a protective series resistance may be necessary. Some types of electronic null detectors may in fact present a very low internal resistance when switched off.

In practice the comparison and calibration procedures depend on the equipment procured and it is necessary to study carefully diagrams and instructions supplied by the manufacturers. The mathematics for evaluating individual and group values of standard cells is straight forward and corresponds to the general procedure in Appendix 2A whereby the cell presenting the lowest voltage may preferably be chosen as the reference so as to obtain all the difference indications with the same (positive) sign on the measuring device.

The procedure described in Appendix 2B can also be applied but requires more time and is generally fitted for measurements on other types of standards where for instance no stable reference is available and when uncertainties in each comparison make it necessary to increase the number of observations.

The computation procedure for the calibration of a compensating potentiometer is explained in Appendix 3.

11.5. The national standard for resistance

The unit of resistance can indirectly be determined by use of calculable changes in capacitance with a lower relative uncertainty than for the unit of voltage.

The level of this unit can in a similar way as for the volt be preserved in a scientific laboratory by use of the quantum Hall effect as developed by von Klitzing. This however requires the adoption on international level of an agreed value of the constant R_H designating the quantized Hall resistance.

In a similar way as for the volt BIPM determines and preserves the mean value of recent determinations of the ohm. Presently the uncertainty of this mean value is estimated to 2 parts in 10^6.

The transfer of the unit of resistance from BIPM or from any of the major national laboratories is usually done by use of a very stable four-terminal 1 ohm resistor. The national reference group standard is frequently composed of three or more of these resistors of which one is used as travelling standard. They are made up of hermetically sealed coils of manganin wire and their stability when properly aged is better than 5.10^{-6} per year. They can generally be sent by air-freight or better in accompanied luggage.

The frequency of recalibration depends much on the stability and the accuracy required.

If the drift behaviour of the chosen type of standard is unknown it may be appropriate to arrange for recalibration after no more than 3 years from the procurement. The result then obtained may help to determine the suitable date for the next calibration.

The value of 1 ohm for the primary reference standard constitutes a suitable level for comparison of resistors and shunts used for industrial applications and in particular for power measurements using AC/DC transfer.

The scaling-down to lower resistance values can then be done by a current comparator bridge, a Kelvin bridge or simply by use of a stabilized current supply, a two-pole switch with low thermal emf and a digital voltmeter used as voltage comparator.

If a current comparator bridge is not available it is possible to scale-up the resistance level from 1 Ω to 100 Ω by use of series-parallel connection of 10 Ω four-terminal resistors in the arrangement proposed by Hamon. The scaling-up to resistances higher than 100 Ω may be done by use of two-terminal resistance boxes and a simple Wheatstone bridge.

The standard resistors with a value lower than 10 kΩ have usually a relatively high temperature dependance which in addition to self-heating effects require operation in an oil bath for best accuracy.

Electrical standards laboratories are frequently climatized so as to obtain a working temperature of 23 °C with admissible variations less than ± 1 °C. In such environments accuracies of the order of 50.10^{-6} can usually be reached for comparisons in air subject of course to equipment limitations and self-heating effects at high currents.

Standard resistors and other resistance measuring equipment with good time stability and very low temperature dependance, in particular around 23 °C can be constructed using other alloys than manganin and known for instance under the trade names of evanohm or zeranin. These alloys do not easily lend themselves for construction of standards as low as 1 Ω but do for values of 10 kΩ constitute excellent laboratory references and travelling standards which do not need to be operated in oil baths. The scaling-down from the 10 kΩ level to values of 1 Ω and below using various techniques does unfortunately currently involve considerable loss in accuracy but the 10 kΩ standard may constitute the reference standard for the range of 1 kΩ to 10 MΩ or more, as frequently required for calibration of electronic equipment. The procurement of a box of such calibrated standards is therefore recommended as a complement to the 1 Ω standard.

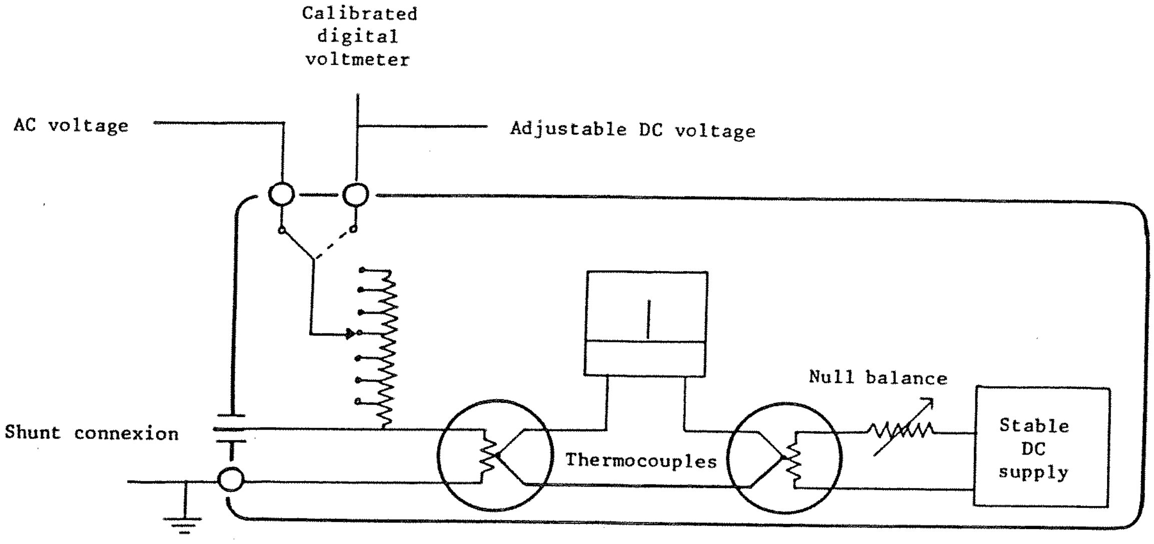

Fig. 9 - Measurement of AC voltage by AC/DC thermal transfer

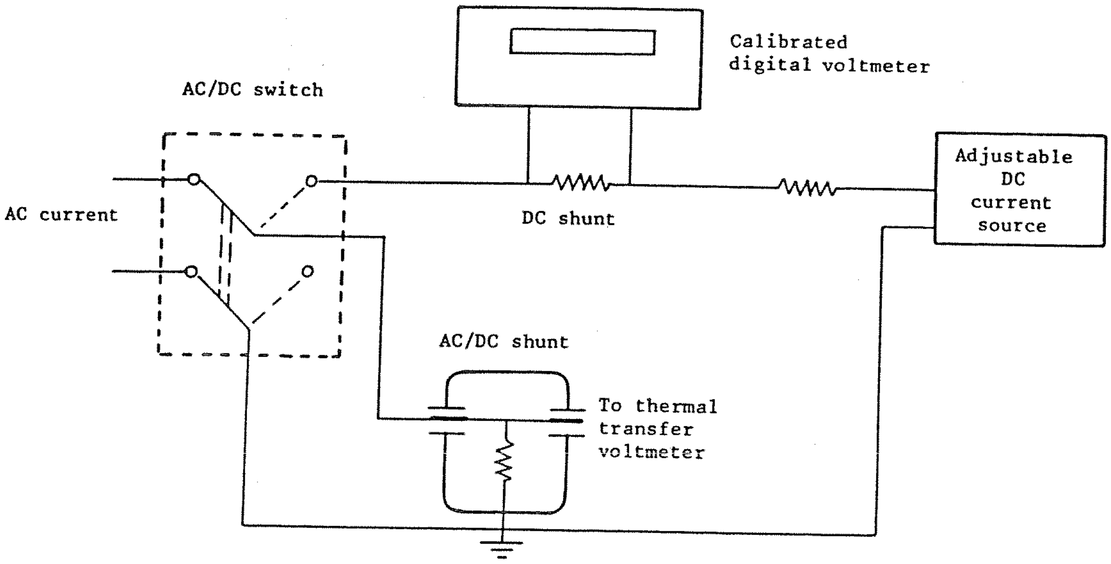

Fig. 10 - Measurement of AC current by AC/DC thermal transfer

The fact that the national resistance scale in this way will be connected to BIPM values at two different points will usually not create problems and may on the contrary constitute a control by redundancy.

