This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: B568 – 98 (Reapproved 2021)

Standard Test Method for Measurement of Coating Thickness by X-Ray Spectrometry[1]

This standard is issued under the fixed designation B568; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the use of X-ray spectrometry to determine thickness of metallic and some nonmetallic coatings.

1.2 The maximum measurable thickness for a given coating is that thickness beyond which the intensity of the characteristic secondary X radiation from the coating or the substrate is no longer sensitive to small changes in thickness.

1.3 This test method measures the mass of coating per unit area, which can also be expressed in units of linear thickness provided that the density of the coating is known.

1.4 Problems of personnel protection against radiation generated in an X-ray tube or emanating from a radioisotope source are not covered by this test method. For information on this important aspect, reference should be made to current documents of the National Committee on Radiation Protection and Measurement, Federal Register, Nuclear Regulatory Commission, National Institute of Standards and Technology (formerly the National Bureau of Standards), and to state and local codes if such exist.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

2.2 *International Standard:*

ISO 3497 Metallic Coatings—Measurement of Coating Thickness—X-ray Spectrometric Methods

3. Terminology

3.1 Definitions of technical terms used in this test method may be found in Terminology E135.

4. Summary of Test Method

4.1 *Excitation*—The measurement of the thickness of coatings by X-ray spectrometric methods is based on the combined interaction of the coating and substrate with incident radiation of sufficient energy to cause the emission of secondary radiations characteristic of the elements composing the coating and substrate. The exciting radiation may be generated by an X-ray tube or by certain radioisotopes.

4.1.1 *Excitation by an X-Ray Tube*—Suitable exciting radiation will be produced by an X-ray tube if sufficient potential is applied to the tube. This is on the order of 35 to 50 kV for most thickness-measurement applications. The chief advantage of X-ray tube excitation is the high intensity provided.

4.1.2 *Excitation by a Radioisotope* —Of the many available radioisotopes, only a few emit gamma radiations in the energy range suitable for coating-thickness measurement. Ideally, the exciting radiation is slightly more energetic (shorter in wavelength) than the desired characteristic X rays. The advantages of radioisotope excitation include more compact instrumentation essentially monochromatic radiation, and very low background intensity. The major disadvantage of radioisotope excitation is the much lower intensities available as compared

[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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with X-ray tube sources. X-ray tubes typically have intensities that are several orders of magnitude greater than radioisotope sources. Due to the low intensity of radioisotopes, they are unsuitable for measurements on small areas (less than 0.3 mm in diameter). Other disadvantages include the limited number of suitable radioisotopes, their rather short useful lifetimes, and the personnel protection problems associated with high-intensity radioactive sources.

4.2 *Dispersion*—The secondary radiation resulting from the exposure of an electroplated surface to X radiation usually contains many components in addition to those characteristic of the coating metal(s) and the substrate. It is necessary, therefore, to have a means of separating the desired components so that their intensities can be measured. This can be done either by diffraction (wavelength dispersion) or by electronic discrimination (energy dispersion).

4.2.1 *Wavelength Dispersion*—By means of a single-crystal spectrogoniometer, wavelengths characteristic of either the coating or the substrate may be selected for measurement. Published data in tabular form are available that relate spectrogoniometer settings to the characteristic emissions of elements for each of the commonly used analyzing crystals.

4.2.2 *Energy Dispersion*—X-ray quanta are usually specified in terms of their wavelengths, in angstroms (Å), or their equivalent energies in kiloelectron volts (keV). The relationship between these units is as follows:

$$(\text{keV})\left(\text{Å}\right) = 12.396$$

where:

keV = the quantum energy in thousands of electron volts, and
Å = the equivalent wavelength in angstroms (10^{-10} m).

In a suitable detector (see 4.3.2), X rays of different energies will produce output pulses of different amplitudes. After suitable amplification, these pulses can be sorted on the basis of their amplitudes and stored in certain designated channels of a multichannel analyzer, each adjacent channel representing an increment of energy. Typically, a channel may represent a span of 20 eV for a lithium-drifted silicon detector or 150 to 200 eV for a proportional counter. From six to sixty adjacent channels can be used to store the pulses representing a selected characteristic emission of one element, the number of channels depending on the width of the emission peak (usually displayed on the face of a cathode ray tube). The adjacent channels used to store the pulses from the material under analysis are called the "region of interest" or ROI.

