INTERNATIONAL STANDARD

ISO 14577-2

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Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 2:
Verification and calibration of testing machines

Matériaux métalliques — Essai de pénétration instrumenté pour la détermination de la dureté et de paramètres des matériaux —

Partie 2: Vérification et étalonnage des machines d'essai

Reference number
ISO 14577-2:2002(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 14577 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14577-2 was prepared by Technical Committee ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 3, *Hardness testing*.

ISO 14577 consists of the following parts, under the general title *Metallic materials — Instrumented indentation test for hardness and materials parameters*:

— *Part 1: Test method*

— *Part 2: Verification and calibration of testing machines*

— *Part 3: Calibration of reference blocks*

Annex B forms a normative part of this part of ISO 14577. Annexes A, C and D are for information only.

Introduction

Hardness has typically been defined as the resistance of a material to permanent penetration by another harder material. The results obtained when performing Rockwell, Vickers and Brinell tests are determined after the test force has been removed. Therefore, the effect of elastic deformation under the indenter has been ignored.

ISO 14577 has been prepared to enable the user to evaluate the indentation of materials by considering both the force and displacement during plastic and elastic deformation. By monitoring the complete cycle of increasing and removal of the test force, hardness values equivalent to traditional hardness values can be determined. More significantly, additional properties of the material, such as its indentation modulus and elasto-plastic hardness, can also be determined. All these values can be calculated without the need to measure the indent optically.

ISO 14577 has been written to allow a wide variety of post test data analysis.

Metallic materials — Instrumented indentation test for hardness and materials parameters —

Part 2: Verification and calibration of testing machines

1 Scope

This part of ISO 14577 specifies the method of verification and calibration of testing machines for carrying out the instrumented indentation test in accordance with ISO 14577-1.

It describes a direct verification method for checking the main functions of the testing machine and an indirect verification method suitable for the determination of the repeatability of the testing machine. The indirect method shall be used in addition to the direct method and for the periodic routine checking of the testing machine in service.

The indirect method of verification of the testing machine shall be carried out independently for each test method.

This part of ISO 14577 is also applicable for transportable testing machines.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14577. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14577 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 376:1999, *Metallic materials — Calibration of force-proving instruments used for the verification of uniaxial testing machines*

ISO 3878, *Hardmetals — Vickers hardness test*

ISO 6508-2, *Metallic materials — Rockwell hardness test — Part 2: Verification and calibration of testing machines (scales A, B, C, D, E, F, G, H, K, N, T)*

ISO 14577-1:2002, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

ISO 14577-3, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 3: Calibration of reference blocks*

ISO Guide to the Expression of Uncertainty in Measurement (GUM) [1)]

1) Published in 1993; corrected and reprinted in 1995.

3 General conditions

3.1 Preparation

The machine shall be designed in such a way that it can be verified.

Before verification and calibration of the testing machine it shall be checked to ensure the conditions laid down in 3.2 to 3.4

3.2 Functional installation

The testing machine shall be configured to operate in compliance with and shall be installed in an environment that meets the requirements of this document and of ISO 14577-1 and, where applicable, ISO 14577-3. The testing machine shall be protected from vibrations. For testing in the micro and nano ranges the testing machine shall also be protected from air currents and temperature fluctuations. This influence shall be checked by repeated measurements of the force/indentation depth curve.

3.3 Indenter

In order to get repeatable measurements of the force/indentation depth data set, the indenter holder shall be firmly mounted into the testing machine

The indenter holder should be designed in such a way that the contribution to the overall compliance is minimized (see annex A).

3.4 Application of the test force

The test force shall be applied and removed without shock or vibration that would significantly affect the test results. It shall be possible to verify the process of increasing, holding and removal of the test force.

4 Direct verification and calibration

4.1 General

4.1.1 The direct verification shall be carried out at a temperature of (23 ± 5) °C.

If a range of operating temperatures is required, then direct verification should be carried out at suitable points over that range to determine the validity of the calibration as a function of temperature. If necessary, a calibration correction function or a set of calibrations valid at specific operating temperatures may be determined.

4.1.2 The instruments used for verification and calibration shall be traceable to National Standards as far as available.

4.1.3 Direct verification involves:

a) verification of the indenter;

b) calibration of the test force;

c) calibration of the displacement measuring device;

d) calibration of the machine compliance;

e) verification of the indenter area function, if the indentation depth is less than 6 μm;

f) verification of the testing cycle.

4.2 Verification of the indenter

4.2.1 General

The indenter used for the indentation test shall be calibrated. Evidence that the indenter complies with the requirements of this part of ISO 14577 shall be fulfilled by a calibration certificate from a qualified calibration laboratory[2)] and evidence from the most recent indirect verification that the indenter area function has not changed. The latter shall be provided using the verification methods described in annex B and suitable certified reference materials. All geometrical values shall be measured and incorporated into the calibration certificate. The indenter performance shall be verified periodically (see clause 6).

If the angle of the indenter deviates from the nominal value for an ideal geometry of the indenter, the average of the certified angles for that indenter should be used in all applicable calculations, e.g. 0,2° error in the Vickers angle of 136° results in a 1 % systematic error in area.

The angle for pyramidal and conical indenters shall be measured within the indentation depth ranges given in Table 1 (and illustrated in Figure 1). Indenters for use in the nano range and in the micro range (indentation depth $\leqslant$ 6 µm) shall have their area function calibrated over the relevant indentation depth ranges of use.

