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Non-magnetic metallic coatings on metallic and non-metallic basis materials — Measurement of coating thickness — Phase-sensitive eddy-current method

Revêtements métalliques non magnétiques sur des matériaux de base métalliques et non métalliques — Mesurage de l'épaisseur de revêtement — Méthode par courants de Foucault sensible aux variations de phase

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Foreword

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 21968:2005), which has been technically revised. The main changes compared with the previous edition are as follows:

- this document has been adapted to the current requirements of ISO/IEC Guide 98-3 (also known as "GUM:1995");
- hints, practical examples and simple estimations of the measurement uncertainty for most important factors have been added;
- repeatability and reproducibility values for typical applications of the method have been added;
- the annex has been expanded with further applications and experimental estimations of factors affecting the accuracy.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Non-magnetic metallic coatings on metallic and non-metallic basis materials — Measurement of coating thickness — Phase-sensitive eddy-current method

1 Scope

This document specifies a method for using phase-sensitive eddy-current instruments for non-destructive measurements of the thickness of non-magnetic metallic coatings on metallic and non-metallic basis materials such as:

a) zinc, cadmium, copper, tin or chromium on steel;

b) copper or silver on composite materials.

The phase-sensitive method can be applied without thickness errors to smaller surface areas and to stronger surface curvatures than the amplitude-sensitive eddy-current method specified in ISO 2360, and is less affected by the magnetic properties of the basis material. However, the phase-sensitive method is more affected by the electrical properties of the coating materials.

In this document, the term “coating” is used for materials such as, for example, paints and varnishes, electroplated coatings, enamel coatings, plastic coatings, claddings and powder coatings.

This method is particularly applicable to measurements of the thickness of metallic coatings. These coatings can be non-magnetic metallic coatings on non-conductive, conductive or magnetic base materials, but also magnetic coatings on non-conductive or conductive base materials.

The measurement of metallic coatings on metallic basis material works only when the product of conductivity and permeability (σ, μ) of one of the materials is at least a factor of two times the product of conductivity and permeability for the other material. Non-ferromagnetic materials have a relative permeability of one.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064, ISO 4618 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
adjustment of a measuring system

set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

Note 1 to entry: Types of adjustment of a measuring system can include zero adjustment of a measuring system, offset adjustment, and span adjustment (sometimes called gain adjustment).

Note 2 to entry: Adjustment of a measuring system should not be confused with *calibration* (3.2), which is a prerequisite for adjustment.

Note 3 to entry: After an adjustment of a measuring system, the measuring system shall usually be recalibrated.

Note 4 to entry: Colloquially the term “calibration” is frequently, but falsely, used instead of the term “adjustment”. In the same way, the terms “verification” and “checking” are often used instead of the correct term “calibration”.

[SOURCE: ISO/IEC Guide 99:2007, 3.11 (also known as “VIM”), modified — Note 4 to entry has been added.]

3.2
calibration

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with *adjustment of a measuring system* (3.1), often mistakenly called “self-calibration”, nor with verification of calibration.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99:2007, 2.39 (also known as “VIM”)]

