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

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

Reference number
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Foreword

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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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3497 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

This third edition cancels and replaces the second edition (ISO 3497:1990), which has been technically revised.

Annex A of this International Standard is for information only.

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

1　Scope

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

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

1.2　The measuring methods to which this International Standard applies are fundamentally those that 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 measuring methods permit simultaneous measurement of coating systems with up to three layers, or simultaneous measurement of thickness and compositions of layers with up to three components.

1.4　The practical measurement ranges of given coating materials are largely determined by the energy of the characteristic X-ray fluorescence to be analysed and by the acceptable measurement uncertainty and can differ depending upon the instrument system and operating procedure used.

2　Terms and definitions

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

2.1
X-ray fluorescence
XRF
secondary radiation occurring when a high intensity incident X-ray beam impinges upon a material placed in the path of the incident beam

NOTE　The secondary emission has wavelengths and energies characteristic of that material.

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

2.3
saturation thickness
thickness that, if exceeded, does not produce any detectable change in fluorescent intensity

NOTE　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.

2.4
normalized intensity

x_n

ratio of the difference in intensity obtained from a coated specimen, x, and an uncoated substrate material, x_0, and the difference obtained from a material of thickness equal to or greater than the saturation thickness, x_s (see 2.3) and an uncoated substrate material, x_0, all measured under the same conditions

NOTE 1   The mathematical relationship is given by:

$$x_\mathsf{n} = \frac{x - x_0}{x_\mathsf{s} - x_0}$$

where

   x is the intensity obtained from the coated specimen;

   x_0 is the intensity obtained from uncoated substrate material;

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

NOTE 2   The normalized intensity is independent of measurement and integration time, and intensity of the excitation (incident radiation). The geometric configuration and the energy of the excitation radiation can influence the normalized count rate. The value of x_n is valid between 0 and 1.

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

NOTE   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 the measurement and can be eliminated for measurement purposes.

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

2.7
basis material
basis metal
material upon which coatings are deposited or formed

[ISO 2080:1981, definition 134]

2.8
substrate
material upon which a coating is directly deposited

NOTE   For a single or first coating the substrate is identical with the **basis material**; for a subsequent coating the intermediate coating is the substrate.

[ISO 2080:1981, definition 630]

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 calibrating 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   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. If this density differs from the density of the calibration standards, a factor that reflects this difference is used and documented in the test report.

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 or a coating consists of more than one element, these elements will generate radiation characteristics for each of them. A suitable detector system can be adjusted to select either one or more energy bands, enabling the equipment to measure thickness and/or composition of 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 generating discrete wavelengths or 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 excitation 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. For some applications the use of a primary filter, located between the X-ray tube and the specimen, decreases the measurement uncertainty.

The chief advantages of this method of excitation are

⎯   the ability to create, by collimation, a very high intensity beam on a very small measurement area;

⎯   the ease of control for personnel safety requirements;

⎯   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 excitation radiation is of slightly higher energy (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 low background intensity.

The major technical disadvantages when compared with the X-ray tube method are

— the much lower intensity that is obtained, which prohibits measurements on small areas;

— the short half-life of some radioisotopes;

— personnel protection problems associated with high intensity radioisotopes (the high voltage X-ray tube can simply be 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. The desired components are separated 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 are 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 kiloelectron-volts (keV), is given by

$$\lambda \times E = 1{,}239\ 842\ 7.$$

3.4 Detection

The type of detector used for wavelength dispersive systems can be a gas-filled tube, a solid state detector or scintillation counter connected to a photomultiplier.

The most suitable detector for receiving fluorescent photons and used in energy dispersive systems 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 different characteristic energies passes into the energy dispersive detector and then on to a multi-channel analyser that is adjusted to select the correct energy band.

3.5 Thickness measurement

3.5.1 Emission method

If the intensity of the characteristic radiation from the coating is measured, the intensity increases with increasing thickness up to the saturation thickness. See Figure 1 a).

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

3.5.2 Absorption method

If the intensity of the characteristic radiation from the substrate is measured, the intensity decreases with increasing thickness. See Figure 1 b).