If in fact at any time there appears an unexplained and consistant divergence between overlapping points of the scales, at for instance the 100 Ω or 1 kΩ level, this will be an indication that a recalibration at a foreign laboratory is necessary for at least one of the standards.

11.6. References for AC current and voltage

Accurate calibration of AC measuring instruments for voltage is nowadays usually made by thermal transfer.

The principle is basically that shown in Fig.9. The AC voltage to be measured creates a current through a heater in the form of a fine wire the temperature of which is sensed by a thermocouple junction. The other junction is heated in a similar way by another current which is adjusted so as to obtain zero reading on a null instrument. The input is thereafter disconnected from the AC circuit and connected to a variable DC supply which is adjusted so as to obtain the same indication as previously in the thermocouple circuit. The supplied DC voltage is then identical to the effective value of the first applied AC voltage within limits which are typically of the order of ± 0.01 %.

AC to DC transfer of current can be done with slightly less accuracy in a similar way by using a special two-pole switch and shunts as shown in Fig.10.

It should be noted that nor the voltage divider nor the shunts used with this device need to be calibrated as long as the DC voltage respectively the DC current are accurately known. The performance in frequency of all the elements i.e. thermal converter, voltage divider or AC/DC shunts must however be known for instance by a type examination certificate supplied by the manufacturer. There are however generally no problems with the frequency response at mains frequencies and up to 10 kHz.

The thermal transfer procedure by manual switching is not used for routine calibrations but mainly for calibration of precision AC voltage sources or signal generators and digital voltmeters specially conceived for AC operation. The latter may in turn be suitable for calibration of voltmeters and ammeters for general use (with the best accuracy of 0.1 %). It may be mentioned in this connection that the majority of four-terminal resistors and shunts below 10 Ω though calibrated in DC can be used for AC calibration of current at mains frequencies.

11.7. References for AC power and energy

The classical and very simple instrument for AC/DC transfer at power frequencies used to be a class 0.1 electrodynamic wattmeter of which versions with light-spot indication were commercially available.

These instruments tend to be replaced by digital electronic wattmeters based on multiplication of samples of voltage and current. Most of these instruments operate only within a limited frequency range and cannot be calibrated in DC. Several manufacturers of test benches for electrical energy meters have however developed thermal transfer comparators which enable AC/DC transfer of power and consequently also calibration with DC of electronic reference energy meters. The accuracy claimed for these comparators is ± 0.01 % within $\cos \varphi = 1$ and 0.5. For more information about electronic energy meters, AC/DC power comparators and test equipment, consult for instance PTB Testing Instructions "Electricity Meters", revised edition 1982, the Annex of which contains information about commercially available equipment.

11.8. Calibration of instrument transformers

Precision measurements of AC current at mains frequencies are usually done by means of instrument current transformers with the secondary winding output rating standardized to 5 A (1 A in special cases).

The testing of such transformers can be greatly facilitated by use of a current comparator test set which by its principle is self-contained and does not require an external standard instrument transformer. Furthermore this device can be connected so as to feed from its secondary circuit (5 A range) the current for the primary circuit (typically up to 2000 A) and thus avoid the need for an adjustable high current supply. This equipment requires simply as external device an IEC standard burden to simulate the impedance of the instruments which are usually connected to the secondary of the tested instrument transformer.

If however, for control purposes a medium-sized calibrated standard instrument transformer is procured it should be remembered that this, or any other current transformer, should never be energized on the primary when the secondary winding is open. This cannot happen when a current comparator test set is used with secondary current feed.

The testing of voltage instrument transformers is less frequent except in countries where such transformers are manufactured. Test installations take in this case more space and are usually combined with testing of other high-voltage components. A medium-sized installation can however be built for voltage transformers up to 35 kV which is normally the highest distribution voltage for transformers supplying industries or consumer areas.

The current comparator principle can also be applied in this case by measuring ratios of currents through low-loss capacitors of which one low voltage capacitor is used as reference. The equipment requires however an adjustable high-voltage supply and as previously standard burdens.

11.9. References for impedance measurement

Impedance measurements in the low-frequency field up to 20 kHz can currently be done with high quality commercial impedance bridges to an accuracy of 0.05 % provided they are verified from time to time by use of decade resistors with known frequency performance and by decade capacitors. The latter can be calibrated with respect to reference capacitors by use of a ratio transformer capacitance bridge. The advantage of the ratio transformer is that basically only one standard capacitor is required which can be calibrated at a foreign laboratory.

An example of this calibration scheme is shown in Fig. 11.

It should be noted that there is not much point in procuring inductance standards as they are usually not very accurate and much influenced by time and by the measuring environment.

Decade inductors may however in some cases be required to calibrate other bridges but they can in this case at each time generally be compared to a capacitor by use of the impedance bridge.

