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Rubber — Guide to the calibration of test equipment

Caoutchouc — Guide pour l'étalonnage du matériel d'essai

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 18899:2004), of which it constitutes a minor revision with the following change:

— inclusion of a code-letter for the classes of calibration interval.

Rubber — Guide to the calibration of test equipment

1 Scope

This International Standard outlines the principles of calibration of rubber test equipment and gives guidance on the general requirements for ensuring measurement traceability, establishing the basis for deciding calibration intervals, and estimating measurement uncertainty.

Methods of calibration for a range of parameters applicable to rubber test equipment are briefly described with reference to relevant standards where appropriate.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9000, *Quality management systems — Fundamentals and vocabulary*

ISO 10012, *Measurement management systems — Requirements for measurement processes and measuring equipment*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, the (metrological) terms and definitions given in ISO 9000 and ISO 10012 and the following apply.

NOTE The terminology used in this International Standard is also in line with the terms and definitions given in ISO Guide 30.

3.1
calibration
process of establishing the relationship between the values of a quantity indicated by a measuring instrument and the corresponding values indicated by a reference instrument

3.2
verification
activity whereby measuring or test equipment is subjected to a specified examination or calibration and found to perform within stipulated tolerances

3.3
metrological confirmation
set of operations required to ensure that an item of measuring equipment is in a state of compliance with the requirements for its intended use

3.4
calibration system
part of a quality system which includes the calibration and metrological confirmation of test equipment and any reference standards held

4 Principles of calibration

Generally, metrological confirmation involves calibration and also any necessary adjustment, repair, recalibration, sealing, or labelling. Confirmation can also involve the verification of a value, for example a length, of some feature of a test apparatus. In common terminology, the whole process of confirmation is considered as the service performed by a calibration laboratory and, frequently, the action of “calibrating” test equipment is more correctly providing metrological confirmation that it meets specified requirements.

Calibration is based on the principle of there being established values of measurement, represented by reference standards (sometimes called transfer standards), against which other measurements can be compared. The calibration value is transferred in turn from an internationally recognized standard to a nationally recognized standard (often called a primary standard), to a series of secondary or transfer standards, and then to the measurement or test equipment. Measurement traceability is being able to relate a measurement through an unbroken chain of comparisons to a primary standard.

Each stage of the transfer of calibration by comparison down the chain results in an increasing uncertainty in the measurement and, hence, a lowering of the accuracy which can be guaranteed. Hence, it is essential to establish that the measurement standard used has a sufficiently small uncertainty for the purpose.

5 Calibration systems

Requirements for measurement processes and measuring equipment are given in ISO 10012, and the general criteria for the operation of test laboratories are given in ISO/IEC 17025.

Where test laboratories carry out their own calibrations, they shall operate a management system conforming to ISO 10012. Where an outside calibration laboratory is used, it shall, wherever possible, be accredited by the relevant national accreditation body.

NOTE Information on accreditation of test and calibration laboratories can be obtained from national accreditation bodies.

6 Traceability

The calibration results for measuring instruments shall be traceable, wherever possible, to national standards.

7 Calibration intervals

Test equipment and measurement standards shall be calibrated at appropriate intervals established on the basis of the stability, purpose, and frequency of use of the equipment/standards. The intervals between calibrations shall be such as to ensure the reliability of the measurements made.

Because of differences in instrument performance, frequency of use, etc., it is not possible to give specific intervals in all cases. However, for the purposes of this International Standard, four classes of calibration interval are recognized for test equipment (a code-letter corresponds to each class):

a) C: requirement to be confirmed but no measurement;

b) N: initial verification only;

c) S: “standard” interval as given in this International Standard;

d) U: in use.

Regardless of the class of interval, equipment shall also be re-verified after any probable change in the accuracy of the equipment such as would be caused by disturbance, relocation, or repairs due to damage or wear.

In-use calibration is calibration at the time of use of the test equipment, usually before making test measurements.

The “standard” interval should preferably be chosen following the guidance given in ISO 10012. Some generally accepted intervals are given in Annex A of this International Standard.

