Designation: A 754/A 754M – 06

Standard Test Method for Coating Weight (Mass) of Metallic Coatings on Steel by X-Ray Fluorescence[1]

This standard is issued under the fixed designation A 754/A 754M; 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.

1. Scope*

1.1 This test method covers the use of X-ray fluorescence (XRF) for determining the coating weight (mass) of metallic coatings on steel sheet. The test method is intended to be used for "on-line" measurements of coating on continuous production lines.

1.2 This test method is applicable to the coatings covered by the following ASTM specifications: A 599/A 599M, A 623, A 623M, A 653/A 653M, A 792/A 792M, A 875/A 875M, A 879/A 879M, A 918, and A 924/A 924M. It may be applicable to other coatings, providing that the elemental nature of the coating and substrate are compatible with the technical aspects of XRF such as the absorption coefficient of the system, primary radiation, fluorescent radiation, type of detection.

1.3 This test method includes the procedure for developing a single standard determination of coating weight (mass).

1.4 This test method includes procedures for both X-ray tube and isotope coating weight (mass) measuring instruments.

1.5 The values stated in either inch-pound units or SI units are to be regarded separately as standard. Within the text, the SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the specification.

1.6 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

A 599/A 599M Specification for Tin Mill Products, Electrolytic Tin-Coated, Cold-Rolled Sheet

A 623 Specification for Tin Mill Products, General Requirements

A 623M Specification for Tin Mill Products, General Requirements [Metric]

A 653/A 653M Specification for Steel Sheet, Zinc-Coated (Galvanized) or Zinc-Iron Alloy-Coated (Galvannealed) by the Hot-Dip Process

A 792/A 792M Specification for Steel Sheet, 55 % Aluminum-Zinc Alloy-Coated by the Hot-Dip Process

A 875/A 875M Specification for Steel Sheet, Zinc-5 % Aluminum Alloy-Coated by the Hot-Dip Process

A 879/A 879M Specification for Steel Sheet, Zinc Coated by the Electrolytic Process for Applications Requiring Designation of the Coating Mass on Each Surface

A 902 Terminology Relating to Metallic Coated Steel Products

A 918 Specification for Steel Sheet, Zinc-Nickel Alloy Coated by the Electrolytic Process for Applications Requiring Designation of the Coating Mass on Each Surface

A 924/A 924M Specification for General Requirements for Steel Sheet, Metallic-Coated by the Hot-Dip Process

3. Terminology

3.1 Definitions—For general definitions of terms relating to metallic-coated steel products, see Terminology A 902.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *averaging time*, *n*—the period over which an electronic measuring instrument acquires samples or "counts" prior to each update of coating weight (mass) output; refer to X1.2 for a more detailed explanation.

[1] This test method is under the jurisdiction of ASTM Committee A05 on Metallic-Coated Iron and Steel Products and is the direct responsibility of Subcommittee A05.07 on Methods of Testing.

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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.

***A Summary of Changes section appears at the end of this standard.**

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3.2.2 *response time*, *n*—the time required for a coating weight (mass) gauge to detect 90 % of a 10 % step change in coating weight (mass).

3.2.3 *sample*, *n*—the area of moving sheet that must be measured under standardized conditions to develop a single determination of coating weight (mass).

3.2.4 *standards*, *n*—the physical standards, either external or internal, that are used to calibrate the measuring instrument.

3.2.5 *substrate*, *n*—the steel sheet upon which the metallic coating is applied.

3.2.6 *time constant*, *n*—an electronic filtering term, unique to the design of each type of measuring instrument, that defines the time taken to respond to a step change in coating thickness; refer to X1.3 for a more detailed explanation.

3.2.7 *X-ray fluorescence*, *n*—the X-rays emitted by an atom when excited to a higher energy state.

4. Basic Principle

4.1 The measurement of coating thickness by XRF methods is based on the combined interaction of the coating and substrate, with an intense beam of primary radiation from an X-ray or isotope source. This interaction results in the generation of X-rays of well-defined energy. These fluorescent X-rays are detected by a radiation detector that can discriminate between selected energy levels in the secondary beam.

4.1.1 The radiation detector can discriminate between specific fluorescent X-rays because the X-rays generated by the interaction between the primary beam and the surface being fluoresced have energy levels that are unique to each element in the targeted material. Each element fluoresces at an energy that is characteristic of that element alone. Thus the fluoresced radiation can be detected separately for either the elements in a coating or the substrate material.

