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Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods

Revêtements métalliques — Mesurage de l'épaisseur — Méthodes par spectrométrie de rayons X

Reference number
ISO 3497:1990(E)

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.

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.

International Standard ISO 3497 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 3497:1976), which has been technically revised.

Annex A forms an integral part of this International Standard.

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Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods

1 Scope

1.1 This International Standard specifies methods for measuring the thickness of metallic coatings by the use of X-ray spectrometric methods.

These methods permit the simultaneous measurement of some 3-layer systems.

1.2 The measuring methods to which this International Standard applies are fundamentally ones which determine the mass per unit area. Using a knowledge of the density of the coating material, the results of measurements can also be expressed as linear thickness of the coating.

1.3 The practical measurement ranges of given coating materials are largely determined by the acceptable measurement uncertainty and may differ depending upon the instrument system and operating procedure used. A table of typical ranges for common materials is given in annex A.

CAUTION — Problems concerning personnel protection against X-rays are not covered by this International Standard. For information on this important aspect, reference should be made to current ISO and national publications, and local regulations, where these exist.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 X-ray fluorescence (XRF): The secondary radiation occurring when a high intensity incident X-ray beam impinges upon a material placed in the path of the incident beam. The secondary emission has wavelengths and energies characteristic of that material.

2.2 intensity of fluorescent radiation: The radiation intensity measured by the instrument, expressed in counts (radiation pulses) per second.

2.3 normalized intensity, I_n: The ratio of the difference in intensity obtained from a coated specimen and an uncoated substrate material, and the difference obtained from a material of thickness equal to or greater than the saturation thickness (see 2.4) and an uncoated substrate material, all measured under the same conditions. This is given by

$$\frac{I_e - I_o}{I_s - I_o}$$

where

I_e is the intensity obtained from the coated specimen;

I_o is the intensity obtained from uncoated substrate material;

I_s is the intensity obtained from a material of thickness equal to or greater than the saturation thickness.

This variable is independent of measurement and integration time, and intensity of the exciting (incident) radiation. The geometric configuration and the energy of the exciting radiation can influence the normalized count rate.

2.4 saturation thickness: The thickness that, if exceeded, will not produce any detectable change in fluorescent intensity.

NOTE 1 Saturation thickness depends upon the energy or wavelength of the fluorescent radiation, density and atomic number of the material and on the angle of incident and fluorescent radiation with respect to the surface of the material.

2.5 intermediate coating: Those coatings that lie between the top coating and the basis material and are of thicknesses less than saturation for each of the coatings.

NOTE 2 Any coating lying between the top coating and the basis material (substrate) and having a thickness above saturation should itself be considered the true substrate since the material under such a coating will not affect measurement and can be eliminated for measurement purposes.

2.6 count rate: The number of radiation pulses recorded by the instrument per unit time (see 2.2).

3 Principle

3.1 Basis of operation

A relationship exists between mass per unit area of the coating (and thus the linear coating thickness if the density is known) and the secondary radiation intensity. This relationship, for any practical instrument system, is first established by calibration using calibration standards having coatings of known mass per unit area. If the coating material density is known, such standards can have coatings given in linear thickness units, provided that the actual density value is also given.

NOTE 3 The coating material density is the density as coated, which may or may not be the theoretical density of the coating material at the time the measurement is made.

The fluorescent intensity is a function of the atomic number of the elements. Providing the top coating, intermediate coating (if present) and the substrate are of different elements they will generate radiation characteristic of each element. A suitable detector system can be adjusted to select either one or more energy bands, enabling the equipment to measure either the top coating or the top and some intermediate coatings simultaneously.

3.2 Excitation

3.2.1 General

The measurement of the thickness of coatings by X-ray spectrometric methods is based on the combined interaction of the coating (or coatings) and substrate with an intense, often narrow, beam of polychromatic or monochromatic X-radiation. This interaction results in the generation of discrete wavelengths and energies of secondary radiation which are characteristic of the elements composing the coating(s) and substrate.

The generated radiation is obtained from a high voltage X-ray tube generator or from suitable radioisotopes.

3.2.2 Generation by a high voltage X-ray tube

Suitable exciting radiation will be produced by an X-ray tube if sufficient potential is applied to the tube and stable conditions apply. Applied voltages are in the order of 25 kV to 50 kV for most thickness requirements but voltages down to 10 kV may be necessary in order to measure low atomic number coating materials. The chief advantages of this method of excitation are the ability to create, by collimation, a very high intensity beam onto a very small measurement area, the ease of control for personnel safety requirements and the potential stability of emission obtainable by modern electronic methods.