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

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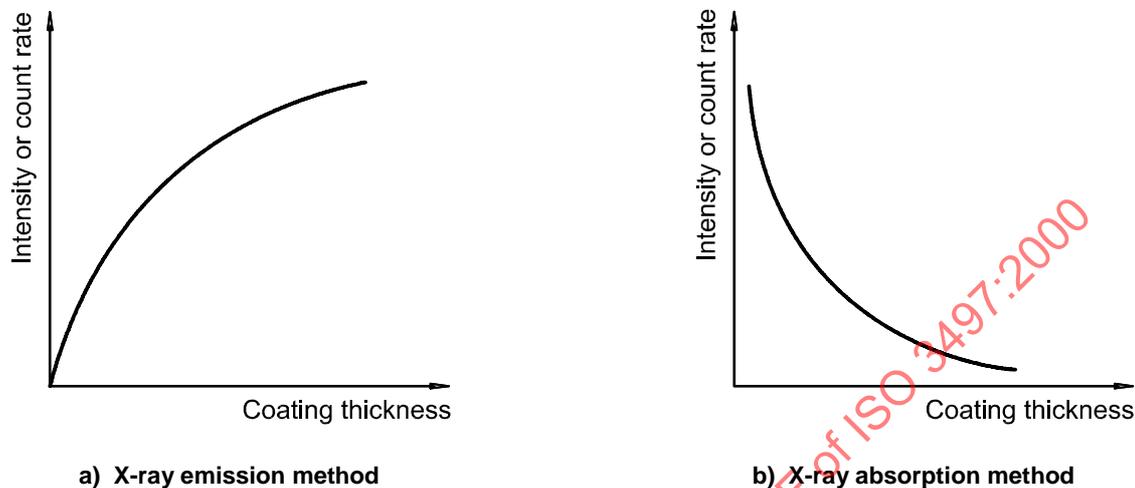

a) X-ray emission method   b) X-ray absorption method

Figure 1 — Schematic illustrations of the relationship between intensity or count rate and coating thickness

3.5.3 Ratio method

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 the ratio method are largely independent of the distance between the test specimen and the detector.

3.5.4 Measurement

For both methods described in 3.5.1 and 3.5.2, the normalized count-rate system is usually used in many commercially available instruments adjusted so that the count-rate characteristic of the uncoated substrate is zero and that of an infinitely thick sample of the coating material is unity. All measurable thicknesses therefore produce count rates that lie within the normalized count-rate range of 0 to 1. See Figure 2.

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 of 0,3 and 0,8. With some equipment other standards may be necessary in order 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 for the thin end of the range by suitable use of standards having thicker coatings but lower uncertainties.
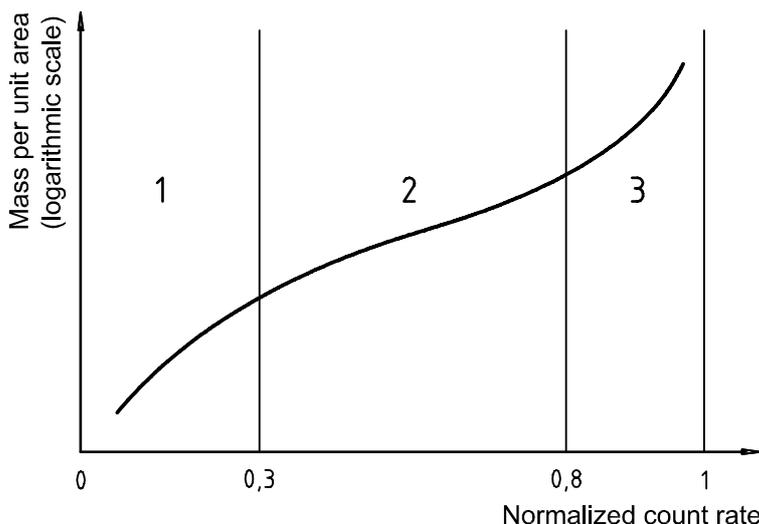
Key

1   Linear range

2   Logarithmic range

3   Hyperbolic range

NOTE   0 = Count rate from saturated (uncoated) substrate material; 1 = count rate from saturated (infinite) coating material.