4.3 *Detection:*

4.3.1 *Wavelength Dispersive Systems*—The intensity of a wavelength is measured by means of an appropriate radiation detector in conjunction with electronic pulse-counting circuitry, that is, a scaler. With wavelength dispersive systems, the types of detectors commonly used as the gas-filled types and the scintillation detector coupled to a photomultiplier tube.

4.3.2 *Energy-Dispersive Systems*—For the highest energy resolution with energy dispersive systems, a solid-state device such as the lithium-drifted silicon detector must be used. This type of detector is maintained at a very low temperature in a liquid-nitrogen cryostat (77K). Acceptable energy resolution for most thickness measurement requirements can be realized with proportional counters, and these detectors are being used on most of the commercially available thickness gages based on X-ray spectrometry. In setting up a procedure for coating-thickness measurement using an energy-dispersive system, consideration should be given to the fact that the detector "sees" and must process not only those pulses of interest but also those emanating from the substrate and from supporting and masking materials in the excitation enclosure. Therefore, consideration should be given to restricting the radiation to the area of interest by masking or collimation at the radiation source. Similarly, the detector may also be masked so that it will see only that area of the specimen on which the coating thickness is to be determined.

4.4 *Basic Principle*—A relationship exists between coating thickness and secondary radiation intensity up to the limiting thickness mentioned in 1.2. Both of the techniques described below are based on the use of primary standards of known coating thicknesses which serve to correlate quantitatively the radiation intensity and thickness.

4.5 *Thickness Measurement by X-Ray Emission*—In this technique, the spectrogoniometer is positioned to record the intensity of a prominent wavelength characteristic of the coating metal or, in the case of an energy-dispersive system, the multichannel analyzer is set to accept the range of energies comprising the desired characteristic emission. The intensity of the coating's X-ray emission (coating ROI) will be at a minimum for a sample of the bare substrate where it will consist of that portion of the substrate fluorescence which may overlap the ROI of the coating and a contribution due to background radiation. This background radiation is due to the portion of the X-ray tube's output which is the same energy as the coating's X-ray emission. The sample will always scatter some of these X rays into the detector. If the characteristic emission energies of the coating and substrate are sufficiently different, the only contribution of the substrate will be due to background. For a thick sample of the solid coating metal or for an electroplated specimen having an "infinitely thick" coating, the intensity will have its maximum value for a given set of conditions. For a sample having a coating of less than "infinite" thickness, the intensity will have an intermediate value. The intensity of the emitted secondary X radiation depends, in general, upon the excitation energy, the atomic numbers of the coating and substrate, the area of the specimen exposed to the primary radiation, the power of the X-ray tube, and the thickness of the coating. If all of the other variables are fixed, the intensity of the characteristic secondary radiation is a function of the thickness or mass per unit area of the coating. The exact relationship between the measured intensity and the coating thickness must be established by the use of standards having the same coating and substrate compositions as the samples to be measured. The maximum thickness that can be measured by this method is somewhat less than what is, effectively, infinite thickness. This limiting thickness depends, in general, upon the energy of the characteristic X-ray and the density and absorption properties of the material under analysis. The typical relationship between a coating thickness and

the intensity of a characteristic emission from the coating metal is illustrated by the curve in the Appendix, Fig. X1.1.

4.6 *Thickness Measurements by X-Ray Absorption*—In this technique the spectrometer, in the case of a wavelength-dispersive system, is set to record the intensity of a selected emission characteristic of the basis metal. In an energy-dispersive system, the multichannel analyzer is set to accumulate the pulses comprising the same energy peak. The intensity will be a maximum for a sample of the uncoated basis metal and will decrease with increasing coating thickness. This is because both the exciting and secondary characteristic radiations undergo attenuation in passing through the coating. Depending upon the atomic number of the coating, when the coating thickness is increased to a certain value, the characteristic radiation from the substrate will disappear, although a certain amount of scattered radiation will still be detected. The measurement of a coating thickness by X-ray absorption is not applicable if an intermediate coating is present because of the indeterminate absorption effect of intermediate layer. The typical relationship between coating thickness and the intensity of a characteristic emission from the substrate is shown in the Appendix, see Fig. X1.2.