3.2.3 Generation by a radioisotope

Only a few radioisotopes emit gamma radiation in the energy band suitable for coating thickness measurement.

Ideally, the exciting radiation is slightly more energetic (shorter in wavelength) than the desired characteristic X-rays. The advantages of radioisotope generation include the possibility of a more compact construction of the instrument, due mainly to there being no need for cooling. In addition the radiation, unlike that from high voltage X-ray generators, is essentially monochromatic and there is a low background intensity.

The major technical disadvantage is the much lower intensity obtainable which prohibits measurements on small areas when compared with the X-ray tube method, the short half-life of the radioisotopes and the personnel protection problems associated with high intensity radioisotopes (the high voltage X-ray tube can be simply switched off).

3.3 Dispersion

3.3.1 General

The secondary radiation resulting from the exposure of a coated surface to X-ray radiation often contains components additional to those required for the measurement of coating thickness. Separation of the desired components is done by either wavelength or energy dispersion.

3.3.2 Wavelength dispersion

The wavelength characteristic of either coating or substrate is selected using a crystal spectrometer. Typical characteristic emission data for commonly used crystals is available in published form from various national authorities.

3.3.3 Energy dispersion

X-ray quanta are usually specified in terms of wavelength or equivalent energies. The relationship between the wavelength, λ, in nanometres, and energy E, in kilo-electronvolts, is given by

$$\lambda \cdot E = 1{,}2396$$

3.4 Detection

3.4.1 The type of detector used for wavelength dispersive systems is usually a gas filled tube or scintillation counter interconnected with a photomultiplier.

3.4.2 The most suitable detector for use in energy dispersive systems to receive the fluorescent photons is selected by the instrument designer according to the application. In the energy band of about 1,5 keV to 100 keV, measurements can be made in normal atmosphere without helium gas or vacuum.

Fluorescent radiation of the different characteristic energies pass into the proportional counter detector tube and then onto a multi-channel analyser which is adjusted to select the correct energy band (or bands).

3.5 Thickness measurement

There are two X-ray methods for measurement of thickness as follows.

a) **Emission method.** If the intensity of the characteristic radiation from the coating is measured, the intensity will increase with increasing thickness up to the saturation thickness. See figure 1a).

b) **Absorption method.** If the intensity of the characteristic radiation from the substrate is measured, the intensity will decrease with increasing thickness. See figure 1b).

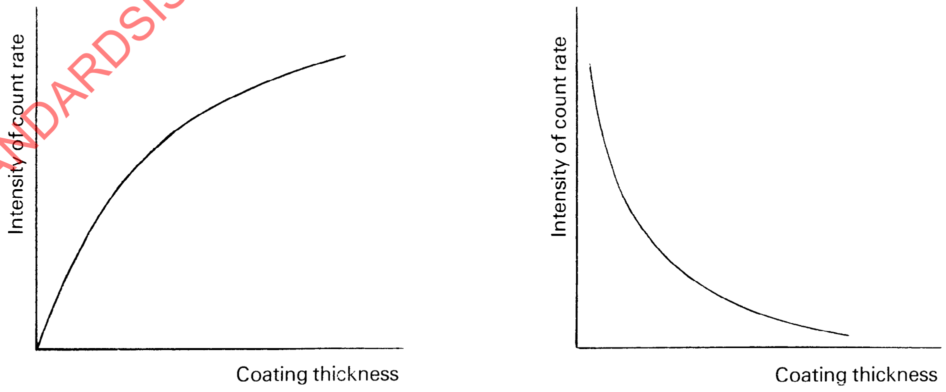
When the X-ray emission method is used, the equipment is adjusted to receive a selected band of energies characteristic of the coating material. Thus thin coatings produce low intensities while thick coatings produce high intensities.

The X-ray absorption method uses the band of energies characteristic of the substrate material. Thus thin coatings result in high intensities while thick coatings produce low intensities. In practice, care has to be taken to ensure that no intermediate coating is present.

It is possible to combine X-ray absorption and emission when coating thicknesses are expressed as a ratio of the respective intensities of substrate and coating materials. Measurements by this ratio method are largely independent of the distance between test specimen and detector.

