CALIBRATION IN CERAMIC TEST LABORATORIES

by J.M[a]. Martín Algarra, J. Gradolí Marco

Calibration and Metrology Laboratory, Comsip Automación S.A, Castellón, Spain
Laboratorio Cerámico Sebastián Carpi, Castellón, Spain

ABSTRACT

At Ceramic Test Laboratories, measurements are performed of length, weight, force, temperature and pressure. These measurements must be carried out with calibrated instruments, in order to be related to the units defined by the International System of Units (SI). Calibration allows "comparing" the readings of measurement and testing apparatus, with the units defined by the SI. Calibration, understood as the comparison and documentation of these results, has come to play an important part in the quality control of tests.

Although in general terms calibration involves a comparison of instrumental readings, very different work techniques are required owing to the variety of magnitudes, ranges and uncertainties that may be obtained.

This study aims to describe the mechanisms that allow relating the measurements and tests performed at the ceramic laboratories; explaining some techniques for calibration, analysis, expression and interpretation of calibration results; reporting the realization of the SI units and their dissemination in industrial activities; as well as how to effect an improvement of product quality as a result of calibrating testing apparatus and production plant on measuring the geometrical and physical characteristics of ceramic tiles.

1. INTRODUCTION

The UNE standards applying to tests for ceramic tiles describe the equipment to be used in testing ceramic materials. UNE 66903 states the need to calibrate inspection, measurement and testing apparatus, a calibration that must in turn be carried out with certified reference instruments, traceable to national references.

The calibration requirements of testing equipment can be complied with at a test laboratory according to two different criteria:

* **External calibration of testing equipment**

Calibration of the testing equipment is performed by an organization external to the test laboratory itself. As a result of the sizes and characteristics of the testing equipment, which make moving it unfeasible, the certifying organization usually sends technical staff and means to the test laboratory, to perform the calibration, issuing a calibration certificate that reports the results and traceability of the measurements.

* **Internal calibration of testing equipment**

The calibration of testing equipment is performed by staff from the test laboratory itself with its own media and procedures, certifying the results obtained and obtaining traceability through calibration of the calibration media.

Of course, the calibration equipment used in the second case must have certified calibration (whether performed internally or externally). We shall briefly list some of the advantages and drawbacks of both options:

* **External calibration**

Advantages

- Normal functioning of the Test Laboratory. Calibration can be considered as an additional activity in maintenance and conservation of the testing equipment.

- As far as documentation is concerned, a historical record is formed of the calibrations performed. No modification is therefore involved of the documentary organization of the Laboratory.

Drawbacks

- Fully dependent on the exterior: the calibration terms must be adapted to the conveniences of the calibrating organization.

- In the case of incidents, discrepancy in results or doubts concerning test results, recourse must be had to external sources to clear up these doubts.

- Constant yearly cost.

* **Internal calibration**

Advantages

- Autonomy in calibrating

- Possibility of extending the services offered in the field of calibration.

- Low yearly cost.

Drawbacks

- Necessary initial investment in equipment, documentation and training.

The most suitable solution must be assessed in each case, and it is a function of the size of the test laboratory, the complexity of tests and means, calibration frequency, possible impact of deviations, the possibility of reinforcing aspects of quality in other areas of the company (production, new products, maintenance, etc.). Certain calibrations are usually worth performing internally, while others are performed more profitably externally.

The following is a review of the measurement and testing instrumentation described by the regulations for tests in the ceramic sector, and the calibration requirements for this instrumentation will be briefly detailed.

2. REGULATIONS AND TESTING EQUIPMENT

UNE-67 098 (EN 98) Determination of dimensions and surface quality

* Length and width measurements

  - Gauge or other suitable instrument for linear measurements

* Thickness measurements

  - Micrometer with a spindle of 5 to 10 mm diameter, or other suitable instrument

* Measurement of straightness of sides

  - Instrument as shown in Figure 1, or other suitable instrument. Calliper A is used to measure straightness of sides.

  - A steel reference plate, of exact dimensions, whose sides are straight and flat

* Rectangularity measurements

  - (As above)

* Measurement of surface flatness (curvature and warpage)

  Tiles larger than 40 mm x 40 mm

  - An apparatus like the one shown in Figure 2 or any other suitable instrument

  - A perfectly flat reference plate, of metal or glass having a minimum thickness of 10 mm, for the instrument described in 6.2.2.1.

