
**Metallic and non-metallic coatings —
Measurement of thickness — Beta
backscatter method**

*Revêtements métalliques et non métalliques — Mesurage de l'épaisseur —
Méthode par rétrodiffusion des rayons beta*



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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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Foreword

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

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

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 3543 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Methods of inspection and coordination of test methods*.

This second edition cancels and replaces the first edition (ISO 3543:1981), which has been technically revised.

Annex A of this International Standard is for information only.

Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method

1 Scope

WARNING Beta backscatter instruments used for the measurement of coating thicknesses use a number of different radioactive sources. Although the activities of these sources are normally very low, they can present a hazard to health, if incorrectly handled. Therefore, reference should be made to current international and national standards, where these exist.

This International Standard specifies a method for the non-destructive measurement of coating thicknesses using beta backscatter gauges. It applies to both metallic and non-metallic coatings on both metallic and non-metallic substrates. To make use of this method, the atomic numbers or equivalent atomic numbers of the coating and the substrate need to differ by an appropriate amount.

NOTE Since the introduction of the X-ray fluorescence method (ISO 3497), the beta backscatter method has been used less and less for the measurement of coating thickness. However, because of its lower cost, it is still a very useful method of measurement for many applications. In addition it has a wider measuring range.

2 Terms and definitions

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

2.1

radioactive decay

spontaneous nuclear transformation in which particles or gamma radiation are emitted or X-radiation is emitted following orbital electron capture, or the nucleus undergoes spontaneous fission

[ISO 921:1997, definition 972]

2.2

beta particle

electron or positron which has been emitted by an atomic nucleus or neutron in a nuclear transformation

[ISO 921:1997, definition 81]

2.3

beta-emitting isotope

beta-emitting source

beta emitter

material, the nuclei of which emit beta particles

NOTE 1 It is possible to classify beta emitters by the maximum energy level of the particles that they release during their disintegration.

NOTE 2 Table A.1 lists some isotopes used with beta backscatter gauges.

2.4

electron-volt

unit of energy equal to the change in energy of an electron in passing through a potential difference of 1 V

NOTE 1 $1 \text{ eV} = 1,602\,19 \times 10^{-19} \text{ J}$

[ISO 921:1997, definition 393]

NOTE 2 Since the electron-volt is too small for the energies encountered with beta particles, the mega-electron-volt (MeV) is commonly used.

2.5

activity

disintegration rate

number of spontaneous nuclear disintegrations occurring in a given quantity of material during a suitably small interval of time divided by that interval of time

[ISO 921:1997, definition 23]

NOTE 1 In beta backscatter measurements a higher activity corresponds to a greater emission of beta particles.

NOTE 2 The SI unit of activity is the becquerel (Bq). The activity of a radioactive element used in beta backscatter gauges is generally expressed in microcuries (μCi) ($1 \mu\text{Ci} = 3,7 \times 10^4 \text{ Bq}$, which represents $3,7 \times 10^4$ disintegrations per second).

2.6

radioactive half-life

time required for the activity to decrease to half its value by a single radioactive decay process

[ISO 921:1997, definition 975]

2.7

scattering

process in which a change in direction or energy of an incident particle or incident radiation is caused by a collision with a particle or a system of particles

[ISO 921:1997, definition 1085]

2.8

backscatter

scattering as a result of which a particle leaves a body of matter from the same surface at which it entered

NOTE Radiations other than beta rays are emitted or backscattered by a coating and substrate and some of these can be included in the backscatter measurement. In this International Standard the term "backscatter" is used to mean the total radiation measured.

2.9

backscatter coefficient (of a body)

R

ratio of the number of particles backscattered to that entering the body

NOTE The value of R is independent of the activity of the isotope and of the measuring time.

2.10 backscatter count

2.10.1 absolute backscatter count

X
number of particles backscattered during a fixed interval of time, and received by a detector

NOTE X depends on the activity of the isotope, the measuring time, the geometric configuration of the measuring system and the properties of the detector. The count produced by the uncoated substrate is generally designated by X_0 , and that of the coating material by X_s . To obtain these values, both these materials have to be available with a thickness greater than the saturation thickness (see 2.13).