4.1.2 The detection system includes the radiation detector in conjunction with suitable electronic discriminating circuitry.

4.1.3 The thickness of a coating can be determined because a quantitative relationship exists between the intensity of the secondary radiation captured by the detector and the thickness of the coating material. The thickness of a sample can be established by comparing the measured intensity and that of a series of standards.

4.1.4 The coating weight (mass) can be calculated from the measured coating thickness for a specific coating type. In practice, the electronics are established to report the coating weight (mass) in commonly used units such as oz/ft^2 [g/m^2].

4.2 *Measurement Techniques*:

4.2.1 Two measurement techniques are used. The first technique involves direct measurement of the intensity of the fluorescent X-rays emitted by the coating itself. With this method, the coating weight (mass) is correlated with the intensity of the fluorescent X-rays emitted by the coating.

4.2.2 The second technique involves the measurement of the attenuation of the fluorescent X-rays emitted by the substrate as they pass through the coating whose weight (mass) is being determined. The correlation in this case is based on the principle that the intensity of the X-rays from the fluoresced substrate is a function of the weight (mass) of the coating for a specific coating type.

4.2.3 Appendix X2 and Appendix X3 contain a more detailed discussion of these two methods of measuring coating weight (mass).

5. Factors Affecting Accuracy

5.1 The equipment used to make a coating weight (mass) measurement using XRF typically consists of a radiation source, a detector, and an electronic system to process the detected signal. The sample absorbs radiation from the source and produces fluorescent radiation. The detector detects this radiation, and the electronic system converts it into coating weight (mass) information. Since an X-ray measurement is basically an accumulation of random events, the accumulation time must be long enough to produce statistically acceptable data. The precision of a coating weight (mass) measurement is determined by the equipment and the data collection time. Without a good calibration curve, however, highly precise equipment cannot produce an accurate result. For example, a very thick coating may produce a very precise X-ray fluorescent signal, but it may be outside the range of the equipment. Therefore, the measurement accuracy depends on the equipment, data collection time, and calibration of the instrument. The environment may also influence the measurement accuracy. Since equipment and coating each have unique characteristics, equipment specifications should be reviewed carefully prior to purchase and installation.

5.2 In order to measure coating weight (mass) accurately, the source must have enough strength to produce fluorescent radiation from the entire sample volume of interest. The sample volume of interest varies, depending on the XRF method used. When the coating weight (mass) is measured using fluorescence from the coating, the sample volume is the entire layer of the coating. When fluorescence from the substrate is used, the sample volume of interest is the lesser of the entire substrate or 5/μ (μ is the absorption coefficient of the substrate for the primary beam energy) thickness of the substrate under the coating. The radiated spot size must be large enough to cover a sample area as described in the procedure (refer to Table 1). The range of coating weight (mass) for which the measuring instrument can be used depends on the strength of the source and the coating composition. If a coating is thicker than 5/μ (μ of the coating for the fluorescent beam energy), XRF produced underneath the 5/μ

TABLE 1 Control Variables to Define a Single Data Point (Single Spot)

Variable	Value[A]	
Type of Gauge	X-ray Tube	Isotope
Area of fluorescence	1.5 to 5 in.^{2} [970 to 3200 mm^2]	5 to 14 in.^{2} [3200 to 9000 mm^2]
Traverse scan speed (traverse mode)	1 in./s [25 mm/s] min	1 in./s [25 mm/s] min
Dwell time (dwell mode)	4 s max	4 s max
Time constant (X-ray) or averaging time (isotope)	min 2.5 in. [65 mm] travel to allow 3 or more "time constants" to elapse	1 to 4 s

[A] Both X-ray tube and isotope coating weight gauges, when used for determining conformance to coating weight (mass) specifications, have diminished accuracy above 1.3 oz/ft^2/side [400 g/m^2/side] for zinc coatings, 0.066 oz/ft^2/side [20 g/m^2/side] for tin coatings, and 0.82 oz/ft^2/side [250 g/m^2/side] for aluminum coatings.

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thickness cannot emerge from the coating due to absorption. A coating thickness of 5/μ is defined as the critical thickness. If a coating is very thin, there may not be enough signal from the coating.