The absorption characteristic is similar to the inverse of the emission characteristic.

In all methods, the intensity of secondary radiation is recorded as pulses, usually taken over a preselected fixed time period. The normalized count-rate system is used in many commercially available instruments adjusted so that the count-rate characteristic of the uncoated substrate is zero and that from an infinitely thick sample of the coating material is unity. All measurable thickness therefore produce count rates which lie within the normalized count-rate range of 0 to 1. See figure 2.



a) X-ray emission method

b) X-ray absorption method

Figure 1 — Relationship between intensity of count rate and coating thickness

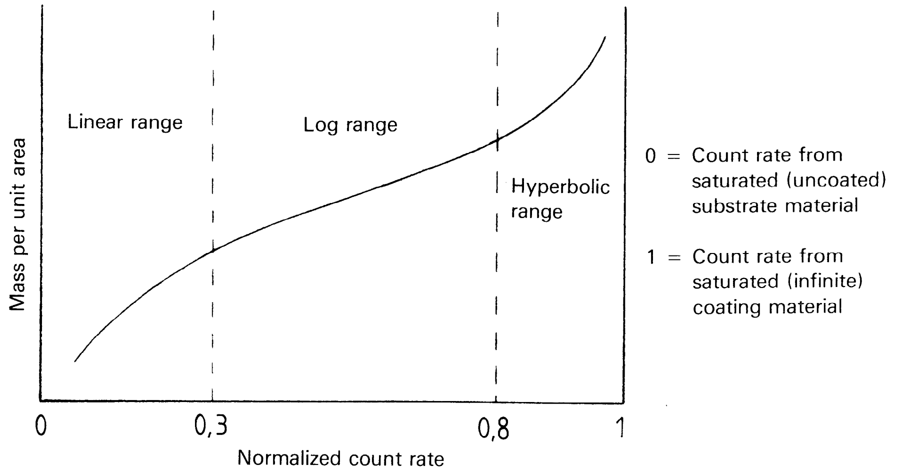


Figure 2 — Relationship between mass per unit area and normalized count rate

In all cases, the best or most sensitive range of measurement lies approximately between 0,3 and 0,8 on the normalized count-rate scale. Thus for best measurement accuracy over the whole thickness range, it is advantageous to use calibration standards having count-rate characteristics at 0,3 and 0,8. Other standards may be necessary with some equipment to ensure precision at other thicknesses. Since the relative uncertainty of calibration of standards increases as thickness decreases, it is essential to establish the correct mathematical relationship at the thin end of the range by suitable use of standards having thicker coatings but lower uncertainties.

When measuring coating/substrate material combinations that have widely differing energies (energy dispersive systems), the ratio of saturated coating to uncoated substrate count-rate characteristics is very high (10 : 1 is typical). In such cases, it is not always essential to have calibration standards having a similar or the same substrate (since the substrate material will not radiate in the same energy band as the coating material). Where the uncoated substrate/infinite coating count-rate ratio is 3 : 1 (for coating/substrate combinations having similar energies) it is often necessary to use an "absorber" selected to absorb the radiation of one of the materials, usually that of the substrate material. This absorber is usually placed manually or automatically between the surface being measured and the detector.

3.6 Multilayer measurements

It is possible to measure more than one coating layer provided that the characteristic X-ray emissions of the inner layers are not completely absorbed by the outer layers. In an energy dispersive system the multi-channel analyser is set to receive two or more distinct energy bands characteristic of two or more materials.

3.7 Alloy composition/thickness measurement

Certain alloys and compounds, for example tin-lead, can be measured simultaneously for composition and thickness. Since the thickness measurement of an alloy or compound is dependent upon alloy composition, it is mandatory either to know or assume the composition before thickness measurement or to be able to measure composition.

NOTE 4 Assumed compositions can introduce errors in thickness measurements.

Some coatings can form alloys by interdiffusion with the substrate. The presence of such alloy layers can add to the measurement uncertainty.

4 Apparatus

Fluorescent X-ray equipment suitable for measuring coating thickness in accordance with this International Standard is available commercially. Equipment designed specifically for coating thickness measurement is of the energy dispersive kind and usually comes with a microprocessor for converting the intensity measurement to mass per unit area or thickness, for storing calibration data, and for computing various statistical measurements. See figure 3.

