Calibration Technology

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Calibration Technology

Basics, reference instruments for pressure and temperature, professional calibration

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Basics, reference instruments for pressure and temperature, professional calibration

Christian Elbert

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Introduction

Every measuring instrument is subject to ageing as a result of mechanical, chemical or thermal stress and thus delivers measured values that change over time. This cannot be prevented, but it can be detected in good time by calibration.
The Egyptians already knew this almost 5000 years ago. The workers calibrated their yardsticks by comparing them with a "royal cubit" (approx. 52.36 cm) made of stone and thus managed to achieve, for example, side lengths on the Cheops pyramid of 230.33 m which differ from each other by only about 0.05 per cent.

Definition

In the process of *calibration*, the displayed value of the measuring instrument is compared with the measuring result of a different measuring device which is known to function correctly and accurately and which itself has been made to coincide directly or indirectly with a national (or international) reference instrument (standard) (Fig. 1). One talks about *verification* when the calibration has been carried out or supervised by an official body. Both of these variants are purely intended for determining the quality of the displayed values. No intervention to the measuring instrument itself is allowed. With *adjustment*, it is understood that there is an intervention to the measuring device in order to minimise a detected measuring deviation. Typically, adjustment is followed by a further calibration, in order to check and document the final state of the measuring instrument following the intervention.

Period of validity

In contrast to verification, which will lose its validity after a period of time set by law, the validity period of a calibration is subject to practical specifications such as manufacturer's

Fig. 1: Accredited calibration laboratory for the measurement parameter "temperature"

instructions, requirements of a quality assurance standard or in-house and customer-specific regulations. Calibration must also be carried out when the measuring instrument is used for the preparation of products which are subject to official supervision such as drugs or foodstuffs.

Significance

As part of a survey of 100 management executives of international companies, the Nielsen Research Company established in 2008 that due to faulty calibrations, manufacturing companies were losing more than 1.7 million dollars annually on average, and those companies with a turnover of more than a billion dollars as much as 4 million dollars.

In connection with limitations on resources and the required increase in efficiency of manufacturing processes, calibration is increasingly gaining in importance. Increasing the measuring accuracy may result in raw material savings and fewer emissions of pollutants, for example, by supplying exactly the right

Fig. 2: Calibration of pressure measuring instruments with a portable calibrator

amount of oxygen during a chemical reaction. The calibration of measuring instruments can sometimes also be relevant to safety: if pressure or temperature sensors (in the chemical industry, for example) do not provide correct values, the incorrect control of chemical processes may even result in a risk of explosion (Fig. 2). At the very least, the importance of calibration can be seen in everyday examples such as in household gas or water meters and in fuel gauges at petrol pumps.

In this book, the basics of calibration and calibration technology will be presented. It will describe which rules, methods and reference instruments are suitable for professional calibration. Pressure and temperature measuring instruments will serve as application examples.

Traceability and calibration hierarchy

Hierarchy of the standards and calibration services

To be able to compare measuring results, they must be “traceable” to a national or international standard via a chain of comparative measurements. To this end, the displayed values of the measuring instrument used or a measurement standard are compared over one or several stages to this standard. At each of these stages, calibration with a standard previously calibrated with a higher-ranking standard is carried out. In accordance with the ranking order of the standards – from the working standard or factory standard and the reference standard to the national standard – the calibration bodies have a calibration hierarchy. This ranges from the in-house calibration laboratory to the accredited calibration laboratory and to the national metrological institute (Fig. 3).

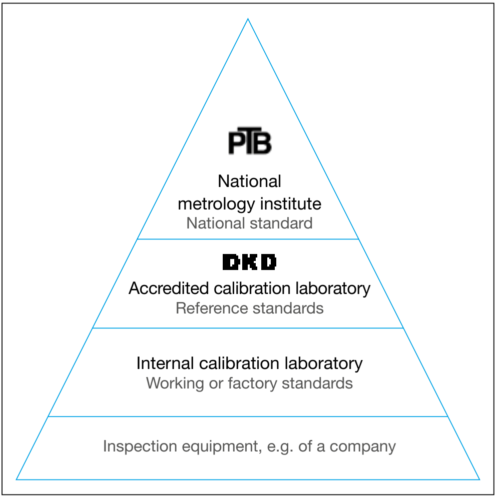


Fig. 3:
Calibration hierarchy described by the example of Germany

Traceability in practice

The *German Calibration Service* DKD (Deutscher Kalibrierdienst) designates the following as essential elements of traceability:

- The chain of comparison must not be interrupted.
- In each stage of the calibration chain, the measurement uncertainty must be known so that the total measurement uncertainty can be calculated. As a rule, a higher-ranking measuring instrument should have a measuring accuracy three to four times higher than the instrument calibrated by it.
- Each stage of the calibration chain must be documented as must the result.
- All bodies carrying out a stage in this chain must prove their competence by means of accreditation.
- Depending on the required measuring accuracy and technical requirements, calibrations must be repeated at appropriate intervals.

Calibration on an international level

BIPM

On an international level, the BIPM (*International Bureau of Weights and Measures*, abbreviation of French: *Bureau International des Poids et Mesures*) coordinates the development and maintenance of primary standards and the organisation of international comparative measurements. Decisions about the representation of the primary standards are made by the CGPM (*General Conference for Weights and Measures*, abbreviation of French: *Conférence Générale des Poids et Mesures*). The participants of the conferences, which take place every four to six years, are the representatives of the 51 signatory states of the international Metre Convention and the representatives of those 26 associated member states without full voting rights.

National metrological institutes

On a national level, institutes are responsible in most cases for metrology. They maintain the national standards to which all calibrated measuring instruments can be traced and ensure that these primary standards are comparable on an international level.

National or private institutes

In most countries, the top metrological institutes are state agencies or authorities. Thus, the national metrology institute of the Federal Republic of Germany PTB (*Physikalisch Technische Bundesanstalt*) in Braunschweig and Berlin is directly responsible to the Federal Ministry of Economy. In the USA, the NIST (*National Institute of Standards and Technology*) in Gaithersburg (Maryland) operates under the authority of the US Department of Commerce. However, in some countries, the national institutes are privatised. Thus in the U.K., the "guardian of measures" belongs to the SERCO Group.

The best-known of the standards stored at PTB are without doubt the atomic clocks, which

Fig. 4: Atomic clock of the PTB (German national metrology institute)

serve among other things as the basis for the time signal in radio clocks and watches (Fig. 4). As the national metrological institute, the PTB also has the legislative mandate to offer scientific and technical services in the area of calibration to science and commerce. For this, it uses a network of accredited institutions.

Accredited calibration laboratories

Accredited calibration laboratories often take on calibration as external service providers for those companies that do not have the required equipment themselves. However, they themselves can also be part of a company and calibrate all measuring instruments within it. To this end, they are equipped with their own working or factory standards which are calibrated at the proper time intervals with the smallest possible measurement uncertainty using the reference standard of the appropriate national metrological institute or other accredited calibration laboratories.

Coordination by the EA

To ensure the same level of all calibrations, the laboratories carrying them out must be accredited in accordance with internationally recognised rules (DIN EN ISO IEC 17025). Within the EU, this is coordinated by the EA (*European co-operation for Accreditation*). In the member states, calibration laboratories can obtain a certificate from the national agencies responsible for them which certifies that they are working in accordance with these regulations. In Germany, the responsible body is the DAkkS (abbreviation of German: *Deutsche Akkreditierungsstelle*), which took over this mandate from the DKD from 17.12.2009. As an expert committee of the PTB and an association of calibration laboratories in industrial companies, inspection institutions and technical authorities, the DKD nowadays works exclusively on specialist grass-roots projects such as the development of standards and directives (Fig. 5).

Fig. 5: Logo of the German Calibration Service

DAkkS carries out a complete evaluation of every accredited calibration laboratory every five years and also pays monitoring visits every 18 months, to ensure that the high demands of measuring processes are met. In addition to the described process monitoring of laboratories, there are also expert committees to ensure technical development and knowledge transfer.

EU-wide recognised calibration certificates

Since all European bodies that accredit calibration laboratories collaborate in the EA, the work procedures of the laboratories have been matched to each other. This is why calibration certificates issued by any calibration laboratory are also recognised in all other European countries.

In-house calibration

It is up to each individual company how to set up its in-house calibration system, but all measurement and test equipment must be calibrated with its own reference standards, which in turn must be traceable to the national standard. As proof, in-house calibration certificates are issued.

Professional calibration

The professional execution of calibrations is governed by various standards, regulations and directives. For a measuring instrument to be calibrated in the first place, it must fulfil certain basic requirements. The physical conditions under which calibration can be carried out must also be known and taken into account. Under these conditions, it is possible to select a calibration procedure suitable for the requirements.

Standards, regulations and calibration directives

In essence, regulations for the calibration of measuring instruments take effect whenever a company decides to observe a standard or directive for its calibration or when it manufactures products whose production is subject to legal regulations.

Quality assurance standards

Of great importance for quality assurance are standards and directives such as the ISO 9000 series of standards, which is being implemented more and more frequently in all industrialised nations. In Clause 7.6 "Control of monitoring and measuring equipment" of the ISO 9001:2008 standard "Quality management systems – Requirements", there is a specific requirement that any inspection equipment that directly or indirectly affects the quality of the products must be calibrated. This includes, for example, test equipment used as a reference in measurement rooms or directly in the production process. The ISO 9000 standards do not stipulate a validity period for calibrations – which would not make a lot of sense given the different

Fig. 6:
Calibration sticker on a mechanical pressure gauge

technologies of measuring devices – but they do specify that any inspection equipment must be registered and then a distinction must be made as to whether or not it must be regularly calibrated. Inspection plans must be drawn up in which the scope, frequency, method and acceptance criteria are defined. Individual calibrations are to be documented in detail. Labels on the measuring instruments (Fig. 6) or appropriate lists must show when each piece of inspection equipment needs to be recalibrated. It is essential to recalibrate when a measuring instrument has been altered or damaged during handling, maintenance or storage.

Requirements for measurement management systems

Closely related to the ISO 9000 series of standards in terms of its structure is the ISO 10012:2004 standard "Measurement management systems – Requirements for measurement processes and measuring equipment". It defines the requirements of the quality management system that can be used by companies in order to establish confidence in the measurement results obtained. In measurement management systems, it is not only the measuring device but the entire measuring process that is considered. This means that those responsible not only have to determine the measurement uncertainty during calibration, but also have to verify and evaluate the measurement uncertainty in use. To this end, statistical methods are also used.