8 Records

Records shall be kept for all measuring equipment and all calibrations carried out, as specified in ISO 10012.

NOTE 1 A calibration certificate for one parameter of an apparatus, even if from an accredited laboratory, does not constitute confirmation of the whole test equipment.

NOTE 2 Guidance on certificates for reference materials is given in ISO Guide 31.

9 Guide to the expression of uncertainty

However skilfully the calibration is performed, the result will always be subject to an associated uncertainty. An estimate of this uncertainty is required for each calibration in order that compliance with the specified requirements can be confirmed. The estimate of the uncertainty shall be made using accepted methods of analysis, combining the random and systematic errors, and shall include errors that are attributable to the measurement standard and those attributable to personnel, procedures, and the environment.

NOTE Guidance on the expression of uncertainty is given in ISO/IEC Guide 98-1 and ISO/IEC Guide 98-3.

A useful guide for test laboratories is that the uncertainty of measurement for transfer standards should be at least five times smaller than that required of the test equipment being calibrated.

10 Conditioning

Measurement standards and measuring equipment shall be calibrated and used in an environment controlled to the extent necessary to ensure valid measurements. Due consideration shall be given to temperature, rate of change of temperature, humidity, lighting, vibration, cleanliness (including dust control), and other factors affecting measurement. Where pertinent, these factors shall be monitored and recorded and, when necessary, compensating corrections shall be applied to the measurement data.

Generally, the ambient temperature for polymer testing is required to be (23 ± 2) °C, and calibrations would normally be carried out at that temperature. However, the usual practice in calibration is to calibrate at an ambient temperature of (20 ± 2) °C. Calibration of polymer-testing equipment at this temperature will be satisfactory for testing in the normal range. The apparatus to be calibrated and the measurement standard shall be conditioned at the calibration temperature for sufficient time for temperature equilibrium to be reached.

11 Procedures

Calibration is carried out following a defined procedure. Each parameter of an apparatus has its own procedure, but these procedures may be combined into a single procedure for the whole apparatus. This International Standard gives the methodology used for each parameter, arranged in sections according to the type of measurement involved (e.g. force, electrical). The information given is intended as guidance for test laboratories. Individual laboratories will have to formulate specific working procedures for the particular calibration equipment and transfer standards to be used, the method to be followed, and the records to be kept.

The number of replicate measurements to be made for each calibration will depend on the particular circumstances and has to be specified in the detailed procedures. Typically, between one and five replicates will be required. An estimate of the component of uncertainty due to the measurement process will require at least three, and preferably five, repeats, but, where this uncertainty has been estimated from a separate trial, a single measurement may be considered adequate.

Attention is drawn to the difference between calibrating a measuring instrument and verifying a quantity (e.g. the difference between a dial gauge and the specified length of a component of the test equipment). In general, the procedures given apply to measuring instruments or devices which form part of the apparatus, e.g. a voltmeter or pressure gauge. However, where appropriate, the procedure may also discuss the measurement of a quantity. A quantity is normally verified using a measuring instrument.

12 Expression of results

If necessary, corrections shall be applied to the readings obtained. When two instruments (the instrument being calibrated and a reference instrument) are being compared, the differences between the two sets of readings shall be tabulated with respect to the reading of the reference instrument. If required, the differences shall be plotted to produce a calibration curve. Where a quantity has been measured, the readings shall be recorded. The estimate of uncertainty shall be calculated.

NOTE There is sometimes confusion between the error in the indicated value and the correction to be made. For example, if the error is − 3 units, then the correction is + 3 units.

13 Calibration records

The information recorded shall include the following information:

a) a reference to this International Standard (i.e. ISO 18899);

b) a description and unique identification of the equipment calibrated;

c) the parameters measured;

d) the measurement procedures used;

e) a unique identification of the calibration standards used and reference to their traceability to an internationally recognized standard, e.g. a calibration certificate;

f) the date on which each measurement was completed;

g) the calibration results obtained after and, where relevant, before any adjustment or repair;

h) the assigned calibration interval;

i) the designated limits of permissible error;

j) the relevant environmental conditions and a statement of any corrections made necessary by these conditions;

k) the estimated uncertainty of the measurement results;

l) details of any servicing, adjustment, repairs, or modifications carried out;

m) identification of the person(s) performing the measurement;

n) identification of the person(s) responsible for ensuring the correctness of the recorded information.