4.7 *Thickness and Composition Measurement by Simultaneous X-ray Emission and Absorption (Ratio Method)*—It is possible to combine the X-ray absorption and emission techniques when coating thicknesses and alloy composition are determined from the ratio of the respective intensities of substrate and coating materials. Measurements by this ratio method are largely independent of the distance between test specimen and detector.

4.8 *Multilayer Measurements*—Many products have multilayer coatings in which it is possible to measure each of the coating layers by using the multiple-energy-region capability of the multichannel analyzer of an energy-dispersive system. The measuring methods permit the simultaneous measurement of coating systems with up to three layers. Or the simultaneous measurement of thickness and compositions of layers with up to three components. Such measurements require unique data processing for each multilayer combination to separate the various characteristic emissions involved, to account for the absorption by intermediate layers, and to allow for any secondary excitation which may occur between layers. Typical examples of such combinations are gold on nickel on copper and nickel on copper on steel.

4.9 *Mathematical Deconvolution*—When using a multichannel analyzer 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 (for example, characteristic radiation from Au and Br). This method sometimes is described as *numerical filtering* in order to distinguish from the technique of setting fixed Region of Interest (ROI) channel limits in the multichannel analyzer.

5. Significance and Use

5.1 This is a sensitive, noncontact, and nondestructive method for measuring the coating thickness (and in some cases, coating composition) of metallic and some nonmetallic coatings over a range of thicknesses from as little as 0.01 µm to as much as 75 µm depending on the coating and substrate materials. It can be used to measure coating and base combinations that are not readily measured by other techniques.

5.2 The coating thickness is an important factor in the performance of a coating in service.

6. Factors Affecting Accuracy

6.1 *Counting Statistics*—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 which is inherent in all radiation measurements. In consequence, an estimate of the counting rate based on a short counting interval (for example, 1 or 2 s) may be appreciably different from an estimate based on a longer counting period, particularly if the counting 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 being used it should be recognized that a significant portion of an intended counting period may be consumed as dead time, that is, time during which the count-rate capacity of the system is exceeded. It is possible to correct for dead-time losses. The manufacturer's instructions for accomplishing this with his particular instrumentation should be followed.

6.1.1 The standard deviation, s, of this random error will closely approximate the square root of the total count; that is, $s = \sqrt{N}$. The "true" count will lie within N ± 2 s 95 % of the time. To understand the significance of the precision, it is helpful to express the standard deviation as a percent of the count, $100\sqrt{N}/N = 100/\sqrt{N}$. Thus, 100 000 would give a standard deviation indicating 10 times the precision (one-tenth the standard deviation) obtained from 1000 counts. This is because $\left(100/\sqrt{1000}\right)/\left(100/\sqrt{100\,000}\right) = 10$. This does not mean that the result would necessarily be ten times as accurate (see 7.2).

6.1.2 A counting interval should be chosen that will provide a net count of at least 10 000. This would correspond to a statistical error in the count rate of 1 %. The corresponding standard deviation in the thickness measurement is a function of the slope of the calibration curve at the point of measurement. Most commercially available instruments display the standard deviation directly in units of thickness.

6.2 *Coating Thickness*—The precision of the measurement will be affected by the thickness range being measured. In the curve shown in the Appendix, see Fig. X1.1, the precision will be best in the portion of the curve from approximately 0.25 to 7.5 µm. The precision rapidly becomes poorer in the portion of the curve above approximately 10 µm. The situation is similar for the absorption curve shown in the Appendix, see Fig. X1.2. At coating thicknesses greater than approximately 10 µm, the intensity changes very little with the coating thickness and, therefore, the precision in that region is poor. These limiting thicknesses are, in general, different for each coating material.

6.3 *Size of Measuring Area*—To obtain satisfactory counting statistics (see 6.1) in a reasonably short counting period, the exposed area of the significant surface should be as large as practicably consistent with the size and shape of the specimen. Caution must be exercised, however, to see that the use of a large sample area in conjunction with high power input to the X-ray tube does not result in a signal so large as to exceed the count-rate capacity of the detection system.