Industry-specific directives

In addition to such universal standards, individual sectors of industry have their own directives for the quality assurance of measuring devices, for example the automotive industry. American automobile manufacturers have developed the QS 9000 directive in which the ISO 9000 standards have been substantially supplemented by industry and manufacturer-specific requirements, and in part tightened. In the meantime, the American QS 9000, the German VDA 6.1 and other country-specific regulations have been combined to some extent in the international ISO/TS 16969 standard. This saves many suppliers multiple certifications.

Legal provisions

Quality assurance standards and directives must only be observed by companies that want to be certified. The situation is completely different when, for example, drugs, cosmetics or foodstuffs are being manufactured. Here legal regulations, whose compliance is controlled by state agencies, often apply. Due to international trade relations, the regulations of the American Food and Drug Administration (FDA) are important. Thus, the *Code of Federal Regulation* (CFR) requires the "calibration of instruments, apparatus, gauges, and recording devices at suitable intervals in accordance with an established written program containing specific directions, schedules, limits for accuracy and precision, and provisions for remedial action in the event accuracy and/or precision limits are not met". European laws have similar stipulations.

Requirements for calibration services

Apart from the regulations mentioned, which all aim at the necessity of calibration, there are also regulations for calibration itself. These must be strictly adhered to by the body performing the calibration in order to achieve legal acceptance of the results. The most important of these is DIN EN ISO/IEC 17025 "General requirements for the competence of testing and calibration laboratories" (Fig. 7). It describes the process-oriented requirements of calibration laboratories and serves as basis for accreditation.

Fig. 7: Accredited calibration laboratory for the measurement parameter "pressure"

Additional directives

National institutes and affiliated technical committees publish directives and documents to supplement this standard, in which concrete technical and organisational procedures are described which serve as a model to the calibration laboratories for defining internal processes. These directives can in turn be part of quality management manuals for companies entrusted with calibration. They guarantee that the instruments to be calibrated are treated identically and that the work of the bodies entrusted with calibration can be verified. In Germany, this function is performed by the DKD.

Calibration capability

Before a measuring instrument can be calibrated, it must be checked as to whether it is suitable for this. The decisive factor is whether the condition of the instrument conforms to generally recognised rules of technology and to the manufacturer's documentation. If calibration is possible only after maintenance or adjustment, this work must be agreed upon beforehand between the customer and the calibration laboratory (Fig. 8). Any subsequent intervention to the instrument will void a prior calibration.

Testing the measuring instrument

To determine whether a measuring instrument is fit to be calibrated, its general condition and function are first checked. When calibrating mechanical pressure measuring instruments,

Fig. 8: Adjustment of a Bourdon tube pressure gauge

the DKD directive DKD-R 6-1 designates, among other things, the following main tests:

- visual inspection for damage (pointer, thread, sealing face, pressure channel)
- contamination and cleanliness
- visual inspection with regard to labelling, readability of the displays
- check whether the documents required for calibration (specifications, instruction manual) are available
- proper functioning of the operating elements (e.g. adjustability of the zero point)
- adjusting elements in a defined position
- torque dependence (zero signal) during mounting.

For other measuring instruments, similar requirements apply in most cases.
In connection with the calibration capability, very elementary questions also arise such as: Is the measuring instrument accessible? Can the measuring instrument be dismounted? Is it possible to calibrate the measuring instrument under conditions similar to those in which it is used?

Ambient conditions

Measured values often depend to a greater or lesser extent on ambient conditions such as temperature, humidity, exposure to dust and electromagnetic radiation. Thus, the Egyptians' royal cubit made of granite expanded at an increase in temperature of 30°C, not unusual for a desert climate, by 0.05 per cent – this alone would already explain the dimensional deviations during the construction of the Cheops pyramid. This is why measuring instruments should be calibrated if possible under the same ambient conditions in which they will be used.

Ideal: calibration under operating conditions

Fig. 9: Temperature control during calibration

Temperature is a particularly important influence quantity in pressure measurement (Fig. 9). This is why for the accuracy classes 0.1, 0.25 and 0.6, calibration must take place in a range of ±2°C, and for all others in a range of ±5°C relative to the reference value. Prior to calibration, the measuring instrument must be given enough time to adopt the ambient temperature or to warm up to a constant operating temperature. In particular therefore, mechanical devices of large mass such as tube pressure gauges should be acclimatised overnight in the calibration room.

Example: calibrating a pressure balance

The equation represented in Figure 10 shows which influencing factors must be taken into account, for example, when calibration is made with a pressure balance (see *Reference standards for the calibration of pressure measuring instruments*, p. 26 ff.). With this calibration instrument, pressure is produced mainly by metal weights which generate a load on a known piston area. The piston area changes with temperature because the piston

$$p = \left[\frac{m \cdot \overbrace{\left(1 - \frac{\rho_l}{\rho_m}\right)}^{\text{Buoyancy}} \cdot \overbrace{g_l}^{\text{Local value of gravitational acceleration}}}{A_0 \cdot \left[1 + \underbrace{(\alpha+\beta)\cdot(T-T_0)}_{A = f(T)} + \underbrace{\lambda \cdot p}_{A = f(p)}\right]} + (\overbrace{\rho_{Fl} - \rho_l)\cdot g_l \cdot \Delta h}^{\text{Height difference}} \right] \cdot 10^{-5}$$

Fig. 10: Influences on the measurement results of a pressure balance:

p	*Pressure in bar*
m	*Mass load*
ρ_l	*Air density*
ρ_m	*Density of the masses*
g_l	*Local value of gravitational acceleration*
A_0	*Piston area under standard conditions*
α, β	*Temperature coefficients*
T	*Piston temperature*
T_0	*Reference temperature (293.15 K)*
λ	*Pressure distortion coefficient*
ρ_{Fl}	*Density of the pressure medium*
Δh	*Height difference*

material can expand thermally. Due to their volume, the weights experience a certain buoyancy which depends on their own density, but also on the density of air. The latter is affected by the ambient temperature and the humidity. The value of the acceleration due to gravity (also called gravitational acceleration or gravitational constant) is not a constant at all. Due to the earth's rotation around its axis and its topography and the inhomogeneous mass distribution in the earth interior, the gravitational constant varies regionally by a few per mille. In addition, it will decrease the farther you move away from the earth's surface. Thus, the gravitational constant decreases by about 0.001 per cent when going up to the next floor in a factory building. This is an order of magnitude that is no longer negligible for high-quality pressure balances.

The ambient conditions must be checked even after a calibration has been concluded. The reason for this is that, for example, sudden changes in temperature can affect the measuring instrument in such a way that calibration becomes void, which is why a calibrated measuring instrument must be professionally stored and transported.

Calibration sequences

Selection of the calibration points

In general when a measuring instrument is used, its measuring range is only partially utilised. The calibration points must be selected such that they are within the working range of the inspection equipment. In calibration, at least the upper and lower process points must be considered. To check the linearity of the test item, at least one more checkpoint lying between these end points is measured. To compare the measured values of the measuring instrument and the reference or working standard, the measurement parameter can, in many cases, be adjusted either according to the displayed values of the test item or according to the readings of the standard.
In many cases, the number of measuring points for the calibration of a measuring instrument is not defined in a standard. In this case, close coordination between the calibration laboratory and the user is required in order to define suitable test points.

Calibration of pressure measuring instruments

The calibration of pressure measuring instruments is regulated in great detail. The document DKD-R 6-1 of the German Calibration Service DKD describes three calibration sequences for different accuracy classes (Table 1). The calibration sequence designated by A for the accuracy class < 0.1 prescribes three loadings up to the full scale value, to be completed

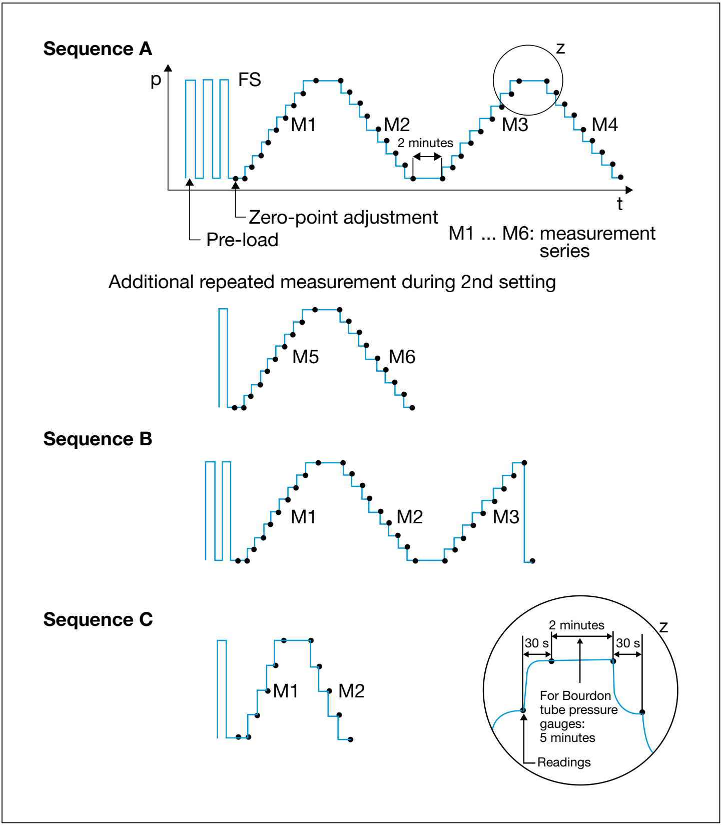
Table 1: Calibration sequences as per DKD-R 6-1 (Source: DKD-R 6-1, p. 11)

Calibration sequence	Desired accuracy class	Number of measuring points	Number of pre-loads	Number of measurement series	
				up	down
A	< 0.1	9	3	2	2
B	0.1 to 0.6	9	2	2	1
C	> 0.6	5	1	1	1

prior to the actual measurement series (Fig. 11 top). The maximum pressure is held for at least 30 seconds and is then completely released again. Over the course of the first measurement series, nine measuring points evenly distributed over the measuring range are measured by gradually increasing the pressure from "low to high". The zero point counts as the first measuring point, but is only included in the statistical evaluation if it is free-moving, i.e. if the pointer movement is not limited by a

Calibration sequence A

Fig. 11: Illustration of the calibration sequences as per DKD-R 6-1 (Source: DKD-R 6-1, p. 12)
FS Full scale value



stop pin. The pressure must be increased at such a low rate that the intended measuring point is not exceeded, since due to hysteresis (see p. 51 ff.), this could lead to a falsification of the results. If necessary, the pressure must be sharply reduced again to ensure that the measuring point is reached from below in all instances. The value reached is read after a holding time of at least 30 seconds. This holding time is necessary as the value displayed for the test item is not adopted directly, but only reached after a certain relaxation time (Fig. 11 bottom right). With tube pressure gauges, the housing should be given a light knock prior to reading in order to minimise friction effects on the movement. The final value at the last test point is also read and documented after 30 seconds. After two minutes at constant pressure, this value is read a second time. With tube pressure gauges, the maximum pressure should even be held for five minutes. In contrast, with piezoelectric sensors, the holding times can be reduced. In the second measurement series, the same measuring points are measured from “top to bottom” starting with the maximum pressure. To do so, the pressure is lowered such that it does not fall below the respective intended value. At the end of this first measurement cycle, the zero point is measured. For this, the instrument should remain depressurised for two minutes.