5.3 The detector must be able to discriminate between signals originating from the coating and the substrate. When the sample contains elements having similar atomic mass or similar X-ray characteristics, detected signals are difficult to discriminate and the measurement accuracy is affected adversely. The measurement accuracy may also be affected adversely when fluorescence from one element influences fluorescence from another. Equipment capable of measuring XRF from several elements simultaneously, including compensating for variations in coating composition, is required when the coating composition is unknown (for example, % Zn in Zn-Al or Zn-Ni coating), or the coating contains elements that are present in the substrate (for example, Zn-Fe coating on Fe), or the coating consists of multiple layers of metal or alloy.

5.4 The required data collection time is determined by the strength of the source, sensitivity of the detector, and coating weight (mass). A stronger source and a more sensitive detector typically require a shorter data collection time. The data collection time shall be long enough to achieve the required precision. For example, if N is the number of counts detected by a counter in a given time interval, the inherent error in radiation detection is equal to $\sqrt{N}$. As a guideline, the data collection time should be long enough to record 10 000 counts for a desired precision of ±1 %.

5.5 The calibration of the equipment has a very significant impact on the accuracy of the measurement. The coating composition of the material to be measured must be similar to that of the calibration standard. If the substrate has any influence on the X-ray signals, then both substrates must be similar. Significant differences in surface roughness and coating component segregation may also affect the accuracy of the measurement adversely. The coating weight (mass) range of the standards must exceed that of the material to be measured and must be within the useful range of the equipment.

5.6 Additional precautions are necessary for measurements made on-line or in a mill environment.

5.6.1 *Cleanliness*—The measurement instrument window must be kept clean to avoid any interference with the X-ray signal. A film of mill dust containing metal powder is normally more deleterious than that of oil and moisture.

5.6.2 *Stability*—The equipment should be maintained at a steady temperature to avoid any instability due to temperature. The influence of variations in air temperature in the gap between the instrument and the material on X-ray measurements must be compensated. The gap between the instrument and the sheet must be uniform and within the specifications of the equipment. Excessive variations in coating weight (mass) readings may be the result of variability in the strip pass-line due to such conditions as strip off-flatness (for example, wavy edges).

5.6.3 *Averaging Time*—During an on-line measurement, the equipment must be operated using an averaging time suitable for detecting variations in the coating weight (mass) without affecting measurement accuracy adversely. A very long averaging time will mask variations in the coating, resulting in a misleading indication of average coating weight (mass). A very short averaging time will yield unreliable results. (Refer to Table 1 for acceptable combinations.)

6. Calibration

6.1 *General*—When taking instrument readings for the purpose of establishing an instrument calibration, exactly the same instrumental conditions should be used as those that will be used on material being measured. The measuring time for calibration standards may be longer than that on material being measured in order to reduce the effect of statistical fluctuations.

6.2 *Standards*—Reliable standards must be used in the calibration of any type of X-ray equipment if accurate results are to be obtained. It should be understood that prolonged counting periods will not compensate for unreliable standards. Calibration standards that are certified for weight (mass) per unit area are reliable for coatings that have the same composition. The same density is not necessary for weight (mass) per unit area measurement. Calibration standards should be produced using the same material for coatings and substrates and the same coating technique as the material being measured. When correlating to standard weigh-strip-weigh techniques, great care must be exercised in selecting the sample because the coating is destroyed in the weigh-strip-weigh test procedure. Recommended sampling is to choose a uniform area approximately 9 by 9 in. [230 by 230 mm]. This can be measured by using an XRF instrument to find areas of uniform signal, from which five weigh-strip-weigh samples are cut in a cross-like pattern, wherein the center sample is in line with two other samples in the longitudinal direction and with two other samples in the cross-sheet direction. If chemical determinations of the coating weights (masses) of the four “satellite” samples agree to within 3 %, the center sample can be assumed to have a coating weight equal to the average of the four samples and can be considered a good calibration standard. If standards representing a particular type of coating and substrate are not available from any reliable source, their preparation may be undertaken, but only if trained personnel are available.

6.3 A minimum of three standard samples covering the range of coatings to be measured should be used for calibration. In general, more standards should be used than there are parameters in the calibration curve. Errors of interpolation may occur between calibration points because the calibration curve is nonlinear. These errors can be minimized by having many closely spaced calibration points. Extrapolation beyond the range of calibration points may also result in serious errors and should not be tolerated.