4 Principle of measurement

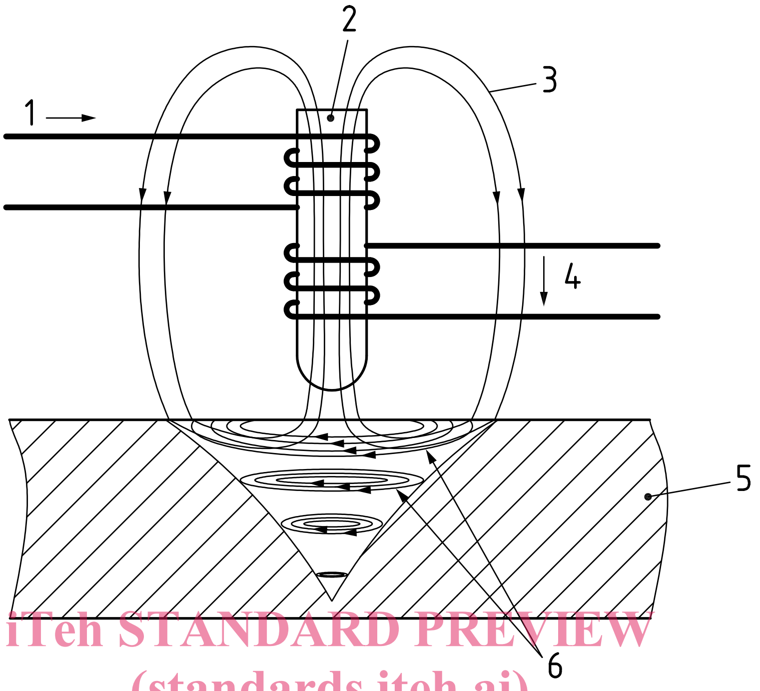
Phase-sensitive eddy-current instruments work on the principle that a high-frequency electromagnetic field generated by the probe system of the instrument will produce eddy currents in the coating on which the probe is placed and in the base material beneath the coating in case this base material is conductive (see Figure 1). These induced currents cause a change of the electromagnetic field surrounding the probe coil system and therefore result in a change of the amplitude and the phase angle of the probe coil impedance. The induced eddy-current density is a function of the coating thickness, the conductivity of the coating material, the used frequency of the probe system and the base metal conductivity. If the thickness of a coating of constant conductivity is increased for a given frequency, the impedance vector describes a so-called local function of the thickness in the impedance plane (see Figure 2). Each point of this local curve connects a phase angle of the impedance vector with the respective coating thickness. Consequently, this impedance angle (phase shift) can be used as a measure of the thickness of the coating on the conductor by means of a calibration with reference standards (see also Annex A).

In order to measure a change of the coil impedance phase angle, the test coil is usually part of a coil system and is coupled with the exciting coil on one ferrite core such as in a transformer (see Figure 1). The changes of phase angle and amplitude due to the impact of the induced eddy currents can be measured, for example, using a lock in amplifier. These values are usually pre-processed by digital means and the resulting thickness is then calculated and displayed.

The probe and measuring system/display may be integrated into a single instrument.

NOTE 1 Annex C describes the basic performance requirements of the equipment.

NOTE 2 Factors affecting measurement accuracy are discussed in Clause 5.