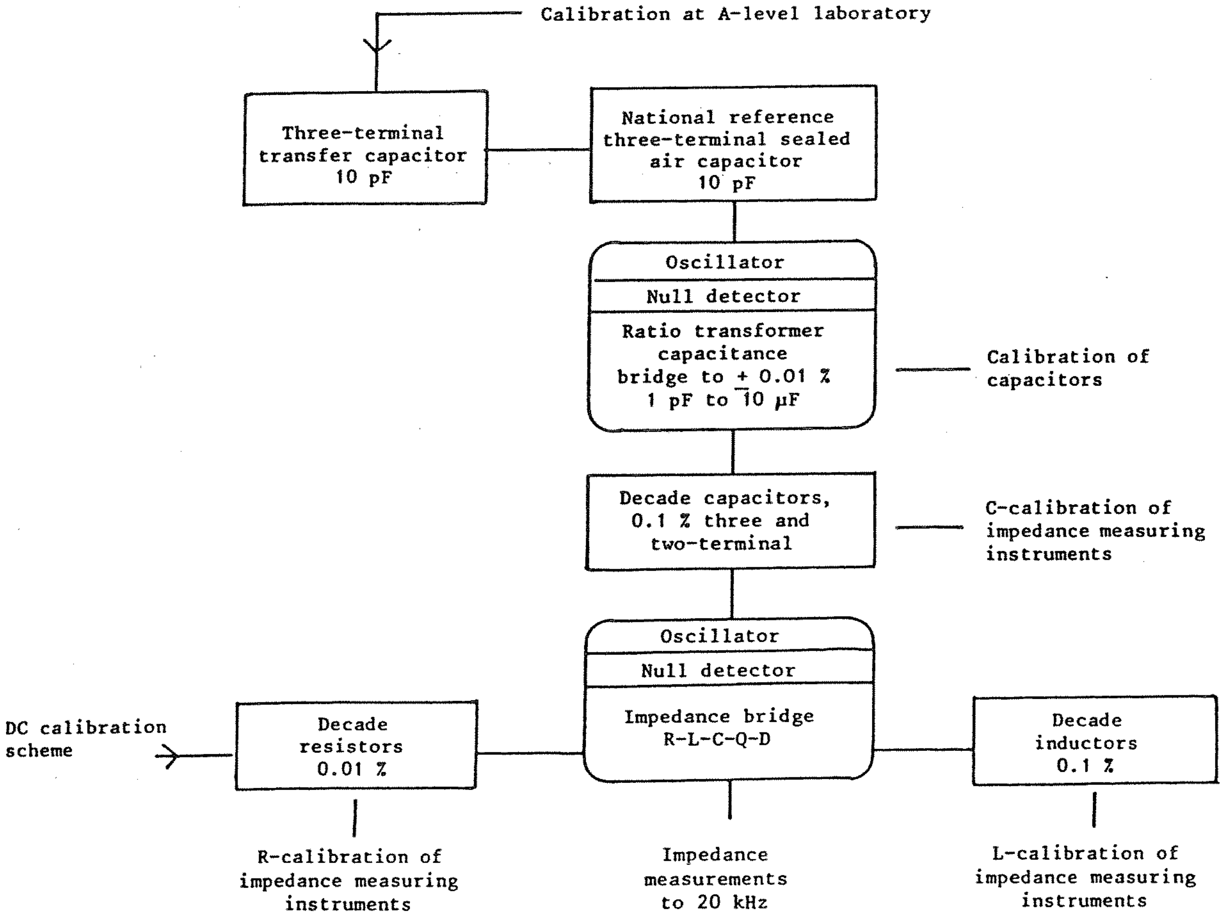

Fig. 11 - Low frequency capacitance, inductance and impedance calibration

12. Photometry

12.1. Requirements

Photometry does in most cases not form part of legal metrology though aspects such as minimum illuminance levels at work places or in traffic may be subject to legal prescriptions.

In many countries lamps, and especially incandescent lamps, are tested on sampling basis for the purpose of government quality control and consumers' protection.

The scheme shown in Fig. 12 illustrates the equipment which may be used in a photometry laboratory concerned with more or less routine tests on incandescent and flourescent lamps.

We have also in the synoptic diagram indicated the possibility of checking illuminance (and luminance) meters using luminous intensity lamps. The complete calibration of such meters must normally comprise the determination of their curve of spectral response using either a special spectrophotometer or simply a disc with narrow-band filters and a comparison receiver of known spectral characteristics. These somewhat scientific measurements and calculations are usually out of the scope of routine practical metrology work. However, illuminance meters at least approximately maintain their spectral characteristics but may show temperature dependance or fatigue effects resulting in a diminishing over-all response which thus may have to be checked.

It may thus be worth to procure a few luminous intensity standard lamps and an adjustable support for their vertical and rotational alignment preferably mounted on a simple optical bench so as to enable such verifications.

Precision illuminance meters if ordered for the service should however be certified as regards spectral response by a recognized official national laboratory.

In order to avoid as much as possible the influence of divergencies in spectral response of the photocells used it is preferable to choose the colour (or appearant) temperature of the standard lamps to be as close as possible to that of the illuminants to be measured. Incandescent filament lamps for room lighting are usually gasfilled and operate at a colour temperature above 2800 K. Vacuum incandescent lamps exhibit a greater stability for use as standards but their colour temperature is at maximum 2400 K and the corresponding light-output is much lower.

12.2. National reference standards

These may be constituted as follows :

<u>Luminous flux</u>

Standard lamps for luminous flux, gas-filled with welded filaments, clear bulbs, nominal wattage 200 W, working voltage 100 V (or 220 V).

The lamps are all to be sent for burn-in and calibration at BIPM or another A-level laboratory. They shall thereafter partly be transported hand-carried partly sent as accompanied luggage to the project site.

Required quantity : 25, whereof 10 shall after burning-in and stability tests be selected by the calibrating laboratory to constitute the national reference group for luminous flux, adjusted to a colour temperature close to 2850 K.

Note : The group of secondary standard lamps may be constituted by those lamps which were not selected to constitute the national reference group.

Working standard incandescent lamps for luminous flux, ratings 25 to 500 W are usually selected from best type of lamps available on the local market. They should be of the same type and size as the corresponding lamps tested.

Luminous intensity

Standard lamps for luminous intensity, gas-filled with flat-bed filament, high stability construction, low voltage type (less than 50 V). The lamps will be used at a voltage such that the colour temperature approaches 2850 K.

Quantity : 10 (whereof 5 shall be selected to constitute a group of national reference standards).

All lamps shall be sent by the supplier to the calibrating laboratory. They shall afterwards be forwarded to the project as hand-carried parcels.

Note : The working standard lamps for luminous intensity may be constituted by the remaining lamps which were not selected as national reference.

12.3. The integrating sphere

The simplest device for measurement of luminous flux is an integrating Ulbricht sphere used together with a photoelectric receiver.

Measurements are made by successive substitutions between the standard lamp and the lamp to be calibrated. The essential characteristic of the physical receiver is first to have a spectral response very close or identical to the day vision of the human eye or rather to the conventional so-called V_λ - curve established by the International Commission on Illumination (CIE). Secondly it should be linear over all the useful measuring ranges so as to enable the establishment of correct ratios of illuminance.

The size of the Ulbricht's sphere should be choosen so large (diameter 2.5 m) as to easily accommodate fluorescent lamps.

The absolute accuracy with which fluorescent lamps can be compared to an incandescent standard will much depend on the fitting of the characteristics of the receiver to the V_λ curve and on the selectivity of the sphere window and its internal paint.

The paint applied by manufacturers of integrating spheres is rarely in conformity with the requirements for the calibration of lamps in particular fluorescent ones. A repainting therefore usually has to be done "in situ" and remade every two or three years depending on conditions of humidity and dust.

Calculations show that if the coefficient of reflectivity of the paint is held below 87 % the influence of lack of spectral uniformity of the paint will be kept within reasonable limits. On the other hand the reflectivity should not be less than let us say 80 % as otherwise the integrating action of the sphere would not be sufficient for lamps with irregular geometrical light distribution.

If the sphere has any corrosion spots when delivered it must first be carefully cleaned and repainted with a corrosion protecting white paint. Thereafter the following paint may be applied through spraying (as experienced by BIPM and several other laboratories) :

Three layers of a mixture of 100g Ti 0_2, 5g CMC, 500 cm^3 H_2O

Two layers of 100g ZnO (medical), 5g CMC, 500 cm^3 H_2O

Three layers of 100g ZnO (medical), 3g CMC, 350 cm^3 H_2O

CMC = Carboxymethyl-cellulose (a commonly used binder).

It is expected that the reflectivity coefficient according to this procedure will approximately be within the said limits.