Calibration sequence A requires that the described cycle of increasing and decreasing the pressure by degrees is repeated once. If clamping effects are observed in pressure measuring instruments with large measuring ranges or exposed flush diaphragms, a third measurement cycle can be carried out in order to detect any dependence of the zero signal on the tightening torque – an effect often observed with low-cost electrical sensors.

Calibration sequence B

Calibration sequence B (Fig. 11 centre), which is used for pressure measuring instruments of accuracy classes 0.1 to 0.6, requires less effort: There are only two preloads up to the maximum value, and the third measurement series ends shortly after the pressure has reached the maximum value. From there, the pressure is discharged immediately down to the zero value.

Calibration sequence C

Calibration sequence C can be applied to pressure measuring instruments of accuracy class > 0.6 (Fig. 11 bottom). It only requires one preload up to the maximum value and only one measurement series, which, including the zero value and the maximum value, consists of five measuring points and in which the pressure is increased by degrees and then lowered again.

Calibration of temperature measuring instruments

Generating an accurately defined temperature is much more complicated than generating a particular pressure. This is why in the calibration of temperature measuring instruments, such extensive measurement series are usually not performed.

Calibration at a fixed point ...

In many cases, thermometers are only calibrated at a single fixed point such as the triple point of water. To adjust the temperature measuring instrument, its characteristic curve (usually very well known) is shifted upwards or downwards in such a way that the instrument displays the correct value at the fixed point.

... or in a measurement series

Measurement series are feasible when a temperature measuring instrument is not to be calibrated at a fixed point, but by comparison with a higher-quality measuring instrument. This is possible, for example, in immersion baths or ovens.

When performing such a measurement series, make sure that:

- enough time is available for the test item to adopt the temperature of the reference instrument
- the environment supplies a homogeneous temperature distribution over space and time, so that the temperature at the reference instrument has the same value as at the test item
- the immersion depth is sufficient and at least ten times the sensor tube diameter, to prevent heat from escaping to the environment.

Software validation

Since the measured data is increasingly recorded by computers, it is necessary also to check the software when calibrating a measuring chain. In particular, malfunctions may occur when modifications are made to the system environment. However, this is not referred to as calibration, but validation, i.e. an inspection that checks whether a process can fulfil the intended purpose.

Reference instruments

Depending on the application and measuring accuracy of the measuring instrument to be calibrated, a suitable reference instrument is required. This chapter describes reference instruments which can be used as reference standards or working standards and as portable calibration instruments using the measurement parameters pressure and temperature as an example.

Primary standard

The highest metrological requirements are met by primary standards whose measured values are recognised without reference to other standards, for example, by determining the measurement parameter via the parameters specified in its definition or via fixed points given by a natural law. Primary standards are usually used by national metrological institutes or accredited calibration laboratories. They serve as reference standards that can be used for calibrating the working standards or factory standards used in in-house calibration laboratories.

Hierarchy according to measurement uncertainty

It must be possible for each measuring instrument to be traced to a national standard by means of a chain of comparative measurements. In this chain, the measurement uncertainty of each reference instrument should be three to four times less than that of the measuring instrument to be calibrated. This also means that in each intermediate step, a loss in accuracy takes place and the measuring accuracy of the measuring instruments used in production is limited by their position in the calibration hierarchy. While, for example, pressure measuring instruments equipped with a pressure balance and used in the national institutes or calibration laboratories as primary standards achieve a measuring accuracy of 0.001 per cent of the measured value, portable pressure measuring instruments typically only achieve 0.2 per cent. In addition to more complicated measurement technology, a higher measuring accuracy also requires more effort and time during calibration.

Reference standards

Reference standards for the calibration of pressure measuring instruments

Pressure balances

The reference standards used for the calibration of pressure measuring instruments are pressure balances which are also referred to as deadweight testers (Fig. 12). They measure the physical quantity pressure with respect to its definition as force per unit area. From that point of view, pressure balances are considered as primary standards.

The core of the pressure balance is a very accurately manufactured piston/cylinder system with exactly measured cross-section. As the pressure transmission medium, the cylinder contains

Fig. 12: Deadweight tester

either a dry, purified, particle-free gas (such as nitrogen or synthetic air) or a hydraulic fluid. Since the gap between the piston and the cylinder is smaller than 1 µm, the system is sealed, even under high pressure, and has good running characteristics. Both the piston and the cylinder are manufactured from highly resistant tungsten carbide. This guarantees that the piston surface does not change in relation to temperature or pressure. A solid, stainless steel housing protects the piston/cylinder system effectively from contact, impacts or dirt from outside.

Operating principle

Via the piston, a force acts on the medium inside the cylinder from which the pressure inside the medium can be calculated – due to the known cross-section of the piston. This force is made up of the weight of the piston, the weights of the masses (calibrated with high accuracy and which are stacked on top of a bell-shaped device or a plate on the piston) and the weight of the bell or the plate (Fig. 13). The

Fig. 13:
Mass load, including trim masses of a few milligrams

Table 2: Characterisation of the weights of a pressure balance (extract from calibration certificate)

Designation of the weight	No.	Actual mass in kg	Pressure value for the system in bar
Piston	1262	0.08160	0.4002
Overhang (bell jar)	1	0.81560	3.9998
Plate	2	0.05097	0.2499
Mass	3	1.01954	5.0000
Mass	4	1.01954	5.0000
Mass	5	1.01954	5.0000
Mass	6	1.01954	5.0000
Mass	7	1.01954	5.0000
Mass	8	1.01954	5.0000
Mass	9	1.01954	5.0000
Mass	10	1.01953	5.0000
Mass	11	1.01952	4.9999
Mass	12	0.50976	2.5000
Mass	13	0.20391	1.0000
Mass	14	0.20391	1.0000
Mass	15	0.12234	0.6000
Mass	16	0.10196	0.5000
Mass	17	0.07137	0.3500
Mass	18	0.05098	0.2500

bell also serves to displace the centre of gravity of the assembly downwards so that the measurement can be more stable. The total mass made up of the piston, the applied weights and the bell or plate (also referred to as the weight applied) is proportional to the measured pressure.

Each pressure balance includes a set of numbered weights in the form of metal discs. The respective weights and the pressure values resulting from them are listed in the calibration certificate (Table 2). Depending on the desired pressure to be measured, the appropriate weights are selected as required.

Considering the local gravitational acceleration

The fact that the acceleration due to gravity, g, is subject to local fluctuations that are not insignificant can be taken into account when initially preparing the weights applied. If at the place where the pressure balance will be used, g is, for example, 0.25 per cent above the standard value of 9.80665 m/s^2, the weights are made 0.25 per cent "lighter". Alternatively, a deviation in the gravitational constant can also be corrected via the equation shown in Figure 10, p. 19, or else via a simpler equation that exclusively takes the dependence on the gravitational constant into account.

Pressure supply

To apply the exact test pressure predetermined by the weights applied to the system – made up of the pressure measuring instrument (sensor) to be tested and the piston – a pump integrated into the pressure balance or an external pressure supply is used. For fine adjustment, an adjustable volume regulated by a precision spindle is available. The approximate pressure can be read off a test pressure gauge mounted on the pressure balance. Once the desired test pressure has almost been reached, the rotary knob at the spindle is rotated and thus medium from the spindle is fed in until the piston along with the weights applied starts "floating". To minimise frictional forces, the system is carefully set into rotary motion at the weights applied. The pressure acting in the cylinder, and thus also on the pressure measuring instrument to be calibrated, then corresponds exactly to the quotient of the weight of the total mass applied divided by the piston cross-section. It remains stable for several minutes, also allowing any adjustment tasks taking a longer time to be carried out on the test item without problems.

Piston-cylinder systems

To allow calibration in different pressure ranges, different piston/cylinder systems can be selected (Fig. 14) such as pneumatic systems for pressures from 1 bar up to typically 100 bar. For higher pressures of up to typically 1000 bar or in special cases up to 5000 bar, hydraulic systems are used. In this case, the oil

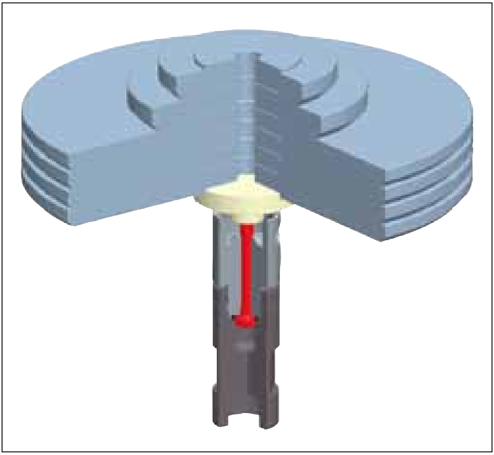


Fig. 14: Piston-cylinder system of a pressure balance

used serves simultaneously as a lubricant for the piston/cylinder unit and optimises the running characteristics.

Connecting the calibration item

Depending on the manufacturer, a wide range of pressure measuring instruments can be connected to the pressure balance via a standard male thread or a quick-release connector. This makes it possible to cover large pressure ranges with an accuracy of up to 0.004 per cent of the measured value. Thus, pressure balances are the most accurate instruments for the calibration of electronic or mechanical pressure measuring instruments. Due to their excellent long-term stability of five years – according to the recommendations of the DKD – they have been in use as reference standards for many years, not only at national metrological institutes, but also in calibration and factory laboratories.

Reference standards for the calibration of temperature measuring instruments

Temperature scales are established against fixed points for certain substances. It is well known that the Celsius scale is defined by the melting point and boiling point of water and a further 99 equidistant scale values lying in-between.