2.10.2 normalized backscatter count

X_n
quantity that is independent of the activity of the isotope, the measuring time and the properties of the detector and defined by the equation:

$$X_n = \frac{X - X_0}{X_s - X_0}$$

where

X_0 is the absolute backscatter count of the saturation thickness of the substrate material;

X_s is the absolute backscatter count of the saturation thickness of the coating material;

X is the absolute backscatter count of the coated specimen;

each of these counts being taken over the same interval of time

NOTE 1 The value of X_n is valid between 0 and 1.

NOTE 2 For simplicity, it is often advantageous to express the normalized backscatter count as a percentage by multiplying X_n by 100.

2.11 normalized backscatter curve

curve obtained by plotting the coating thickness as a function of X_n

2.12 equivalent (apparent) atomic number

for a material, which can be an alloy or a compound, the atomic number of an element that has the same backscatter coefficient R as the material

2.13 saturation thickness

minimum thickness of a material that, if exceeded, does not produce a change in backscatter

NOTE Figure A.1 shows saturation thickness, s , plotted as a function of density for different isotopes.

2.14

sealed source

radioactive source sealed in a container or having a bonded cover, the container or cover being strong enough to prevent contact with and dispersion of the radioactive material under the conditions of use and wear for which it was designed

[ISO 921:1997, definition 1094]

NOTE Also referred to as "sealed isotope".

2.15

aperture

opening of the mask abutting the test specimen and that determines the size of the area on which the coating thickness is to be measured

NOTE This mask is also often referred to as a platen, an aperture platen or a specimen support.

2.16

source geometry

spatial arrangement of the source, the aperture and the detector, with respect to each other

2.17

dead time

time period during which a Geiger-Müller detector is unresponsive to the receipt of further beta particles

2.18

resolving time

recovery time of the Geiger-Müller detector tube and associated electronic equipment during which the counting circuit is unresponsive to further pulses

2.19

basis material

basis metal

material upon which coatings are deposited or formed

[ISO 2080:1981, definition 134]