Table 1 — Values for the measuring ranges for the angle of pyramidal and conical indenters

Dimensions in micrometres

Indentation depth	Macro range	Micro range
h_1	6	0,2
h_2	200	120

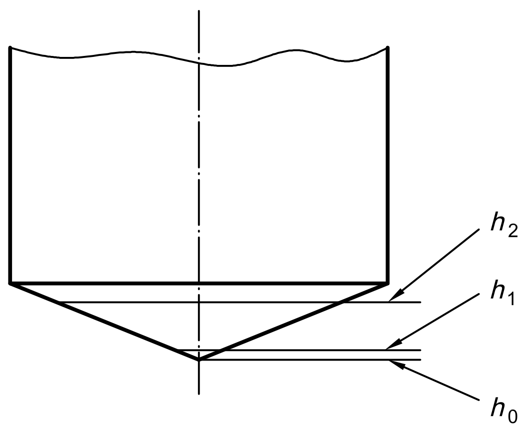


Figure 1 — Illustration of measuring ranges given in Table 1

4.2.2 Vickers indenter

4.2.2.1 The four faces of the right square-based diamond pyramid shall be smooth and free from surface defects and contaminants. For notes on cleaning of the indenter surface see also annex D in ISO 14577-1:2002.

The surface roughness of the indenter has a similar effect on measurement uncertainty as test piece roughness. When testing in the nano range, the indenter surface finish should be taken into consideration.

2) See ISO/IEC 17025 [1].

4.2.2.2 The angle between the opposite faces of the vertex of the diamond pyramid shall be 136° ± 0,3° (see Figure 2).

The angle shall be measured in the range between h_1 and h_2 (see Table 1 and Figure 1). The geometry and finish of the indenter shall be controlled over the whole calibrated indentation depth range, i.e. from the indenter tip, h_0, to the maximum calibrated indentation depth, h_2.

4.2.2.3 The angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall not exceed 0,5°.

4.2.2.4 The four faces shall meet at a point. The maximum permissible length of the line of conjunction between opposite faces is given in Table 2 (see also Figure 3).

4.2.2.5 The radius of the tip of the indenter shall not exceed 0,5 µm for the micro range (see Figure 4).

4.2.2.6 The verification of the shape of the indenter shall be carried out using microscopes or other suitable devices.

If the indenter is used for testing in the micro or nano range a verification by an atomic-force-microscope (AFM) is recommended.

Table 2 — Maximum permissible length of the line of conjunction

Range of the indentation depth µm	Maximum permissible length of the line of conjunction µm
$h > 30$	1
$30 \geqslant h > 6$	0,5[a]
$h \leqslant 6$	$\leqslant$ 0,5[b]

[a] This may be assumed to have been achieved when there is no detectable conjunction when the indenter is verified by an optical microscope at 400 × magnification.

[b] The correction of the shape of the indenter is taken into account, see C.2 in ISO 14577-1:2002.

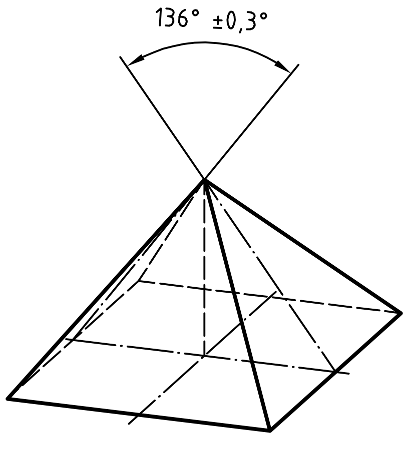
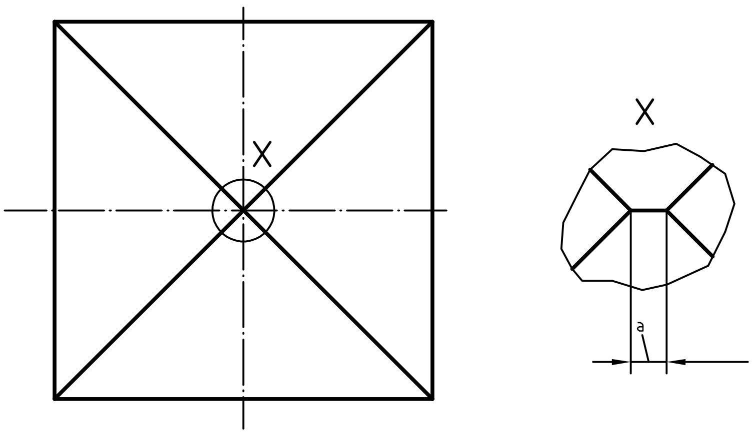


Figure 2 — Angle of the Vickers diamond pyramid



a Line of conjunction

Figure 3 — Line of conjunction on the tip of the indenter, schematically

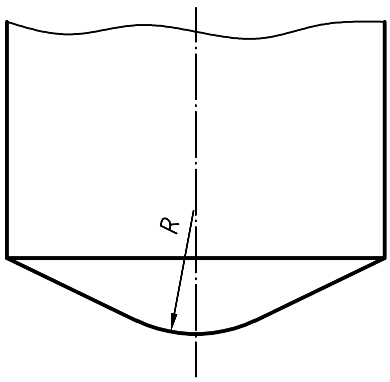


Figure 4 — Radius of the tip of the indenter

4.2.3 Berkovich, modified Berkovich and corner cube indenters

4.2.3.1 General

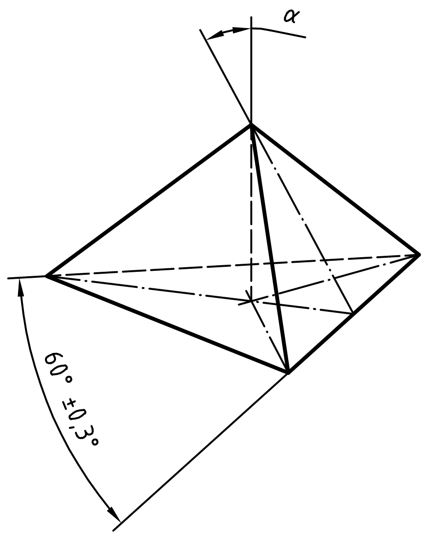
In practice there are two types of Berkovich pyramidal diamond indenters in common use. The Berkovich indenter [2] is designed to have the same surface area as a Vickers indenter at any given indentation depth. The modified Berkovich indenter [3] is designed to have the same projected area as the Vickers indenter at any given indentation depth.