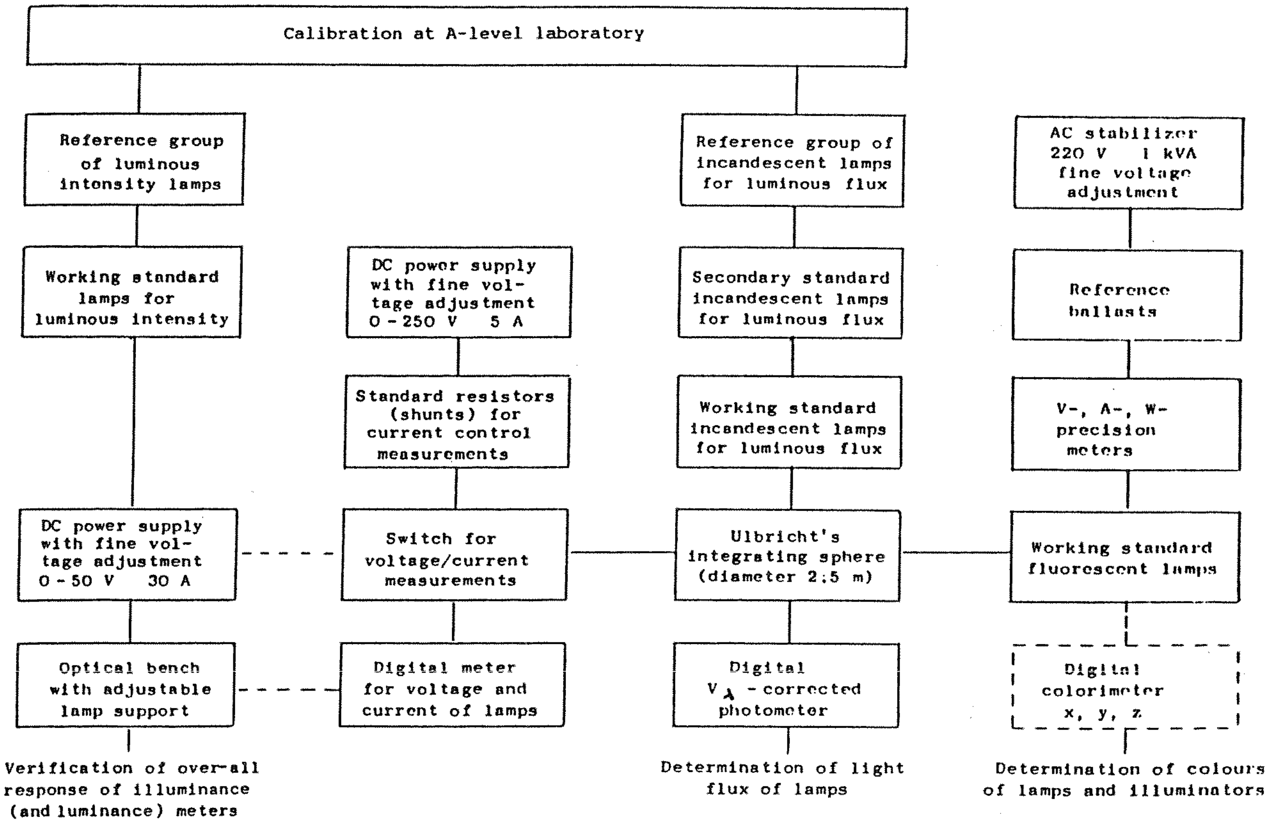

Fig. 12 - Scheme for photometric calibration

APPENDIX 1

Scaling up and scaling down of the magnitudes of mass standards from the basic value of 1 kg

The (national) reference set of weights should originally be adjusted to OIML class E2. The usual composition of this set is from 1 mg to 2 x 10 kg in the 1, 2, 2*, 5 or 1, 1*, 2, 5 series of multiples.

The exact value of each of these weights should be determined locally at regular intervals, for instance every two or three years by a group weighing procedure sometimes simply called "calibration of a set of weights".

This operation shall take place in a clean room without air draught and direct sunlight in which the temperature variations can be kept small (less than 0.5 °C per hour).

Equal-armed balances or mechanical substitution balances may be used as mass comparators but electronic force-compensating balances are becoming popular and provide the facility of obtaining printed results so as to speed up measurements and avoid mistakes.

Note that all the balances must have sufficiently large weighing pans so as to accommodate for a group of weights in the 1 + 2 + 2* + 5 series and that attention must be paid to problems of centering the resulting point of gravity. Some types of special mass comparators may not be suitable in these respects and require for the scaling-up operations the use of special weights which can be stacked.

All mass comparators require the use of an additional set of milligramme weights of class E2 for calibrating the difference range or to determine the sensitivity.

The resolution and the repeatability of the balances used for the fundamental comparisons (as well as for calibration of class F1 weights) should preferably be as follows

Maximum load		Readability *	Standard deviation
10 (or 20)	kg	1 mg	2 mg
2		0.1	0.2
200	g	0.01	0.02
20		0.001	0.002

* For digital indication = one scale interval
for analog indication = 1/5 of scale interval

A number of methods have been devised for calibrating sets of weights by internal comparisons of various possible weight combinations.
The aim of these methods is generally to attempt to distribute the global influence of the uncertainties resulting from the various comparisons in a

manner so as to affect the result for each weight in proportion to its mass.

Several methods involve for each decade of weights a greater number of comparisons than the strict minimum and thus require a longer time during which it may be more difficult to maintain stable environmental conditions.

The method exposed here as an example is probably the simplest one as it requires the minimum of comparisons for computing the conventionally true value (or the error) of each weight. However, and this is important, the full series of comparisons shall be repeated at least twice so as to enable estimates of the accuracy and checks for mistakes in measurements and calculations.

The set of comparisons should usually start and end with a comparison of the (national) secondary standard kilogramme with the 1 kg weight of the reference set.

Weight sets with the usual denominations in the series of 1, 2, 2*, 5, 10 will anyway require an extra weight in the scaling-up or scaling down operations. The comparison of the 1 kg denomination of the reference set to the secondary standard kilogramme can therefore be integrated in the scaling-up scheme as shown below :

$$[1]\ \text{kg} = [1]_c + d_1$$

$$[2] = [1]_c + [1] + d_2$$

$$[2]^* = [2] + d^*_2$$

……

$$[5] = [1] + [2] + [2]^* + d_5$$

$$[10] = [1] + [2] + [2]^* + [5] + d_{10}$$

$$[10]^* = [10] + d^*_{10}$$

……

$$[20] = [10] + [10]^* + d_{20}$$

where $[1]_c$ designates the exact value of the secondary standard and d_1 through d_{20} the mean differences in mass measured from (at least) two comparisons. It can be seen that the exact computation of the values of the various weights is in this case straight forward by appropriate additions starting from the top of the table.

In the scaling-down operation the arithmetic may at first seem more elaborate as only the value of the weight with highest mass will be known. In this case the operations can be easily facilitated by using an assumed value of the error of the first weight in each decade and then correct the computed values for each weight by applying adjustments proportionally to each weight.

The procedure is probably best illustrated by the numerical example in table below for the scaling down from 1 kg to 100 g.

An extra well distinguishable weight of 100 g is required, the exact value of which does not need to be known. However if this additional weight has been calibrated independently (at another laboratory) this may, within limits of uncertainty, constitute a convenient check of the correctness of the results of the group weighings. We may designate this weight by $[100]_F$ and assume at first that its error is zero.

The computation table will furnish the value of mass for the 100 g weight which can be used in a similar operation for the scaling down to 10 g and so forth. The table will also furnish the values of the extra weights used. However it is important to note that, below 1 kg, even if the value of the lowest weight in a decade is known it shall not be used as a reference for computing the values of the other weights as this procedure will usually increase the uncertainty of the results.

The calibration of working mass standards used for verification of heavy weighing equipment such as platform scales and weighbridges can be done by comparing first a specially adjusted brass or bronze weight of 20 kg nominal value, used a transfer standard, to the sum of the two 10 kg stainless steel weights of the class E2 reference set.

This weight is then used for the calibration of a large number of 20 kg rectangular weights which may generally conform to OIML R 2 as regards shape but preferably be adjusted to better than ± 1 g and which can be used as a means of transfer to standards with higher mass in the range of 100 to 1000 kg.

The calibration of the 20 kg weights can be greatly facilitated by using an electronic balance with 10 mg scale interval and an associated printer.

The comparison of the 100 to 1000 kg weights to a set of 20 kg rectangular weights requires special equipment, some information is given in the BIML brochure "Mobile equipment for the verification of road weighbridges".