14 Electrical measurements

14.1 Current

The measurement of current is largely confined to electrical and chemical tests. A variety of types of ammeter might be encountered and the range of current level and accuracy required is quite wide. In particular, some methods require the measurement of very small currents, and specialized procedures and calibration standards are needed to achieve the necessary low level of uncertainty.

The ammeter is compared to a standard instrument or to a standard current source.

Relevant International Standards: IEC 60051-1 and IEC 60051-9.

14.2 Voltage

The measurement of voltage is largely confined to electrical and chemical tests. A variety of types of voltmeter might be encountered and the range of voltage level and accuracy required is quite wide. In particular, some methods require the measurement of very small voltages, and specialized procedures and calibration standards are needed to achieve the necessary low level of uncertainty.

The voltmeter is compared to a standard instrument or to a standard voltage source.

Relevant International Standards: IEC 60051-1 and IEC 60051-9.

14.3 Frequency and bandwidth

The usual situation involving frequency is where a frequency generator requires verification, but there might also be a need to calibrate a frequency meter. The principle is the same in both cases.

Comparison is made with a standard frequency meter.

Relevant International Standards: IEC 60051-1 and IEC 60051-9.

14.4 Resistance

The measurement of resistance is largely confined to electrical tests. The usual situation is that a resistor or resistor network requires verification which is carried out using a calibrated resistance meter. As an alternative to using a resistance meter, the resistance may be measured by means of standard resistors in a bridge circuit.

The instrument is used to measure standard resistors.

Relevant International Standards: IEC 60051-1 and IEC 60051-9.

14.5 Wattage

Measurement of power consumption is occasionally required in testing but is more commonly used to monitor processing equipment. As an alternative to using a wattmeter or watt-hour meter, the voltage and current may be measured.

The wattmeter is compared to a standard instrument.

Relevant International Standards: IEC 60051-1 and IEC 60051-9.

14.6 Chart recorders

Chart recorders are used in a variety of applications, and a number of different types and sensitivities might be encountered. In some cases, the recorder scales will be calibrated integrally with, for example, a force transducer with which it is associated.

The recorder is compared to a standard signal or to a standard instrument.

Relevant International Standards: IEC 61143-1 and IEC 61143-2 for X-*t* recorders and IEC 61028 for X-Y recorders.

15 Dimensional measurements

15.1 Length-measuring instruments

The type of instrument used in polymer testing ranges from a microscope with graticules to a tape measure and, consequently, the range of magnitudes and accuracies to be covered is large. The International Standard used to transfer traceable calibration is chosen accordingly.

The measuring instrument is compared to a standard instrument, standard gauge blocks, or the equivalent.

Relevant International Standards:

— coordinate-measuring machines	ISO 10360-2
— dial gauges	ISO 463
— height gauges	ISO 7863
— micrometers (external)	ISO 3611
— roundness-assessment instruments (stylus type)	ISO 4291
— vernier callipers	ISO 13385-1

15.2 Linear dimensions

Depending on the circumstances, the magnitude of the dimension, and the accuracy needed, callipers, a coordinate-measuring machine, a height gauge, a travelling microscope, a dial gauge and rig, a ruler, etc., may be used to verify a dimension. Although, in most cases, this will be straightforward, in some instances it might be difficult to obtain access to the measurement location with an instrument of adequate accuracy.

15.3 Profiles

A variety of profiles will require verification, and a suitable instrument and technique has to be decided on in each case. In many instances, a projection system with a standard template is convenient.

15.4 Extension, compression, and deflection

Several types of extensometer are in common use, covering a considerable strain range. Additionally, dial gauges or other transducers, or the crosshead movement of the test machine, may be used to monitor extension, compression, or deflection.