4.2.3.2 The three faces of the triangular based diamond pyramid shall be smooth and free from surface defects and from contaminations. For notes on cleaning of the surface see also annex D in ISO 14577-1:2002.

The surface roughness of the indenter has a similar effect on measurement uncertainty as does test piece roughness. When testing in the nano range the indenter surface finish should be taken into consideration.

4.2.3.3 The radius of the tip of the indenter shall not exceed 0,5 µm for the micro range and shall not exceed 0,2 µm for the nano range (see Figure 4).

4.2.3.4 The angle between the axis of the diamond pyramid and the three faces is designated α. The angle between the three faces of the diamond pyramid shall be 60° ± 0,3° (see Figure 5).



α = 65,03° ± 0,3° for Berkovich indenter

α = 65,27° ± 0,3° for modified Berkovich indenter

α = 35,26° ± 0,3° for corner cube indenters

Figure 5 — Angle of the Berkovich and corner cube indenters

4.2.3.5 The verification of the shape of the indenter shall be carried out using microscopes or suitable devices.

If the indenter is used for testing in the micro and nano range a measurement by an atomic-force-microscope (AFM) should be carried out. For the nano range this measurement is recommended.

4.2.4 Hardmetal ball indenters

4.2.4.1 The characteristics of the hardmetal balls shall be the following:

— hardness: the hardness shall be not less than 1 500 HV 10, when determined in accordance with ISO 3878;

— density: ρ = 14,8 g/cm^3 ± 0,2 g/cm^3.

The following chemical composition is recommended:

— cobalt (Co) 5,0 % to 7,0 %

— total carbides other than tungsten carbide 2,0 %

— tungsten carbide (WC) balance

4.2.4.2 The balls shall have a certified geometry. Batch certification methods are sufficient. The certificate shall show the diameter of the average value of at least three measured points of different positions. If any value differs from the permissible values of the nominal diameter (see Table 3), the ball shall not be used as an indenter.

Table 3 — Tolerances for ball indenters

Dimensions in millimetres

Ball diameter	Tolerance
10	± 0,005
5	± 0,004
2,5	± 0,003
1	± 0,003
0,5	± 0,003

4.2.5 Spherical tipped conical indenters

The characteristics of spherical tipped conical indenters shall be as given in Table 4 (see also Figure 6).

Table 4 — Tolerances for sphero-conical indenters

Feature	Tolerance
$R_{av} \leqslant 50$ µm	$\pm 0{,}25\ R_{av}$
500 µm $> R_{av} >$ 50 µm[a]	$\pm 0{,}1\ R_{av}$
Cone included angle, 2 α	
120°	± 5°[a]
90°	± 5°
60°	± 5°
Cone flank angle α	
60°	± 5°
45°	± 2,5°
30°	± 2,5°
NOTE Centerline of cone to centerline of mount is within 0,01 mm.	
[a] Rockwell diamond indenters (see ISO 6508-2) fulfill this requirement	

The instantaneous radius of curvature [$R(h)$] of the spherical cap at any indentation depth h measured from the point of first contact shall not vary by more than a factor of two from the average radius, thus:

$$0{,}5 \leqslant \left| R(h)/R_{av} \right| \leqslant 2$$

Indenters with a spherical tipped cone shape are useful for many applications. These indenters are normally made from diamond but may also be made from other materials, e.g. ruby, sapphire or hardmetal. They are intended to indent only with the spherical tip. If Hertzian contact mechanics are to be used to interpret the indentation response, the value used for the indenter radius is critical. It is therefore recommended that the shape of each indenter be determined directly, by a suitable measurement system, or indirectly by indentation into a reference material with known properties.

Surface roughness Ra should be minimized. Roughness causes an uncertainty in the definition of the first contact point in the actual area of contact of the indenter with the test piece. Asperities have radii of contact vastly different from the average radius of the spherical cap and therefore behave very differently. If possible, the Ra of the diamond surface should be less than 1/20 of the usual indentation depth for an indenter.

NOTE Geometry suggests that the depth of spherical cap h_s on a cone of included angle 2 α and radius R_{av} is given by:

$$h_s = R_{av}\,[1 - \sin(\alpha)]$$

In practice, there is a gradual transition from spherical cap to cone geometry which is hard to specify. Given this and the uncertainties in R_{av} and α allowed (see Table 4), caution should be exercised whenever the depth exceeds 0,5 h_s.