Gas-actuated thermometers

In metrological institutes, the temperature can be determined by gas thermometers. These measuring instruments are based on the fact that the vapour pressure of liquids depends very strongly on temperature. To measure an absolute temperature, either the temperature-dependent difference in volume at constant pressure or the temperature-dependent pressure differential at constant volume relative to the corresponding value at a calibration point is determined. However, this method of temperature measurement is fairly complicated.

Fixed point cells

This is why cells in which fixed points of high-purity substances can be set are more suitable for the calibration of temperature measuring instruments. As a function of temperature and pressure, substances exist in the three classical physical states – solid, liquid and gas. Phase transitions, for example from solid to liquid, can be used for calibration since at constant pressure, the temperature of a substance remains constant until the phase transition is complete, i.e. until all the ice of a water/ice mixture has become liquid.

Triple point cells

Apart from the phase transitions between two states of matter, for some substances triple points are also used as fixed points for calibration. At the triple point, the three conventional phases (solid, liquid and gas) of a high-purity substance are present in a thermal equilibrium. Figure 15 shows the phase diagram of water and the position of its triple point. Triple points can be set very accurately and reproduced at any time. Moreover, they can be maintained over a longer period of time.

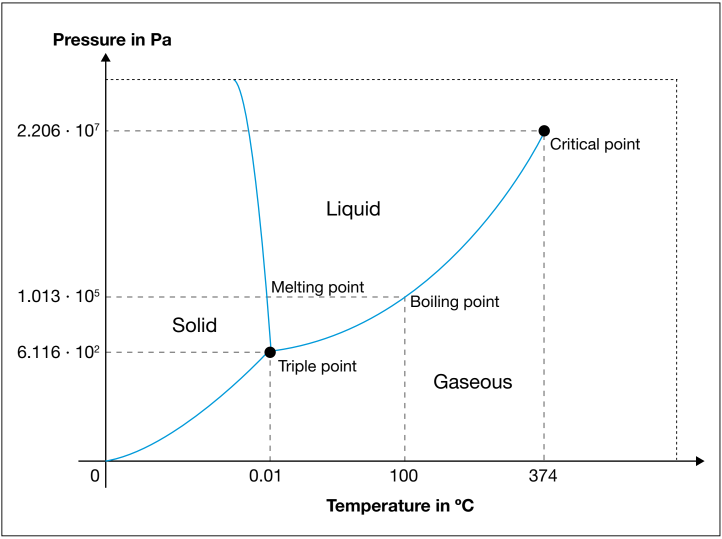


Fig. 15:
Phase diagram of water

For the purpose of calibration, in 1990 the CIPM (*International Committee for Weights and Measures*, abbreviation of French: *Comité International des Poids et Mesures*) established the ITS-90 (*International Temperature*

Table 3:
Selected fixed points as per ITS-90

Fixed point	Temperature in °C
Triple point of hydrogen	–259.3467
Triple point of neon	–248.5939
Triple point of oxygen	–218.7916
Triple point of argon	–189.3442
Triple point of mercury	–38.8344
Triple point of water	0.01
Melting point of gallium	29.7646
Melting point of indium	156.5985
Melting point of tin	231.928
Melting point of zinc	419.527
Melting point of aluminium	660.323
Melting point of silver	961.78
Melting point of gold	1064.18
Melting point of copper	1084.62

Fig. 16: Calibration with the water triple point cell

Scale of 1990), which defines temperatures via predefined temperature fixed points (Table 3).

Calibration with the water triple point cell

For the calibration of thermometers, the water triple point cell has the greatest importance (Fig. 16). To be able to use this fixed point cell for calibrating thermometers, it first must be cooled down to –6°C in a liquid bath. If this is done slowly enough, the water does not form ice in the cell, but rather remains liquid. Only after shaking the cell carefully does the water freeze completely from top to bottom within seconds. A closed ice jacket is formed around the inner tube. The water triple point cell is placed into a Dewar vessel which has been filled with crushed ice to a point just below the upper edge of the inner tube to prevent it from heating up too quickly. After a certain time, over a period of up to eight hours – depending on the number of calibrated thermometers – a constant temperature of +0.01°C (273.16 K) is obtained in the cell.

To calibrate thermometers by means of the triple point cell, a few millilitres of alcohol are poured into the inner tube for better heat transfer. Then, for example, platinum resistance thermometers can be inserted into the inner tube. After a few minutes, they will have adopted the temperature of the triple point, at which point the recording of the measured values displayed by them can begin.

Working standards

Working standards are usually calibrated by means of a reference standard. They are used for the quick and effective calibration of measuring instruments in calibration laboratories and factory laboratories.

Working standards for the calibration of pressure measuring instruments
The cost-effectiveness of the calibration of pressure measuring instruments and of their documentation depends on the level of automation possible (Fig. 17). The process can be automated to a high degree with measuring in-

Fig. 17: Automated calibration of pressure measuring instruments

Fig. 18: Example of a precision pressure controller

struments that have an electronic output, as in this case, a higher-ranking evaluation instrument can read the signal of the measuring instrument and communicate simultaneously with a pressure controller (Fig. 18), operating as a working standard.

Pressure controllers

Pressure controllers are devices controlled via a keyboard or PC connection that are capable of quick and stable delivery of a desired pressure. Depending on their design, these instruments work in the high-precision pressure range from approx. 25 mbar and high pneumatic pressure ranges of up to 400 bar up to ultra-high pressure applications of 2000 bar and more. Owing to the high compressibility of gases, a hydraulic system is used from approx. 400 bar upwards.

Pressure controllers generally consist of a reference pressure sensor (typically a piezo-resistive sensor) and a valve unit regulated via electronic control. To ensure that the output of a pressure controller is capable of delivering an exactly defined pressure, the instrument must be supplied from outside with an initial pressure above the desired maximum final value. If pressures below atmospheric pressure are also to be measured, an external vacuum sup-

ply, for example a pump, must also be available. Since the pressure measurement takes place electronically, no complicated mathematical corrections are necessary, in contrast to pressure balances. Pressure controllers achieve measurement uncertainties of at least 0.008 per cent of the measuring span.
The valve unit can have different designs: a simple 2-stage system, which only has the positions “open” and “closed”, and a more precise needle system, which enables a continuously variable larger or smaller aperture for the gas (Fig. 19). Although the desired pressures to be measured can usually be obtained a little more quickly using the 2-stage system, the continuously variable control has several advantages: applying the pressure more slowly reduces the mechanical stress on the measuring instrument to be calibrated. Since the speed of pressure build-up can be reduced near the measuring point, the risk of overshooting the desired pressure value is reduced. Its very precise control also makes it possible to compensate any slight losses in pressure caused by leaks in the tube system. The control charac-

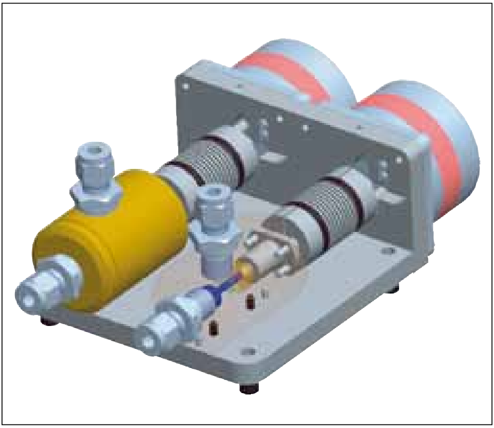


Fig. 19: Valve unit with needle system

teristics can be affected not only by the needle geometry, but also by the selection of materials. Suitable ceramic valve systems ensure, for example, that control is achieved independently of temperature and thus in a more stable manner.

Pressure controllers for automated calibration

It is not only in industrial plant that pressure measuring instruments need to be calibrated. Pressure sensors have also found their way into motor vehicles and washing machines. What is required in such calibration applications is less high accuracy, but rather high speed. Accordingly, there are also pressure controllers that have adaptive control algorithms, which by means of the temperature-insensitive ceramic valve system, are capable of moving to measuring points fully automatically within a few seconds. This allows five measured values of a pressure sensor whose job it is to determine the filling level of a washing machine, for example, to be checked within one minute (Fig. 20). Pressure controllers of this type still reach accuracies of up to 0.025 per cent. Frequently, overshoot-free control is sacrificed in favour of speed.

Fig. 20: Pressure controller with temperature-independent ceramic valve system

Working standards for the calibration of temperature measuring instruments

The calibration of thermometers at fixed points is very complicated. A much easier and quicker method is that of comparative measurement. This is done by exposing the thermometers to be calibrated together with a calibrated thermometer as a working reference to a constant temperature. As soon as thermal equilibrium has been reached, the temperature values can be read and any measuring deviations determined. Most calibration laboratories and factory laboratories usually use this method, also because it allows them to calibrate several thermometers simultaneously.

Standard resistance thermometers

The reference thermometers used are in general resistance thermometers. For the temperature range from −189°C to 660°C, accredited calibration laboratories require at least two 25-ohm standard resistance thermometers, depending on the operating range:

- one for temperatures from −189°C to 232°C which was calibrated at the triple points of argon and mercury listed in ITS-90 and at the fixed points of indium and tin
- one for temperatures from 0.01°C to 660°C calibrated at the fixed points of tin, zinc and aluminium.

Additionally, there are encapsulated thermometers that are used in cryostats down to −259°C and high-temperature designs up to 962°C.

Platinum resistance thermometers

However, in daily use the working standards used are not the standard resistance thermometers, but for example, platinum resistance thermometers which were calibrated by comparative measurements on these thermometers. The measuring resistor of these thermometers is a winding made of very pure platinum (Fig. 21). As their resistance

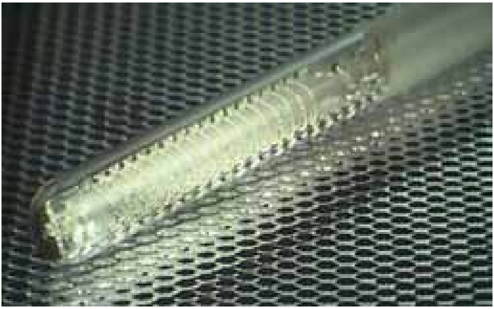


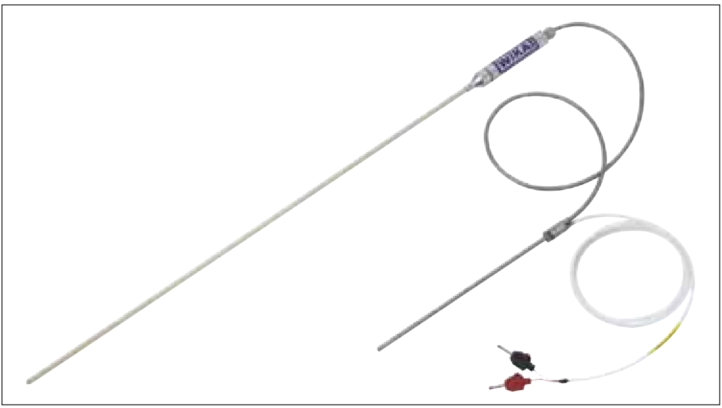
Fig. 21:
Platinum coil for platinum resistance thermometers

changes with temperature in a defined way, the temperature can be determined via the resistor. The precision of the resistance measurement greatly affects the measurement uncertainty of the thermometer calibration. The comparison resistor required in the measurement bridge is temperature-controlled to approx. 23°C. This allows measurement uncertainties below 1 mK to be reached.