The measurement device is compared to a standard instrument or a length standard.

Relevant International Standard: ISO 5893.

15.5 Finish, roughness, and flatness

Where a surface finish is specified, it is verified with a surface roughness meter, but, to measure flatness on a larger scale, a coordinate-measuring machine, a straight edge and gauge blocks or feeler gauges or a dial gauge may be used to map the surface profile.

Surface roughness meters are normally calibrated and adjusted in use by means of standard reference blocks.

Relevant International Standards: surface plates ISO 8512, surface texture ISO 4287.

15.6 Sieves, mesh, and pore size

A considerable range might be encountered from relatively coarse sieves to very fine pore sieves, which might necessitate more than one approach to calibration.

The relevant dimensions are measured using an appropriate calibrated instrument.

Relevant International Standards: sieves ISO 3310-2, microscope with graticules ISO 9344.

15.7 Area

Areas are usually verified by calculation from measurements of the relevant dimensions, but in some cases, comparison with a template as for profiles might be convenient.

15.8 Volume

Volumes other than that of glassware and similar containers are normally verified by calculation from measurements of the relevant dimensions or by weighing the amount of a liquid, e.g. water, that they hold. See also 19.1 Glassware.

15.9 Angle

Many angles encountered in test equipment are fixed and form part of a profile or might be verified by calculation from measurements of relevant dimensions. Where variable angles are involved, a scale will probably require calibration.

The indicated angles are measured using a calibrated protractor or the equivalent.

15.10 Levelling

The usual situation is that an apparatus requires to be levelled.

The level and/or level indication are verified by means of a standard level.

15.11 Centre of percussion

Specification of the centre of percussion is virtually restricted to impact testers and is found by calculation from the appropriate parameters of the pendulum, using standard formulae. Guidance is given in some test method standards.

16 Fluids: flow, pressure, viscosity, and density measurements

16.1 Flow meters

Flow meters can be for air, other gases, or liquids, and the calibration shall be valid for the fluid whose flow rate is to be measured.

The flow meter is compared to a standard instrument.

Relevant International Standard: ISO 7066-2.

16.2 Devices producing a specified flow rate

The device is verified using a calibrated flow meter or, if appropriate, by measurement of the quantity of fluid which flows in a given time.

16.3 Air exchange rate

Air exchange rate is specified for environmental chambers such as ageing ovens. The preferred method of verification is by measurement of the flow rate into the oven using a flow meter. However, for some designs this is not possible because there are multiple or inaccessible inlet points. Alternative procedures which might give estimates of the accuracy are measurement of the power consumption of the oven with normal air flow and with the air inlets blocked off or measurement of the time to fill a flexible sack attached to the air outlet.

16.4 Pressure transducers

Pressure gauges are much more commonly found in processing equipment than in test apparatus but, when required, might be for gases or liquids. A wide range of capacities may be encountered.

The pressure transducer is compared to a standard instrument.

16.5 Manometers

Where manometers are used rather than a pressure transducer, they are usually calibrated by measurement of the relevant dimensions, but they may also be compared to a standard pressure gauge.

16.6 Devices producing a specified pressure

In some test methods, it is specified that a pressure be produced by a device, for example, by using gravity. Such devices require verification by direct measurement of the pressure produced, using a jig and calibrated pressure transducer appropriate to the particular apparatus. These devices might require frequent reverification because of, for example, friction effects. In some cases, it might be acceptable to calculate the pressure by measurement of relevant parameters of the device.

16.7 Density

Density measurement usually involves the use of some form of balance, plus, in some cases, the use of a container of known volume or the measurement of dimensions. Therefore, the instruments to be calibrated are the balance and, possibly, also a container and dimension-measuring instruments. Specially designed density balances are commonly found which will additionally require calculations to verify the readings or a procedure involving comparison of weights of known relative mass.

Density columns are calibrated in use using standard reference floats.