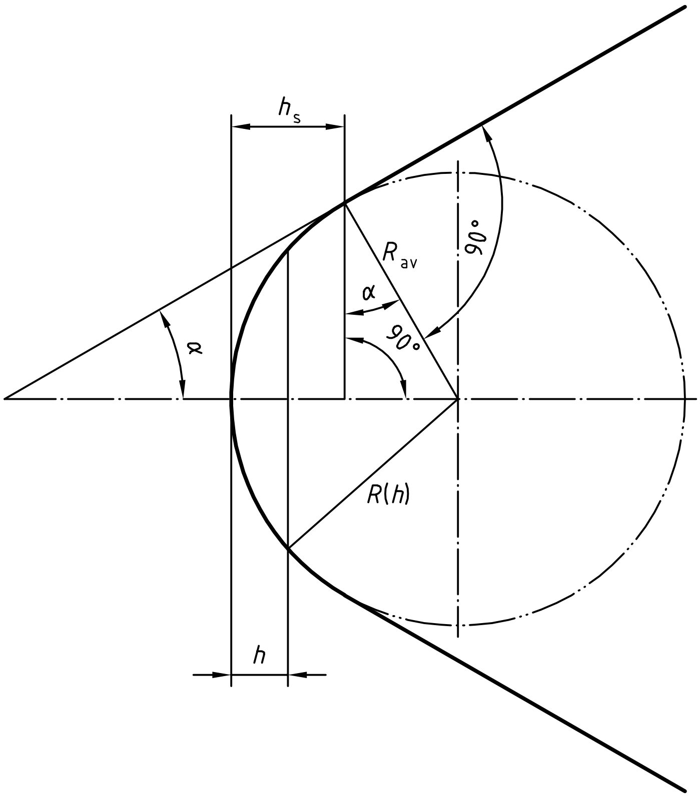


Figure 6 — Representation of the features of spherical indenters

4.3 Calibration of the test force

4.3.1 Each range of force used shall be calibrated over the whole force range for both application and removal of the test force. A minimum of 16 evenly distributed points in the test force range shall be calibrated, i.e. 16 during application and 16 during removal of the test force. The procedure shall be repeated three times.

4.3.2 The test force shall be measured by a traceable method, e.g.:

a) measuring by means of an elastic proving-device in accordance with class 1 of ISO 376:1999;

b) balancing against a force, accurate to within ± 0,2 % applied by means of calibrated masses with mechanical advantage;

c) electronic balance with a suitable accuracy of 0,1 % of maximum test force or 10 µN for the nano range.

4.3.3 The repeatability of the test force shall be within the tolerance of the nominal value of the test force as given in Table 5.

Table 5 — Tolerances for test forces

Range of the test force N	**Tolerances** %
$F \geqslant 2$	± 1,0
$0{,}1 \leqslant F < 2$	± 1,5
$0{,}001 \leqslant F < 0{,}1$	± 2,5[a]

[a] For the nano range the tolerance of ± 1 % is strongly recommended.

4.4 Calibration of the displacement measuring device

4.4.1 The estimation capability required of the displacement measuring system depends on the size of the smallest indentation depth to be measured. For the micro range this value is 0,2 µm, for the macro range ⩾ 2 µm.

The scale of the displacement measuring device shall be graduated to permit an estimation capability of indentation depth in accordance with Table 6.

4.4.2 The displacement measuring device shall be calibrated on the testing machine for every range used by means of a suitable method and a corresponding system. The device shall be calibrated at a minimum of 16 points in each direction evenly distributed throughout its travel. The procedure shall be repeated three times.

4.4.3 Changes in temperature are commonly a dominant source of displacement drift. To minimize thermally induced displacement drift, the temperature of the instrument shall be maintained such that the maximum displacement drift does not exceed the maximum permissible displacement error specified in Table 6, over the time period of one calibration cycle. The drift rate shall be measured either during, immediately before or immediately after each calibration cycle, e.g. by monitoring displacement during a suitable hold period at constant applied test force.

The maximum permissible error for each range is given in Table 6. Notes for different methods for the direct verification of the displacement measuring device are given in annex C.

Table 6 — Estimation capability and maximum permissible error of the displacement measuring device

Range of application	Estimation capability of the displacement measuring device nm	Maximum permissible error
Macro	⩽ 100	1 % of h
Micro	⩽ 10	1 % of h
Nano	⩽ 1	2 nm[a]

[a] For the nano range the tolerance of ± 1 % of h is strongly recommended.

4.5 Verification and calibration of the machine compliance

4.5.1 General

See Annex C in ISO 14577-1:2002.

This verification and calibration shall be carried out after the test force and the displacement measuring system have been calibrated in accordance with 4.3 and 4.4.

4.5.2 Procedure

The verification and calibration of machine compliance is carried out by measurement of hardness and/or indentation modulus at least at five different test forces.

For indentation depths ⩾ 6 µm it is not necessary to take into account the real contact area function. For the verification and calibration of the machine compliance a reference material with certified hardness value and/or certified indentation modulus, independent from the indentation depth, shall be used (e.g. Vickers hardness reference blocks, steel for large indentation depths, glass BK 7 for small indentation depths). The range for the test force is defined by the minimum test force which correlates to 6 µm indentation depth and the maximum possible test force of the testing machine.

For indentation depths < 6 μm the above mentioned method shall be applied where the indenter has a calibrated area function and the measurement is carried out on two different reference materials that have a significant difference in hardness, i.e. a factor of 2 at least, e.g. fused silica and tungsten. In general, the use of reference blocks with a large $E/\sqrt{H_{\text{IT}}}$ is recommended.

The measurement is carried out on each material at equal indentation depths. The range for the test forces is defined by the indentation depths > 0,5 μm and the maximum test force of the testing machine or the maximum test force for which no unusual test piece response (e. g. cracking of ceramics or glasses) occurs.

The deviation of the measured values as a function of the relevant test forces shall not exceed the values for the maximum percentage error of the testing machine given in Table 7, Table 8 and 5.2.6.