Figure 2 —Schematic illustration of the relationship between mass per unit area and normalized count rate

3.6   Absorber for secondary radiation

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). When the uncoated substrate/infinite coating count-rate ratio is 3:1 (for coating/substrate combinations having similar energies) it is often helpful 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.

3.7   Mathematical deconvolution

When using a multi-channel analyser a mathematical deconvolution of the secondary radiation spectra can be used to extract the intensities of the characteristic radiation. This method can be used when the energies of the detected characteristic radiations do not differ sufficiently, e.g. characteristic radiation from Au and Br. This method is sometimes described as 'numerical filtering' in order to distinguish it from the filtering method (see 3.6).

3.8   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.

## 3.9   Alloy composition thickness measurement

Certain alloys and compounds, for example Sn-Pb, can be measured simultaneously for composition and thickness. In some cases this method can also be used under the conditions described in 3.8, e.g., Au on Pd/Ni on a Cu alloy substrate. Since the thickness measurement of an alloy or compound is dependent upon alloy composition, it is essential either to know or assume the composition before thickness measurement or to be able to measure the composition.

NOTE   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

See Figures 3, 4 and 5.
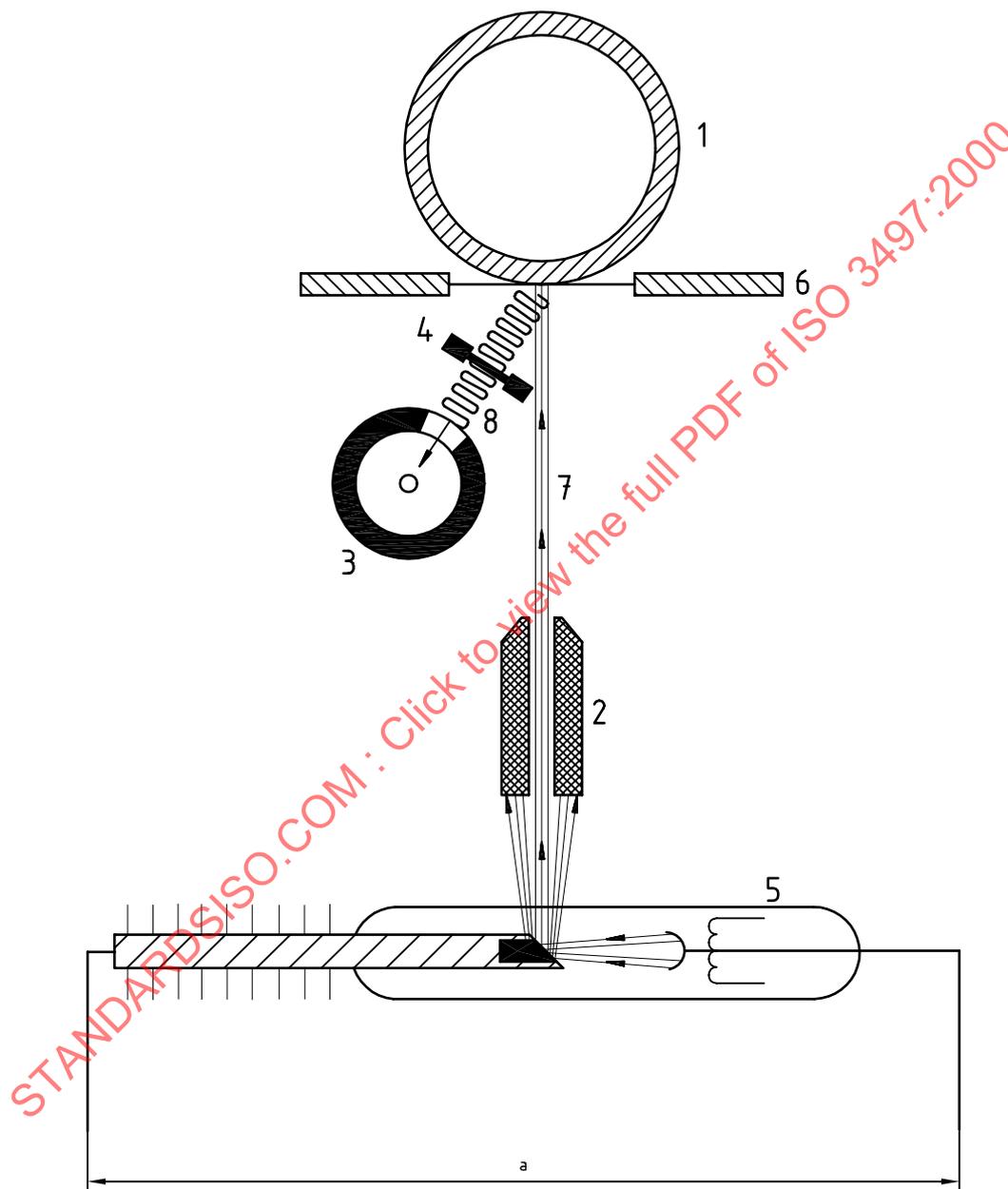
Key

1	Test specimen	4	Absorber	7	Incident X-ray beam
2	Collimator	5	X-ray generator	8	Characteristic fluorescent X-ray beam for detection and analysis
3	Detector	6	Specimen support		

a   High voltage
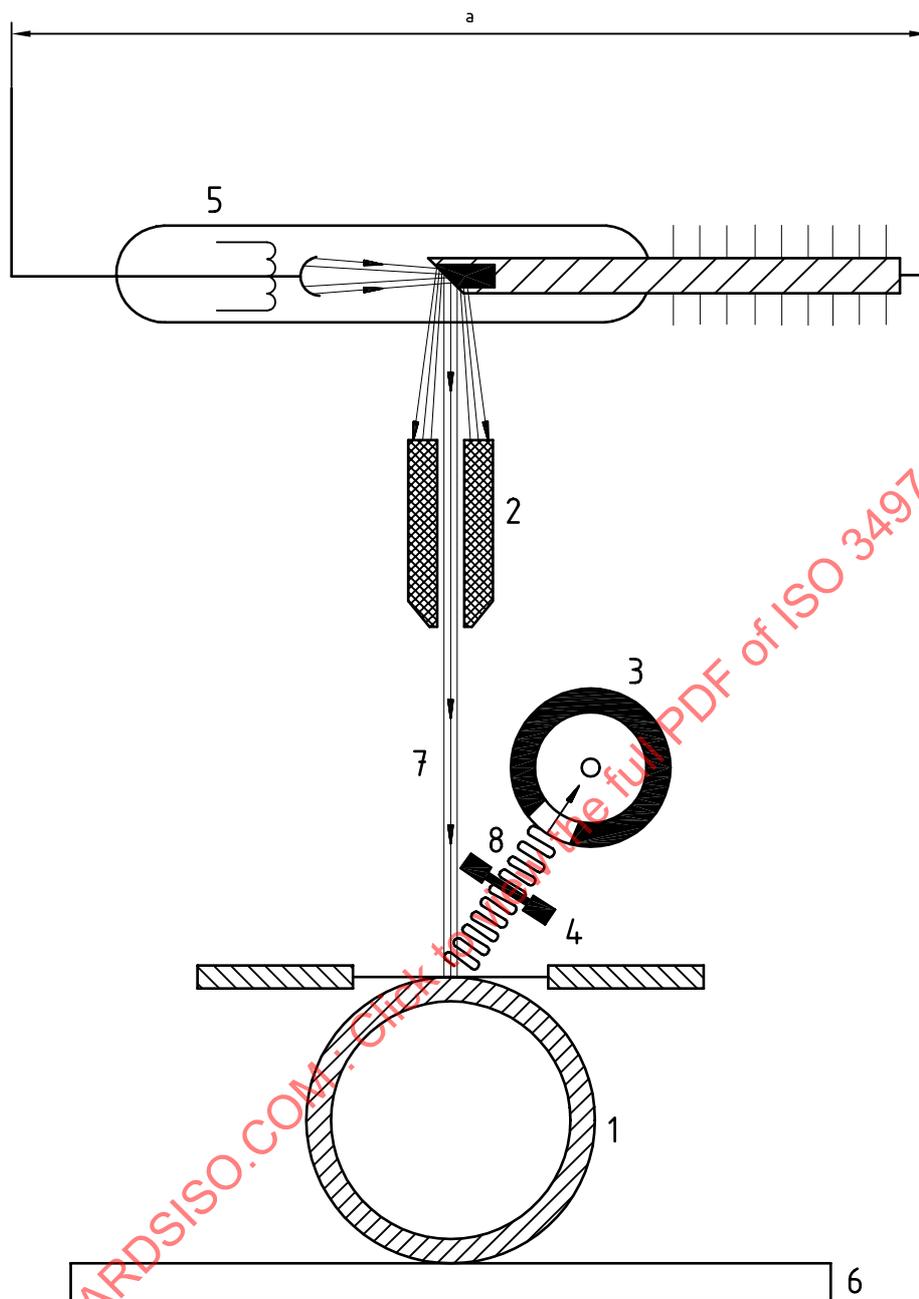
Figure 3 — Schematic representation of an X-ray tube