6.4 The instrument shall be calibrated with weight (mass) standards having the same coating and substrate materials as those being measured.

6.5 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 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 the same but not produced under conditions known to be the same as the coating being measured, the X-ray

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properties may be assumed to be the same for weight (mass) per unit area measurements, provided that the specimen properties discussed in 5.3 are verified to be the same for the standard and the specimen.

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

6.7 In the X-ray absorption technique, the substrate thickness of the specimen and the calibration standards should be the same unless the critical thickness, as defined in 5.2, is exceeded.

6.8 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 specimen shall be the same.

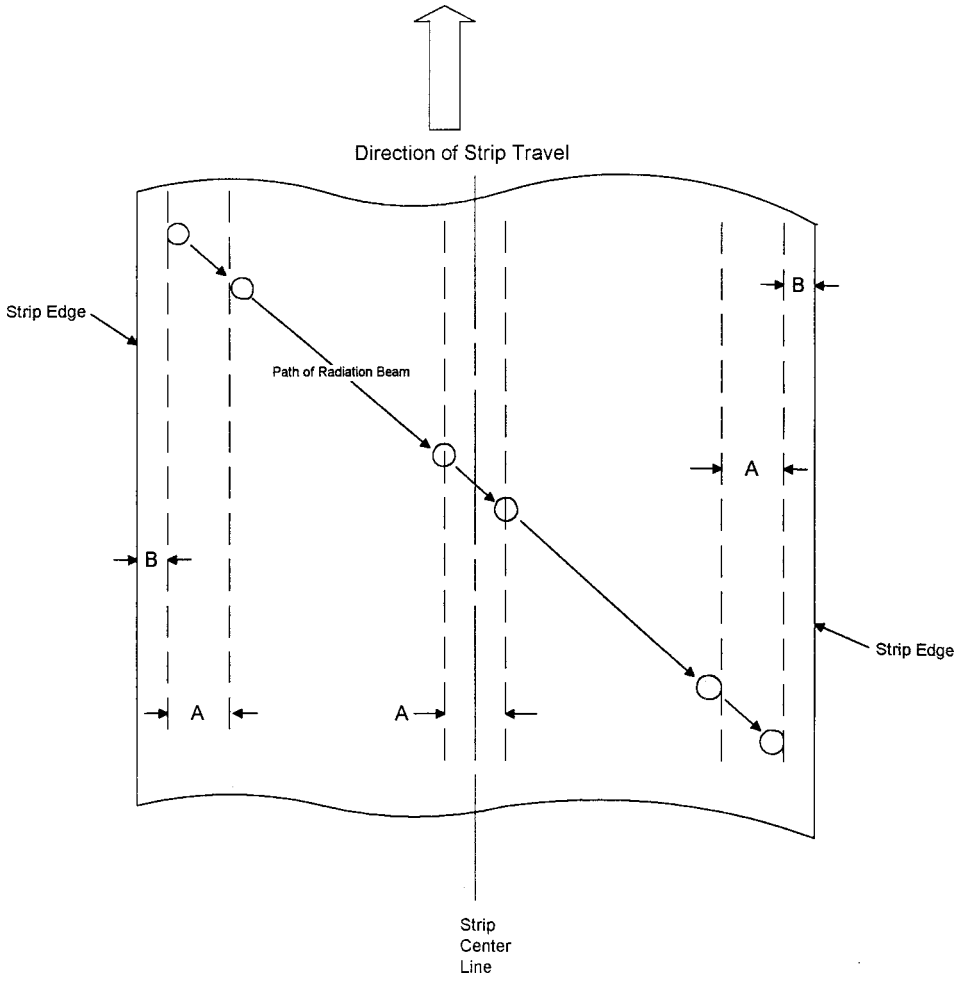
7. Procedure

7.1 Operate each instrument in accordance with the manufacturer's instructions, heeding the factors listed in Section 5. Calibrate the instrument in accordance with Section 6.

7.2 *Definition of a Single Data Point*— The instrument shall be operated in a manner that meets the requirements of Table 1.

7.2.1 *Area of Fluorescence*—The coating area fluoresced shall be between 1.5 and 5 in.^{2}[970 and 3200 mm^2] for X-ray tube gauges and 5 and 14 in.^{2}[3200 and 9000 mm^2] for isotope gauges.

