International Standard 3543

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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 bêta

First edition — 1981-07-15

UDC 669.058 : 531.717 : 537.533.74 **Ref. No. ISO 3543-1981 (E)**

Descriptors : coatings, metal coatings, non metallic coatings, dimensional measurement, thickness, non destructive tests, beta backscatter method.

Price based on 9 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3543 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in May 1978.

It has been approved by the member bodies of the following countries:

Australia	India	Sweden
Czechoslovakia	Israel	Switzerland
Egypt, Arab Rep. of	Italy	United Kingdom
France	Japan	USA
Germany, F.R.	Mexico	USSR
Hungary	South Africa, Rep. of	

No member body expressed disapproval of the document.

Printed in Switzerland

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

1 Scope and field of application

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 employ this method, the atomic numbers or equivalent atomic numbers of the coating and the substrate must differ by an appropriate amount.

CAUTION — Beta backscatter instruments used for the measurement of coating thicknesses employ 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, all rules and regulations of local or national authorities must be observed.

2 Definitions

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

2.1 radioactive decay : A spontaneous nuclear transformation in which particles or gamma radiation are emitted or X-radiation is emitted following orbital electron capture or in which the nucleus undergoes spontaneous fission.*

2.2 beta particle : An electron, of either positive or negative charge, which has been emitted by an atomic nucleus or neutron in a nuclear transformation.*

2.3 beta-emitting isotope; beta-emitting source; beta emitter : A material the nuclei of which emit beta particles.

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

2.4 electron-volt : A unit of energy equal to the change in energy of an electron in passing through a potential difference of 1 V. (1 eV = 1,602 10 × 10^{-19} J)*

Since this unit is too small for the energies encountered with beta particles, the mega-electronvolt (MeV) is commonly used.

2.5 activity : The 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.*

Therefore, in beta backscatter measurements, a higher activity corresponds to a greater emission of beta particles.

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 µCi = 3,7 × 10^4 Bq, which represents 3,7 × 10^4 disintegrations per second).

2.6 half-life, radioactive : For a single radioactive decay process, the time required for the activity to decrease to half its value by that process.*

2.7 scattering : A 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.*

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 may be included in the backscatter measurement. Whenever the term "backscatter" is used in this International Standard, it is to be assumed that reference is made to the total radiation measured.

* Definition taken from ISO 921, *Nuclear energy glossary.*

2.9 backscatter coefficient, R (of a body) : The ratio of the number of particles backscattered to that entering the body.

This number 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 : The number of particles backscattered during a fixed interval of time, and received by a detector.

X will, therefore, depend 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, it is necessary that both these materials are available with a thickness greater than the saturation thickness (see 2.13).

2.10.2 normalized backscatter count, x_n : A quantity which 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.

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 : The 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 which has the same backscatter coefficient R as the material.

2.13 saturation thickness : The minimum thickness of a material which produces a backscatter which is not changed when the thickness is increased. (See also annex C.)

2.14 sealed source : A 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.*

(Also called sealed isotope.)

2.15 aperture : The opening of the mask abutting the test specimen, which determines the size of the area on which the coating thickness is to be measured. (This mask is also often referred to as a platen, an aperture platen, or a specimen support.)

2.16 source geometry : The spatial arrangement of the source, the aperture, and the detector, with respect to each other.

2.17 dead time : The time period during which a Geiger-Müller tube is unresponsive to the receipt of further beta particles.

2.18 resolving time : The recovery time of the Geiger-Müller tubes and associated electronic equipment during which the counting circuit is unresponsive to further pulses.

3 Principle

When beta particles impinge upon a material, a certain portion of them 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, that is, 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 $0 \leqslant x_n \leqslant 0{,}35$ the curve is essentially linear. In the range $0{,}35 \leqslant x_n \leqslant 0{,}85$ 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 $0{,}85 \leqslant x_n \leqslant 1$ the curve is nearly hyperbolic.

* Definition taken from ISO 921, *Nuclear energy glossary*.