Key

1	Test specimen	4	Absorber	7	Incident X-ray beam
2	Collimator	5	X-ray generator	8	Characteristic fluorescent X-ray beam for detection and analysis
3	Detector	6	Specimen support		

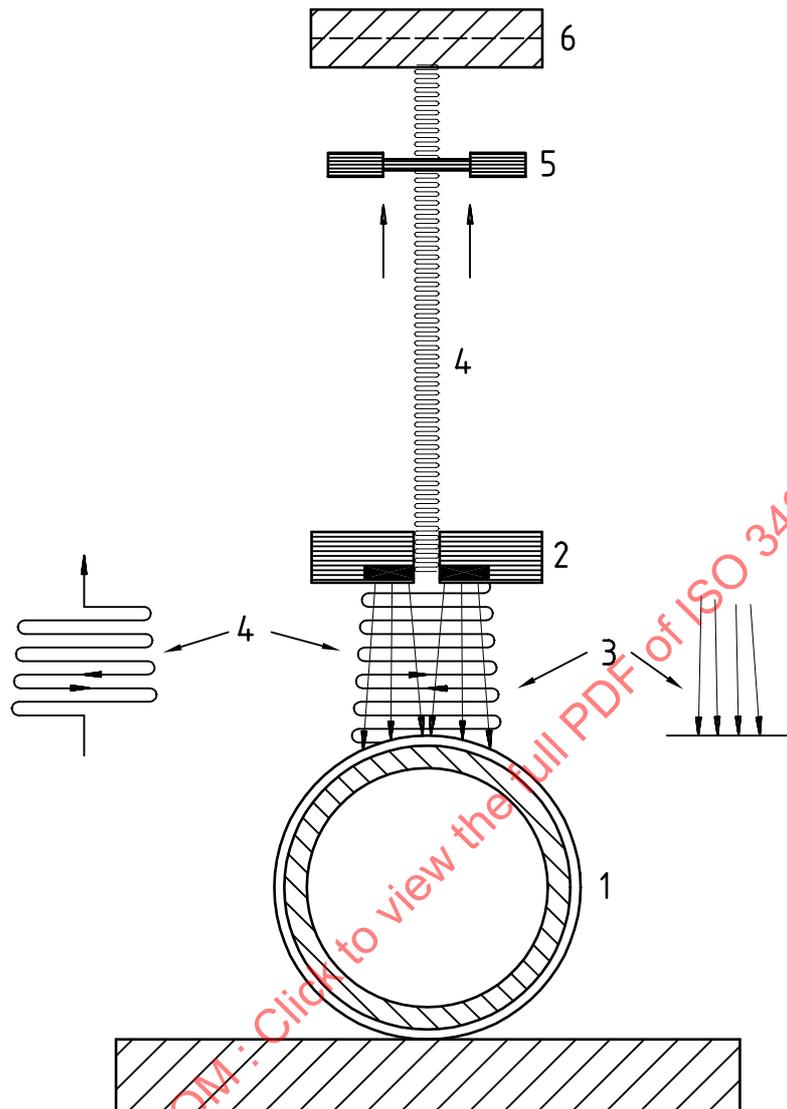a   High voltage

Figure 4 — Schematic representation of an X-ray tube with a solid specimen support

Key

1	Test specimen	4	Characteristic fluorescent X-ray beam for detection and analysis
2	Radioisotope and collimator	5	Absorber
3	Incident X-ray beam	6	Detector

Figure 5 — Schematic representation of a radioisotope as primary X-ray source

4.1 Primary X-ray source, being either an X-ray tube or a suitable radio isotope either of which shall be capable of exciting the fluorescent radiation to be used for measurement.