7.2.2 *Traverse Scan Speed*—When the instrument is operated in a traversing mode over a moving strip, the scan speed shall be that speed which will result in less than a 1 % test error, measured over a maximum of 4 in. [100 mm] of strip width (see Fig. 1 for test locations across the strip width), based on a standard test condition. The standard test condition is illustrated in Fig. 2; namely, for a 10 % step in coating weight (mass), the coating weight (mass) value measured and assigned to the data point shall be the initial value plus a minimum of 90 % of the step value. Conformance to this measurement standard may be determined by calculations based on the time constant being used by the instrument or scan speed or by actual measurement. In no instance shall the traverse scan speed be less than 1 in./s [25 mm/s] nor the elapsed time to determine one data point be longer than 4 s.



NOTE—(A) Sampling width (2.5 to 4.0 in. [65 to 100 mm]); (B) distance from strip edge (2 in. [50 mm] ± 0.25 in. [6 mm]).

FIG. 1 Edge, Center, Edge Data Point Locations

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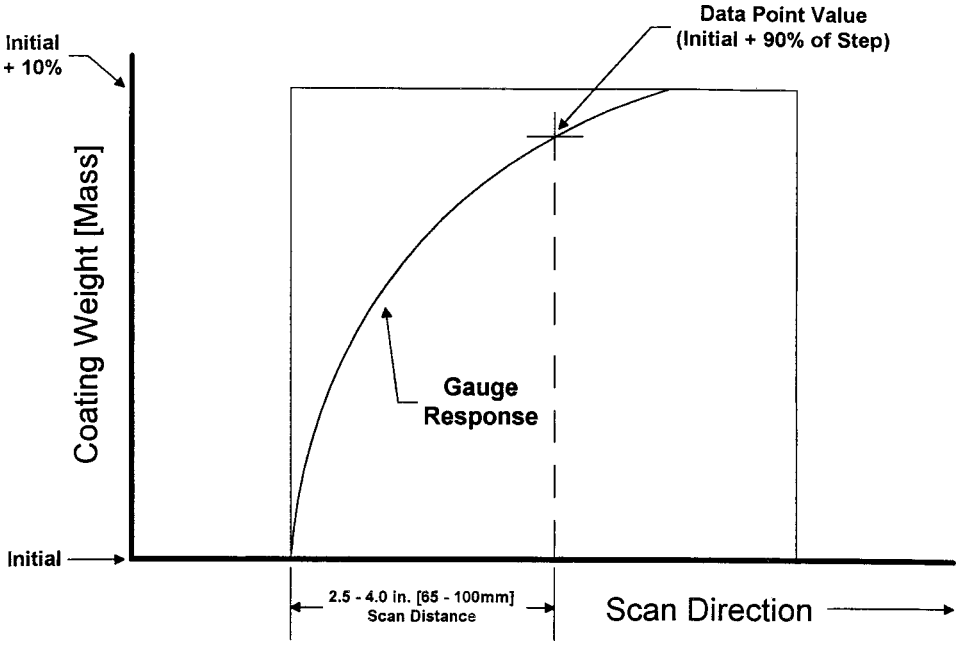

FIG. 2 Gauge Response for Standard Test Condition

7.2.3 *Dwell Time*—When the instrument is operated in the dwell mode, the requirements of the standard test condition of 7.2.2 shall apply and the maximum time used to determine one data point shall be 4 s.

7.3 *Coating Weight (Mass) Sampling Width and Location*—The instrument shall be operated in a manner that meets the requirements of Table 2. The triple spot technique for characterizing coating weight (mass) shall be used and may be acquired in either "scan" or "dwell" mode.

7.3.1 *Sampling Width*—For X-ray gauges, the width distance sampled at each of the edge locations by the radiation beam, to obtain a single data point, shall be between 2.5 and 4.0 in. [65 and 100 mm], measured from the outer edges of the beam. The width distance sampled at the center location shall be measured from the center of the beam. In the case of isotope gauges, no minimum sampling distance applies, only a maximum of 4 in. [100 mm], with the same sampling locations as for X-ray gauges. Refer to Fig. 1 and Table 2.