If, after applying the currently valid correction for machine compliance and indentation area function, a measured value from a reference block deviates from the certified value of the test piece by more than the maximum permissible amount and repetition of the procedure using a newly verified and certified indenter (and valid machine compliance correction corresponding to that indenter) also fails to reproduce the certified value, the testing machine shall be serviced and a full, direct calibration be performed. Current procedures for the machine compliance correction are also given in [3] and [4].

The compliance of the testing machine may be affected by the particular construction and mounting of an indenter and also the method used to mount a sample. For instance, mounting in plastics (e.g. PVC) may introduce an extra compliance into the measurement loop. The verification and calibration of machine compliance should be performed using the indenter that will be used for subsequent measurements.

4.6 Verification of the indenter area function

4.6.1 General

See Annex C in ISO 14577-1:2002.

4.6.2 Procedure

Procedures for the determination of indenter area function are given in annex B.

The verification of the indenter area function consists of a comparison of the measured indenter area function with a documented indenter area function determined for the newly certified and calibrated indenter.

NOTE Indenter area function and machine compliance correction can be determined simultaneously using an iterative procedure and multiple reference materials [4].

If the certified property value for a reference material cannot be obtained within the limits set out in 5.2 and if a newly certified indenter with corresponding valid machine compliance correction does not obtain the reference value, the original indenter shall have failed its verification and shall be recalibrated. If the difference in area between the measured and certified area functions (obtained as described in annex B and expressed at each measured indentation depth as a percentage of the original certified area value) exceeds 30 % at any indentation depth in the range of calibration of an indenter, that indenter shall be discarded.

4.7 Verification of the testing cycle

The testing cycle (application of the test force, holding of the maximum test force and removal of the test force) shall be measured with a tolerance of 0,1 s. Duration of each part of the testing cycle shall meet the requirements of ISO 14577-1.

5 Indirect verification

5.1 General

The indirect verification shall be carried out periodically or before tests requiring high accuracy. If the testing machine is used in the micro or nano range it is recommended to carry out the indirect verification at shorter intervals, at least weekly.

Indirect verification should be carried out at a temperature of (23 ± 5) °C by means of reference blocks calibrated in accordance with ISO 14577-3. These reference blocks can be calibrated for hardness, indentation modulus and/or other materials parameters.

5.2 Procedure

5.2.1 The indirect verification shall be carried out at least at the two test forces most frequently used. For tests with indentation depths < 6 µm this provides some verification of the contact area function. For each test force two different reference blocks, which span the range of application as widely as possible, shall be chosen.

EXAMPLE 1

$$\text{HM}, H_{\text{IT}} \leqslant 2\,500\ \text{N/mm}^2$$

$$2\,500\ \text{N/mm}^2 < \text{HM}, H_{\text{IT}} \leqslant 7\,000\ \text{N/mm}^2$$

$$7\,000\ \text{N/mm}^2 < \text{HM}, H_{\text{IT}}$$

EXAMPLE 2

$$50\,000\ \text{N/mm}^2 < E_{\text{IT}} \leqslant 100\,000\ \text{N/mm}^2$$

$$100\,000\ \text{N/mm}^2 < E_{\text{IT}} < 450\,000\ \text{N/mm}^2$$

5.2.2 If a testing machine is only used at one test force, it shall only be verified at this test force on at least two reference blocks with certified values that span the range of values of the test pieces to be tested.

5.2.3 On each reference block, five measurements are recommended in accordance with ISO 14577-1. For indentation depths < 6 µm at least ten measurements at each test force on each block are recommended to reduce the uncertainty in repeatability of the measurement mean.

5.2.4 For each reference block the arithmetic mean value $\overline{q}$ from the n values $q_1, ..., q_n$ is calculated:

$$\overline{q} = \frac{q_1 + \ldots + q_n}{n} \tag{1}$$

The experimental standard deviation shall be calculated as a parameter to describe the scatter of the measurement values:

$$s(q) = \sqrt{\frac{\sum_{i=1}^{n} (q_i - \overline{q})^2}{n-1}} \tag{2}$$

Where q represents the materials parameters.

The relative scattering of the measured values is the coefficient of variation expressed as a percentage:

$$V = \frac{s(q)}{\overline{q}} \times 100 \qquad (3)$$

5.2.5 The repeatability of the testing machine under the particular verification conditions is determined by the coefficient of variation of the measured value.

The repeatability of the testing machine is considered satisfactory if it satisfies the conditions given in Table 7.

Table 7 — Repeatability of the testing machine

Material parameter	Micro range		Macro range
	$0{,}2\ \mu m \leqslant h \leqslant 1\ \mu m$	$h > 1\ \mu m$	
HM, H_{IT}	5 %	2 %	2 %
E_{IT} [a]	5 %	5 %	5 %

[a] For nearly perfect plastic materials these limits may not be achievable.

5.2.6 The error of the testing machine is characterized by the difference

$$\left| \overline{q} - q \right|$$

where

$\overline{q}$ is the arithmetic mean value calculated from single measurements;

q is the specified value of the reference block used.

and is standardized using the t statistic.

$$t = \frac{\overline{q} - q}{s(q)} \times \sqrt{n}$$

The value of t returned from the testing machine shall not exceed the critical value t_c for a two-tailed test, with $(n - 1)$ degrees of freedom, at the 95 % confidence level (see Table 8 for example values).

Table 8 — Example values for n and t_c

n	5	10	20
t_c	2,78	2,26	2,09

More accurately:

$$\frac{s(q)}{\sqrt{n}} = \left[\frac{(n-1)s^2 + (n_{cal} - 1)s_{cal}^2}{n + n_{cal} - 2} \right]^{1/2} \times \left(\frac{1}{n} + \frac{1}{n_{cal}} \right)^{1/2}$$

where

s_{cal} is the experimental standard deviation (determined during calibration);

n_{cal} is the number of calibrations averaged for the certified reference block value.