  Tiles smaller than 40 mm x 40 mm

  - A metal ruler

  - Thickness gauges

* Surface quality

  - Fluorescent light with colour temperature of 6000 K at 6500 K.

  - A ruler measuring one metre, or any other suitable means for measuring distance.

  - Photometer

Summing up, we can classify the dimensional equipment required in three groups (we purposely omit the equipment needed for testing surface quality):

* Length measurement instruments: Gauge, callipers, micrometer, ruler.

* Length definers: Reference plates

* Equipment required: Figure 1, Figure 2.

Calibration of the equipment listed in the first group requires **generating** a known (certified) length, and comparing the indication obtained from the instrument reading with the true (certified) value of the reference length. The agreement of the instrument indication with the generated length requires the most perfect possible definition of length for the class of instrument involved, as well as suitable behaviour with regard to repeatability and stability in the face of ambient variations by the calibration instrument. Calibration assesses instrument behaviour under optimum measuring conditions.

Calibration of these instruments can be performed at test laboratories that have suitable reference lengths (reference blocks) which define the geometrical characteristic being evaluated. The use of reference blocks in calibration may require some accessories in function of the instrument to be calibrated (gauge, micrometer, callipers, etc.), and the kind of measurement performed (interior, exterior, etc.). It should also be mentioned that using these instruments in calibration requires training workers in handling, maintaining, and using the equipment (as described in the documents accompanying the instruments), as well as having methods or calibration procedures which allow the calibration results to be expressed with their corresponding uncertainties (there are publications in this respect, see Documentos SCI published by MINER).

The reference plates do not, however, help to measure as the other instruments mentioned above do, but **realize** (**define**, **generate**) a given geometrical characteristic (flatness, straightness, rectangularity, etc.). To enable this **definition** to be used in testing, it must be measured and certified. The definition (surface flatness, rectangularity of two planes, etc.), requires multiple measurements of length (basic magnitude), and composition in order to obtain a mathematical and statistical definition of the characteristics being considered (e.g. theoretical plane, distance to plane, etc.), which adds a certain amount of information to be obtained to a complexity of calculations and actions. There are currently specialized instruments (measuring three coordinates), which perform measurements of these and other geometrical characteristics semi-automatically or automatically. An instrument of this kind allows calibrating the reference plates. If such an instrument is not available (owing to cost, complexity in use, and maintenance), the targeted characteristics must be certified at a laboratory equipped with such facilities.

The equipment brought together in the third epigraph are auxiliary elements, which facilitate the measuring operations laid down in the regulations. Although their use does not generally affect measurement, they must be kept and used suitably in order to obtain the performance stipulated by the regulations.

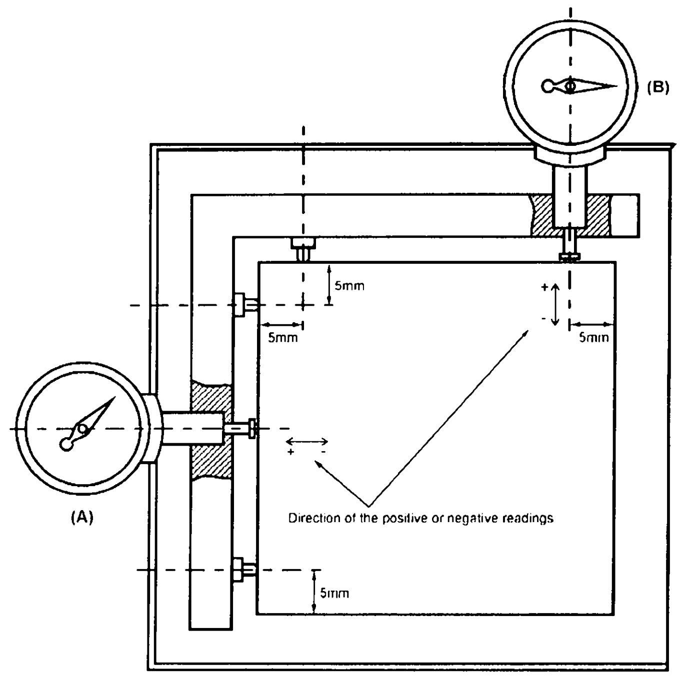

Figure 1. Apparatus for measuring straightness of sides and rectangularity (EN 98)