4.2 Collimator, in 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, for receiving the fluorescent radiation from the measured specimen and converting this into an electrical signal that 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, for processing the incoming data according to its software program and thus determining the mass per unit area or coating thickness of the test specimen.

NOTE   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.

The essential components of an X-ray fluorescence coating thickness measuring apparatus include a primary X-ray source, a 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 to each other. If the atomic numbers of the coating and substrate materials are very close, it may be necessary to introduce an absorber that will absorb the characteristic fluorescent energy of one of the materials, e.g. the substrate.

5   Factors that influence the measurement results

5.1   Counting statistics

5.1.1   The production of X-ray quanta is random with respect to time. This 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 that 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) can 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 the particular instrumentation.

5.1.2   The standard deviation, s, of this random error closely approximates to the square root of the count rate and the accumulation time; i.e.,

$$s = \sqrt{\frac{X}{t_{\text{meas}}}}$$

where

  X is the count-rate;

  t_{meas} is the accumulation time (measuring time) in seconds.

95 % of all measurements lie in the interval:

$$X - 2s \leqslant X \leqslant X + 2s$$

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 X-ray fluorescence thickness instruments display the standard deviation in micrometres or as a percentage of mean thickness.

Using the method of deconvolution (numerical filtering) an additional contribution to the standard deviation of the count rate comes from the mathematical algorithm.

5.2   Calibration standards

Thickness standards for calibration measurement are available. However, it cannot be guaranteed that the accuracy of such standards is higher than 5 % (the value is lower in specific cases). Due to roughness, porosity and diffusion it is more difficult to maintain 5 % accuracy for thin coatings. The calibration thickness standard can only be used if it provides a normalized count rate between 0,05 and 0,9.

In addition to the trueness of the calibration standards, the repeatability of the measurements during calibration affects the reproducibility of coating thickness results taken with different apparatus and from different laboratories.

The mass per unit area, density, thickness and composition of the standards shall be certified. Traceability to national, international or other acceptable standards is essential. Acceptability shall be agreed between the supplier and the customer.

5.3 Coating thickness

The measurement uncertainty under repeatable conditions will be affected by the thickness range being measured. In the curve shown in Figure 2, the relative accuracy will be best in the portion of the curve between approximately 30 % and 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 generally 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 cases, the area to be measured may be smaller than the beam area (see 5.11). In this case a change of the area to be measured shall be adequately corrected.

Caution is also 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).

5.5 Coating composition

Mass/unit area measurements can 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. Therefore, if possible, the thickness and composition shall be measured simultaneously (see 3.7).

In addition, thickness measurements are 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 give rise to variations in 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 then there is 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, m, is in units of mass per unit area, the linear thickness, d, is obtained by dividing this measurement by the coating density, ρ:

$$d = \frac{m}{\rho}$$

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

$$d = d_m \times \frac{\rho_{standard}}{\rho_{coating}}$$

where

d is the linear thickness, in micrometres;

d_m is the linear thickness readout, in micrometres;

$\rho_{standard}$ is the density of the coating material of the calibration standards, in grams per cubic centimetre;

$\rho_{coating}$ is the density of the coating material of the test specimen, in grams per cubic centimetre;

m 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.

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

5.8 Substrate thickness

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

For the X-ray absorption or intensity ratio method, it is essential that the substrate 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 cases, the emission method shall be used.

5.11 Specimen curvature

If it is necessary to make measurements on curved surfaces, the collimator or beam-confining aperture shall be selected 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   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 the measurements shall be made 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 be stable enough to provide identical excitation characteristics both in calibration and measurement. For example, a change in X-ray tube current would change the primary intensity of tube radiation.