In general $s(q) \approx s$ to a first approximation and this is more valid if n and n_{cal} are greater. See also annex K of ISO GUM:1995 for the use of Welch-Satterthwaite equation to better define the effective number of degrees of freedom to use when determining the critical t-statistic.

6 Intervals between verifications

6.1 Direct verification

6.1.1 A full direct verification shall be carried out when the machine is new.

6.1.2 A limited direct verification in accordance with 4.3, 4.4 and 4.7 shall be carried out:

a) when the machine is installed or after its dismantling and reassembly or relocation;

b) when the result of the indirect verification is not satisfactory;

c) at least at intervals not exceeding 3 a.

6.1.3 In both cases (6.1.1 and 6.1.2), each direct verification shall be followed by an indirect verification.

6.2 Indirect verification

The period between two indirect verifications shall not exceed one year. It is recommended that more frequent indirect verifications be performed depending on how frequently the machine is used.

6.3 Routine checking

Before any series of tests, or periodically (e.g. daily) within each series, a test at two different test forces shall be performed on a test piece of known materials parameter. The result of this test shall be recorded on a suitable chart, for example see annex D. If the results are outside the normal range of reproducibility an indirect verification shall be performed.

It is good practice that test indentations be performed both before and after each test series.

For routine checking of the condition of the indenter, see annex D in ISO 14577-1:2002.

7 Verification report/Calibration certificate

The verification report/calibration certificate shall include at least the following information:

a) reference to this part of ISO 14577, i.e. ISO 14577-2;

b) method of verification (direct and/or indirect);

c) identification data of the testing machine;

d) means of verification (reference blocks, elastic proving devices etc.);

e) test forces;

f) verification temperature;

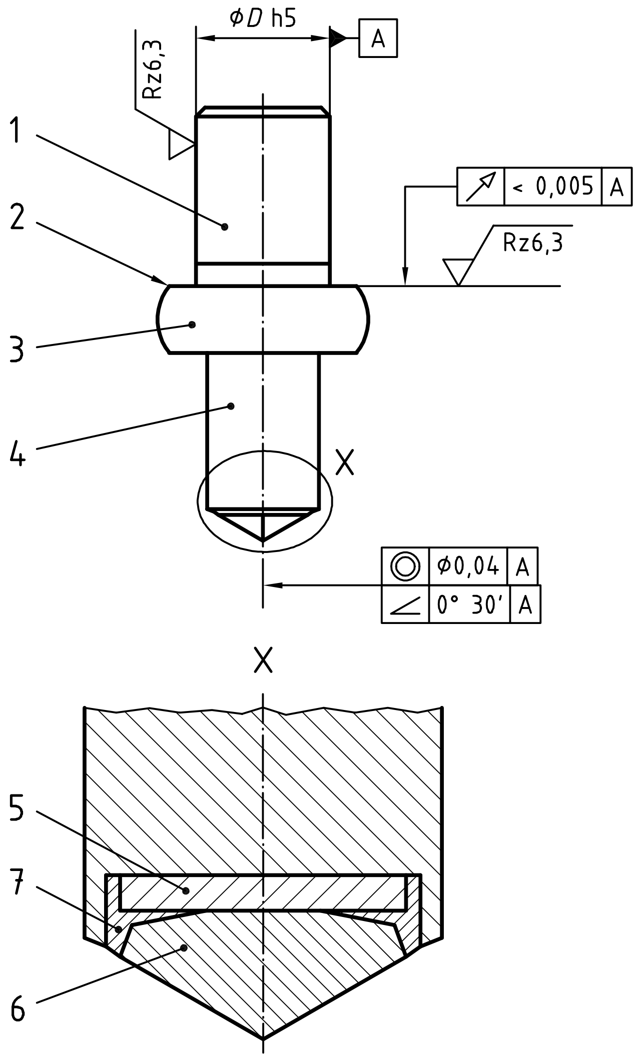
g) the results obtained, reported in the format required in ISO 14577-1;

h) date of verification and reference to the verification institution.

Annex A
(informative)

Example of an indenter holder

Dimensions in millimetres,
surface roughness values in micrometres



Key

1 Neck
2 Seating face
3 Seating
4 Shank
5 Support for the indenter (hardmetal)
6 Indenter
7 Embedding material

Figure A.1 — An example of a suitable design for the indenter holder

Annex B
(normative)

Procedures for determination of indenter area function

B.1 General

The following methods for determining the indenter area function are valid. Each of these methods results in the same indenter area function within the permissible tolerances.

B.2 Direct measurement method

The most appropriate method of direct measurement will depend on the intended use of the indenter. A traceably calibrated atomic force microscope (AFM) is ideal for high resolution shape characterization of the last 1 µm or so of the indenter. Care must be taken to ensure that the AFM measurements take account of the various sources of error and uncertainty [5], [6]. Traceably calibrated electron or optical microscopy methods may be more convenient if only larger indentation depths are intended.

B.3 Indirect measurement methods

B.3.1 These methods rely on using the test machine to perform indentation cycles on a material of known properties. It therefore requires that all instrument calibrations required by this part of ISO 14577 be completed satisfactorily and that the machine compliance correction has been determined as in 4.5 or that an iterative indentation modulus based procedure is to be used [4]. Once the force/displacement data have been corrected for frame compliance (and for thermal or other systematic drift) the following two methods may be applied.

B.3.2 If a material of known Martens hardness as a function of the test force is used as a test piece A_s (h) can be derived for each specific indentation depth h measured at test force F. This method is normally not useful for small indentation depths e.g. less than 0,2 µm.