6.4 *Coating Composition*—Thickness determinations by X-ray methods can be affected by the presence of foreign materials such as inclusions, co-deposited material, and alloying metals as well as by voids and porosity. The sources of error will be eliminated by the use of calibration standards electroplated in the same type of solution under the same conditions as those used in the production of the coatings to be measured. If pores or voids are present, X-ray methods will give an indication of coating mass per unit area but not of thickness.

6.5 *Density*—If the density of the coating materials differs from that of the calibration standards, there will be a corresponding error in the thickness measurement. Commercially available X-ray fluorescence instruments allow the use of a density correction procedure to compensate for small differences between the density of the coating material to be measured and the density of the calibration standards coating material. This procedure is commonly used for the measurement of hard gold coatings having a density of 17.5 g/cm^3 with calibration standards having a soft (pure) gold coating, which has a density of 19.3 g/cm^3. Variations in density can result either from variations in composition or from variations in plating conditions (see 6.4).

6.6 *Substrate Composition*—The effect of differences in substrate composition will be relatively minor on thickness measurements made by the X-ray emission method if an intensity ratio is used and if the X rays emitted by the substrate do not excite or overlap the radiation being measured. However, when thickness measurements are made by the X-ray absorption method the substrate composition of the test specimens must be the same as that of the standards.

6.7 *Substrate Thickness*—The effect of a thin substrate will be slight on thickness measurements by X-ray emission provided that an intensity ratio is used and if the X rays emitted by the substrate are not energetic enough to excite the radiation being measured. Care must be taken that the coating and substrate are thick enough to prevent the primary X-ray beam from reaching and fluorescing the material on which the sample is supported. However, when thickness is to be determined by the X-ray absorption technique, the thickness of the substrate must exceed a certain minimum or critical thickness. It must be established experimentally that the minimum thickness requirements have been met for a particular substrate-source combination, although it is sometimes possible to back up the test specimen substrates with a sufficient thickness of materials of the same composition. The X-ray absorption method cannot be used when one or more intermediate coating layers are present.

6.8 *Surface Cleanliness*—Foreign materials such as dirt, grease, or corrosion products will lead to inaccurate thickness determinations. Protective coatings such as lacquer or chromate conversion coatings over the coating to be measured will also affect the results.

6.9 *Specimen Curvature*—Thickness measurements should be made on flat surfaces if practical. In those cases where the measurement of thickness on curved surfaces cannot be avoided, a collimator should be used on the excitation beam, reducing the measurement area to a size that will minimize the effects of curvature. Spatial relationships between the curved surface, the excitation beam, and the detector are particularly important, and variations in these relationships can introduce errors in measurement. Calibration standards having the same radius of curvature as that of the test specimens can also be used to eliminate curvature effects.

6.10 *Excitation Energy*—The intensity of the characteristic secondary radiation from either the coating or the substrate is strongly affected by any variation in the excitation energy, that is, by changes in potential applied to the X-ray tube or changes in the tube current, or both. In general, the radiation intensity varies directly with the current and the square of the potential. Therefore, in any method based on a simple relationship between intensity and thickness, the final adjustment of excitation energy must be made with reference to the observed intensity from a standard sample used to construct the working curve. However, if the method is based on intensity ratios rather than absolute intensities, minor variations in excitation energy are compensated for.

6.11 *Detector*—Errors can be introduced by erratic operation of the detector system which includes the associated scaling circuitry as well as the detector tube itself. If instability is suspected, a series of twenty or more count measurements should be made on the same specimen without moving the specimen and the standard deviation of the series calculated. Most modern industrial X-ray instrumentation will perform this calculation automatically. The value should not be significantly greater than the square root of one measurement, $\sqrt{N}$. Some forms of instability become evident if the same specimen is measured periodically.

6.11.1 All radiation-detection/pulse-processing systems have limitations with respect to reliable count-rate capability. Operation of the gas-filled and scintillation types above their count-rate capabilities will result in loss of counts and erroneously low readings. Operation of an energy-dispersive system at high-input pulse rates will require an excessively long time to obtain a statistically valid total, even with "dead-time" compensation (see 6.1).

6.12 Any extrapolation beyond the thickness range covered by the calibration standards excluding infinite thickness can result in serious measurement errors; therefore, it is necessary to take additional steps for measurements outside this range.