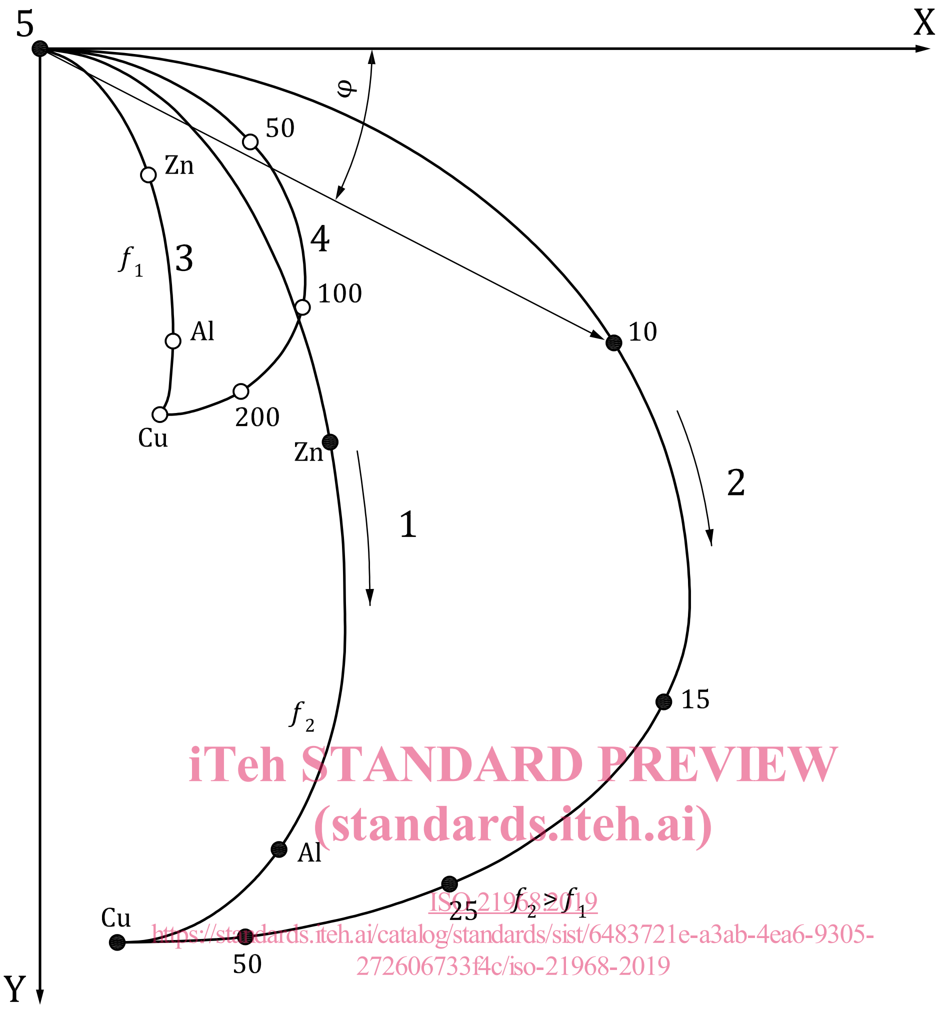


Key

1	exciting current
2	ferrit core of probe
3	high-frequency alternating magnetic field
4	measured signal $U = f(t(\varphi))$
5	base material (conductive)
6	induced eddy currents

Figure 1 — Phase-sensitive eddy-current method

Dimensions in micrometre



Key

1 conductivity local curve for the frequency f_2
2 thickness local curve of Cu for the frequency f_2
3 conductivity local curve for the frequency f_1
4 thickness local curve of Cu for the frequency f_1
5 coil in air (unaffected)
X real part
Y imaginary part

Figure 2 — Thickness local curves of Cu in the normalized impedance plane for two frequencies f_1 and f_2

For each instrument, there is a maximum measurable thickness of the coating.

Since this thickness range depends on both the applied frequency of the probe system and the electrical conductivity of the coating, the maximum thickness should be determined experimentally, unless otherwise specified by the manufacturer.

An explanation of eddy-current generation and the calculation of the maximum measurable coating thickness, t_{max}, is given in Annex A.

However, in the absence of any other information, the maximum measurable coating thickness, $t_{\max}$, can be estimated using Formula (1):

$$t_{\max} \approx 0{,}8 \cdot \delta_0 \tag{1}$$

where δ_0 is the standard penetration depth of the coating material (see Annex A).

5 Factors affecting measurement uncertainty

5.1 Basic influence of the coating thickness

The sensitivity of a probe, i.e. the measurement effect, depends on the used frequency, the conductivity of the coating and the base material, and the properties of the probe system. Besides the properties of the probe system, the resulting uncertainty of the thickness also depends on the sample materials, such as the homogeneity of the coating and base metal conductivity and roughness.

5.2 Electrical properties of the coating

The conductivity of the coating as well as the base material determine the induced eddy-current density for a given probe system and frequency. Consequently, the coating and base metal conductivity cause the measurement effect for this method. The relationship between coating thickness and the measured value depends strongly on the conductivity of both the coating and base material. Therefore, calibration procedures and measurements shall be made on the same material. Different materials with different conductivities as well as local fluctuations of the conductivity or variations between different samples can cause (more or less) errors in the thickness reading.

5.3 Geometry — Base material thickness

In cases of a conductive base material (base metal), the generation of eddy currents by the coil's magnetic field in the depth of the base metal is obstructed if the base metal thickness is too small. This influence can only be neglected above a certain critical minimum base metal thickness.

Therefore, the thickness of the base metal should always be higher than this critical minimum base metal thickness. An adjustment of the instrument can compensate for errors caused by thin base metal. However, any variation in thickness of the base metal can cause increased uncertainty and errors.

The critical minimum base metal thickness depends on both the probe system (frequency, geometry) and the conductivity of the coating and the base metal. Its value should be determined experimentally, unless otherwise specified by the manufacturer.

NOTE A simple experiment to estimate the critical minimum base metal thickness is described in Annex D.