TABLE 2 Coating Weight (Mass) Sampling Width and Location Specifications

Variable	Value[A]	
Type of Gauge	X-ray Tube	Isotope
Sampling width at Edge Center Edge positions	2.5 to 4.0 in. [65 to 100 mm]	4.0 in. [100 mm] max
Edge readings—distance of beam outer edge from strip edge	2.0 ± 1/4 in. [50 ± 6 mm]	2.0 ± 1/4 in. [50 ± 6 mm]
Center readings—distance from center of strip width	±1 in. [±25 mm]	±1 in. [±25 mm]

[A] Both X-ray tube and isotope coating weight gauges, when used for determining conformance to coating weight (mass) specifications, have diminished accuracy above 1.3 oz/ft^2/side [400 g/m^2/side] for zinc coatings, 0.066 oz/ft^2/side [20 g/m^2/side] for tin coatings, and 0.82 oz/ft^2/side [250 g/m^2/side] for aluminum coatings.

7.3.2 *Edge Readings*—The outer 2 in. [50 mm] ± 0.25 in. [6 mm] of the strip edges, measured from the outer edge of the radiation beam, shall not be used for determining coating weight (mass).

7.3.3 *Center Reading*—The midpoint of the center reading shall be within 1 in. [25 mm] of the center of the strip width.

8. Precision and Bias

8.1 *Precision*—The precision of the procedure in this test method for "on-line" measurements of coating weight (mass) on steel sheet is being determined.

8.2 *Bias*—Since there is no accepted reference material for determining the bias for the procedure in this test method, bias has not been determined.

9. Keywords

9.1 aluminum zinc-coating; coating; coating weight (mass); coatings-metallic; metallic coated; sheet; steel sheet; tin mill products; X-ray fluorescence; zinc coating; zinc-5 % aluminum coating

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APPENDIXES

(Nonmandatory Information)

X1. BASIC PRINCIPLES

X1.1 *Calibration*—There are a number of design parameters unique to each machine and its particular installation or operation practice, or both, that require careful development of calibration curves to establish the correlation between the secondary X-rays being detected and coating weight (mass). Factors that need to be considered include, but are not limited to the following: the apparent absorption coefficient, which depends on the type of primary radiation and atomic numbers of the coating and substrate, as well as the physical arrangement of the radiation source and detection system; the uniformity of the distance between the specimen and radiation and detection sources; the sample curvature; the lower acceptable limit of the substrate; the effect of very localized coating nonuniformities; and all of the factors that influence the stability of the X-ray source and detection equipment.

X1.2 *Averaging Time*—Some coating weight (mass) measuring instruments acquire sampling information ("counts") over a defined period, or averaging time, prior to each update of their output. The "isotope" column of Table 1 establishes the limits when this type of output is used.

X1.3 *Time Constant*—Some coating weight (mass) measuring instruments process their output through an analog filter. The amount of filtering is traditionally quantified by a "time constant." One time constant is defined as 63 % of the final value when a step input is applied. Refer to Fig. X1.1. The output approaches 100 % of the input after several time constants. Paragraph 7.2.2 and the "X-ray" column of Table 1 establish the limits when this type of filtering is used.