6.12.1 When making measurements in the range between the highest thickness standard and the saturation (or infinite thickness) standard, especially in the so-called hyperbolic range, one must always use additional thickness standards with

values slightly above and below the presumed thickness of the test specimen. Instrument measurement precision will rapidly decrease with increasing thickness in the hyperbolic range. For this reason, significantly longer measurements times are usually required for measurement applications using the hyperbolic range.

6.12.2 The use of additional standards between the substrate standard and the lowest thickness standard will also improve the accuracy of the measurement in the lower range, which is also called the linear range.

6.13 *Filter to Absorb Secondary Radiation*—When measuring coating/substrate combinations having similar characteristic emission energies it is often helpful to use an absorber or filter made from an appropriate material to absorb the characteristic X-ray emission of the substrate or coating material to improve measurement accuracy and precision. In most commercially available XRF systems this absorber is a thin metal foil which is manually or automatically placed between the detector and the test specimen.

7. Calibration

7.1 *General*—In taking instrument readings for the purpose of establishing an instrument calibration, exactly the same instrumental conditions, including collimator size, voltage, and tube current, shall be used as those which will be used on test specimens.

7.2 *Standards*—Prolonged counting periods will not compensate for inaccurate standards. Standards representing various thickness ranges of a number of coatings on different substrates are generally available from thickness gage manufacturers. Those that are certified for thickness (as opposed to mass per unit area) are suitable provided they are used for coatings of the same density and composition. Calibration standards for gold coatings, certified as to mass of gold per unit area, with an accuracy of ±5 %, are available.[3] If standards representing a particular type of coating and substrate are not available, their preparation may be undertaken only if thoroughly competent personnel in the fields of electroplating and analytical chemistry are available.

7.2.1 Calibration standards must be used in such a manner as to minimize wear and abrasion of the plated surface. If the standards are visibly scratched or abraded they should be replaced. It is recommended that two sets of standards be maintained, that is, a set of primary standards and a set of working standards. These should be used only to calibrate and periodically check the condition of the working standards. At the first signs of wear or discrepancy, the working standards should be replaced.

7.3 The instrument shall be calibrated with thickness standards having the same coating and substrate materials as those being measured. Exceptions are allowed if the intensity of the characteristic coating material emission is not influenced by the characteristic emission of the substrate material. An example of this situation is the measurement of silver on copper. The instrument calibration may be made with standards of silver on nickel. The intensity of the characteristic silver emission is not influenced by the characteristic emission of nickel or copper.

7.4 The coating of the calibration standards must have the same X-ray emission (or absorption) properties as the coating being measured. If the coating of the standards is electrodeposited from the same bath and under the same conditions as the coating to be measured, the X-ray properties may be assumed to be the same. If the coating on the standard is gold, but not electroplated under conditions known to be the same as the coating being measured, the X-ray properties may be assumed to be the same for mass per unit area measurements. Under such circumstances, thickness measurements must be corrected for density differences, unless density differences can be shown to be insignificant.

7.5 If the thickness is to be determined by the X-ray absorption technique, the substrate of the thickness standards shall have the same X-ray emission properties as that of the test specimen. This shall be verified by comparing the intensities of the selected characteristic radiations of both uncoated substrate materials.

7.6 In the X-ray absorption technique, the substrate thickness of the test specimen and the calibration standards shall be the same unless the critical thickness, as defined in 6.7, is exceeded.

7.7 If the curvature of the coating to be measured is such as to preclude calibration on a flat surface, the curvature of the standard and that of the test specimen shall be the same.

8. Standard Less Techniques by Fundamental Parameter Computer Simulation

8.1 If the software of the XRF instrument is capable to model the true physical properties of the coating and basis material characteristic X-ray emission (by fundamental parameter based computer simulation) then a measurement of coating thickness and coating composition is obtained which is not derived from an instrument calibration with standards. This standard less measurement shall be corrected by means of calibration standards. The standards correction procedure (calibration) performs the same way as the procedure used for establishing empirical instrument calibrations alone.

8.2 In cases when the coating(s) to be measured and the available calibration standard do not meet the conditions of 7.3, then the computer simulation based on the fundamental parameter technique will cover such situations, if the following conditions are fulfilled:

8.2.1 The composition of the coating(s) of standard and part to be measured does not differ considerably, and

8.2.2 If the characteristic radiation of substrate components influences the radiation intensities which are used for calculating the thickness and composition of the coating, the composition of the substrate of standard and specimen shall not differ considerably.