Example of Computation Table

Weight		Compared to	mean measured difference d mg		computed assumed error mg		proportional adjustment mg		final values mg
$[100]_F$ g	=	100 g (nominal)			0	+	4.40 x 1/10	=	+ 0.44
[100]	=	$[100]_F$	- 0.32	+	= - 0.32	+	4.40 x 1/10	=	+ 0.12
[200]	=	$[100]_F$ + [100]	- 0.41	+ +	= - 0.73	+	4.40 x 2/10	=	+ 0.15
[200]*	=	[200]	+ 0.15	+	= - 0.58	+	4.40 x 2/10	=	+ 0.30
[500]	=	$[100]_F$ + [200] + [200]*	- 0.49	+ + +	= - 1.80	+	4.40 x 5/10	=	+ 0.45
[1] kg	=	[100] + [200] + [200]* + [500]	+ 0.43	+ + + +	= - 3.00	+	4.40	=	+ 1.40

From earlier comparisons : [1] kg = 1 kg + 1.40 mg

APPENDIX 2

Intercomparison of standards

The purpose of this Appendix is to explain the mathematics of two simplified schemes used for the establishment of the difference between the values in a group of more than two standards.

If more than 2 standards of similar type and magnitude are to be intercompared in order to establish differences in between them or to an assigned mean value, there are mainly two methods :

A. comparison of each standard X_1 X_2 X_3 X_4 etc. to a stable reference (or better standard) K

B. direct intercomparisons of the standards X_1 X_2 X_3 X_4 etc.

A. Comparison to a reference

In the first case the scheme of comparison is simply

$$X_1 - K = a_1$$
$$X_2 - K = a_2$$
$$X_3 - K = a_3$$
$$X_4 - K = a_4 \qquad \text{etc.}$$

where a_1 a_2 a_3 a_4 represent the observations made. The arithmetics here is straightforward. If for instance X_1 is considered as a calibrated standard with an assigned value, one obtains the other difference as

$$X_2 - X_1 = a_2 - a_1$$
$$X_3 - X_1 = a_3 - a_1$$
$$X_4 - X_1 = a_4 - a_1 \qquad \text{etc.}$$

An important case is when the mean value $X_m = \frac{\Sigma X}{n}$ represents an assigned value and one wants to express X_1, X_2, X_3, etc. in terms of differences to the mean value. We then obtain by summation of all equations

$$\frac{\Sigma X}{n} - K = \frac{\Sigma a}{n}$$

$$K = X_m - \frac{\Sigma a}{n}$$

and

$$X_1 - X_m = a_1 - \frac{\Sigma a}{n}$$

$$X_2 - X_m = a_2 - \frac{\Sigma a}{n} \qquad \text{etc.}$$

A typical application for this method is the comparison of standard cells or standard resistors by substitution.

Note : The electrical DC standards are constituted by groups of standard cells and groups of standard resistors. Each group has an assigned mean value obtained from calibration at BIPM or other laboratories.

However, the substitution method is based on the stability of the comparison value K throughout the measurements. Drift of K with time may be compensated by repeating the measurements in reverse order (Go-Return procedure) and taking the mean values of a_1, a_2, etc. The procedure is generally suitable whenever the time for each measurement can be reduced. The random component of the uncertainty may in this case only be reduced by repeating the measurement series in which case another comparison standard K' may be used to change measurement conditions.

B. Direct intercomparison of the standards

This is a method widely used which obviates for the need of a stable reference (K). It is most warmly recommended for comparisons where highest precision and accuracy are required and especially when the standards compared are of identical type. The calibrations of subdivisions (autocalibration) can, in simplified cases, be given a similar mathematical treatment as the intercomparison of several standards.

If a set of standards X_1, X_2, X_3, X_4, etc. are to be intercompared, one first establishes a set of comparison equations, where care is taken so as to include all possible combinations but each one only once (a comparison $X_4 - X_3$ is identical mathematically to $X_3 - X_4$).

We may write these equations as follows :

$$\begin{array}{llll}
X_1 - X_2 = a_1 & & & \\
X_2 - X_3 = a_2 & X_1 - X_3 = a'_1 & & \\
X_3 - X_4 = a_3 & X_2 - X_4 = a_2' & X_1 - X_4 = a_1'' & \\
X_4 - X_5 = a_4 & X_3 - X_5 = a_3' & X_2 - X_5 = a_2'' & X_1 - X_5 = a_1''' \\
 & & & \text{etc.} \\
X_{n-1} - X_n = a_n & X_{n-2} - X_n = a_n' & X_{n-3} - X_n = a_n'' & X_{n-4} - X_n = a_n'''
\end{array}$$

Generally there will be $N = \frac{n\,(n-1)}{2}$ equations.

For 10 standards to be intercompared (n = 10), there will thus be 45 different comparisons. In many cases this number represents a practical maximum. If n is still higher it is preferable to make the comparisons by groups of maximum 10 equations in each. Groups of 5 give 10 equations and constitute in many cases a reasonable number of comparisons.

If the results of measurements (a_1, a_2, etc.) are of comparable precision, we see that we may give equal importance to all the equations. The mean results out of all equations therefore represents the best value for the differences $X_1 - X_m$; $X_2 - X_m$; etc.

These best values may be calculated as follows :

Adding all equations containing X_1 gives

$$\begin{array}{l} X_1 - X_2 = a_1 \\ X_1 - X_3 = a_1' \\ X_1 - X_4 = a_1'' \\ \underline{X_1 - X_5 = a_1'''} \end{array}$$

$$5X_1 - \Sigma X = a_1 + a_1' + a_1'' + a_1'''$$

or with $X_m = \frac{\Sigma X}{n}$ and n = 5

$$X_1 = X_m + \frac{a_1 + a_1' + a_1'' + a_1'''}{5}$$

Adding all equations containing X_2 gives

$$\begin{array}{l} X_2 - X_3 = a_2 \\ X_2 - X_4 = a_2' \\ X_2 - X_5 = a_2'' \\ \underline{-(X_1 - X_2) = -a_1} \end{array}$$

$$5X_2 - \Sigma X = -a_1 + a_2 + a_2' + a_2''$$

$$X_2 = X_m + \frac{-a_1 + a_2 + a_2' + a_2''}{5}$$

Adding all equations containing X_3 gives :

$$\begin{aligned} X_3 - X_4 &= a_3 \\ X_3 - X_5 &= a_3' \\ -(X_2 - X_3) &= -a_2 \\ \underline{-(X_1 - X_3)} &\underline{= -a_1'} \\ 5X_3 - \Sigma X &= -a_1' - a_2 + a_3 + a_3' \end{aligned}$$

$$X_3 = X_m + \frac{- a_1' - a_2 + a_3 + a_3'}{5}$$

Adding all equations containing X_4 gives :

$$\begin{aligned} X_4 - X_5 &= a_4 \\ -(X_3 - X_4) &= -a_3 \\ -(X_2 - X_4) &= -a_2' \\ \underline{-(X_1 - X_4)} &\underline{= -a_1''} \end{aligned}$$

$$5X_4 - \Sigma X = - a_1'' - a_n' - a_3 + a_4$$

$$X_4 = X_m + \frac{- a_1'' - a_2' - a_3 + a_4}{5}$$

Adding all equations in X_5 gives :

$$\begin{aligned} -(X_4 - X_5) &= -a_4 \\ -(X_3 - X_5) &= -a_3' \\ -(X_2 - X_5) &= -a_2'' \\ \underline{-(X_1 - X_5)} &\underline{= -a_1'''} \\ 5X_5 - \Sigma X &= -a_1''' - a_2'' - a_3' - a_4 \end{aligned}$$

$$X_5 = X_m + \frac{- a_1''' - a_2'' - a_3' - a_4}{5}$$

Generally one finds that the deviation of X_1, X_2, etc. from the accepted or assigned mean value X_m can be easily determined by arranging the results from the comparison equations in a simple tabular form as illustrated below :