NOTE 5 The essential components of an X-ray fluorescence coating thickness measuring apparatus include a primary X-ray source collimator, a support for the test specimen, a detector and an evaluating system. The source, collimator and detector are usually in a geometrically fixed relation with each other. If the atomic numbers of the coating and substrate materials are very close, it may be necessary to introduce an absorber which will absorb the characteristic fluorescent energy of one of the materials, for example the substrate.

It may be necessary to introduce special software, electronic filtering or a physical absorber. The use of these will separate, filter or absorb characteristic fluorescent energy of one or more of the materials present. The introduction of such devices enables fluorescence from the material being measured to be enhanced, so decreasing measurements uncertainty.

4.1 Primary X-ray source

This is either an X-ray tube or a suitable radial isotope. Both shall be capable of exciting the fluorescent radiation to be used for measurement.

4.2 Collimator

This takes the form of a precisely dimensioned aperture or apertures which, in theory, can be of any shape. The aperture size and shape determines the incident X-ray beam dimensions at the surface of the coating being measured. Current commercial instruments have collimator apertures that are circular, square or rectangular.

4.3 Detector

The detector receives the fluorescent radiation from the measured specimen and converts this into an electrical signal which is passed on for evaluation. The evaluating unit is set to select one or more energy bands characteristic of the top, intermediate, and/or substrate materials.

4.4 Evaluating unit

This processes the incoming data according to its software programme and thus determines the mass per unit area or coating thickness of the test specimen.

5 Factors that influence the measurement results

5.1 Counting statistics

5.1.1 General

The production of X-ray quanta is random with respect to time, which means that during a fixed time interval the number of quanta emitted will not always be the same. This gives rise to the statistical error which is inherent in all radiation measurements. In consequence, an estimate of the count rate based on a short counting period (e.g. 1 s or 2 s) may be appreciably different from an estimate based on a longer counting period, particularly if the count rate is low. This error is independent of other sources of error, such as those arising from mistakes on the part of the operator or from the use of inaccurate standards. To reduce the statistical error to an acceptable level, it is necessary to use a counting interval long enough to accumulate a sufficient number of counts. When an energy dispersive system is used, it should be recognized that a significant portion of the intended counting period may be consumed as dead time, i.e. time during which the count-rate capacity of the system is exceeded. It is possible to correct for dead time losses by following the manufacturer's instructions for his particular instrumentation.

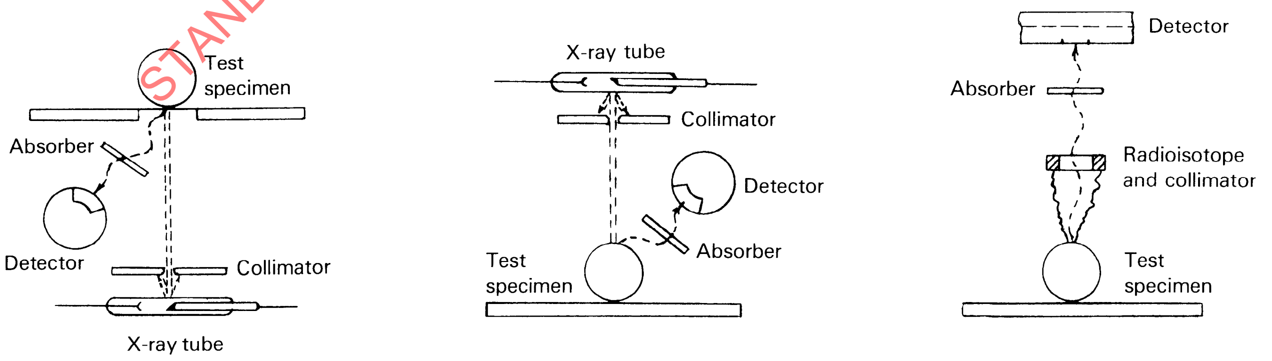
5.1.2 The standard deviation, s, of this random error closely approximates the square root of the total count; that is

$$s \approx \sqrt{N}$$

where N is the count in a given time.

95 % of all measurements lie within

$$N\left(1 \pm \frac{2}{\sqrt{I}}\right)$$



a) **X-ray tube**

b) **X-ray tube**

c) **Radioisotope as primary X-ray source**

Figure 3 — Schematic representations of energy dispersive systems with their essential major components

5.1.3 The standard deviation of the thickness measurement is not the same as the standard deviation of the count rate but is related to it by a function that is dependent upon the slope of the calibration curve at the point of measurement. Most commercially available XRF thickness instruments display the standard deviation in micrometres or as a percentage of mean thickness.