9. Referee Test

9.1 If a referee test is required in order to resolve a disagreement, it shall be performed by using suitable Standard

[3] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

Reference Material (SRM)[4] thickness standards from the National Institute of Standards and Technology (NIST), if such standards are available. A suitable SRM standard is an SRM standard of the same substrate/coating combination for which the XRF gage was calibrated, the thickness of which is within the range of the calibration, preferably close to that of the test specimens being measured. The SRM shall be measured five times, each measurement being made under the same conditions as used for the measurement of the test piece. If the average of the five measurements of the SRM differs from the certified mass per unit area of equivalent thickness by more than 10 %, the calibration is not valid.

NOTE 1—SRMs are issued by NIST and include *coating thickness* SRMs for some coating systems. The stated mass per unit area of each *coating thickness* SRM is certified to be within 5 % of the true mass per unit area.

10. Procedure

10.1 Operate each instrument in accordance with the manufacturer's instructions, paying attention to the factors listed in Section 6. Calibrate it in accordance with Section 7.

10.2 *Calibration Checks*—The instrument calibration shall be checked periodically or before a test series, by remeasuring one of the calibration standards or a reference specimen with known mass per area or thickness. If there is a change of the measured thickness that is large enough to preclude meeting the requirements of Section 11, recalibrate the instrument.

10.3 Observe the following precautions:

10.3.1 *Substrate Thickness*—If the X-ray absorption technique is used, verify that the substrate thickness of the test specimens exceeds the critical thickness. If not, make sure that the calibration has been made with a substrate having the same thickness and emission properties as the test specimens.

10.3.2 *Measurement Area*—The size of the measurement area will depend on the size of the collimator used to restrict and control the size of the excitation beam. In no case shall the measurement area be larger than the coated area available on the test specimen. Suitable means must be provided to perfectly align the test specimen relative to the excitation beam.

10.3.3 *Surface Cleanliness*—Remove all foreign materials, such as dirt, grease, lacquer, oxides, and conversion coatings from the surface before the measurement by cleaning without removing any of the coating material. Avoid specimen areas having visible defects such as flux, acid spots, and dross in making measurements.

10.3.4 *Measuring Time*—Use a sufficient measuring time to obtain a repeatability that will yield the desired accuracy (see 6.1).

10.4 *Computation of Results*—Convert the intensity readings to thickness units. The conversion is made automatically by most commercial coating thickness instruments.

11. Precision and Bias

11.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10 % at 95 % confidence level.

11.2 Although an uncertainty of less than 10 % may be achieved consistently for a great number of applications, the uncertainty may be greater when the coating thickness is less than 1 µm.

11.3 Instruments suitable for compliance with 11.1 are available commercially. For many coating systems the instruments are capable of making measurements with an uncertainty of less than 5 % at 95 % confidence level.

11.4 The measurement bias is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than, and attributable to: (*1*) the calibration error of the instrument, and (*2*) the quality of the calibration standard used to calibrate the instrument.

12. Report

12.1 The report shall include the following information:

12.1.1 Type of instrument used,

12.1.2 Size of collimator aperture,

12.1.3 Measurement time,

12.1.4 Description of test specimen and measurement area,

12.1.5 If applicable a statement that a correction for density was made,

12.1.6 Type of calibration standards and the measurement mode used,

12.1.7 Thickness of coating as determined from the measurements,

12.1.8 Statistical measurement parameters of the measurement series as required,

12.1.9 Identification of testing facility and operator, and

12.1.10 Date of measurements.

13. Keywords

13.1 absorption; collimator; emission; filter; ratio; X-ray

[4] SRMs may be purchased from the Office of Standard Reference Materials, National Institute of Standards and Technology, Gaithersburg, MD 20899.