However, in the absence of any other information, the required minimum base metal thickness, $t_{\min}$, can be estimated from Formula (2):

$$t_{\min} = 3 \cdot \delta_0 \tag{2}$$

where δ_0 is the standard penetration depth of the base metal (see Annex A).

In cases of a non-conductive and non-magnetic base material, the base material thickness does not affect the measurement results and consequently it shall not be considered as an influencing factor.

5.4 Geometry — Edge effects

The induction of eddy currents is obstructed by geometric limitations of the coating (such as edges, drills and others). Therefore, measurements made too near to an edge or corner may not be valid unless the instrument has been specifically adjusted for such measurements. The necessary distance in order to avoid an impact of the edge effect depends on the probe system (field distribution).

NOTE 1 A simple experiment to estimate the edge effect is described in Annex D.

NOTE 2 Amplitude-sensitive eddy-current instruments as described in ISO 2360 can be substantially more affected by edge effects.

5.5 Geometry — Surface curvature

The propagation of the magnetic field and consequently the induction of eddy currents are affected by the surface curvature of the coating and the base material. This influence becomes more pronounced with decreasing radius of the curvature and decreasing coating thickness. In order to minimize this influence, an adjustment should be performed on a sample with the same geometry.

The influence of surface curvature depends considerably on the probe geometry and can be reduced by reducing the sensitive area of the probe. Probes with very small sensitive areas are often called “microprobes”.

NOTE 1 There are instruments and probes available that are capable of automatically compensating the sample surface curvature influence if the curvature diameter is known. They can avoid the resulting thickness error.

NOTE 2 A simple experiment to estimate the effect of surface curvature is described in Annex D.

5.6 Surface roughness

Measurements are influenced by the surface topography of the coating and can also be influenced by the surface topography of a conductive base metal. Rough surfaces can cause both systematic and random errors. Random errors can be reduced by making multiple measurements, each measurement being made at a different location, and then calculating the average value of that series of measurements.

In order to reduce the influence of roughness, a calibration should be carried out with reference parts with a roughness equivalent to the coated sample.

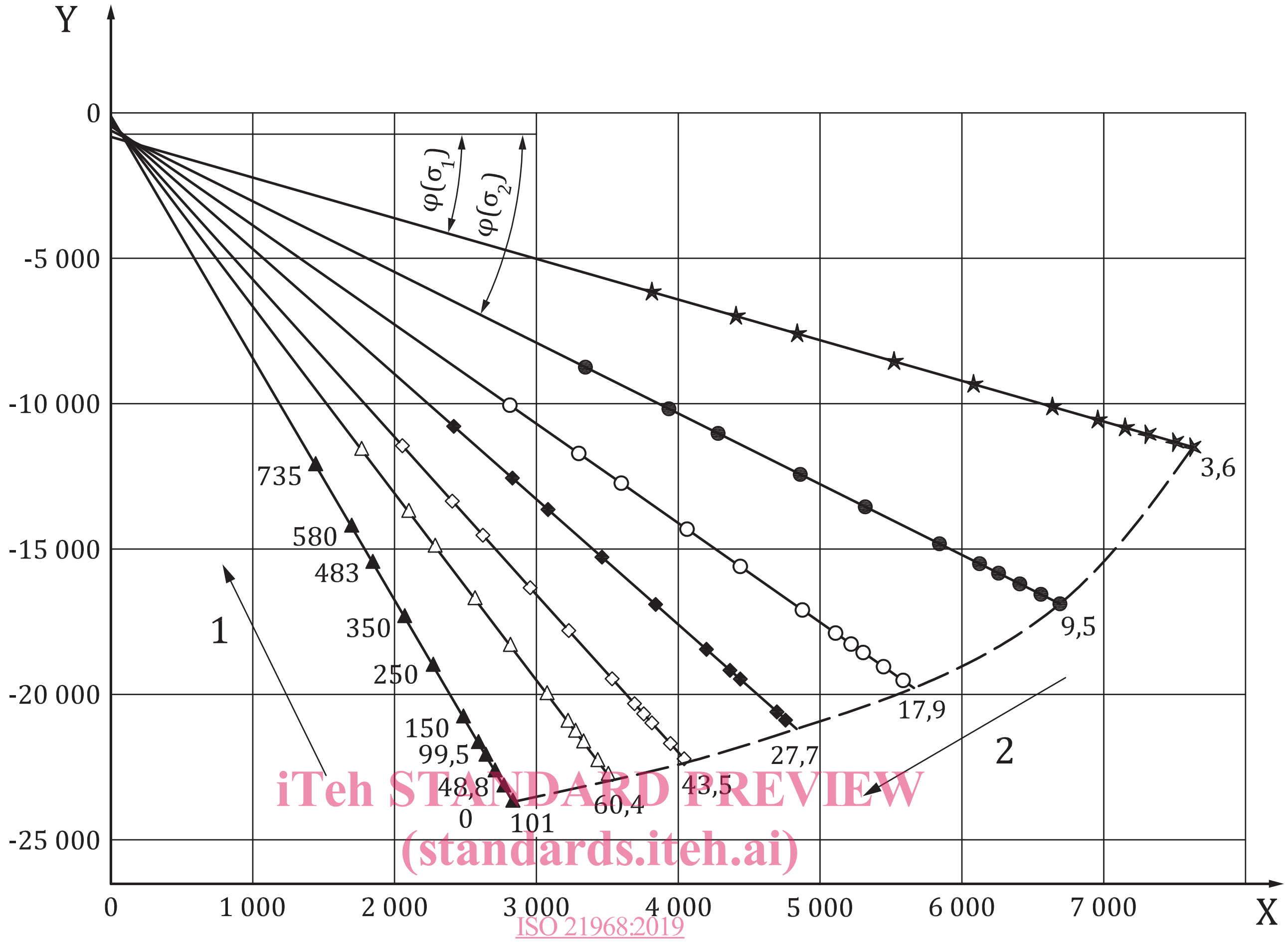
If necessary, the definition of the average coating thickness that is used should be stated between supplier and client.