5.2 Calibration standards

Thickness standards for calibration measurement are available. However, such standards are not guaranteed to better than 5 % (this value is lower in specific cases). It is more difficult to maintain 5 % for thin coatings, for example, due to roughness, porosity and diffusion. The calibration thickness standard can only be used if it provides a normalized count rate between 0,05 and 0,9.

5.3 Coating thickness

The measurement uncertainty will be affected by the thickness range being measured. In the curves shown in figure 2, the accuracy will be best in the portion of the curve from about 30 % to 80 % saturation. The accuracy for a given measuring time rapidly decreases outside this range. The situation is similar for the absorption curve. The limiting thicknesses are, in general, different for each coating material.

5.4 Size of measuring area

To obtain satisfactory counting statistics (see 5.1), in a reasonably short counting period, select the collimator aperture to provide the largest possible measuring area consistent with the size and shape of the specimen. In most cases, the relevant or representative area to be measured shall be larger than the collimated beam area (the collimated beam area at the measured surface is not necessarily the same as the collimator aperture dimensions). However, in some special cases, the area to be measured may be smaller than the beam area (see 5.11).

Caution may also be necessary if the measuring area produces a count rate that saturates or exceeds the capacity of the detector (some commercial instruments may limit this count rate automatically but this should be verified with the relevant manufacturer).

The calibration must then be carried out on an area of the same dimension.

5.5 Coating composition

Mass/unit area measurements may be affected by the presence of foreign materials such as inclusions, co-deposited material or alloy layers formed by diffusion at the coating/substrate interface. Thickness measurements are, in addition, affected by voids and porosity. Some sources of error can be eliminated by the use of representative calibration standards, i.e. standards produced under the same conditions and having representative X-ray characteristics. Since inclusions, pores or voids can create a different density, coatings with such imperfections are best measured in mass per unit area units. If known, the actual coating density value can often be introduced into the measuring equipment in order to enable a correction to be made (see 5.6).

5.6 Coating density

If the density of the coating differs from that of the calibration standard there will be a corresponding error in the thickness measurement. When the density of the coating material is known, the thickness can be obtained (see 3.1).

If the instrument measurement is in units of mass per unit area, the linear thickness is obtained by dividing this measurement by the coating density:

$$d = \frac{\rho_A}{\rho_2} \times 10$$

If the measurement is in linear units, a density correction may be applied as in the following equation:

$$d = d_m \times \frac{\rho_1}{\rho_2}$$

where

d is the linear thickness, in micrometres;

d_m is the linear thickness readout, in micrometres;

ρ_1 is the density of the coating material of the calibration standards, in grams per cubic centimetre;

ρ_2 is the density of the coating material of the test specimen, in grams per cubic centimetre;

ρ_A is the mass per unit area of coating of the test specimen, in milligrams per square centimetre.

5.7 Substrate composition

If the emission method is used, the effect of differences in the substrate composition is negligible provided that

a) fluorescent X-rays from the substrate do not encroach into the energy band selected for the characteristic coating energy (if encroachment does occur, special procedures are needed to eliminate their effect);

b) fluorescent X-rays from the substrate material are incapable of exciting the coating material;

c) the intensity ratio method is used (see 3.5).

If the absorption method is used, it is essential that the composition of the substrate of the calibration or reference standard(s) is identical with that of the substrate of the test specimen.

5.8 Substrate thickness

For measurements by the X-ray emission method, it is necessary for the substrate of double-sided material to be thick enough to prevent interference from any underlying material.

For use of the X-ray absorption method, it is necessary for the substrate to be equal to or greater than its saturation thickness. If this criterion is not met, it is essential to calibrate the apparatus with reference standards of identical substrate thickness (see 6.3).

5.9 Surface cleanliness

Foreign material present on the surface can lead to inaccurate measurements. Protective coatings, surface treatments or lacquers can also lead to inaccuracies.

5.10 Intermediate coatings

The absorption method cannot be used in the presence of intermediate coatings, the absorption properties of which are not known. In such a case, the emission method is advised.