B.3.3 From the curve segment of the removal of the test force the contact compliance may be related to the indentation modulus of the test piece by the equation:

$$\left|\frac{\mathrm{d}h}{\mathrm{d}F}\right|_{F_{\max}} = C = \frac{\sqrt{\pi}}{2E_R}\frac{1}{\sqrt{A_p}} \tag{B.1}$$

in which

$$\frac{1}{E_R} = \frac{1-\nu_s^2}{E_s} + \frac{1-\nu_i^2}{E_i} \tag{B.2}$$

where

C is the compliance of the contact, $\mathrm{d}h/\mathrm{d}F$, at maximum applied force (reciprocal of the contact stiffness);

E_R is the Young's modulus, reduced;

A_p is the projected contact area, value of the indenter area function at the contact depth defined in the same way as for the calculation of hardness according to A.4 in ISO 14577-1:2002;

ν_s is the Poisson's ratio for the test piece;

ν_i is the Poisson's ratio for the indenter (for diamond 0,07);

E_s is the Young's modulus of the material;

E_i is the Young's modulus of the indenter (for diamond $1,14 \times 10^6$ N/mm^2).

Thus, if a material of known indentation (or Young's) modulus is used as a test piece, A_p can be derived for each specific contact depth and h_c, by rearranging the above relationships (see A.4 of ISO 14577-1:2002 for the determination of contact depth). Use of an iterative method and multiple reference materials permits the simultaneous measurement of indenter area function and machine compliance correction [4].

The force range shall be selected to encompass the full range of likely indentation depths. For force controlled indentations some preliminary experiments are required to establish the force range needed to produce appropriate displacements in the reference material. A series of at least ten different forces shall be chosen to span the range of interest and, for each force, at least ten indentations shall be made in the reference material and the mean value used to determine A_p. Thus a plot of A_p versus indentation contact depth, h_c, is obtained. One advantage of using a modulus as the reference property is that the elastic response of the test piece is not sensitive to work hardening or thermal treatment or to the exact amount of creep that has occurred. All that is required is that the creep rate during unloading be negligible with respect to the unloading rate of the indentation experiment. Another advantage is that Young's modulus may be determined independently by non-indentation techniques. This eliminates circularity in the calibration traceability.

This method may also be adapted to the use of reference materials certified for different parameters, e.g. hardness, H_{IT} (see A.4 of ISO 14577-1:2002) or Martens Hardness, HM (see A.2 of ISO 14577-1:2002). If HM is to be used to determine an area function, the rearranged equations do not give the projected area but yield A_s (the surface area at maximum indentation displacement) in an equivalent manner. In this case it is the surface area function that is derived from a plot of A_s versus the maximum displacement, h_{max}. The two functions are not the same and are not interchangeable.

The area function is normally expressed as a mathematical function relating the projected or surface area to the distance from the tip of the indenter. At indentation depths where a relatively simple (cubic or polynomial) mathematical function is unable to describe the area function, an estimate may be made either graphically or by using a look-up table. Alternatively a different mathematical function can be used to describe different parts of the indenter or a spline function adopted.

The two methods described in B.3.2 and B.3.3 are the most generally applied.

Annex C
(informative)

Examples for direct verification of the displacement measuring system

The following methods are recommended for the measurement of the relative displacement of the indenter:

a) Laser interference method.

b) Inductive method.

c) Capacitive method.

d) Piezotranslator method.

Annex D
(informative)

Examples for the documentation of the results of indirect verification

It is a benefit to chart the results of the routine checks and indirect verifications in order to monitor the performance of the testing machine over time. Each indirect verification should include a minimum of 3 or 5 force/indentation depth curves. The mean value and the standard deviation of the determined material parameter are recorded and documented in the form of a $\overline{q}/s$ chart (see Figures D.1 and D.2). Examples of suitable parameters to chart are indentation modulus or indentation depth at two predetermined test forces e.g. F_{max} and 0,1 F_{max}.

Any instability over time of the test force and displacement unit or contamination of the indenter is seen in both $\overline{q}/s$ charts. Wear of the indenter tip is most sensitively detected by the $\overline{q}/s$ chart of the results measured with 0,1 × F_{max}.