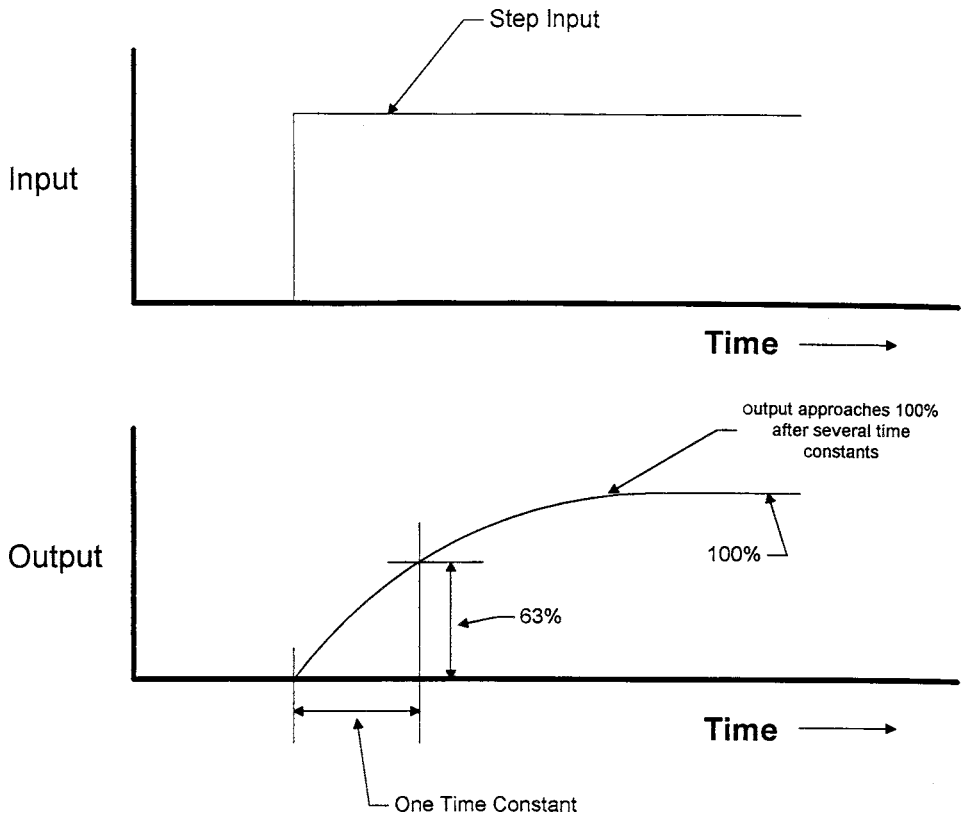

FIG. X1.1 Definition of Time Constant

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X2. WEIGHT (MASS) PER UNIT AREA MEASUREMENTS BY EMISSION OF FLUORESCENT X-RAYS OF COATING

X2.1 With this technique, the detection system is set up to count the number of X-rays in an energy region characteristic of X-rays from the coating element. Moreover, a specific, well-defined discrete energy level is usually selected from the various energy levels emitted by the coated element, such as the zinc Kα peak of a zinc (galvanized) coating.

X2.2 If the coating contains more than one element, the detection system is typically set up to count the number of X-rays of a specific energy level from the element that has the highest concentration in the coating. The count rate intensity of the discrete energy level being detected will be at a minimum for a sample of the bare substance, where it consists only of scattered (background) radiation. For a thick sample of the solid coating metal, or for a sample having an "infinite" coating weight (mass) per unit area, the intensity will have its maximum value for a given set of conditions. For a sample having a coating weight (mass) per unit area less than an infinite weight (mass) per unit area, the intensity will have an intermediate value between the above two end points.

X2.3 In general, the intensity of the emitted secondary X radiation depends on the excitation energy, atomic numbers of the coating and substrate, area of the specimen exposed to the primary radiation, and weight (mass) per unit area 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 weight (mass) per unit of the coating.

X2.4 The exact relationship between the measured intensity and corresponding coating weight (mass) per unit area must be established by the use of standards having coating and substrate compositions similar to those of the samples to be measured. In general, the limiting weight (mass) per unit area depends on the atomic number of the coating and the arrangement of the measuring apparatus.

X3. WEIGHT (MASS) PER UNIT AREA MEASUREMENTS BY ABSORPTION OF THE FLUORESCENT X-RAYS OF SUBSTRATE

X3.1 With this technique, the detection system is set up to record the intensity of a selected energy emitted by the substrate material. The intensity will be a maximum for a specimen of the uncoated substrate material and will decrease with increasing coating weight (mass) per unit area. This is because both the exiting and secondary characteristic radiations undergo attenuation in passing through the coating.

X3.2 Depending on the atomic number of the coating, when the coating (mass) per unit area is increased to a certain value, the characteristic radiation of the substrate will disappear, although a certain amount of background radiation will be detected. The measurement of a coating weight (mass) by X-ray absorption is not applicable if an intermediate coating is present because of the indeterminate absorption effect of the intermediate layer.

X3.3 As for measurement by the emission method, if all of the other variables are fixed, the intensity of the characteristic secondary radiation is a function of the weight (mass) per unit area of the coating.

X3.4 The exact relationship between the measured intensity and corresponding coating weight (mass) must be established by the use of standards having coating and substrate compositions similar to those of the samples to be measured.

SUMMARY OF CHANGES

Committee A05 has identified the location of selected changes to this standard since the last issue, A 754/A 754M - 96(2004), that may impact the use of this standard. (June 1, 2006)

(*1*) Deleted A 591/A 591M and revised A 879 to A 879/A 897M.

(*2*) Corrected a typographical error in Table 1.

(*3*) Corrected a typographical error in 5.4.

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