APPENDIX

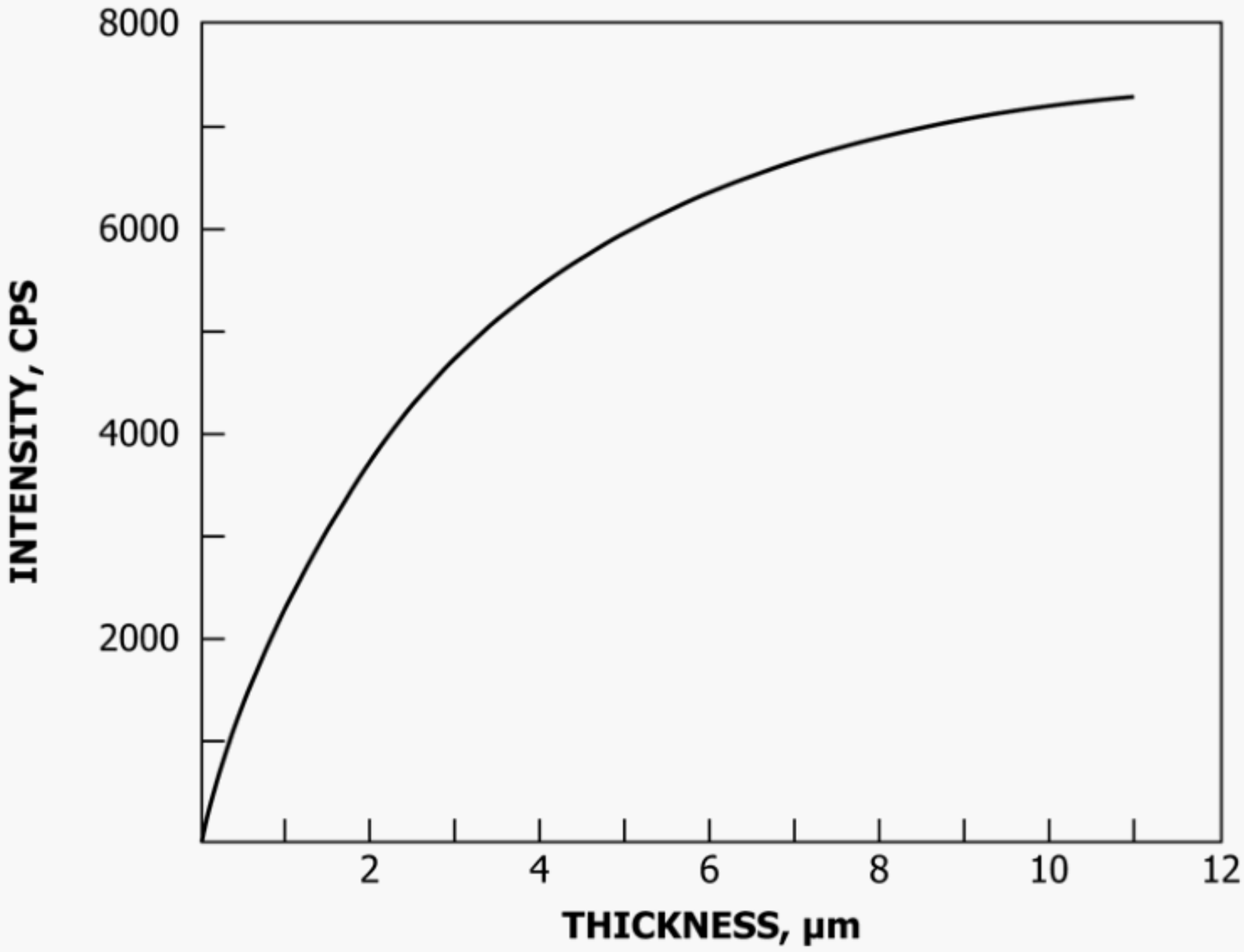
(Nonmandatory Information)

X1. CALIBRATION CURVES

X1.1 Since commercially available X-ray spectrometry thickness gages give a direct display in thickness units, it is not necessary for the user of such equipment to generate or to use calibration curves. However, the general shapes of calibration curves in the emission mode and in the absorption mode of operation are given here as general information.