2.20

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

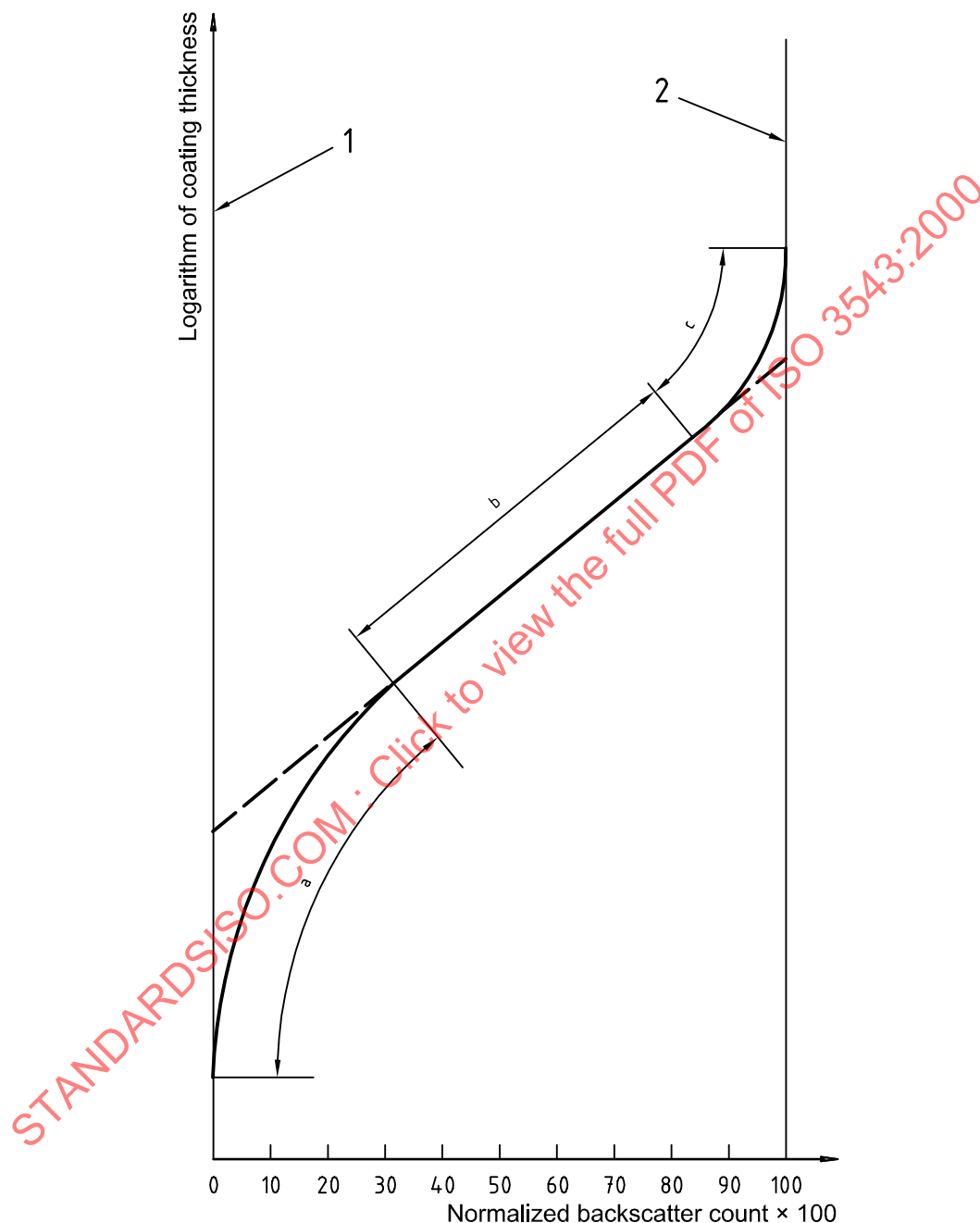
[ISO 2080:1981, definition 630]

3 Principle

When beta particles impinge upon a material, a certain portion of particles is backscattered. This backscatter is essentially a function of the atomic number of the material.

If the body has a surface coating, and if the atomic numbers of the substrate and of the coating material are sufficiently different, the intensity of the backscatter will be between two limits: the backscatter intensity of the substrate and that of the coating. Thus, with proper instrumentation and, if suitably displayed, the intensity of the backscatter can be used for the measurement of mass per unit area of the coating, which, provided that it is of uniform density, is directly proportional to the thickness, i.e., to the mean thickness within the measuring area.

The curve expressing coating thickness versus beta backscatter intensity is continuous and can be subdivided into three distinct regions as shown in Figure 1, on which the normalized count, X_n , is plotted on the x-axis, and the logarithm of the coating thickness on the y-axis. In the range $0 \leq X_n \leq 0,3$ the curve is essentially linear. In the range $0,3 \leq X_n \leq 0,8$ the curve is nearly logarithmic; this means that, when drawn on semi-logarithmic graph paper, as in Figure 1, the curve approximates a straight line. In the range $0,8 \leq X_n \leq 1$ the curve is nearly hyperbolic.



Key

- 1 Substrate with saturation thickness
- 2 Coating with saturation thickness
- a Approximately linear
- b Approximately logarithmic
- c Approximately hyperbolic

Figure 1 — Typical normalized backscatter curve

4 Apparatus

4.1 Beta backscatter gauge, comprising:

- a) a radiation source (isotope) emitting mainly beta particles having an energy appropriate to the coating thickness to be measured;
- b) a probe or measuring system with a range of apertures that limit the beta particles to the area of the test specimen on which the coating thickness is to be measured, and containing a detector capable of counting the number of backscattered particles, for example a Geiger-Müller counter (or tube);
- c) a readout instrument where the intensity of the backscatter is displayed;
- d) a readout instrument display, which can be in the form of a meter reading or a digital readout, either proportional to the absolute count or to the absolute normalized count or to the coating thickness expressed either in thickness units or in mass per unit area.