A B	X_1	X_2	X_3	X_4	X_5
$- X_1$	0	$- a_1$	$- a_1'$	$- a_1''$	$- a_1'''$
$- X_2$	a_1	0	$- a_2$	$- a_2'$	$- a_2''$
$- X_3$	a_1'	a_2	0	$- a_3$	$- a_3'$
$- X_4$	a_1''	a_2'	a_3	0	$- a_4$
$- X_5$	a_1'''	a_2''	a_3'	a_4	0
	S_1	S_2	S_3	S_4	S_5
$X_X - X_m =$	M_1	M_2	M_3	M_4	M_5

where S_1, S_2, etc. represent the vertical columns sums for X_1, etc. and M_1, M_2, etc. the mean values = $\frac{S}{n}$. The differences a_1, a_2, etc. in the original equations are introduced in this table with signs so that A - B represents a positive difference and hence results with correct signs are obtained by adding each vertical column and dividing by the number of unknowns. The lower left triangular part in the table therefore contains observation values $a_1 = X_1 - X_2$, etc. without sign reversal, whereas the upper triangular part contains the same observations with sign reversal.

If one adds the horizontal lines one obtains the same results but with reversed sign. This summation may be done as a control of the calculation.

Final value of X_1, X_2, etc

The procedure outlined above enables the calculation of differences of X_1, X_2, etc. with respect to the mean X_m. Any of the values can, however, be calculated and expressed in absolute terms either with knowledge of X_m which may be an assigned value for the group of standards, or by a comparison of any of the quantities used X_1, X_2, etc. to a known standard. By taking successive differences between the results of the vertical columns in the table above, we eliminate X_m and obtain the calculated mean values for all the differences $X_1 - X_2$; $X_2 - X_3$; etc. or if X_1 is standardized : $X_2 - X_1$; $X_3 - X_1$; $X_4 - X_1$; etc.

APPENDIX 3

Calibration of a compensating potentiometer or an electronic digital voltage supply

It is not necessary to know the total or individual resistances of a potentiometer. It is however important to determine its deviations from linearity by autocalibration which is the term used for the intercomparison of the intervals of the decades.

To enable autocalibration it is necessary that the potentiometer has decades with at least 10 steps (and not 9 as may be the case in some lower grade equipment).

The equipment necessary for autocalibration is

- a sensitive null detector (galvanometer amplifier) (See Note below)
- a finely adjustable and stable voltage source (power supply or another potentiometer) which does not need to be calibrated.

The deviations from linearity are measured by the null detector the sensitivity of which is calibrated in all the measurements by creating a known small voltage difference in the circuit (using some of the lowest dials of the potentiometer).

It should be remembered that the highest dials are always the most critical from the time stability point of view and the last dials simply have to be checked for defects in contacts or in resistors after transport, bad handling or after a long period of non-use.

The first dial is controlled by comparing each of its steps to the full value (10x) of the next lowest dial. Readings on the null detector are converted to µV by calibration of its scale using the last dials of the potentiometer which may correspond to 1 µV or 10 µV depending on the type of potentiometer, required resolution and null detector sensitivity.

After the usual standardization procedure of the potentiometer has been done and found to be stable with time, the adjustable external voltage source (power supply or potentiometer) is adjusted so that null balance is obtained when dial 1 is at 0 and dial 2 on 10 times.

The null detector is thereafter disconnected using its command key (on the potentiometer or external) and dial 1 is switched to position 1 and dial 2 to position 0. The first reading on the null detector is then noted (a_1). The operation is repeated with new balance when dial 1 is on position 1 and dial 2 on position 10. The reading of the unbalance of dial 1 position 2 is noted (a_2), etc.

Note : Whenever possible it is preferable that the null detector has a scale with only positive numbers, i.e. is graduated for instance from 0 to 100 divisions with its effective mechanical and electrical zero set to around 50 divisions. This decreases the risk for the introduction of observation and computation mistakes.

The following independant equations can then be set up :

$(1.1) - (2.10) = a_1$

$(1.2) - (1.1 + 2.10) = a_2$ a_1 , a_2 etc are expressed in μV

$(1.3) - (1.2 + 2.10) = a_3$

$(1.4) - (1.3 + 2.10) = a_4$

etc

Note that for the first dial the sensitivity of the null detector may vary due to variable resistance of the circuit and the detector readings must be calibrated for each step and expressed in μV instead of in divisions of the scale.

These equations may now be written

$(1.1) = (2.10) + a_1$

$(1.2) = 2(2.10) + a_1 + a_2$

$(1.3) = 3(2.10) + a_1 + a_2 + a_3$

$(1.4) = 4(2.10) + a_1 + a_2 + a_3 + a_4$

etc

$(1.10) = 10(2.10) + a_1 + \ldots\ldots\ldots + a_{10}$

For linearity calculations it is customary to set the full value of the main dial compared (dial 1 position 10) to be exactly U (or 1 volt in most cases).
(The procedure is the same for a resistance box but the full value is then set to the value of total resistance obtained by external comparison to a standard).

This means that (1.10) = U and

$$(2.10) = \frac{U}{10} - \bar{a} \qquad \text{where} \quad \bar{a} = \frac{1}{10}\sum_{1}^{10} a_x$$

The same equations may thus be written

$$(1.1) = \frac{U}{10} + a_1 - \bar{a}$$

$$(1.2) = \frac{2U}{10} + a_1 + a_2 - 2\bar{a}$$

$$(1.3) = \frac{3U}{10} + a_1 + a_2 + a_3 - 3\bar{a}$$

etc

or generally for any position x of the first dial

$$(1.x) = \frac{xU}{10} + \sum_{1}^{x} a_x - x\bar{a}$$

As $\frac{xU}{10}$ represents the nominal voltage (or should-be voltage) of the dial position we find that the linearity deviations for the first dial is

$$d_x = \sum_{1}^{x} a_x - x\bar{a}$$

A computation table using this formula can easily be established.

The next lower dials may be calibrated using the same procedure as above. It will be seen however that the nominal voltage of the next dial has to incorporate the true value of voltage resulting from the previous calibration of the higher dials so that for dial number 2 at position x we have

$d''_{x_t} = d''_x - \frac{x\bar{a}}{10}\text{(dial 1)}$ where d''_x is measured as above and

d''_{x_t} is the total deviation from linearity

For dial 3 we would have

$$d'''_{x_t} = d'''_x - \frac{x\bar{a}}{10}\text{ (dial 2) } - \frac{x\,\bar{a}}{100}\text{ (dial 1)}$$

etc

An example of a computation table for a potentiometer having a full range of 2 V is reproduced on the next page.

The resulting errors above the usually standardized value (in this case 1 V) can be comparatively important as shown in the table. The errors of the third dial or lower are however frequently quite small in absolute terms and typically of the same order as the accuracy of the calibration method which depends on the detector, earthing problems and other influences. A rapid calibration of the lower dials should however, as already mentioned be done as a check for abnormal performance.

It is good practice to repeat each full calibration twice with a few days interval before making use of the resulting corrections i.e. the mean tabulated errors taken with a reverse sign.

The method described can of course also be used for calibrating precision voltage supplies (voltage calibrators) such as used for direct calibration of digital voltmeters and similar instruments.