NOTE Amplitude-sensitive eddy-current measurement as described in ISO 2360 can be more affected by base metal roughness.

5.7 Lift-off effect

If the probe is not placed directly onto the coating, the gap between probe and coating (lift-off) will affect the measurement of the metal coating thickness. The strength of the lift-off effect depends on the probe design and the resulting field geometry. By using appropriate electronic circuit design and/or a mathematical algorithm in the instrument, lift-off compensation can be applied for gaps of up to 1 mm.

The strength of the lift-off effect can be small for some probe designs. In this case, an increase of the lift-off height results mainly in a reduction of the impedance amplitude but only in a small change of the phase angle as a measure of the thickness (see Figure 3). The remaining influence can be compensated by an appropriate mathematical algorithm using the measured amplitude and phase angle information.



Key

—▲—	sample conductivity σ = 101,088 % IACS
—△—	sample conductivity σ = 60,407 % IACS
—◇—	sample conductivity σ = 43,48 % IACS
—◆—	sample conductivity σ = 27,688 % IACS
—○—	sample conductivity σ = 17,85 % IACS
—●—	sample conductivity σ = 9,534 % IACS
—★—	sample conductivity σ = 3,551 % IACS
1	increasing lift-off height in µm
2	increasing σ in % IACS
X	real part
Y	imaginary part

Figure 3 — Lift-off effect

Lift-off compensation shall be verified in accordance with the manufacturer’s instructions by using electrically nonconductive shims of known thickness, which are inserted between the probe and the coating.

NOTE 1 A simple experiment to estimate the lift-off effect is described in Annex D.

NOTE 2 Instead of lift-off compensation, the thickness of non-conductive coatings on top of conductive base metals can be measured by using the amplitude change as measuring effect (see Annex A).

5.8 Probe pressure

The pressure with which the probe is applied to the test specimen shall be made constant as it can affect the instrument reading.

NOTE The phase-sensitive eddy-current measurement can be substantially less affected by the pressure with which the probe is placed onto the sample than the amplitude-sensitive eddy-current method given in ISO 2360. Contactless measurements are possible.

5.9 Probe tilt

Unless otherwise instructed by the manufacturer, the probe should be applied perpendicularly to the coating surface as tilting the probe away from the surface normal can cause measurement errors.

The possibility of tilt inadvertently occurring can be minimized by probe design or by using a probe holding jig.