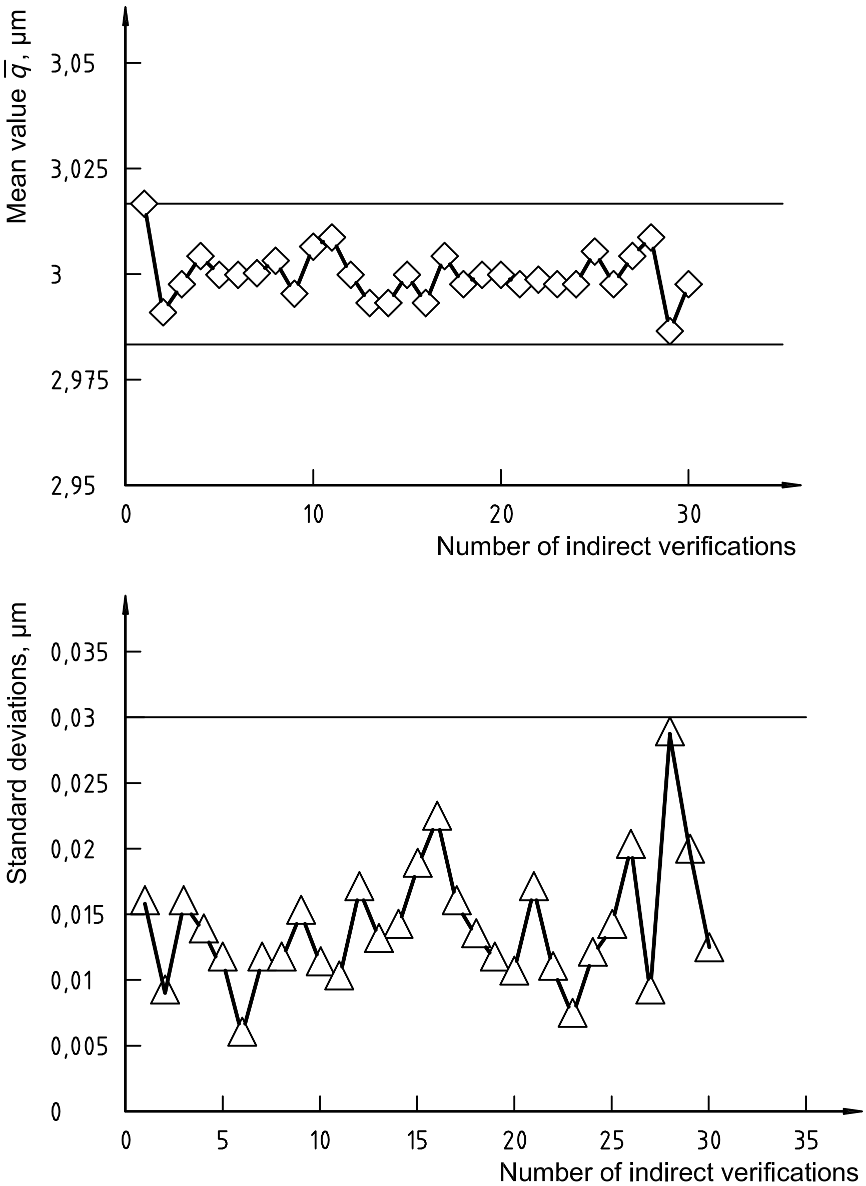


Figure D.1 — Example for $\overline{q}$ /s chart, indentation depth at F_{max}

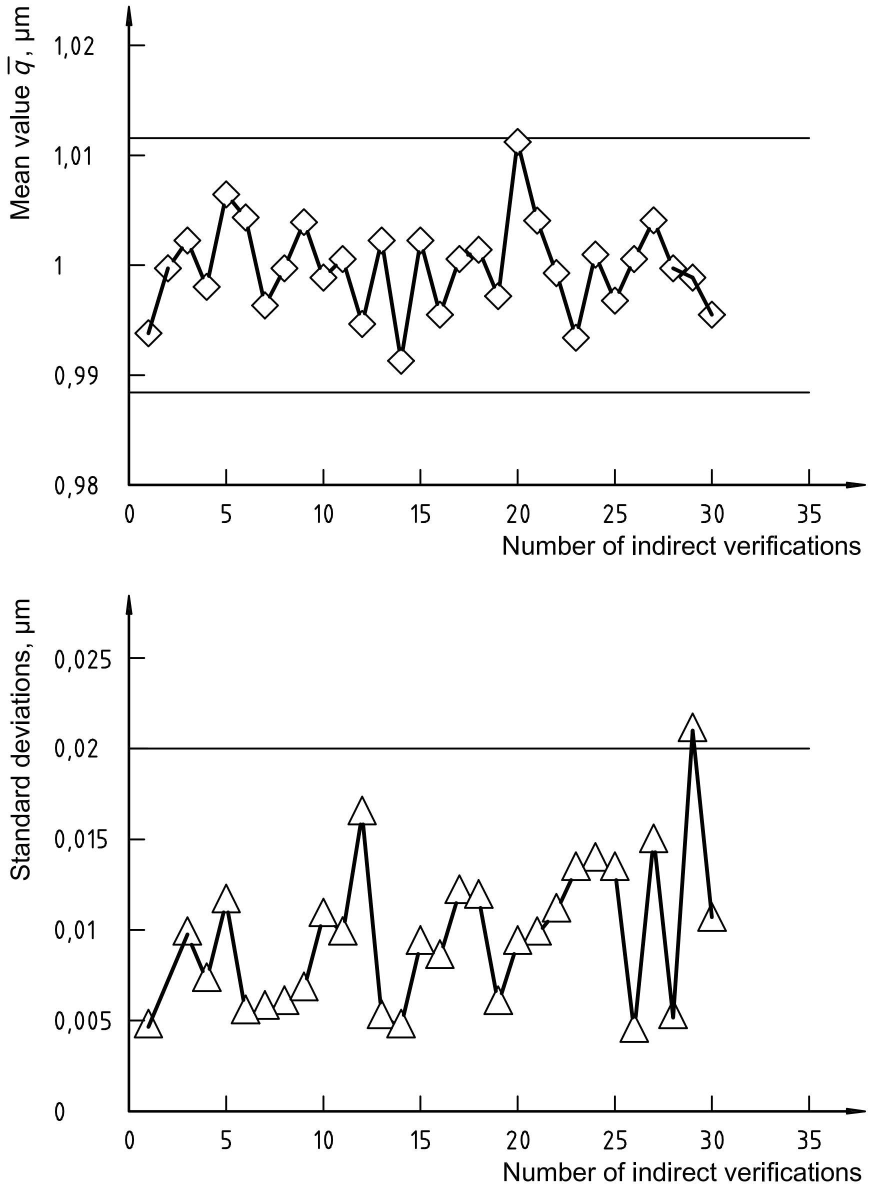


Figure D.2 — Example for $\overline{q}$ /s chart, indentation depth at 0,1 F_{max}

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