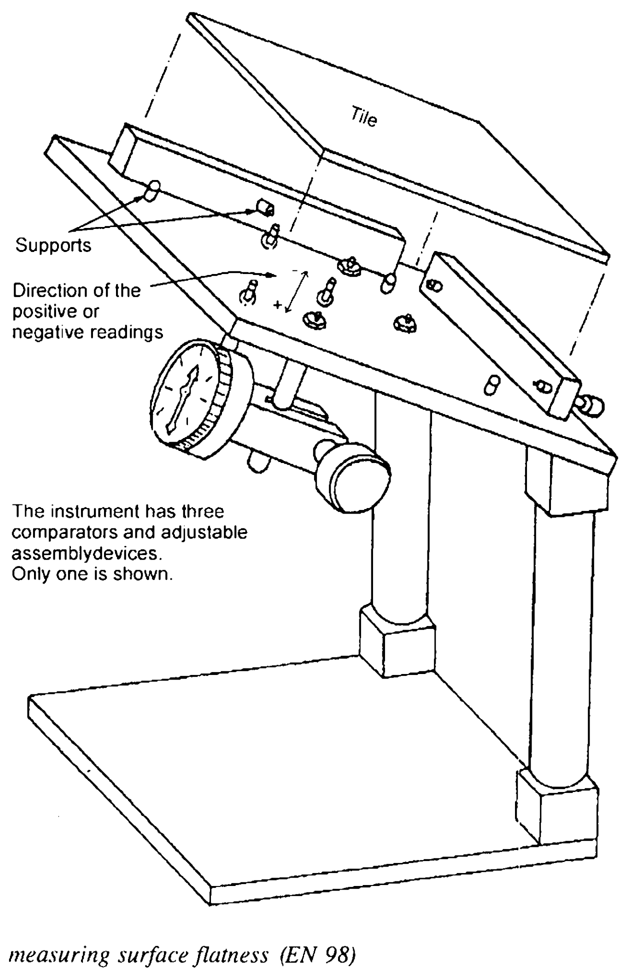

Figure 2. Apparatus for measuring surface flatness (EN 98)

In view of the above, it is to be inferred that at least some dimensional calibrations can be performed internally, without involving excessive investment. The documentary part of the calibration must parallel that of the recording of test results (UNE 66903).

UNE 67 099 (EN 99) Determination of water absorption

* Equipment

  - Drying oven capable of operating at (110 ± 5°C)

  - A heating facility built of suitable inert material, in which boiling takes place.

  - A balance capable of weighing the piece to be tested with an accuracy of 0.01%

Temperature can be measured with an (internally or externally) calibrated thermometer. To perform the calibration, temperature uniformity must be assessed in the chamber (temperature distribution in the testing volume), and its stability in time. This involves measuring temperature (concurrently if possible), at various points throughout the volume of the oven at regular time intervals in order to assess temperature stability. Temperature calibration follows below.

Calibrating the balance takes place by applying weights of certified values, similarly to what was described for dimensional measurements, verifying their indications, their repeatability, and sensitivity in the face of small variations (slight weight increments, if possible equal to the sensitivity of the balance).

UNE 67 100 (EN 100) Determination of the modulus of rupture

* Equipment

  - Drying oven capable of operating at (110 ± 5°C)

  - A monitoring-recording instrument having an accuracy of 2%

  - Two metal rods

  - A centre rod of the same kind

The standard is not very explicit here. The equipment is understood to monitor-record the force applied to the tile placed between the rollers, and to have a force-applying mechanism. Calibration of the monitoring-recording apparatus of an applied force can be carried out by having an externally calibrated force measurement device put in the place of the tile, and comparing the readouts of the recorder with those of the force measurement device. As was the case with regard to calibration of the length measurement instruments, stability and repeatability of the comparison must be assessed during this verification.

UNE 67 105 (EN 100) Determination of crazing resistance

* Equipment

- A steam autoclave, to maintain a pressure of (500 ± 20 KPa), that is, a water vapour temperature of (159 ± 1°C)

The pressure that the autoclave maintains can be measured by means of a calibrated pressure gauge.

The remaining regulations that do not refer to reference materials, deal with already described equipment (dimensional measurements, temperature, weight, force, pressure measurement). The above allows the conclusion to be drawn that calibration of some testing equipment can be performed at the laboratory itself, using minimum resources. Other calibrations cannot be performed so easily or feasibly internally, and it will be necessary to resort to external calibration.