COMPUTATION TABLE

Test : Potentiometer   Ser No.   Date

First dial (x0.1 V) compared to (10x0.01 V) on second dial   t = 23 °C

Step x 0.1 V	Galv reading in divisions		a µV Sens:0.1µV per div	$\sum_{1}^{x} a$	$x\bar{a}$	$\sum_{1}^{x} a - x\bar{a}$	Errors dial 1 µV (rounded)
	on 10x0.01	on step					
1	50	93	4.3	4.3	4.37	- 0.07	- 0.1
2	38	80	4.2	8.5	8.74	- 0.24	- 0.2
3	28	82	5.4	13.9	13.11	+ 0.79	+ 0.8
4	20	60	4.0	17.9	17.48	+ 0.42	+ 0.4
5	10	65	5.5	23.4	21.85	+ 1.55	+ 1.5
6	35	66	3.1	26.5	26.22	+ 0.28	+ 0.3
7	33	82	4.9	31.4	30.59	+ 0.81	+ 0.8
8	21	60	3.9	35.3	34.96	+ 0.34	+ 0.3
9	17	61	4.4	39.7	39.33	+ 0.37	+ 0.4
10	23	63	4.0	43.7	43.70	0 (set	point)
Mean	$\bar{a} = \frac{\Sigma a}{10} =$		+ 4.37				
11	20	46	2.6	46.3	48.07	- 1.77	- 1.8
12	9	20	1.1	47.4	52.44	- 5.04	- 5.0
13	16	36	2.0	49.4	56.81	- 7.41	- 7.4
14	19	46	2.7	52.5	61.18	- 8.68	- 8.7
15	15	42	2.7	54.8	65.55	- 10.75	- 10.8
16	16	30	1.4	56.2	69.92	- 13.72	- 13.7
17	13	80	6.7	62.9	74.29	- 11.39	- 11.4
18	26	53	2.7	65.6	78.66	- 13.06	- 13.1
19	8	28	2.0	67.6	83.03	- 15.43	- 15.4
20	8	42	3.4	71.0	87.4	- 16.40	- 16.4

Second dial (x0.01 V) compared to (10x0.001 V) on third dial

Step x 0.01 V	Galv reading in divisions		a µV Sens:0.1 µV per div	$\sum_{1}^{x} a$	$x\bar{a}$	$\sum_{1}^{x} a - x\bar{a}$	$\frac{x\bar{a}(\text{dial 1})}{10}$	Errors dial 2 µV
	on 10x0.001	on step						
1	42	32	- 1.0	- 1.0	- 1.0	0	- 0.4	- 0.4
2	30	22	- 1.8	- 2.8	- 2.0	- 0.8	- 0.9	- 1.7
3	20	12	- 0.8	- 3.6	- 3.0	- 0.6	- 1.3	- 1.9
4	11	1	- 1.0	- 4.6	- 4.0	- 0.6	- 1.7	- 2.3
5	27	18	- 0.9	- 5.5	- 5.0	- 0.5	- 2.2	- 2.7
6	47	37	- 1.0	- 6.5	- 6.0	- 0.5	- 2.6	- 3.1
7	36	27	- 0.9	- 7.4	- 7.0	- 0.4	- 3.0	- 3.4
8	25	16	- 0.9	- 8.3	- 8.0	- 0.3	- 3.5	- 3.8
9	12	35	- 0.7	- 9.0	- 9.0	0	- 3.9	- 3.9
10	33	23	- 1.0	-10.0	-10.0	0	- 4.4(4.37)	- 4.4
	Mean dial 2:		$\bar{a}$ = - 1.0					

APPENDIX 4

Density of air free distilled water as a function of temperature

Values are expressed in kg/m^3

t_{68} °C	0,0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
0	999,839	999,846	999,852	999,859	999,865	999,871	999,877	999,882	999,888	999,893
1	999,898	999,903	999,908	999,912	999,917	999,921	999,925	999,929	999,933	999,936
2	999,939	999,943	999,946	999,948	999,951	999,954	999,956	999,958	999,960	999,962
3	999,964	999,965	999,967	999,968	999,969	999,970	999,970	999,971	999,971	999,971
4	999,972	999,971	999,971	999,971	999,970	999,969	999,968	999,967	999,966	999,965
5	999,963	999,962	999,960	999,958	999,956	999,953	999,951	999,948	999,945	999,943
6	999,939	999,936	999,933	999,929	999,926	999,922	999,918	999,914	999,910	999,905
7	999,901	999,896	999,891	999,886	999,881	999,876	999,870	999,865	999,859	999,853
8	999,847	999,841	999,835	999,828	999,822	999,815	999,808	999,801	999,794	999,787
9	999,780	999,772	999,764	999,757	999,749	999,741	999,732	999,724	999,716	999,707
10	999,698	999,689	999,680	999,671	999,662	999,653	999,643	999,633	999,623	999,614
11	999,603	999,593	999,583	999,572	999,562	999,551	999,540	999,529	999,518	999,507
12	999,496	999,484	999,473	999,461	999,449	999,437	999,425	999,413	999,400	999,388
13	999,375	999,362	999,350	999,337	999,323	999,310	999,297	999,283	999,270	999,256
14	999,242	999,228	999,214	999,200	999,186	999,171	999,157	999,142	999,127	999,112
15	999,097	999,082	999,067	999,051	999,036	999,020	999,005	998,989	998,973	998,957
16	998,941	998,924	998,908	998,891	998,875	998,858	998,841	998,824	998,807	998,790
17	998,772	998,755	998,737	998,720	998,702	998,684	998,666	998,648	998,630	998,611
18	998,593	998,574	998,556	998,537	998,518	998,499	998,480	998,461	998,441	998,422
19	998,403	998,383	998,363	998,343	998,323	998,303	998,283	998,263	998,242	998,222
20	998,201	998,181	998,160	998,139	998,118	998,097	998,076	998,054	998,033	998,011
21	997,990	997,968	997,946	997,924	997,902	997,880	997,858	997,836	997,813	997,791
22	997,768	997,745	997,722	997,699	997,676	997,653	997,630	997,607	997,583	997,560
23	997,536	997,512	997,488	997,464	997,440	997,416	997,392	997,368	997,343	997,319
24	997,294	997,269	997,244	997,220	997,195	997,169	997,144	997,119	997,093	997,068
25	997,042	997,017	996,991	996,965	996,939	996,913	996,887	996,861	996,834	996,808
26	996,781	996,755	996,728	996,701	996,674	996,647	996,620	996,593	996,566	996,538
27	996,511	996,483	996,456	996,428	996,400	996,372	996,344	996,316	996,288	996,260
28	996,231	996,203	996,174	996,146	996,117	996,088	996,059	996,030	996,001	995,972
29	995,943	995,913	995,884	995,854	995,825	995,795	995,765	995,735	995,705	995,675
30	995,645	995,615	995,584	995,554	995,523	995,493	995,462	995,431	995,401	995,370
31	995,339	995,308	995,276	995,245	995,214	995,182	995,151	995,119	995,087	995,056
32	995,024	994,992	994,960	994,928	994,896	994,863	994,831	994,798	994,766	994,733
33	994,701	994,668	994,635	994,602	994,569	994,536	994,503	994,469	994,436	994,402
34	994,369	994,335	994,302	994,268	994,234	994,200	994,166	994,132	994,098	994,064
35	994,029	993,995	993,960	993,926	993,891	993,856	993,821	993,787	993,752	993,717
36	993,681	993,646	993,611	993,576	993,540	993,505	993,469	993,433	993,398	993,362
37	993,326	993,290	993,254	993,218	993,181	993,145	993,109	993,072	993,036	992,999
38	992,962	992,926	992,889	992,852	992,815	992,778	992,741	992,704	992,666	992,629
39	992,592	992,554	992,516	992,479	992,441	992,403	992,365	992,327	992,289	992,251
40	992,213									

Note : This table is generally in line with OIML Recommendation R 22 "International Alcoholometric Tables" and constitutes an abridged version of a table published by H. Wagenbreth and W. Blanke in PTB-Mitteilungen No.6, 1971, whereby the fourth decimal place has been omitted (without rounding). The third decimal place has been maintained but should not be considered as significant. It is in fact about ten times smaller than the uncertainty of the best determinations of the density of bidistilled water.