5 Factors relating to measurement uncertainty

5.1 Counting statistics

Radioactive decay takes place in a random manner. This means that, during a fixed time interval, the number of beta particles backscattered will not always be the same. This gives rise to statistical errors inherent in radiation counting. In consequence, an estimate of the counting rate based on a short counting interval (for example, 5 s) can be appreciably different from an estimate based on a longer counting period, particularly if the counting rate is low. To reduce the statistical error to an acceptable level, the counting interval has to be long enough to accumulate a sufficient number of counts.

For counts normally made, the standard deviation, σ , will closely approximate the square root of the absolute count, that is $\sigma = \sqrt{X}$; in 95 % of all cases, the true count will be within $X \pm 2 \sigma$. To judge the significance of the precision, it is often helpful to express the standard deviation as a percentage of the count, that is $100\sqrt{X} / X$, or $100/\sqrt{X}$. Thus, a count of 100 000 will give a value 10 times more precise than that obtained with a count of 1 000. Whenever possible, a counting interval shall be chosen that will provide a total count of at least 10 000, which would correspond to a standard deviation of 1 % arising from the random nature of radioactive decay.

Direct-reading instruments are also subject to these statistical random errors. However, if these instruments do not permit the display of the actual count rate, one way to determine the measuring precision is to make a large number of repetitive measurements at the same location on the same coated specimen, and to calculate the standard deviation by conventional means.

NOTE The precision of a thickness measurement by beta backscatter is always less than the precision described in 5.1, as it also depends on the other factors described in 5.2 to 5.17.

5.2 Coating and substrate materials

As the backscatter intensity of a measurement depends on the atomic numbers of the substrate and of the coating, the uncertainty of the measurement will depend to a large extent on the difference between these atomic numbers; thus, with the same measuring parameters, the greater this difference, the more accurate the measurement will be.

As a guide, for most applications, the difference in atomic numbers should be at least 5. For materials with atomic numbers below 20, this difference may be reduced to 25 % of the higher atomic number; for materials with atomic numbers higher than 50, this difference should be at least 10 % of the higher atomic number. Most unfilled plastics and related organic materials (for example photoresists) may be assumed to have an equivalent atomic number close to 6.

NOTE Table A.2 gives the atomic numbers of some typical coatings and substrate materials.

5.3 Aperture

Despite the collimated nature of the sources used in commercial backscatter gauges, the backscatter recorded by the detector is nearly always the sum of the backscatter produced by the test specimen exposed through the aperture and that of the specimen support. It is therefore, advantageous to use for the platen construction a material with a low atomic number, and to select the largest aperture possible. However, measuring errors will still occur if the edges of the aperture opening are worn or damaged, or if the test specimen does not properly contact these edges.

Because the measuring area on the test specimen has to be constant to prevent the introduction of another variable, namely the dimensions of the test specimen, the aperture shall be smaller than the area of the surface on which the measurement is made.

5.4 Coating thickness

5.4.1 In the logarithmic range, the “relative measuring error” is nearly constant, and has its smallest value.

5.4.2 In the linear range, the “absolute measuring error”, expressed in mass per unit area or thickness, is nearly constant, which means that as the coating thickness decreases, the relative measuring error increases. At, or near, $X_n = 0,3$, the relative errors of the linear and logarithmic ranges are about the same. This means that the relative error at this point can, for all practical purposes, be used to calculate the absolute error over the entire linear range.

5.4.3 In the hyperbolic range, the measuring error is always large, because a small variation in the intensity of the beta backscatter will produce a large variation in the measured value of coating thickness.