We should not like to close this section without mentioning the difficulties involved for the persons executing and responsible for testing equipment calibrations, in as much as these are to be assessed on the basis of criteria established by the regulations. The acceptance criteria for instruments and testing conditions are badly defined in the regulations, which entails difficulties in interpreting and carrying out the calibrations. This unsuitable definition, which is not only the case of ceramic test standards, is being improved in some newly drawn up regulations based on definitions of classified testing equipment. We believe that the striving for standardization and rationalization in this sense is fully justified and that it will give rise to enhanced product quality.

3. CALIBRATION CERTIFICATES

Calibration certificates reflect the results and conditions of the performed calibration. They must clearly identify the calibrated measuring equipment or instrument, state the results of the calibration with the relevant uncertainty, the applied procedure or measuring technique, and the traceability of the references employed. In accordance with ISO-TAG-4, this must also be accompanied by the information (e.g. covariance, coefficients of correlation, degrees of freedom, etc.), which allows subsequent propagation of the uncertainties involved to the calibrations performed with the calibrated equipment.

The end result of the measurement is determined in function of the average value of the measurements performed, as this is the best statistical assessment value.

The expression of uncertainty reflected in the calibration certificates, is obtained on the basis of the uncertainty of the references used, the calibration technique employed, and the performed calibration itself. The references used trace the measurement, that is, allow relating the measurement to the measurements carried out according to the International System of Units through an uninterrupted chain of calibrations. The measuring technique determines the functional dependence between the results and the measurements of the references used, and also determines the law of propagation of reference uncertainties to results. Performing a calibration gives rise to a random experiment that determines a sample of possible results on the basis of which the calibration results are formed. Recommendation INC-1 of the BIPM establishes the use of variances to characterize measurement uncertainties, the law of the propagation of uncertainties therefore being a quadratic function.

Measurement traceability is accredited by an explicit declaration on behalf of the organization issuing the calibration certificate confirming the existence of a chain of calibrations leading back to the units defined by the SI. This declaration can be endorsed by the numbers and dates of issue of the calibration certificates of the references used, by customer audits at the certifying organization, or by third party audits, which as independent bodies, accredit the suitability of the calibration in respect of other requirements. This has given rise to calibration laboratory networks, backed by prestigious organizations, which avoid the drawbacks and costs stemming from the audits of each customer.

4. THE INTERNATIONAL SYSTEM OF UNITS: REALIZING AND DISSEMINATING UNITS

The International System (SI) of Units stems from a striving for rationalization and standardization in performing measurements in different countries. The SI adopts definitions of units, which it attempts to keep independent of a specific realization, facilitating a high degree of reproducibility among the laboratories that carry out the tests, while to date, the unit of weight remains the only one without an absolute definition: the unit is still the iridium-platinum cube in Sèvres at the BIPM.

The definition of the unit redounds to the excellence of reproducibility and therefore to international agreement, but is often unsuitable for direct industrial application, thus requiring dissemination before achieving industrial application. The dissemination of measurements on the basis of the definition realized at the National Laboratories takes place through calibration, which currently occurs via the National Networks of Test and Calibration Laboratories, and the manufacturers that base themselves on these. All industrial test quality is upheld by the degree of compatibility achieved as a result of the measurements calibration has made possible.

However, the highly desirable consistency and quality of industrial tests is not the only objective that calibration allows achieving. Consistency and quality can also be achieved in different production processes (even in the ceramic sector), enabling reproducibility to be reached of manufacturing conditions resembling what has already been attained in other production sectors. As far as application in the industrial production process is concerned, calibration reaches the factory through corrective, preventive and predictive maintenance plans, facilitating reproducibility of manufacturing conditions.

5. EXAMPLE

To illustrate a practical situation that may be encountered in many Test Laboratories, the calibration of industrial thermometers will be described. This example has been chosen because of the ease with which the user can obtain some defining points of the scale. The ITS-90 definition of a temperature scale will be briefly described, as well as some alternatives for calibrating an industrial thermometer.