4 Instrumentation

In general, a beta backscatter gauge will comprise :

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. The display, which can be in the form of a meter reading or a digital readout, is 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 accuracy

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) may 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, it is necessary to use a counting interval 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 ten 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.

IMPORTANT NOTE — The precision of a thickness measurement by beta backscatter is always less than the precision described above, inasmuch as it also depends on other factors which are listed below.

5.2 Coating and substrate materials

As the backscatter intensity of a measurement depends on the atomic numbers of the substrate and the coating, the accuracy of the measurement will depend to a large degree 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 rule of thumb, for most applications, it can be stated that 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.

(Annex B gives atomic numbers of commonly used coating 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, $x_n = 0{,}35$, the relative errors of the linear and logarithmic ranges are about the same. This means that the relative error at this point may, 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 of the Geiger-Müller tube (see 2.17), 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 instructions furnished by the manufacturer for mounting the source shall be followed exactly.

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 about 50 µm or if the calibration is made using standards with the same curvature as the test specimen. By the use of 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.

The relationship between maximum aperture size and specimen surface curvature is peculiar, in most cases, to the individual instrument design. These details are, therefore, best 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 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 this intermediate layer is less than the saturation thickness, and varies, this test method will not yield 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, corrosion products, etc., can produce erroneous readings. Natural oxide coatings which 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, it is necessary that the backscatter produced by the substrate materials of the test specimen and that of the calibration standard 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

The beta backscatter test method is basically 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 must be corrected for this 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 may be negligible with 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 is not constant over the entire range of measurement for a given isotope, but is 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 measurements fall into the range $0{,}35 \leqslant x_n \leqslant 0{,}85$ of the normalized curve.

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

5.14 Measurement time

Too short a measurement time will yield a poor measurement precision. The selection of the measurement time will, therefore, depend on the measurement precision which is required. 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 may 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 numbers of the coating and 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 the effect is, generally, 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 with standards before measurements are made, and also each time the measurement conditions are changed. During use, this calibration should be checked at least every 4 h, and at one calibration point, generally that of the bare substrate material, at least once an 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 are required, 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. Sometimes, it is also possible to use, for calibration, foils of the coating material which are placed on, and in contact with, the substrate. When using such foils, it is necessary to ensure that they are clean, smooth, uniform in thickness, and in intimate contact with the substrate.

The substrate of the calibration standards 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, that is standards made of a different material but having the same beta backscatter characteristics, are used for the calibration of the instrument, their suitability shall be established prior to the measurements.

6.4 Substrate thickness

The substrate thickness for the test specimen and the calibration should be the same, unless the saturation thickness defined in 5.8.1 is exceeded. If they are different, appropriate corrections have to 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, paying attention to the factors listed in clause 5. It 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 put into service, and at frequent intervals, in accordance with 6.1, during use.

7.2 Precautions

The following precautions shall be observed.

7.2.1 Substrate thickness

The substrate thickness shall exceed the saturation thickness. If it does not, make sure that the calibration has been made with a substrate having the same thickness and properties as the test specimen, or correct the readings by the procedures mentioned 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 with standards having the same curvature as the test specimen, verify that the calibration is applicable to the measurement. This can be done in the following manner.

7.2.3.1 Test specimens

Two test specimens are required, one being a curved specimen, the other being a flat specimen of the same material.

7.2.3.2 Procedure

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 this error is negligible when compared to 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 case of a significant difference, the manufacturer's instructions regarding corrections shall be followed, or new standards shall be used which agree with the test specimen.

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 in making measurements.

7.2.6 Measuring time

A sufficient measuring time to obtain a repeatability of readings that will yield the desired precision shall be used.

7.2.7 Continuous measurements

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

8 Measuring accuracy and precision

8.1 Gauges capable of measuring coatings with an accuracy 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

9.1 The report of the thickness measurements shall be accompanied by a statement on the reliability and certification of the calibration standards and of the standard deviation for each measurement, as calculated from actual repetitive measurements.

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.

9.2 The report of the thickness measurement shall be accompanied, wherever appropriate, by the following statements, or their equivalent (see 6.3) :

a) the thickness of the coating has not been corrected for density;

b) the coating does not have the same composition as that of the calibration standards;

c) the substrate is not the same as that of the calibration standard, and (no) correction was made for the difference.