Thermocouples

At temperatures above the operating range of resistance thermometers, the working standards used are usually thermocouples made of platinum and a platinum-rhodium alloy (Fig. 22). They are suitable for a tem-

Fig. 22:
Thermocouple made of platinum and platinum-rhodium alloy



perature range from 0°C to 1300°C. Platinum-gold thermocouples are even more stable due to the fact that they hardly age.

Liquid baths

A constant temperature over time and space is essential for the calibration of thermometers. Amongst others, this can be generated by liquid baths or heating ovens. Stirred liquid baths offer the most homogeneous spatial temperature distribution and a constant temperature over a long period of time (Fig. 23). Their properties depend on the geometry of the bath, the bath liquid used and the temperature range to be investigated. Each bath should have sufficient immersion depth for the thermometers and the highest possible volume in order to be able to keep a certain temperature constant over a longer period of time, even if thermometers are immersed and removed in the mean-

Fig. 23:
Stirred liquid bath

Temperature range in °C	Medium
–80 to 0	Methanol and ethanol
–35 to 70	Water-glycol-mixture (60% glycol)
10 to 80	Water
50 to 200	Silicone oil
180 to 500	Saltpetre

Table 4: Suitable temperature ranges of different tempering media

time. Good insulation in relation to the surroundings is also important. Pumps and stirrers are not allowed to introduce any heat into the bath. The temperature control fluid must have a high thermal conductivity and low viscosity. It must be inert in the temperature range under consideration and have low vapour pressure. Typical examples are listed in Table 4.

Each liquid bath is calibrated prior to its first use. This is done by measuring the temperature in axial direction – from bottom to top – and radial direction – from inside to outside – at three temperatures, i.e. at the initial, middle and final value of the measuring range, using a resistance thermometer as the working standard.

In the actual calibration, the resistance thermometers as the working standards and the thermometers to be calibrated are introduced into the liquid (by means of a positioning device) in such a way that they are very close to each other. This allows the effect of inhomogeneities to be minimised, achieving measurement uncertainties of below 5 mK.

Tube furnaces

The calibration of thermometers in a temperature range from 500°C to 1200°C requires tube furnaces (Fig. 24). The heating tube, well-insulated in relation to the outside of such furnaces, is heated electrically. The heated interior of typical tube furnaces has a diameter of 7 cm and is 50 cm long. At its centre is a compensation block made of

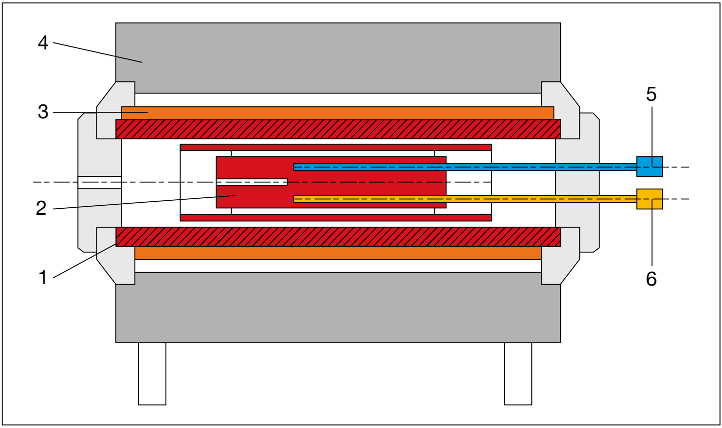


Fig. 24:
Schematic design of a tube furnace:
1 Ceramic heating pipe
2 Metal block with bore holes
3 Heating coil
4 Thermal insulation
5 Reference (standard)
6 Calibration item

nickel or steel containing six to eight concentric bores. The test items and the working standards are inserted into these bores. The mass and structure of this metal block and the good insulation of the furnace ensure that the temperatures in all bores are identical and remain stable over time. Nevertheless, a tube furnace must also be calibrated in the same way as a liquid bath. A particularly critical feature is the axial temperature profile, because a temperature gradient can easily form in the direction of the inlet openings of the bores.

In calibration, care must be taken that the bore diameter is not more than 0.5 mm larger than the diameters of the thermometers, since air gaps would hinder heat transfer. This is no longer so important above approx. 600°C, since heat transfer takes place mainly by heat radiation in this case.

As in the case of liquid baths, the test items and the standard resistance thermometers should be immersed into the block as deeply as possible. Ideally their measuring resistors are at the same height. Sensors that are shorter than 7 cm are best calibrated in a liquid bath, because the temperature of the furnace is no longer sufficiently constant over whole immersion depth.

Portable pressure calibration instruments

Dismounting a measuring instrument, packing it and sending it to an external calibration laboratory is often a complex and time-consuming task. In many cases, for example, if a production process would have to be interrupted while the instrument is removed, it would not even be possible. In this case, on-site calibration is the only option. Moreover, this has the advantage that the measuring instrument can be calibrated more easily as part of the entire measuring chain and its mounting position can also be taken into account.
Suitable instruments are also available for on-site calibration. Although their measurement uncertainty is lower than that of the reference and working standards described above, they are fully adequate for most industrial processes.

Portable instruments for on-site calibration of pressure measuring instruments

On-site calibration of pressure measuring instruments requires a working standard and a pressure source (Fig. 25). This can be a compressed nitrogen gas cylinder or an external hand pump which is available for pressures below 40 bar in a pneumatic version and up to 1000 bar in a hydraulic version. The most convenient solutions are portable pressure calibrators. They combine pressure generation and the working standard in a portable instrument. This avoids the cumbersome and time-consuming assembly of the instrument

Hand-held calibrators ...

*Fig. 25:
Portable pressure calibrator with accessories in a service case*

from several components and reduces the risk of leaks in the pressure system. Depending on the version, the pressure in the instrument is built up by means of a manual or electric pump. Calibration instruments of this type make it possible to achieve measure-

*Fig. 26:
Calibration of a process transmitter with portable calibrator*

ment uncertainties of up to 0.025 per cent of the measuring span.

Many models not only measure pressures, but also ambient temperatures and the electric signals output by the measuring instruments to be calibrated (Fig. 26). This makes it possible, for example, to check the correct functioning of pressure switches or process transmitters. Pressure switches close and open a valve upwards of a certain pressure value. Process transmitters convert the measurement parameter of pressure into a proportional electric output signal.

When calibrating a process transmitter, first the pressure connection and the electrical connection between the measuring instrument and the calibration instrument are established. To avoid contamination of the portable standard, a dirt trap can be connected in-between, as otherwise, the measuring instrument would operate with contaminated media. This is followed by performing a zero point adjustment with the valves open. The individual pressure test points can then be established by means of the integrated pump, and the delivered electric signals can be measured.

… with integrated data logger

Portable pressure calibrators are equipped with integrated data loggers. The measured values are recorded on site. The data can be read out later at the laboratory or office by connection to a PC and displayed in a log. Some are also equipped with an electronic evaluation system, which performs an error calculation and shows the result on the display immediately after calibration.

Portable instruments for on-site calibration of temperature measuring instruments

On-site calibration of thermometers is usually carried out with dry-well calibrators and micro calibration baths (Fig. 27). They are

Fig. 27:
Dry-well calibrator

basically smaller transportable versions of the heating ovens and liquid baths already described. In both, the reference thermometer used as a working standard is permanently built in and serves simultaneously to control the oven or bath temperature and to calibrate the test items.

Dry-well calibrators

Inserts with bores of different diameters can be built into the metal block of dry-well calibrators, enabling the testing of thermometer models of different types. In on-site calibration, a thermometer measuring insert can be taken out of its thermowell fitted in the plant and placed in the calibrator, without having to interrupt the electrical connections to the electronic evaluation system. Temperature sensors are often only calibrated at a single temperature – that of the process they monitor. Cali-

bration is carried out at a measurement uncertainty of 0.1 K to 3 K, depending on the temperature range and the properties of the calibration item.
Since all dry-well calibrators are closed at the bottom and open at the top, a temperature gradient inevitably forms in the bores, which may lead to measuring errors, especially if the temperature sensors to be calibrated are too short. They should be inserted into the calibrator as deeply as possible, i.e. about 15 cm. If the temperature sensors cannot be inserted into the bore of the calibrator by more than 7 cm, it is better to use an external reference thermometer as the working standard instead of the permanently built-in one (Fig. 28). For sensor lengths below 7 cm, micro calibration baths should be used.

*Fig. 28:
Portable calibrator with an external temperature sensor as reference thermometer*

Fig. 29: Micro calibration bath

Micro calibration baths

In contrast to the large-sized versions, micro calibration baths (Fig. 29) can only accommodate one or two test items, because the temperature of the relatively small amount of liquid cannot be kept constant as efficiently due to the heat dissipation of the thermometers. Despite the homogeneous temperature distribution in the bath, the thermometers to be calibrated must permit a minimum immersion depth. Depending on their design, this should be ten to fifteen times the sensor diameter. In the range from –35°C to 255°C, micro calibration baths achieve measurement uncertainties of 0.1 K to 0.3 K.

Calibration characteristics

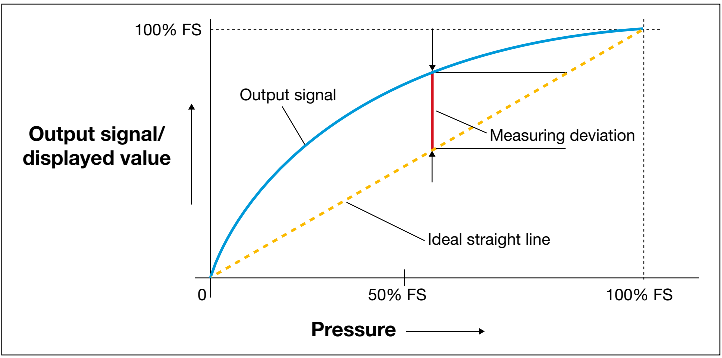
An analysis of the measured values obtained during the calibration sequence allows characteristic features of the measuring instrument to be determined which can be taken as an indication of its quality and suitability for the measuring process. The determination of typical characteristics such as measuring deviation, hysteresis and repeatability is described below.