5.11 Specimen curvature

If it is necessary to make measurements on curved surfaces, select the collimator or beam confining aperture to minimize the effects of the surface curvature. Surface curvature effects are minimized by the use of an aperture of small dimension compared to the radius of curvature of the surface under test.

NOTE 6 The use of rectangular apertures may be advantageous in measuring cylindrical surfaces.

If calibration is made with calibration standards of the same size and shape as the test specimen, surface curvature effects are eliminated, but make the measurement in the same position and plane and on the same measuring area. In such cases, a collimator aperture larger than the test specimen can sometimes be used.

5.12 Excitation energy and excitation intensity

Since fluorescent radiation intensity depends upon both excitation energy and intensity, it is essential that the apparatus used is stable enough to provide identical excitation characteristics both in calibration and measurement.

5.13 Detector

Errors in measurement can be introduced due to erratic or unstable operation of the detector system. Before use, check the apparatus for stability.

Stability checks are either

a) automatically applied by some instruments; or

b) manually applied by the operator.

In both cases, a single reference or sample piece is placed in the X-ray beam and is not moved during the check. A series of single count-rate measurements is made over a short period of time and the standard deviation of this series should not be significantly greater than the square root of the mean of the series. To establish stability over a longer period of time, the above results are compared with those previously obtained (or stored in the instrument in automatic checking) at some other time. The time taken for a single measurement series or required between two separate series will establish stability for that time period.

5.14 Radiation path

Keep the radiation path as short as possible, since losses can increase measurement uncertainty. Elements having atomic numbers below 20 do not radiate sufficiently strongly for the type of apparatus shown in figure 3. It is therefore necessary to use vacuum or helium spectrometers if lower atomic number materials are to be measured.

5.15 Conversion of count rate to mass per unit area or thickness

Modern commercial apparatus use microprocessors for the conversion of count rate to mass per unit area or thickness. The microprocessor usually has a mathematically derived master programme which is modified to local requirements using calibration or reference standards. The reliability of the conversion depends on the validity of the calibration curve, equation, algorithm, or on whatever other conversion method is used. It also depends on the quality of the calibration standards and the number and thicknesses of the calibration points relative to the thicknesses being measured.

When one coating layer causes additional fluorescence of another layer, it is essential for the conversion method to take this into account.

Extrapolation beyond the thickness range established by calibration standards can result in serious errors.

5.16 Tilt of specimen surface

If the tilt of the test surface with respect to the X-ray beam differs from that used during calibration, there may be a significant change in the count rate. For example a 10° difference in tilt may cause a 4 % change in the count rate.

6 Calibration of instrument

6.1 Introduction

6.1.1 General

Make the calibration in accordance with the instrument manufacturer's instructions. Give appropriate attention to the factors listed in clause 5 and to the requirements of clause 8.

The instrument shall be calibrated for the coating-substrate system and the thicknesses or masses per unit area to be measured. The calibration shall be checked at least once each day that the measurements are made, by remeasuring one of the calibration standards. If there is a change of the measured thickness that is great enough to preclude meeting the requirements of clause 8, recalibrate the instrument.

6.1.2 Linear range calibration

For measurements on very thin coatings in the linear range, i.e. for coatings that produce normalized count rates below 0,3 (30 % of full scale), it is advisable to make a calibration using an uncoated sample of the substrate material and a single coating standard of known thickness within the linear range. It is necessary for the user to ascertain that the thickness being measured and that of the calibration standard are within the linear range.

6.1.3 Logarithmic range calibration

For measurements within this range it is necessary, in most cases, to use a set of at least four standards: one sample of uncoated substrate, one sample of the coating material of at least saturation thickness, one coated standard at or very near to the lower end of the logarithmic range of thickness and one coated standard having a coating near to the top end of the logarithmic thickness range.

6.1.4 Entire measurable range

To measure from zero into the hyperbolic range, it will probably be necessary to use additional coated standards to more closely define the extremities of the thickness range.

Some instruments that are calibrated for these additional coated standards are capable of interpolation between zero value and the minimum thickness standard. Extrapolation beyond the highest standard is not recommended because it is likely to yield unreliable results (see also 5.2).

6.2 Standards

6.2.1 General

Use reliable reference standards to calibrate the apparatus. Final measurement uncertainty is directly dependent on the measurement uncertainty of the calibration standards and the measurement precision.