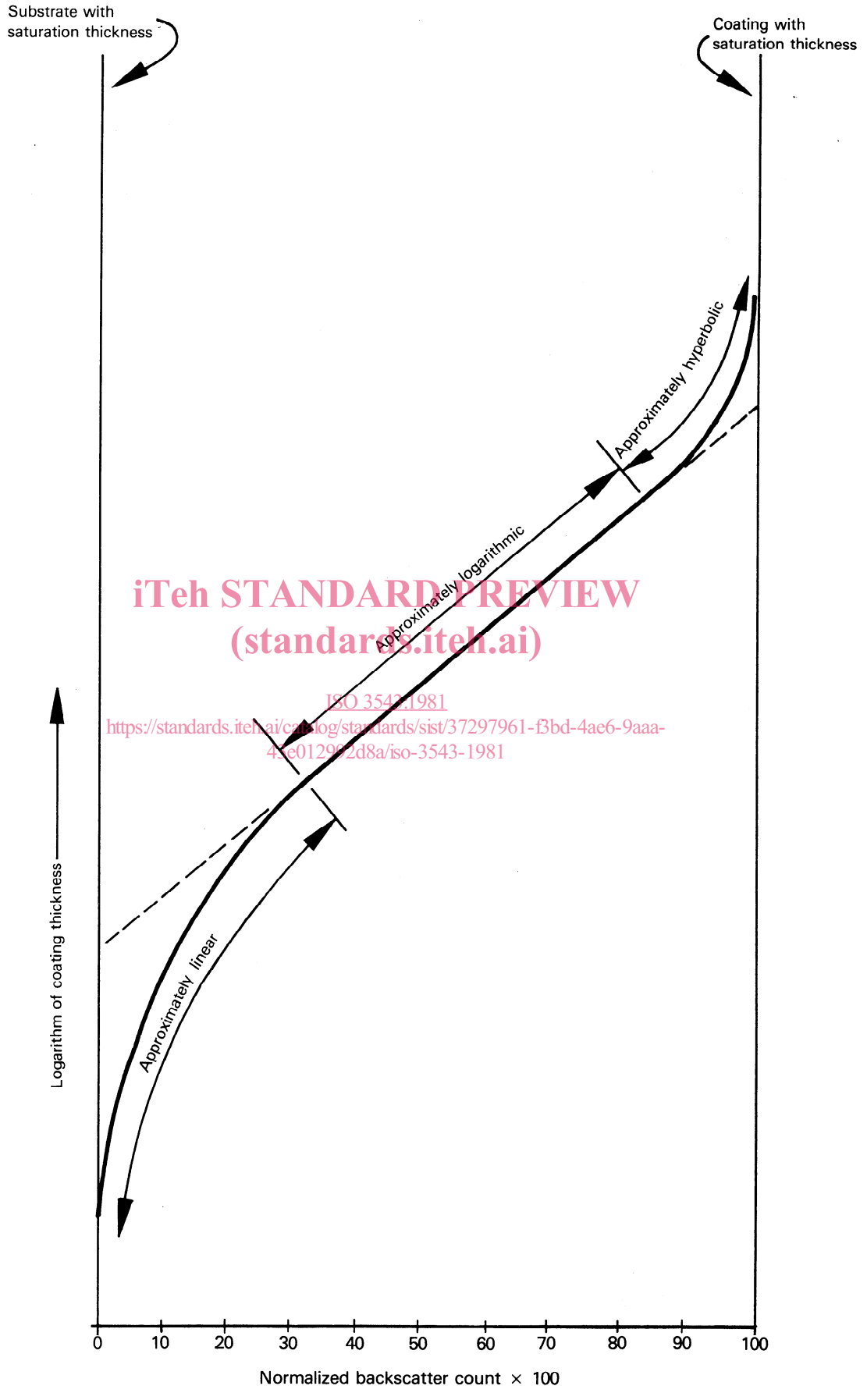

Figure 1 — Typical normalized backscatter curve