Measuring deviation

Deviation from the "correct" value

The measuring deviation (also referred to as *measuring error*) indicates how far a value v_d displayed or output by a measuring instrument deviates from the "true" value v_t or from the "correct" value v_c (Fig. 30). The true value of a measurement parameter is an imaginary value. To determine this value, a measuring instrument of infinite accuracy and display would have to be available. The correct value is the value that is measured with an error-free or – in practice – high-accuracy measuring instrument such as a primary standard.

Fig. 30:
Definition and determination of the measuring deviation:
FS Full scale value



In general it should be noted that the measured value can deviate from the displayed value. Thus high-resolution digital displays may give the impression that a measuring instrument is particularly accurate. However, the number of displayed digits and the accuracy of the measuring instrument do not have to coincide. Ideally the accuracy of the measuring instrument is reflected in its resolution. It should be five to ten times higher than the desired accuracy. The display of an electronic pressure measuring instrument that can determine pressure with an accuracy of ±0.1 bar should in this case produce a digital value with one or two decimal places in the unit "bar".

According to DIN 1319, mechanical measuring instruments are divided into accuracy classes. Such an accuracy class includes all measuring instruments that meet predetermined metrological requirements, so that the measuring deviations of these instruments are within established limits. Thus, for example, the display of a pressure measuring instrument of accuracy class 0.6 deviates from the correct value by no more than 0.6 per cent. In contrast to instruments with a digital display, mechanical measuring instruments do not have a "fixed" resolution. Depending on the spacing of the individual division marks on the measurement scale, an interpolation between 1/3 und 1/5 of the scale graduation can take place.

Absolute and relative measuring deviation

The measuring deviation is specified either as absolute or relative error. The absolute error is indicated with a sign and has the same unit as the measurement parameter.

$$F = v_d - v_c$$

The relative measuring deviation refers to the correct measured value. Thus it is also indicated with a sign, but has no unit:

$f = (v_d - v_c)/v_c \cdot 100\%$

Systematic and random measuring deviations

A distinction must be made between systematic and random measuring deviations. Systematic measuring deviations are unidirectional, i.e. they have a magnitude not equal to zero and a sign. This is why systematic deviations always result in errors in the measuring result. Although known systematic measuring deviations can be compensated by means of mathematical methods or empirical data, a "calibration without adjustment" of this type is carried out only rarely in practice, so that its contribution to the measurement uncertainty (see p. 55 ff.) is usually calculated. Random measuring deviations are not unidirectional. Even if a measurement is repeated under the same conditions, the displayed measured values are scattered around the correct value in magnitude and sign. Thus, random measuring deviations "only" lead to uncertainties in the result. If it were possible to carry out an infinite number of repetitions, the random measuring deviations would average out, allowing the true value to be determined. In practice, the measurement uncertainty of the measuring instrument can be determined from an infinite number of repetitions by means of statistical methods.

Hysteresis

In connection with measuring instruments, the term hysteresis (according to DIN 1319, also referred to as *reversal error*) means that the value displayed by the test equipment not only depends on the input parameter (for example the applied pressure or the prevailing temperature), but also on the values of the input parameter that were measured previously. Figure 31 shows the values output by a pres-

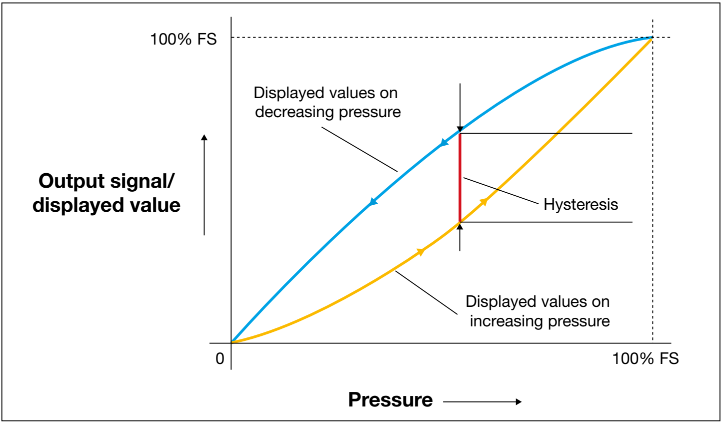


Fig. 31:
Definition and determination of the hysteresis:
FS Full scale value

sure measuring instrument both on increasing pressure (yellow) but also on decreasing pressure (blue). The latter were determined only after reaching the maximum value. Despite otherwise identical conditions, the measuring instrument shows different values at identical pressures. The correct measured value is in the range between two linear compensating curves and cannot be specified in more detail. One reason for hysteresis can be too slow a relaxation process in the measuring instrument. Such a process takes place, for example, when the Bourdon tube of a mechanical pressure measuring instrument does not react completely elastically to an elongation.

Evaluation of the hysteresis

If an evaluation of hysteresis is desired, it must be ensured that the intended pressure to be measured is approached “asymptotically”. The measured value must be reached without exceeding the nominal value. Otherwise, the hysteresis effects would be falsified. If accidentally a measured value is exceeded, the test pressure must first be reduced well below the nominal value before approaching the pressure to be measured again from the correct test direction.

Repeatability

Repeatability is a measure of how well a measuring result can be reproduced if it is determined again under the same conditions. In contrast to hysteresis, the input parameter must also follow the same curve. Figure 32 shows two measurement series on the same pressure measuring instrument. In both, the pressure is gradually increased at a uniform rate. Nevertheless, the displayed values differ from one another.

Consideration of clamping effects

The characteristic repeatability is also helpful when the values of the test item depend on the mounting state. In particular when the display of a pressure measuring instrument depends on the tightening torque of the threaded connection, repeatability can take into account the contribution of uncertainty in the later application.

For the determination of repeatability, a high *repeatable accuracy* is required. To determine it, the same measurement procedure must be repeated several times by the same person on the same measuring instrument and in the same place under the same experimental con-

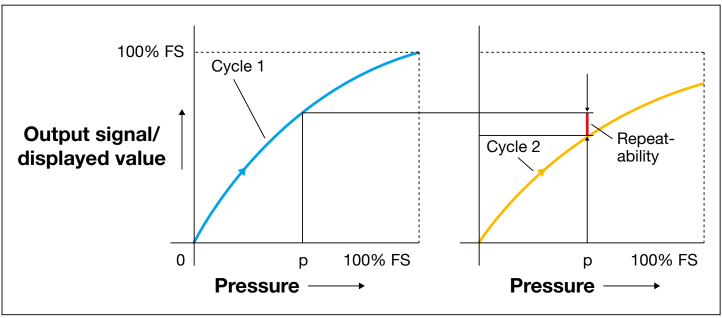


Fig. 32: Definition and determination of the repeatability: FS Full scale value

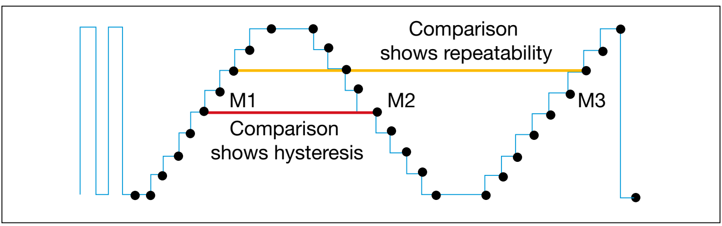
ditions; moreover, the independent individual measurements must be carried out within a very short time span. The higher the repeatable accuracy, usually the smaller the scatter of the measured value, and thus the measurement uncertainty of the instrument.

Determination of the characteristics in practice

Suitable calibration sequences

With a suitable calibration sequence, it is possible to determine the measuring deviation Δp, the hysteresis h and the repeatability b. The measuring deviation at a certain pressure corresponds to the difference between the mean value of all measured values of the test item and the pressure displayed by the reference instrument (reference or working standard). The hysteresis at a certain pressure is obtained from the difference between the individual measured values measured at increasing pressure and decreasing pressure. The repeatability at a certain pressure corresponds to the difference between the two measured values determined at increasing and decreasing pressure, respectively. The three calibration sequences described on page 20 ff. allow different evaluations. Sequence A allows hysteresis and repeatability to be determined twice each, sequence B gives repeatability and hysteresis once (Fig. 33), while sequence C gives hysteresis only once, but not repeatability.

Fig. 33: Calibration sequence B as per DKD-R 6-1 with evaluation



Measurement uncertainty

The measurement uncertainty can be determined from measuring results by statistical methods using technical scientific analyses. Manufacturers of measuring instruments often prefer the term *accuracy*, which has a positive connotation. However, this is a purely qualitative characterisation. A quantitative characterisation, i.e. by means of a numeric value, can only indicate by how much the values displayed on an instrument can deviate from the correct value.

Basics according to GUM

Definition

The term *measurement uncertainty* is defined by the International Vocabulary of Metrology as a characteristic assigned to the result of a measurement, for example in calibration, and which characterises the range of values that can be sensibly attributed to the measurement parameter by the completed measurement. Figuratively speaking, the measurement uncertainty indicates a range around the measured value in which the correct value lies with high probability, i.e. including how well the result obtained reflects the value of the measurement parameter. This can be of importance, for example, when products are to be checked as to whether they meet an established specification. If, for example, a component must not be longer than 10.0 mm, and the measurement gives 9.9 mm at a measurement uncertainty of ±0.2 mm, then the requirement might not be fulfilled.

In general, measured values are always scattered – even under identical measurement conditions – around an empirical mean value.

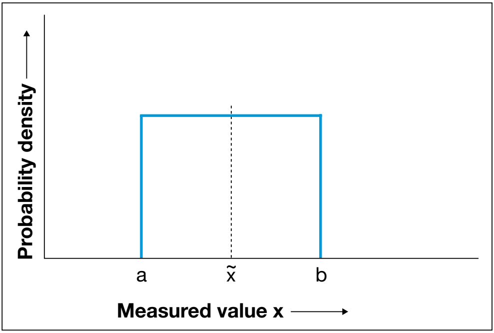


Fig. 34:
Illustration of a rectangular distribution:
x̃ Mean value
a Lower limit value
b Upper limit value

This arithmetic mean is calculated by adding up the individual measured values and dividing the result by the number of measurements.