5.10 Temperature effects

As temperature changes affect the characteristics of the probe, it should be used under approximately the same temperature conditions as when the instrument was calibrated.

The influence of temperature variations can be reduced by a temperature compensation of the probe. The manufacturer's specification shall be taken into account.

Most metals change their electrical conductivity with temperature. Because the measured coating thickness is influenced by changes in the electrical conductivity of the coating and the base material, large temperature changes should be avoided (see 5.2).

NOTE Temperature differences between the probe, the electronics of the instrument, the environment and the sample can cause large thickness errors. One example is the thickness measurement of hot coatings.

5.11 Intermediate coatings

The presence of an intermediate coating can affect the measurement of the coating thickness if the electrical characteristics of that intermediate coating differ from those of the coating or the base metal. If a difference does exist, then the measurements will, in addition, be affected by an intermediate coating thinner than t_{min}. If the intermediate coating is thicker than t_{min} and non-magnetic, it can be treated as the base metal (see 5.3).

5.12 External electromagnetic fields

The measurement results can be influenced by strong electromagnetic interfering fields. When observing unexpected results or a strong variation of results, which cannot be explained by other factors, this influence should be taken into account. In this situation, a comparison measurement should be carried out at a location without interfering fields.

6 Calibration and adjustment of the instrument

6.1 General

Before use, every instrument shall be calibrated or adjusted according to the instructions of the manufacturer by means of suitable thickness reference standards, base material and a reference standard with sufficient coating thickness as a saturation material. The material, geometry and surface properties of the base metal used for calibration or adjustment should be similar to those of the test specimens in order to avoid deviations caused by the factors described in Clause 5. Otherwise, these influences shall be considered in the estimation of the measurement uncertainty.

During calibration or adjustment, the instruments, the standards and the base material should have the same temperature as the test specimens to minimize temperature-induced differences.

In order to avoid the influence of instrument drift, periodic control measurements with reference standards or control samples are recommended. If required, the instrument shall be re-adjusted.

NOTE Most instruments automatically adjust themselves during a function called “calibration”, carried out by the operator, whereas the result of the calibration is often not obvious.

6.2 Thickness reference standards

Thickness reference standards for calibration and adjustment are either coated base materials or, if available, metal foils, which are placed onto uncoated base materials.

Metal foils and coatings shall have the same conductivity as the coatings to be measured. Thickness values of the reference standards and their associated uncertainties shall be known and unambiguously documented. The surface area for which these values are valid shall be marked. The thickness values should be traceable to certified reference standards.

The uncertainties shall be documented with their confidence level, for example *U* (95 %), i.e. the probability, that the “true” value is within the reported uncertainty interval around the documented thickness value, is minimum 95 %.

Prior to use, metal foils and coatings are to be checked visually for damage or mechanical wear as this would cause an incorrect adjustment and thus systematic deviation of all measurement values.

The use of metal foils as reference standards, compared to selected coated base metals, benefits from the possibility of placing the foils directly on each base material. The geometry influence and other factors are then already considered during the adjustment.

6.3 Methods of adjustment

Adjustment of the coating thickness gauges is executed by placing the probes on uncoated and/or one or more coated pieces of base metal with known coating thickness and a coated piece with a sufficiently high coating thickness, so that it can be used as a saturation standard. Depending on the instrument types, the instructions of the manufacturer and on the functional range of the instrument under use, adjustments can be carried out on the following items:

a) a piece of uncoated base material;

b) a piece of coated base material with a sufficiently high thickness (saturation standard);

c) a piece of uncoated base material and a piece of coated base material with a defined coating thickness;

d) a piece of uncoated base material and several pieces of coated base material with defined, but different, coating thicknesses;

e) a piece of uncoated base material, one or several pieces of coated base material with defined, but different, coating thicknesses and a piece of coated base material with a sufficiently high thickness (saturation standard);

f) several pieces of coated base material with defined, but different, coating thicknesses;

g) several pieces of coated base material with defined, but different, coating thicknesses and a piece of coated base material with a sufficiently high thickness (saturation standard).

According to 6.2, the term “coated base material” includes metal foils placed onto uncoated base material.