APPENDIX 5

The calibration services of the BIPM

The BIPM undertakes a limited range of calibrations for the designated national laboratories of Member States of the Convention du Mètre. In each of the fields listed below only the national primary standards for the quantity in question are accepted for calibration. The principal calibrations offered at present are the following :

Mass - 1 kg prototype mass standards of platinum-iridium
- 1 kg mass standards of stainless steel.

Length - Iodine-stabilized lasers at the following wavelengths : 633 nm, 612 nm and 515 nm,
- methane-stabilized lasers at a wavelength of 3,39 µm,
- iodine and methane absorption cells for the above-mentioned lasers,
- 1 m line standards (principal interval),
- end gauges of length between 500 and 1000 mm.

Electricity - Standard Weston cells in temperature-controlled enclosures,
- small groups (of up to four) bare cells at 20 °C,
- Zener diodes at 1.018 V,
- 1 Ω and 10 000 Ω resistance standards
- 10 pF capacitance standards.

Photometry - Incandescent lamps used as standards of luminous intensity and luminous flux at a colour temperature of 2 800 K.

Ionizing radiations

Measurement standards for the following quantities :
- absorbed dose to graphite (^{60}Co γ-rays),
- air kerma (^{60}Co γ-rays and X-rays), tissue kerma (14.65 MeV neutrons),
- neutron fluence rate at 2.50 MeV and 14.65 MeV
- Radioactive neutron sources (emission rate),
- Solutions of τ-ray emitters, in standardized ampoules, measured by a calibrated ionization chamber in the SIR system.

<u>Information</u>

Governmental organisations in States which are not Members of the Convention du Mètre may also apply for calibration of items in accordance with this list.

Information about the possibility of acceptance for calibration, cost and customs formalities can be supplied on request addressed to

The Director
Bureau International des Poids et Mesures
Pavillon de Breteuil
F-92312 Sèvres, France

APPENDIX 6

Calibration at other laboratories than BIPM

Excellent facilities for calibration of national primary and secondary standards exist in a large number of countries. The usual procedure is to apply for the initial calibration of standards of highest level at the national metrology laboratory in the country of its manufacture.

The addresses of the scientific metrology (A-level) laboratories which maintain direct liaison with BIPM and have extensive facilities for such calibration are given below.

Current calibrations at slightly lower level (B-level), suitable in particular for high-grade secondary standards, can be undertaken by official or officially accredited laboratories in a large number of industrialized countries, information can in this case generally be supplied through the CIML-members to which the mailing addresses are given in each issue of the OIML Bulletin.

A-level laboratories

Australia
CSIRO Division of Applied Physics
National Measurement Laboratory
Bradfield Road, West Lindfield
Sydney NSW

Canada
National Research Council
Division of Physics
Montreal Road
Ottawa, Ontario, K1A OC9

People's Republic of China
Coordination body :
State Bureau of Technical Supervision
P.O. Box 2112
Beijing

France
Mechanical quantities : Laboratoire National d'Essais
1 rue Gaston Boissier
75015 Paris

Electrical quantities : Laboratoire Central des Industries Electriques
33 avenue du Général Leclerc
BP n° 8, 92260 Fontenay-aux-Roses

Coordination body : Bureau National de Métrologie
22 rue Monge
75005 Paris

Fed. Rep. of Germany
Physikalisch-Technische Bundesanstalt
Bundesallee 100
3300 Braunschweig

German Democratic Republic
Amt für Standardisierung, Messwesen und Warenprüfung
Fürstenwalder Damm 388
1162 Berlin

India
National Physical Laboratory
Hillside Road
110012 New Delhi

Italy
Mechanical quantities : Istituto di Metrologia G. Colonnetti
Strada delle Cacce 73
10135 Torino

Electrical quantities : Istituto Elettrotecnico Nazionale Galileo Ferraris
Strada delle Cacce 91
10135 Torino

Japan
Mechanical quantities : National Research Laboratory of Metrology
1-4 Umezono 1-Chome, Tsukuba
Ibaraki 305

Electrical quantities : Electrotechnical Laboratory
1-4, 1-Chome, Umezono, Tsukuba
Ibaraki 305

United Kingdom
National Physical Laboratory
Teddington, Middlesex TW11 OLW

United States of America
National Institute of Standards and Technology (formerly NBS)
Gaithersburg, Maryland 20899

U.S.S.R.
Coordination body : Gosstandart
Leninsky Prospect 9
117049 Moscow

APPENDIX 7

Publications on available calibration facilities

Many national laboratories have issued special publications concerning available facilities and the practical formalities relative to applications for calibration. Most of this information is usually only available in the national language. For up-to-date information it is adviced to write directly to the national laboratory where the calibration is planned to be made.

The following publications are however of general technical interest as they also contain information on characteristics and limitations of the standards to be calibrated :

- NIST Calibration Services
  Users Guide 1989 Edition, NIST Spec.Publ. 250 Jan. 1989, 206 pages + separate supplement Fee Schedule, March 1989, 67 pages
  Available from : US Government Printing Office
  Washington D.C.20402
  (Contains bibliography to other NIST, formerly NBS, publications concerning calibration)

- NPL Measurement Services (separate brochures)
    - Mass & Density
    - Engineering Dimensional Metrology
    - Pressure and Vacuum
    - Hardness Measurement
    - Temperature
    - Direct Current and Low Frequency Electrical Measurements
    - Optical Metrology
    - Colorimetry, Spectrophotometry, Photometry and Radiometry
    - Acoustics

  NAMAS Concise Directory

  Published by National Physical Laboratory
  Teddington, Middlesex TW11 OLW
  United Kingdom

- Flow Measurement Facilities at NEL (latest report and leaflets)
  Published by National Engineering Laboratory
  East Kilbride
  Glasgow
  Scotland G75 OQU

- Tests and Measurements 1984, 85 pages
  Published by CSIRO Division of Applied Physics
  National Measurement Laboratory
  Bradfield Road West Lindfield
  Sydney NSW, Australia

- Testing and Measurement in the Physikalisch-Technische Bundesanstalt, 1988/1989.
  (This report contains an extensive list of items which can be calibrated at PTB in Braunschweig and in Berlin as well as the uncertainty with respect to the national standards which can be attained for standards of highest quality)

- The Bureau National de Métrologie in France has published a list of primary and officially accredited laboratories in a brochure with the title
  "Liste des laboratoires d'étalonnage accrédités du service d'étalonnage du BNM", 1988, 12 pages
  Available from Bureau National de Métrologie, 22 rue Monge, 75005 Paris.

- The capabilities of calibration in a large number of countries participating in the Asia-Pacific Metrology Programme and including Australia, China, India, Japan and U.K. are indicated in
  APMP Directory of National Measurement Systems,
  compiled by Wen Shanlin and Yang Xiaoren of the National Institute of Metrology, Beijing, China.
  CSC Technical Publications No.224, CSC(87) ISP 25, April 1987, 274 p.
  Published by Commonwealth Science Council
  Commonwealth Secretariat
  Marlborough House, Pall Mall
  London, SW1Y, 5HX
  United Kingdom