Typical density distributions

To describe the scatter of the measured values in a good approximation, typically rectangular or Gaussian distributions are used. In a rectangular distribution (Fig. 34), the probability of measuring the parameter x in the interval from a to b is constant. Outside the interval it is zero. In a Gaussian distribution typical for random measuring deviations with its bell-shaped density function (Gaussian curve), the farther away from the mean value x̃, the smaller the probability of obtaining a measured value x (Fig. 35). The characteristic parameter of the Gaussian distribution is the standard deviation

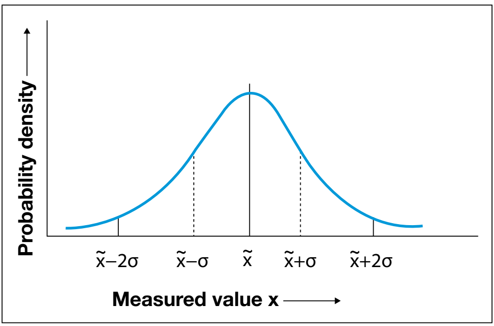


Fig. 35:
Illustration of a Gaussian distribution:
x̃ Mean value
σ Standard deviation

or its square, the variance. Both are a measure of how broad the distribution is and thus how large the scatter of the individual measurements is around their mean value.

Standard deviation = measurement uncertainty

The measurement uncertainty u is identical to the standard deviation. In the Gaussian distribution, the standard deviation is designated with σ and amounts to $1/\sqrt{3} \cdot (b-a)/2$. The *expanded measurement uncertainty* is obtained from the measurement uncertainty by multiplication by a factor k. In industry, this factor is 2 in most cases. In a Gaussian distribution, more than 95 per cent of all measuring results are in the interval $\tilde{x} - 2\sigma$ to $\tilde{x} + 2\sigma$.

Determination according to GUM

To enable accredited calibration laboratories to determine the measurement uncertainty according to identical aspects, the ISO/BIPM (International Bureau of Weights and Measures) published a guideline that establishes a procedure that has meanwhile also given its name to it: GUM (*Guide to the expression of Uncertainty in Measurement*). The GUM was translated by the German Calibration Service DKD under the name DKD-3 (German title: “Angabe der Messunsicherheit bei Kalibrierungen”) as a guideline for German calibration laboratories and was made tangible, for example in the DKD-R 6-1 guideline by means of application examples specifically for pressure metrology.

The basic idea of the GUM is to establish a model that describes the measurement with sufficient accuracy. This model sets the measurement parameter in relation to the input parameters, in order to determine the total measurement uncertainty u_{total} from the individual measurement uncertainty contributions u_i. A simple example is the pressure as the quotient of force and area, in which force is defined as the product of the mass, for example the applied weights of a pressure balance, and the acceleration due to gravity g. None of the three parameters (area, mass, g) can be determined accurately. This is why all contribute to the total measurement uncertainty. A more detailed analysis can include, as shown in Figure 10, p. 19, more ambient conditions such as the temperature.

Two methods according to GUM

The GUM describes two methods of determining the measurement uncertainty of the input parameters. Type A is a statistical analysis in which under identical conditions, at least ten measurements are carried out and the arithmetic mean and standard deviation are determined from the measured values. In type B, the measurement uncertainties of the input parameters are determined from scientific knowledge, for example from the following information:

- data from previous measurements
- general knowledge and experience regarding the properties and the performance of measuring instruments and materials
- manufacturer's instructions
- calibration certificates or other certificates
- reference data from manuals.

Measurement uncertainty budget

Sources of measurement uncertainties during calibration

Possible sources of measurement uncertainties during calibration are, for example, inherent in the:

- procedure: method, instrument type, number and duration of measurements, measurement aids ...
- calibration item: repeatability, quality of calibration, evaluation software ...
- ambient conditions: temperature, atmospheric pressure, gravitational constant ...
- reference instrument: accuracy, use under application conditions ...
- operator: experience, care, handling ...

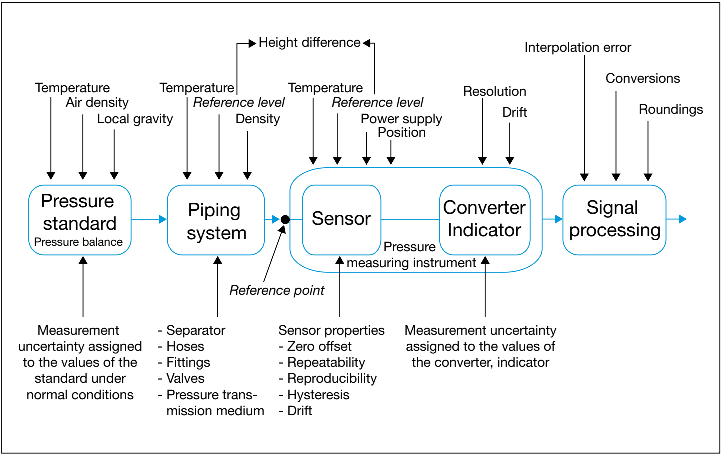


Fig. 36: Influencing parameters during calibration of a pressure measuring instrument (Source: DKD-R 6-1, p. 16)

Figure 36 shows all influence quantities that play a role in the calibration of a pressure measuring instrument.

Once all sources of measurement uncertainties during calibration have been established and evaluated quantitatively, a measurement uncertainty budget is drawn up. It contains the relevant standard deviations and a note of the method used to determine them in tabular form.

Typical relevant characteristics for the analysis of the measurement uncertainty are the measuring deviation, hysteresis and repeatability discussed in *Calibration characteristics*, p. 49 ff. They also include display fluctuations and zero offsets.

Determination of the total measurement uncertainty

To determine the total measurement uncertainty for non-correlating (i.e. independent) input parameters, the squares of the individual measurement uncertainties are added up and the square root is taken from the sum. If individual input parameters depend on one another, complicated analytical and numerical calculation processes are required.

Example calculation

The calculation of the measurement uncertainty will be discussed in more detail below, using the example of calibrating a pressure measuring instrument according to sequence B described in *Calibration of pressure measuring instruments*, p. 20 ff. Table 5 lists the pressure values displayed by the reference instrument and the mean values of the measurements in the test item, as well as the measuring deviation, repeatability b and hysteresis h. From the last-named, the individual measurement uncertainties u(b) and u(h) are obtained.

Table 5: Evaluation of the calibration parameters

Reading of calibrator in bar	Mean value μ in bar	Measuring deviation in bar	Repeatability b in bar	Hysteresis h in bar
0.0000	0.0000	0.0000	0.0000	0.0000
2.0000	2.0047	–0.0047	0.0006	0.0000
4.0000	4.0028	–0.0028	0.0007	0.0040
6.0000	6.0027	–0.0027	0.0003	0.0042
8.0000	8.0037	–0.0037	0.0002	0.0035
10.0000	10.0036	–0.0036	0.0006	0.0054
12.0000	12.0009	–0.0009	0.0018	0.0034
14.0000	14.0022	–0.0022	0.0000	0.0017
16.0000	16.0028	–0.0028	0.0007	0.0006

Determination of the individual measurement uncertainties

For the evaluation, the uncertainties due to the limited resolution of the display u(r) and the constant zero offset u(f0) are also included. It is assumed that these four uncertainties follow a rectangular distribution. In the case of hysteresis, this gives, for example:

$$u(h) = 1/\sqrt{3} \cdot h/2$$

Other measurement uncertainty contributions to be included are those of the standard u(N) and possibly measurement uncertainties due to the procedure u(V), e.g. when a digital multimeter was used for the evaluation of a pressure

	Calibration item				Standard	Procedure		
	u(K)				u(N)	u(V)	U_abs	W_abs
Portion	u(b)	u(h)	u(f0)	u(r)				
Factor	0.2887	0.2887	0.2887	0.2887	0.5			
bar	bar	bar	bar	bar	bar	bar	bar	bar
0.000	0.0000	0.0000	0.0000	0.0014	0.0013	0.00000	0.0038	0.0038
2.000	0.0002	0.0000	0.0000	0.0014	0.0013	0.00000	0.0038	0.0085
4.000	0.0002	0.0012	0.0000	0.0014	0.0013	0.00000	0.0045	0.0074
6.000	0.0001	0.0012	0.0000	0.0014	0.0013	0.00000	0.0045	0.0072
8.000	0.0001	0.0010	0.0000	0.0014	0.0013	0.00000	0.0043	0.0080
10.000	0.0002	0.0016	0.0000	0.0014	0.0013	0.00000	0.0050	0.0086
12.000	0.0005	0.0010	0.0000	0.0014	0.0013	0.00000	0.0044	0.0053
14.000	0.0000	0.0005	0.0000	0.0014	0.0013	0.00000	0.0039	0.0062
16.000	0.0002	0.0002	0.0000	0.0014	0.0013	0.00000	0.0039	0.0067

Table 6: Evaluation of the measurement uncertainty

sensor with electrical output signal. For these effects, the expanded uncertainties can usually be taken from manufacturer's instructions or existing calibration certificates. They are incorporated into the measurement uncertainty budget as simple uncertainty contributions, i.e. divided by the expansion factor k = 2.

Total measurement uncertainty and accuracy

Table 6 shows in detail the evaluation for each pressure stage measured value. The expanded total measurement uncertainty is ultimately given by U_abs. To determine this value, the squares of the individual measurement uncertainties are added up and the square root is taken from the sum and then multiplied by the expansion factor k = 2. In order to ultimately obtain a value W_abs for the accuracy of the measuring instrument, the systematic measuring deviation at each measuring point is added to the total measuring uncertainty.

Documentation

DKD calibration certificate

Every calibration must be documented in writing, although there are no generally binding regulations specifying which information has to be recorded. The exceptions are DKD calibration certificates issued by accredited calibration laboratories (Fig. 37). The data entered there apply only to the time of the actual calibration and may not be changed afterwards.


Alexander Wiegand SE & Co. KG

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Deutsche Akkreditierungsstelle GmbH

als Kalibrierlaboratorium im / as calibration laboratory in the

DAkkS

Deutschen Kalibrierdienst

DKD

Kalibrierschein

Calibration certificate

Kalibrierzeichen

Calibration mark

Fig. 37: Example of a DKD calibration certificate (cover sheet)

Minimum data required for a DKD calibration certificate

In accordance with the publication DAkkS-DKD-5 titled "Instructions for issuing a calibration certificate", all calibration certificates issued by an accredited calibration laboratory must contain the following minimum data:

- the title "Calibration certificate"
- the name of the accreditation body
- the accreditation number of the calibration laboratory
- the unique continuous number of the calibration certificate
- the definitions or procedures applied
- the measuring results and the associated measurement uncertainties or a statement on its conformity with an established metrology specification
- the authority according to which the calibration certificate is issued
- the conditions under which the calibrations or measurements were carried out (e.g. ambient conditions)
- a general statement on the metrological traceability of the measuring results
- in the case of adjustment or repair of an instrument to be calibrated, the calibration results before (if available) and after the repair.