5.5 Resolving time of the detector

Because of the dead time (see 2.17) of the Geiger-Müller tube, the count indicated by the readout instrument is always less than the actual number of backscattered beta particles that would otherwise be counted. This does not diminish the measuring accuracy, unless the count rate is excessively high.

5.6 Source geometry

The greatest measuring accuracy is obtained with the source placed in a particular position with respect to the test specimen. This position depends on the collimation of the beam of beta particles from the source, and the location, form and size of the aperture. If possible, most of the backscattered radiation should be from the test specimen, and not from the platen. In general, the measuring uncertainty is reduced to a minimum when the isotope is mounted on the aperture platen, where it can be adjusted to an optimum position. The source shall be mounted in accordance with the manufacturer's instructions.

5.7 Curvature

This test method is sensitive to the curvature of the test specimen. However, the normalized backscatter curve remains the same if the surface of the test specimen does not protrude into the aperture of the platen by more than 50 μm or if the calibration is made using standards with the same curvature as the test specimen. By using specially selected aperture platens or masks, where the isotope is pre-mounted in a fixed optimum position, it is possible to obtain nearly identical readings on flat and curved specimens. This permits the use of flat calibration standards for the measurement of curved specimens.

In most cases, the relationship between maximum aperture size and specimen surface curvature is specific to the individual instrument design. These details shall, therefore, be obtained from manufacturer's data.

5.8 Substrate thickness

5.8.1 Test specimens without intermediate layers between the coating and the basis material

The test method is sensitive to the thickness of the substrate, but for each isotope and material there is a critical thickness, called "saturation thickness", beyond which the measurement will no longer be affected by an increase of the substrate thickness. This thickness depends on the energy of the isotope and on the density of material; it depends very little on the atomic number of the material. If values are not supplied by the manufacturer, they should be determined experimentally.

If the substrate thickness is less than the saturation thickness, but constant, substrate correction will, in general, yield accurate measurements. However, if the substrate thickness is less than the saturation thickness, and varies, this test method will not yield merely a single value for the coating thickness, but a range of values with an upper and lower limit. If the readout instrument is capable of displaying the absolute or normalized backscatter count rate, simplified graphs can be used to determine this range for each substrate thickness, without having actual standards. If they are not available from the manufacturer, this range has to be determined experimentally.

5.8.2 Test specimens with intermediate layers between the coating and the basis material

If the intermediate layer adjacent to the coating is thicker than the saturation thickness, the test method will not be affected by any variations in the substrate thickness, as long as the instrument is calibrated with standards having the intermediate coating as the basis material.

If the thickness of the intermediate layer is less than the saturation thickness, but constant, substrate correction will, in general, yield accurate measurements. However, if the thickness of the intermediate layer is less than the saturation thickness, and varies, this test method will not yield merely a single value for the coating thickness, but a range of values with an upper and lower limit. If the readout instrument is capable of displaying the absolute or normalized backscatter count rate, simplified graphs can be used to determine this range for each substrate thickness, without having actual standards. If they are not available from the manufacturer, this range has to be determined experimentally.

5.9 Surface cleanliness

Foreign material, such as dirt, grease and corrosion products, can produce erroneous readings. The natural oxide coatings that form on some metal coatings also tend to produce low readings, especially if the measurement requires the use of isotopes of which the beta emission has an energy of less than 0,25 MeV.

5.10 Substrate material

In order to obtain accurate thickness readings, the backscatter produced by the substrate materials of the test specimen and that produced by the calibration standard shall be the same. If they are different, other calibration standards shall be used, or appropriate corrections made, in accordance with the manufacturer's instructions.

5.11 Density of coating material

Essentially, the beta backscatter test method is a method for comparing the mass per unit area of the coating material on the test specimen with that of the coating on the standard. If these differ from each other, the thickness readout shall be corrected for the difference. This is done by multiplying the measured thickness by the coating density of the calibration standard and then dividing the product by the coating density of the test specimen. Porosity or voids in the coating material can also change the apparent density of the material.