5.13 Detector

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

Stability checks are either:

a) performed automatically by some instruments or

b) performed manually by the operator.

In both cases, a single reference or sample shall be placed in the X-ray beam and shall not be moved during the check. A series of single count-rate measurements shall be made over a short period of time and the standard deviation of this series shall 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 shall be compared with those previously obtained (or stored in the instrument in automatic checking) at some other time.

NOTE   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

The radiation path shall be kept as short as possible, since losses can increase measurement uncertainty. The instrument designer should have optimized the radiation path according to the application range. Elements having atomic numbers below 20 do not radiate sufficiently strongly for the type of apparatus shown in Figures 3, 4 and 5. It is therefore essential to use vacuum or helium spectrometers if lower atomic number materials have 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 of thickness. The microprocessor usually has a mathematically derived master programme that 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 that the conversion method 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 can be a significant change in the count rate, particularly in the portion of the calibration curve above 0,9 normalized count rate, where a tremendous change of the thickness can occur; e.g a 5° difference in tilt can cause a 3 % change in the count rate and from this results a 12 % change in thickness.

6 Calibration of instrument

6.1 Introduction

6.1.1 General

The instrument shall be calibrated in accordance with the instrument manufacturer's instructions. Appropriate attention shall be paid to the factors listed in clause 5 and to the requirements of clause 8.

The instrument shall be calibrated with standards having the same composition of coating(s) and substrate as the materials being measured. Exceptions are allowed if the changed situation does not affect those radiation properties that are used for calculating the thickness (and composition) reading(s).

Whenever possible the instrument shall be calibrated with standards. In a situation where standards are difficult to obtain, e.g., when either the coating or substrate materials are abnormal, it may be possible to use a standard-free calibration with a fundamental parameter-based computer simulation. Although any standard(s) used for calibration should have the same composition of coating(s) and substrate as the materials being measured, exceptions are possible if changed conditions do not affect those radiation properties that are used for calculating the thickness (and composition).

EXAMPLE

— To be measured: gold over stainless steel;

— For calibration used: gold over nickel;

— The intensity of the Au-L radiation is not influenced by the characteristic radiation of nickel or stainless steel. Using the emission method (see 3.5.1) and if the Au-L intensity is corrected for peak overlapping then the calibration with gold over nickel standards is possible.

One contribution to the uncertainty of the calibration curve results from the selected measurement time in the calibration procedure. Therefore a sufficiently long measurement time shall be selected so that the uncertainty of the count rate measurements on the standards is sufficiently small.

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), a calibration shall be made using an uncoated sample of the substrate material and a single coating standard of known thickness within the linear range. The user shall 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 the logarithmic range it is essential 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 the lower end of the logarithmic range of thickness

— one coated standard having a coating near the top end of the logarithmic thickness range.

6.1.4 Entire measurable range

To measure from zero into the hyperbolic range, additional coated standards shall be used to define more closely 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).

6.1.5 Standard-free techniques by fundamental parameter computer simulation

For standard-free techniques the simulation software has to model accurately the true physical properties of the sample.

The technique allows thickness and composition measurements to be obtained that would otherwise be difficult or impossible. However, the accuracy of such measurements would be improved by the use, if possible, of standards. Corrections to these measurements by the additional use of standard(s) follow the same procedure, and have the same limiting conditions of 6.1.1.

In cases where the specimen and the available standards do not meet the conditions of 6.1.1, the computer simulation based on the fundamental parameter technique will cover these situations if the following conditions are fulfilled:

a)   that the composition of the coating(s) of the standards and of the part to be measured do not differ considerably;

b)   as the characteristic radiation of substrate components influences the radiation intensities that are used to calculate the thickness and composition of the coating, that the composition of the standard's substrate and of the specimen do not differ considerably.

6.2   Standards

6.2.1   General

Use reliable reference standards to calibrate the apparatus. The final measurement uncertainty depends directly 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 that 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 on a particular substrate surface, it is essential to exercise care in order 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 has been 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).