Preparation of the measurement results and measurement uncertainties

Measuring results can be given in the form of equations and formulae or tables and graphics. For each measuring result, the associated measurement uncertainty must be given in all instances. Moreover, under normal circumstances (i.e. if the expansion factor 2 guarantees a 95 per cent coverage), the following note regarding the measurement uncertainties must be included in the calibration certificate: *"The measurement uncertainty given is the expanded* *measurement uncertainty obtained from the standard measurement uncertainty by multiplication by the expansion factor k = 2. It was determined according to DKD-3. The value of the measurement parameter is in the assigned interval of values with a probability of 95 per cent."* (Source: DAkkS-DKD-5, p. 13)

Period of validity of the calibration

The validity period of the calibration does not have to be explicitly stated. On customer request, the calibration laboratory can give a recommendation as to when recalibration should be carried out. However, this must be documented or marked separately.

Graphic evaluation

To illustrate the result of the calibration, it can be represented graphically (Fig. 38). To this end, in most cases the absolute or relative measuring deviations and, in addition, the associated (relative) measurement uncertainties are entered in the form of bars for the points investigated. The relative values are either referred to the associated measured values or the measuring span. Often, the specification limits are also drawn in, allowing you to see immediately if the measuring instrument remains within these limits. If only a certain range of values was investigated in the calibration, these validity values are also drawn in.

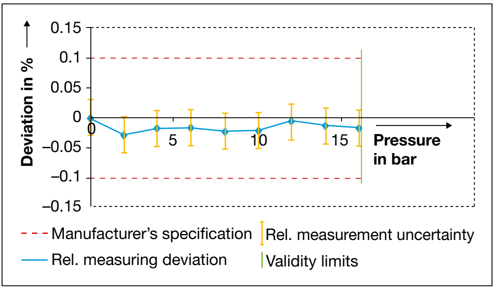

Fig. 38: Graphical evaluation of a pressure gauge calibration

Single-figure indication

Methods for the determination of the single-figure indication

Each measurement uncertainty is always assigned one measured value, which is why calibration produces a table such as the one described above. However, in practice or for assigning a measurement instrument to an accuracy class, a characteristic value is required that illustrates how accurately an instrument can measure: This is referred to as single-figure indication. For this, the so-called deviation span is used. It is made up of the expanded measurement uncertainty and the systematic measuring deviation. There are several ways of obtaining a magnitude for the single-figure indication from the individual values W_abs which are listed, for example, in Table 6 (p. 61). Thus, for example, the maximum value can be used. Other options are, for example, the fixed-point, minimum or tolerance-band methods described in more detail, for example, in DKD-R 3-9, in which the single-figure indication is calculated from the gradient of the characteristic curves determined on the measuring instrument.
The GUM procedure incidentally does not contain a single-figure indication. Instead, the specification limits can be investigated as to whether they are observed in all points of the measuring range, taking into account the systematic measuring deviation and the expanded measurement uncertainties, plus a small safety margin if necessary. In this case, the calibration laboratory can enter a conformity statement in the calibration certificate.

Trends

Increasing demands from users on their measurement technology directly result in continuously higher demands on the reference instruments. The further development associated with this does not of course happen arbitrarily, but is often based on challenges that at first sight are not related at all to calibration. This is proven, for example, by the ever increasing air traffic over Europe: In order to accommodate more aircraft in the airways, the vertical minimum distance between individual aeroplanes at flying heights of between 29,000 and 41,000 feet has been cut in half from 2000 feet originally to 1000 feet (approx. 300 m). The resulting airspace may only be used by aeroplanes whose equipment allows a sufficiently accurate display and a sufficiently accurate observance of the flying height. Thus, the altimeter, which functions on the relationship between height and atmospheric pressure, must be able to measure with an accuracy that is clearly below 0.01 per cent. This is why aeroplane and sensor manufacturers, or the relevant service providers, demand higher-quality reference instruments for the calibration of pressure measuring instruments. At the same time, it must be possible to process calibrations cost-effectively and efficiently, which, given the prescribed test procedures, can only be achieved by a higher degree of automation.

Digital reference standards

To this end, renowned calibration technology manufacturers are working on digital reference standards such as the digital pressure balance (Fig. 39). In contrast to classic pressure balances which have to be operated manually, the time-consuming placement of weights can be omitted. In addition, subsequent mathematical evaluation is no longer required. Its centre-

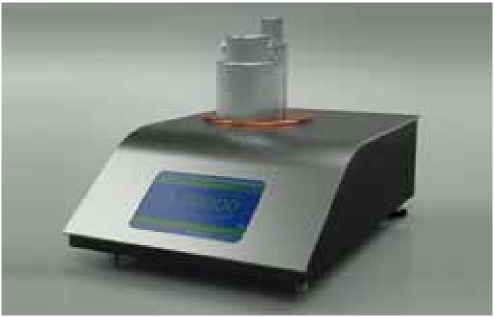

Fig. 39: Digital deadweight tester as an automatic reference standard

piece is the proven piston/cylinder system for the classic pressure balance used as a primary standard. However, the weight force produced by the piston is transferred to a high-resolution load cell. The cell is capable of resolving the weight force produced by a weight of 1 kg to the weight force of a weight of 1 mg, i.e. to the nearest millionth.

Integrated references

Maximum application reliability over a longer period is also offered by integrated reference sensors in which all critical effects on pressure measurement (such as the piston temperature) are measured via a sensor package and corrected fully automatically in real time. These reference instruments also feature a high-precision pressure control. Thanks to its digital measurement technology, these instruments can be remote-controlled, and the results can be processed directly on a PC. The currently available integrated references have measurement uncertainties of only 0.0015 per cent.

But this does not yet exhaust the full potential of calibration technology. Today, in collaboration with national institutes, solutions for future measurement challenges are already being worked on.

Technical terms

Accuracy class According to DIN 1319, a class of measuring instruments that meet predetermined metrological requirements so that the measuring deviations of these instruments remain within established limits

Adjustment Intervention to the measuring instrument in which the deviation from the correct value is minimised

Calibration Comparison of the value determined by a measuring instrument with the correct value

CGPM Abbreviation of French: *Conférence Générale des Poids et Mesures*; General Conference for Weights and Measures

CIPM Abbreviation of French: *Comité International des Poids et Mesures*; International Committee for Weights and Measures

Correct value Measured value determined by means of a high-quality standard

DAkkS Abbreviation of German: *Deutsche Akkreditierungsstelle*; national accreditation body of the Federal Republic of Germany (www.dakks.de)

Deviation span Sum of expanded measurement uncertainty and systematic measuring deviation

DKD Abbreviation of German: *Deutscher Kalibrierdienst*; German Calibration Service (www.dkd.eu)

Expected value Mean value obtained by repeating a random event many times. It corresponds to the empirical, i.e. arithmetic, mean of a distribution.

Fixed points Here: temperatures of phase transitions of certain substances which are suitable for defining temperature scales

Gaussian distribution Probability distribution in the form a bell-shaped curve according to Gauss

Hysteresis According to DIN 1319 also called reversal error: difference of the individual measured values in different measuring directions, for example increasing and decreasing pressure curve

ITS-90 Abbreviation of *International Temperature Scale of 1990*

Measurement uncertainty The measurement uncertainty characterises the measured values of a measurement parameter around its mean value.

Measuring deviation Difference between the measured and the correct value

NIST Abbreviation of *National Institute of Standards and Technology*

Pressure Force per area [N/m^2 or Pa = 10^{-5} bar]

Primary standard Standards that meet the highest requirements are referred to as primary standards. This means that a unit must basically be produced in accordance with the current international definition in such a way that it displays the lowest possible uncertainty according to the current state-of-the-art.

PTB Abbreviation of German: *Physikalisch Technische Bundesanstalt*; national metrology institute of the Federal Republic of Germany based in Braunschweig and Berlin (www.ptb.de)

Reference standard The standard within an organisation (institute, laboratory) with the highest available accuracy. All measurements made by this organisation are traceable to the reference standard.

Repeatability Difference between two measured values determined under identical conditions (e.g. each at increasing and decreasing pressure)

Standard Precise measuring instrument, comparison object or reference material for the calibration of other measuring instruments

Standard deviation Measure of the scattering of results for a measurement parameter around its mean value

Traceability Under traceability conditions, measured results can be referred to (inter) national standards through an uninterrupted chain of calibrations and thus be evaluated.

Triple point Fixed point of a substance defined by temperature and vapour pressure at which its solid, liquid, and gas phases are in a thermodynamic equilibrium

True value Measured value determined by an ideal measuring instrument

Variance Square of the standard deviation

Verification Test by a state institute as to whether a measuring instrument observes the error limits prescribed by the Verification Act

Working standard Standard used routinely by an organisation for calibrating measuring instruments. It is calibrated using a ↑*Reference standard*.

The company behind this book

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Over the past 60 years, WIKA Alexander Wiegand SE & Co. KG has built a reputation as a renowned partner and competent specialist for any task in the field of pressure and temperature measurement. On the basis of steadily growing efficiency, innovative technologies are applied when developing new products and system solutions. The reliability of the products and the readiness to face all challenges of the market have been the key factors for WIKA to achieve a leading position in the global market.

Within the WIKA Group, 7000 employees are dedicated to maintain and improve technology in pressure and temperature measurement. Over 500 experienced employees in the sales department consult customers and users competently on a partnership basis.

More than 300 engineers and technicians are searching continually on behalf of WIKA to provide innovative product solutions, improved materials and profitable production methods. In close cooperation with recognised universities, institutions and companies, solutions for specific applications are developed and designed.

The WIKA quality assurance management system has been certified in accordance with ISO 9001 since 1994. In 2003, WIKA Tronic's development and manufacturing of pressure sensors and pressure transmitters for the automotive industry were certified in accordance with the globally accepted ISO/TS-16949 standard. The quality and safety standards of our company meet the standard systems of several countries.

Alongside high product quality and efficient health and safety at work, comprehensive environmental protection has equal standing as a company goal. In addition to compliance with national and international environmental laws and regulations, the WIKA environmental management system is certified to ISO 14001.

Thinking globally and acting locally: WIKA has numerous subsidiaries and agencies around the world. We are therefore familiar with the respective country-specific requirements, standards and applications. This is how we ensure the individual assistance of our customers.

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