5.12 Composition of coating

Because the composition of a coating affects the mass of coating per unit area, it will also affect the instrument response (amount of backscattered beta radiation). This effect can be negligible for alloying elements having densities close to each other such as cobalt-nickel alloys. Very small quantities of alloying elements, such as those present in high gold alloy deposits, also have little effect.

5.13 Energy of beta particles

Because the precision of the measurement, for a given isotope, is not constant over the entire range of measurement but is at a maximum in the logarithmic portion of the normalized beta backscatter curve (see Figure 1), the isotope should, whenever possible, be selected in such a manner that the expected measurement falls into the range $0,3 \leq X_n \leq 0,8$ of the normalized curve.

In general, instructions for selecting the proper isotope are provided by the manufacturer.

5.14 Measurement time

A measurement time that is too short will yield poor measurement precision. Selection of the measurement time will, therefore, depend on the desired measurement precision. Each time the measurement time is increased by a factor of n , the counting uncertainty is reduced by a factor of $\sqrt{n}$.

5.15 Activity of radioactive source

The count rate is dependent on the activity of the source. An old source can have a low activity, requiring excessive time to make a good measurement (see 5.1). As a practical guide, the source should be replaced before its half-life has elapsed.

5.16 Coating-substrate combination

The precision of measurement depends on the difference between the atomic number of the coating and that of the substrate materials. The greater this difference, the better the precision (see also 5.2).

5.17 Surface roughness

Measurement accuracy can be influenced by the roughness of the coating surface, but generally the effect is negligible, especially if the energy of the beta particles is high, and the atomic number of the coating is low.

6 Calibration of instruments

6.1 Frequency of calibration

Beta backscatter instruments shall be calibrated using standards before measurements are made and each time the measurement conditions are changed. During use, the calibration should be checked at least every 4 h, and at one calibration point, generally that of the bare substrate material, at least once per hour depending on the stability of the instrument. Attention shall be given to the factors listed in clause 5 and the procedures specified in clause 7.

6.2 Method of calibration

In addition to the zero point, the complete calibration curve can be defined either by two points of the logarithmic range, or by a single point, if the slope in the logarithmic range is known. In the first case, two calibration standards shall be used and in the second, only one.

6.3 Calibration standards

The instrument shall be calibrated with standards having a uniform coating thickness. Whenever possible, these standards shall have an accuracy of $\pm 5\%$, or better (see 8.2). The coating and substrate materials of the standard should have the same (or equivalent) atomic numbers as the substrate and coating materials of the test specimen. Standards corresponding to the bare substrate material and the coating material are also considered to be "calibration" standards. For calibration, it is sometimes also possible to use foils of the coating material, which are placed on, and in contact with, the substrate. When used, such foils shall be clean, smooth, uniform in thickness and in intimate contact with the substrate.

The substrate of the calibration standard shall have the same backscatter properties as that of the test specimen. This shall be verified by comparing the backscatter intensity of both uncoated substrate materials.

If coating materials have the same or equivalent atomic numbers, but different densities, the normalized backscatter curves will, for all practical purposes, be parallel. Under these circumstances, thickness measurements shall be corrected for the difference in densities (see also 5.11).

If "equivalent" calibration standards, i.e., standards made of different materials but having the same beta backscatter characteristics, are used to calibrate the instrument, their suitability shall be established prior to measurements being made.

6.4 Substrate thickness

The substrate thicknesses for the test specimen and the calibration shall be the same, unless the saturation thickness (see 5.8.1) is exceeded. If they are different, appropriate corrections shall be made.

6.5 Curvature

The curvature of the calibration standard and of the test specimen shall be the same, except if it can be demonstrated that the readings from a flat or curved specimen are, for all practical purposes, identical. If this is not possible, the readings shall be corrected.

7 Measuring procedure

7.1 Calibration and operation

Each instrument shall be operated in accordance with the manufacturer's instructions, with particular attention being paid to the factors listed in clause 5, and shall be calibrated in accordance with clause 6.