Reference standards shall have uniform coatings of known mass per unit area or thickness and, in the case of alloys, of known composition. The coating anywhere on the available or defined surface of the reference standard shall not deviate from the specified value by more than ± 5 %. Standards which are specified in thickness units (as opposed to mass per unit area) will be reliable, provided that they are used for coatings of the same composition and the same or known density. For the measurement of the composition of an alloy, the composition of the calibration standards need not be the same, but shall be known.

6.2.2 Foils

If standards take the form of foils laid down onto a particular substrate surface, it is essential to exercise care to ensure that the contacting surfaces are clean and without folds or kinks. Any density differences shall be compensated or allowed for in the final measurement.

6.3 Selection of standards

Calibrate the instrument in either mass per unit area or thickness units; in the latter case, the thickness values shall be accompanied by the density of the coating material, or by the assumed density if the thickness of the standard was calculated from a mass per unit area measurement. The standard shall have the same coating and substrate (see 5.7 and 5.8) materials as the test specimen, although some apparatus designs allow some deviation from this ideal (see 3.1).

6.4 X-ray emission characteristics of standards

The coating of the calibration standards shall have the same X-ray emission (or absorption) properties as the coating being measured (see 5.6).

6.5 Substrate X-ray emission characteristics of thickness standards

If the thickness is to be determined by the X-ray absorption method, the substrate of the thickness standards shall have the same X-ray emission properties as those of the test specimen. This shall be verified by comparing the intensities of the selected characteristic radiations of both the uncoated substrate of the test specimen and that of the calibration reference standard.

6.6 Substrate thickness

In the X-ray absorption method, the substrate thickness of the test specimen and the calibration standards shall be the same unless the saturation thickness (defined in 2.4) is exceeded.

If the curvature of the coating to be measured precludes calibration of a flat surface, it is essential either

a) to observe the precaution given in 5.11; or

b) to calibrate using standards having the same curvature as the test specimen.

7 Procedure

7.1 General

Operate the instrument in accordance with the manufacturer's instructions and give appropriate attention to the factors listed in clause 5 and to the accuracy requirements of 6.2 and clause 8.

7.2 Selection and calibration of the collimator or aperture

Select the aperture or collimator in accordance with the shape and size of the available test area on the test specimen. Ensure that the distance between the collimator aperture opening and test specimen remains constant during the measurement.

7.3 Measurement of curved surfaces

It is possible to make measurements on curved surfaces, provided a sufficiently small collimator aperture is selected such that a characteristic curve valid for a flat test surface can be used. If this is not the case, it is essential to take into account the observations of 5.4 and 5.11.

7.4 Calibration (see clause 6)

Check the calibration data at regular intervals in order to be sure that the instrument and measurement conditions have not changed. Since different instrument designs have different requirements, adhere to the manufacturer's instructions.

7.5 Measurement time

As final measurement uncertainty depends upon measurement time, select a measurement time sufficient to produce an acceptably small measurement uncertainty.

7.6 Number of measurements

As measurement uncertainty will be partially determined by the number of measurements made, an increase in the number of measurements will decrease measurement uncertainty. If the number of measurements is increased by n, the measurement uncertainty will be reduced by a factor of $1/\sqrt{n}$.

The standard deviation shall be calculated from at least 10 measurements made by relocating the same measurement area over or under the incident X-ray beam after each measurement.

7.7 Precautions

See 1.3.

7.8 Expression of results

The conversion of the intensity (count rate) values to mass per unit area or thickness units is made automatically by many commercially available instruments. For other instruments, construct curves similar to those of figure 1 using suitable calibration standards. Express results of mass per unit area measurements in milligrams per square centimetre and those of thickness in micrometres, unless otherwise agreed.

8 Measurement uncertainty

The instrument, its calibration and its operation, shall be such that the uncertainty of the measurement is less than 10 %.

To reduce the measurement uncertainty, increase measurement time, number of measurements made and/or the collimator/aperture size (up to the point where the detector becomes saturated).

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) unambiguous identification of the test specimen;

c) date of measurement;

d) the location of the measurement on the test specimen;

e) the number of measurements averaged for each reported measurement;

f) the collimator aperture size and the measured area size, if different;

g) the measured values;

h) the density used for thickness calculation and justification for the value used;

i) a standard deviation which is representative of the reported measurements (see 7.6);

j) any deviations from this ISO test method;

k) any factors that might influence interpretation of the reported results;

l) the name of the operator and that of the testing laboratory.