17 Optical measurements

17.1 Irradiance

Irradiance measurement is required for weathering test methods, which should specify the wavelength range over which the irradiance is to be measured. A calibrated radiometer is used to measure the irradiance at a specified position relative to the light source. Spectrally non-selective radiometers shall have been calibrated to be traceable to the World Radiation Reference and filter radiometers by one of the methods given in CIE Publication No. 64.

17.2 Refractometers

The Abbe refractometer or an immersion method using a microscope are the most frequently employed, although other designs of refractometer are possible. When using a refractometer, the refractometer scale is calibrated using standard reference liquids of known refractive index and, in the microscope method, standard liquids are used during the determination in accordance with the test method standard.

17.3 Colour-measuring instruments

There are currently no International Standards for measurement of the change in colour of rubber products and, consequently, no calibration procedure is given.

18 Temperature measurements

In virtually all test methods, the temperature of the atmosphere in which the test is to be carried out and the test pieces are to be conditioned is specified. A variety of instruments are used to measure the temperature. They may be portable or built into the apparatus. The term thermometer is taken here to cover all types of instrument. There are two separate situations: A) that in which the thermometer is calibrated and B) that in which the atmosphere is verified.

Procedure A — The thermometer is compared to a standard instrument in a carefully controlled enclosure.

Procedure B — A thermometer is used to verify the atmosphere of a room or enclosure at various points covering the whole volume of interest.

19 Chemical analysis and reference materials

19.1 Glassware

Many specifications only require glassware to be of recognized standard grade, and no further calibration is necessary.

NOTE If calibration is required, it can be carried out by the procedures given in ISO 4787.

19.2 pH-meters

A variety of pH-meters and indicators with differing levels of accuracy are in common use. For most purposes, calibration can be carried out adequately by the use of buffer solutions, but, for higher-accuracy applications, calibration involves the input of signals from a standard millivolt source.

The meter is compared with standard buffer solutions or with a calibrated millivolt source.

19.3 Reference materials

A great many chemical-analysis methods rely on standard reagents and reference materials, the use of which effectively calibrates the measurement procedure. However, the certification of reference materials is outside the scope of this International Standard.

20 Relative-humidity measurements

In many test methods, the relative humidity of the atmosphere in which the test is to be carried out and the test pieces are to be conditioned is specified. The instruments used to measure humidity may be portable or built into the test room or test cabinet. There are two separate situations: A) that in which the measuring instrument is calibrated and B) that in which the atmosphere is verified.

NOTE Although some test methods specify tolerance limits of ± 2 %, this is not generally achievable with the instruments and calibration standards available.

Procedure A — The relative-humidity instrument is compared to a standard instrument or a standard atmosphere in a carefully controlled enclosure.

Procedure B — A relative-humidity instrument is used to verify the atmosphere in a room or cabinet at various points covering the whole volume of interest.

21 Force measurements

21.1 Tensile-, flexural-, and compression-testing machines

Machines used to measure tensile, compressive, and flexural properties are calibrated by verification of the essential parameters.

Relevant International Standard: ISO 5893.

21.2 Force transducers

Force transducers are usually incorporated into "tensile" machines but are also found in other types of apparatus. They can have a number of ranges, and some function in both the tensile and the compressive mode.

The transducer is compared to a standard proving device.

21.3 Devices producing a specified force

In some test methods, it is specified that a force be produced by a device, for example by using gravity. Such devices require verification by direct measurement of the force produced, using a jig and calibrated force transducer appropriate to the particular apparatus. These devices might require frequent reverification because of, for example, friction effects. In some cases, it might be acceptable to calculate the force by measurement of relevant parameters of the device.

21.4 Torque

Torque measurement is not commonly required but is used in certain low-temperature and adhesion methods, and some test rigs incorporate torque wrenches. Where the torque is produced by a weight and pulley system, calibration is usually carried out by calculation from the mass of the weight and the dimensions of the system. In ISO 1432 (the so-called Gehman test), a procedure for the calibration of the torque wire used is given in the International Standard.

The torque transducer is compared to a standard instrument.

Relevant International Standard: ISO 6789.