The International Temperature Scale ITS-90

A temperature scale can be defined by applying numerical values to certain temperatures. Fixed points are required (that is, stable and reproducible ones), to which certain temperatures are attributed. It must subsequently be specified how to determine temperature between the fixed points. The usual method involves establishing a correlation between a given physical property (the most common are the length of a rod, the volume of a certain amount of liquid, or the electrical resistance of a wire), of a thermometric substance, and assuming a certain temperature correlation function. This function allows a temperature to be attributed outside the fixed points. Such a scale is defective in that it is wholly arbitrary, depending on the selected property and substance.

A thermodynamically absolute scale can be defined by means of the yield of a fully reversible ideal Carnot cycle, which defines absolute zero for the temperature scale. Having a zero in the scale leaves it perfectly defined by assigning a value to a point. The point chosen for the definition of the thermodynamic scale is the triple point of water, to which the value of 273.16 is assigned by definition. The triple point of water is highly reproducible, which provides an extremely valued property for metrology. The thus defined scale uses the Kelvin (K) as a unit, defined as a 1/273.16 part of the thermodynamic temperature of the triple point of water. This scale is usable and reproducible up to 1350 K.

Another method that is simple to reproduce uses the radiation of hot bodies, as some of the properties of the radiation emitted by a body in thermodynamic equilibrium are directly related to the temperature of the body. This property allows the temperature scale to be extended beyond 1350 K.

The International Temperature Scale of 1990 (ITS-90) is the best fit found between the thermodynamic temperature and the electrical properties of a platinum wire (between 0.65 K and 961.78°C), and the laws of blackbody radiation, yielding great reproducibility, continuity, and accuracy in measurement.

Calibration of industrial thermometers

In normal operation, industrial thermometers are subjected to different influences, such as high temperatures, chemical agents, vibrations and blows, etc. The effect of these factors is that thermometric properties can vary significantly. Therefore, they must be periodically checked and recalibrated or repaired if necessary. The period depends on work conditions and must be determined by plant or laboratory maintenance activities.

Two calibration methods can be distinguished: fixed points or by comparison

Fixed point calibration

Fixed point calibration has the advantage of making external calibration almost redundant, as the values are determined by the definition of the scale (some external calibration must be performed to periodically check the reproducibility obtained). The most widely used fixed points in industry are:

- Triple point of water (0.01°C), and melting point of water (0°C), for the calibration of glass thermometers, heat resistances, references for thermocouples.

- Boiling point of water (100°C)

- Freezing point of metals: tin (232°C), lead (327°C), zinc (419°C), aluminium (660°C)

- Melting point of metals: gold (1064°C), palladium (1554°C), platinum (1772°C)

Fixed point calibrations are highly accurate, but cost time and are very expensive, so that they are infrequently performed in industrial calibrations.

Calibration by comparison

This is the commonly used method in the industrial calibration of thermometers, involving a comparison of the reading of the thermometer being calibrated, with that of another calibrated thermometer used as a reference. This method suffers the drawback that the temperature both thermometers measure is not the same, as they are not in the same physical place at the same time. The thermometers to be calibrated are located in the same bath or kiln as close to each other as possible. The result of the calibration (uncertainty), depends on the calibration of the reference, the uniformity and stability of the temperature in the medium used for comparison, and the experience and technique of the worker performing the calibration.

6. ANNEXES

Some references and definitions are given which may be of interest to the reader.

* BIPM INC-1 (1980) Recommendations of the work group for establishing uncertainties.
* Procesos de Calibración del SCI, Ministry of Industry (Collection of publications, under constant review, on calibration procedures of Industrial Calibration Systems (SCI))
* ISO/IEC/OIML/BIPM ISO/TAG 4/WG 3 (June 1992) Guide to the expression of uncertainty in measurement

VOCABULARY

Taken from ISO/IEC/OIML/BIPM (1984) International vocabulary of basic terms in metrology (under review)

Calibration [6.13]

The whole group of activities which, under specific conditions, establish the relationship between the values indicated by a measurement instrument or measurement system, or values represented by a measured material, to the corresponding known value of a measurand.

Traceability [6.12]

The property of the result of a consistent measurement in being able to relate to suitable references, generally national or international, through an uninterrupted chain of comparisons.

Uncertainty of measurement [3.09]

[1984] An assessment characterizing the range of values in which the true value of a measured magnitude lies.

[Review] A parameter associated with the result of a measurement, which characterizes the scatter in the values, which can reasonably be attributed to the measurand.