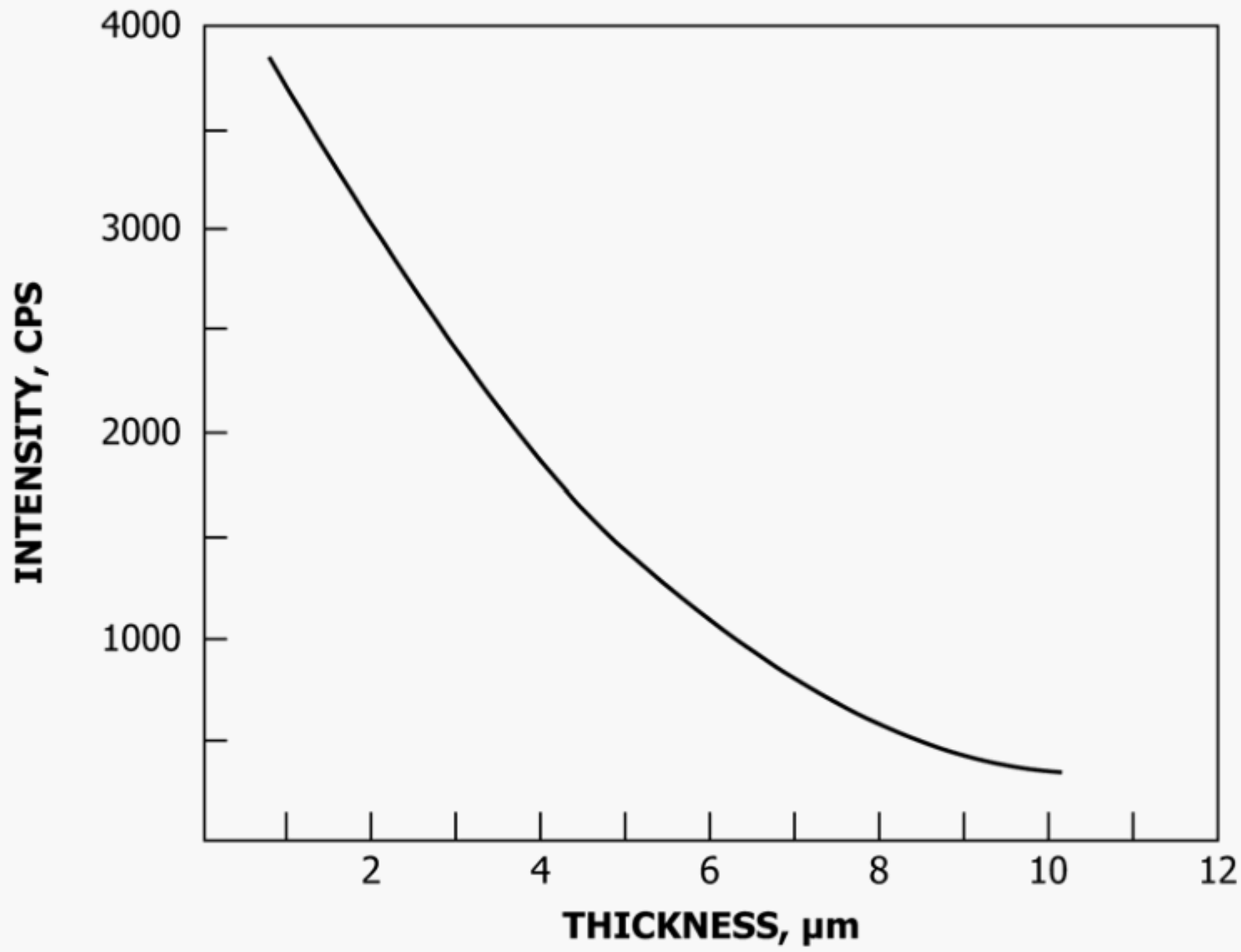
X1.1.1 *Emission*—A typical calibration curve for determining a nickel coating thickness by X-ray emission is shown in Fig. X1.1. The intensities are background-corrected, that is, the intensity for Ni Kα is obtained from a sample of the unplated brass substrate and subsequently substracted for each of the intensity readings obtained from electroplated samples.

X1.1.2 *Absorption*—A representative calibration curve for determining a nickel coating thickness by X-ray absorption is shown in Fig. X1.2. The intensities are background-corrected as they were in the emission technique. The emission now being measured comes from the substrate.



NOTE 1—Intensity is that of the Ni Kα line after a subtraction of background intensity.

FIG. X1.1 Calibration Curve for the Determination of a Nickel Thickness by X-Ray Emission



NOTE 1—Intensity is that of the Cu Kα line from the brass substrate after subtraction of background intensity.

FIG. X1.2 Calibration Curve for the Determination of a Nickel Thickness by X-Ray Absorption

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