21.5 Energy

Energy is specified in tests involving impact. The energy of the impact device is verified by calculation from measurements of the relevant parameters of the apparatus. In some cases, it might be necessary to make corrections for friction.

21.6 Inertia

If the inertia of a component of an apparatus is required, it is verified by calculation from measurements of the relevant parameters of the apparatus.

22 Mass measurements

22.1 Balances

The measurement of mass is required in a large number of test methods and with a wide range of specified accuracies. However, the majority of measurements are made using a general-purpose laboratory balance reading to 1 mg.

The balance is used to measure standard weights.

Relevant International Standard: ISO 9368-1.

22.2 Weights

Weights in various forms are utilized in many test methods. In many cases, the object whose mass is to be verified includes a support and/or is made up of more than one weight. Weights can be calibrated by comparison with standard weights, but, for test purposes, the normal procedure is weighing using a calibrated balance.

23 Miscellaneous measurements

23.1 Timers, clocks, etc.

Timers, clocks, etc., are required in a great many test methods and include a very wide range of accuracies and time intervals. The principle of calibration is the same in all cases, but calibration standards of several different levels might be required. However, for the majority of purposes, comparison with the telephone "speaking clock" is satisfactory.

The timer is compared to a standard time signal over time periods appropriate to the intended use of the timer.

23.2 Time intervals

Time intervals are commonly specified and are normally measured with a timer, clock, etc. Where intervals are set automatically, they are verified by means of a calibrated timing device. In certain cases, the timing device may conveniently be a frequency counter.

23.3 Frequency and counters

A number of test methods involve the frequency of events (including revolutions executed by a rotating component) or counting the number of such events. The general approach taken to calibration is to count the events over a set time period, but exactly how this is done depends on the particular circumstances. For lower frequencies, it can be done manually, but commonly the apparatus will incorporate a mechanical or electrical counter. Such counters can be verified by inputting a known frequency from a mechanical or electrical source, as appropriate, although it may be satisfactory for mechanical devices to simply check for correct operation.

23.4 Velocity

Velocity (speed) is specified in a variety of tests, and the magnitude and accuracy required varies considerably. Verification is usually carried out by calculation from measurements of time and distance, using appropriate instruments of the required accuracy.

Air velocity, in ovens for example, is normally verified by calculation from the volumetric flow rate and the oven dimensions.

23.5 Tachometers

Commonly, a fixed angular velocity is specified which requires verifying with a calibrated tachometer. Where variable speeds are involved, a tachometer will probably be incorporated in the apparatus and hence will require calibration.

The tachometer is compared to a standard instrument.

23.6 Rate of heating or cooling

Heating and cooling rates are verified by measurement of temperature as a function of time. In many cases, the measurements are conveniently displayed graphically to demonstrate variations in the rate over the time interval of interest.

24 Calibration schedules

For the test equipment specified in each test method standard, a calibration schedule should be drawn up which lists the parameters to be verified, the requirements, and a reference to the document specifying the verification procedure and verification frequency.

Annex A
(informative)

Calibration intervals

For most equipment an initial interval of 1 year will be satisfactory, but in cases of doubt an initial interval of 6 months is recommended.

For some measurements, the following maximum intervals have been generally accepted:

Weights: Class F1 and below	1 year
Class E2 and above	2 years
Weighing machines	1 year plus before-use check
Thermometers: Mercury-in-glass	5 years plus annual check
Platinum resistance	1 year
Hardness testers	1 year plus in-use check
Humidity-measuring instruments	1 year
“Tensile” machines	1 year
Extensometers	1 year
Torque transducers	1 year
Steel rules	5 years plus annual check
Hardness blocks	6 months
Analogue meters	1 year
Bridges	1 year
Digital meters	1 year
Voltage standards	1 year
Potentiometers	1 year
Recording instruments	6 months
Signal generators	1 year
Watt-hour meters	3 months
Accelerometers	2 years
Micrometers and vernier and dial gauges	2 years
Mechanical limit gauges (gap, plug, ring, screw, etc.)	3 years
Surface plates	3 years
Pressure and vacuum gauges	1 year