The calibration of the instrument shall be checked at the test site each time the instrument is used, and at frequent intervals, in accordance with 6.1, during use.

7.2 Precautions

7.2.1 Substrate thickness

The substrate thickness shall exceed the saturation thickness. If it does not, ensure that the calibration has been made with a substrate having the same thickness and properties as the test specimen, or correct the reading using the procedure given in 5.8.

7.2.2 Measuring aperture

The size of the measuring aperture depends on the size and shape of the test specimen. The manufacturer's recommendations concerning the choice of a measuring aperture shall be followed. In no case shall the measuring aperture be larger than the coated area available on the test specimen. The test specimen shall be seated firmly and securely against the measuring opening, except in the case of continuous measurements or measurements on large areas.

7.2.3 Curved specimens

Verify that the aperture used for the measurement is correct for the radius of curvature of the test specimen, and, if the calibration has not been made using standards having the same curvature as the test specimen, verify that the calibration is applicable to the measurement as follows.

Two test specimens shall be used, one being a curved specimen, the other being a flat specimen of the same material.

Place the flat specimen over and in intimate contact with the platen aperture. Record the count rate obtained from this specimen using the equipment, isotope and platen in question. Remove and replace this specimen several times, each time recording the count rate. Determine the mean value of the count rate and the associated standard deviation.

Replace the flat specimen by the specimen with the curved surface, and repeat the procedure used for the flat specimen. The mean count rate value obtained from the curved specimen should ideally remain within the limits established with the flat specimen, if the platen used for the test is ideally suitable. In practice, a small error, due to curvature, is acceptable if it is negligible in comparison with the error of the coating measurement (see 5.4).

7.2.4 Substrate material

The backscatter produced by the substrate of the standard shall be the same as that produced by the test specimen. This shall be verified by actual tests. In the case of a significant difference, the manufacturer's instructions regarding corrections shall be followed, or new standards that agree with the test specimen shall be used.

7.2.5 Surface cleanliness

All foreign materials, such as dirt, grease, lacquer, oxides and conversion coatings, shall be removed from the surface prior to the measurement, by cleaning it without removing any coating material. Specimen areas having visible defects, such as flux, acid spots, etc., shall be avoided when taking measurements.

7.2.6 Measuring time

The measuring time used shall be such that repeatability of readings and the desired precision are obtained.

7.2.7 Continuous measurements

The material being measured, the material feed mechanism and the measuring head being used shall together provide conditions that lie within the acceptance limits laid down in accordance with the manufacturer's recommendations.

8 Measurement uncertainty

8.1 Gauges capable of measuring coatings with a measurement uncertainty of a few percent are commercially available.

8.2 The equipment and its operation shall be such that the coating thickness can be determined to within 10 % of its true thickness.

9 Test report

The test report shall contain the following information.

- a) reference to this International Standard, i.e. ISO 3543;
- 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 platen aperture size;
- g) the measured values and the standard deviation for each as calculated from actual repetitive measurements, accompanied by a statement on the reliability and certification of the calibration standards used (see Note). The report of the thickness measurement shall be accompanied, wherever appropriate, by the following statements, or their equivalent (see 6.3):
 - 1) that the thickness of the coating has not been corrected for density;
 - 2) that the coating does not have the same composition as that of the calibration standards;
 - 3) that the substrate is not the same as that of the calibration standard, and (no) correction was made for the difference.
- h) the density used for thickness calculation and the justification used for that value;
- i) any deviations from this International Standard test method;
- j) any factors that might influence interpretation of the reported results;
- k) the name of the operator and that of the testing laboratory;
- l) the latest date of certification of the calibration or other acceptable reference standard(s) used and their traceability.

NOTE Simplified methods to determine the random errors of thickness measurements prior to an actual measurement are available from some manufacturers. If not available, the error can be determined by either of two methods: a) by computing the standard deviation from repetitive thickness determinations; b) by computing the standard deviation of the counting rate from repetitive counts, and calculating the equivalent deviation of the thickness.