



Designation: D7639 – 10 (Reapproved 2018)

# Standard Test Method for Determination of Zirconium Treatment Weight or Thickness on Metal Substrates by X-Ray Fluorescence<sup>1</sup>

This standard is issued under the fixed designation D7639; 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 ( $\epsilon$ ) 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) spectrometry for the determination of the mass of zirconium (Zr) coating weight per unit area of metal substrates.

1.2 Coating treatments can also be expressed in units of linear thickness provided that the density of the coating is known, or provided that a calibration curve has been established for thickness determination using standards with treatment matching this of test specimens to be analyzed. For simplicity, the method will subsequently refer to the determination expressed as coating weight.

1.3 XRF is applicable for the determination of the coating weight as zirconium or total coating weight of a zirconium containing treatment, or both, on a variety of metal substrates.

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

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

1.7 *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.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.53 on Coil Coated Metal.

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## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

## 3. Summary of Test Method

3.1 The test specimen is placed in the X-ray beam, and the resultant peak intensity of the zirconium Ka line (at 0.0786 nm or 15.747 keV) or the zirconium La line (at 0.606 nm or 2.042 keV) is measured. The intensity (in counts or counts per second) is then compared to a previously prepared calibration curve or equation to obtain the coating weight of zirconium treatment in  $\text{mg}/\text{m}^2$  or  $\text{mg}/\text{ft}^2$  (or  $\mu\text{m}$  or  $\text{nm}$ ).

3.2 The exact relationship between the measured number of counts and the corresponding coating weight (or coating thickness) must be established for each individual combination of substrate and zirconium-containing treatment. Usually determined by the treatment supplier, this relationship is established by using primary standards having known amounts of the same treatment applied to the same substrate composition as the test specimens to be measured.

## 4. Significance and Use

4.1 The procedure described in this test method is designed to provide a method by which the coating weight of zirconium treatments on metal substrates may be determined.

4.2 This test method is applicable for determination of the total coating weight and the zirconium coating weight of a zirconium-containing treatment.

## 5. Apparatus

5.1 *X-Ray Fluorescence Spectrometer*, capable of measuring the intensity of zirconium Ka or La line, and establish the

<sup>2</sup> 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.

relationship between peak intensity and coating weight. The spectrometer's design must include, as a minimum, the following features:

5.1.1 *Source of X-Ray Excitation*, X-ray tube with excitation above 2.55 keV if measuring the zirconium La line, or above 18 keV if measuring the zirconium Ka line.

5.1.2 *X-Ray Detector*, with high sensitivity and capable of discriminating between zirconium La or Ka radiation and other X-rays of higher or lower energies.

5.1.2.1 In the case of wavelength dispersive X-ray fluorescence (WDXRF), this can be an analyzing crystal (for example, fixed channel, goniometer) setup to detect the zirconium X-rays (La or Ka line). Germanium 111 has been found to be acceptable for the Zirconium La line and LiF220 or LiF200 for the zirconium Ka line.

5.1.2.2 In the case of energy dispersive X-ray fluorescence (EDXRF), it can be a proportional counter, or a semiconductor such as a PIN diode or a silicon-drift detector.

5.1.3 *Pulse-Height Analyzer*, or other means of energy discrimination.

5.1.4 *Optical Path*, specified by manufacturer. A helium or vacuum path is recommended when measuring the zirconium La line in order to minimize the attenuation of the X-rays by the air in the optical path. The zirconium Ka line has a higher energy and its intensity will not be affected by air.

5.1.5 *Signal Conditioning and Data Handling System*, whereby a coating weight versus X-ray counts curve may be established within the system for the direct readout of coating weight.

5.1.6 *Sample Spinner (optional)*, to reduce the effects of coating weight variation across the test specimen.

## 6. Calibration Standards and Test Specimens

6.1 *Calibration Standards*—should be specimens for which the coating weight has been well characterized by other analytical procedures such as x-ray photoelectron spectroscopy, Auger emission spectroscopy, glow discharge optical emission spectrometry, weigh-strip-weigh method, or other depth-profiling analytical technique.

6.2 *Blank (bare and untreated) Specimen (optional)*, should be of the same metal substrate on which the treatment coating weight is to be determined. It may be necessary to prepare a blank specimen from a treated specimen if an untreated specimen is not available. To best imitate a bare, untreated blank, abrade a treated specimen that is from the same metal specimen as the test specimen using a small abrasive pad.

NOTE 1—The first abrading is made parallel with the rolling direction of the metal, the second abrading is made perpendicular to the rolling direction of the metal, and the third abrading is made parallel with the rolling direction of the metal. This procedure should be repeated until constant readings are obtained. Always use the same side of the metal substrate from which the readings of the treated specimen will be taken.

6.3 *Calibration Standards and Test Specimens*—shall be cut to the required size, if necessary, for measurement by the instrument.

6.4 All calibration standards and test specimens shall be flat in the area of measurement and free of burrs and distortions that would prevent proper seating in the analysis chamber or

the specimen holder, or proper seating of the handheld analyzer on the standard's surface.

6.5 The treatment on the substrate should be uniform in the area of measurement. If the coating weight might vary across the surface, it is recommended to analyze the test specimen in three different areas and use the average reading as the result.

6.6 The area of measurement should be maintained free of foreign materials. The test specimen shall be handled only by the edges that are outside of the area to be measured.

6.7 The coated area of the test specimen should be larger than the measured area.

6.8 The calibration standards and test specimens should be measured over the X-ray port using the same rolling direction of the metal. This is not necessary for instruments operating with a sample spinner.

## 7. Calibration Procedure

7.1 Set up the instrument calibration and operating parameters according to the chemical supplier and instrument manufacturer's recommendations.

7.2 Establish calibration curve by carefully determining the intensity of the emitted zirconium radiation from each of the calibration standards (a minimum of five standards is recommended). Obtain three readings for each standard (measured across the standard's surface if it is suspected that the zirconium coating weight might be varying).

7.3 Construct a calibration by using the software and algorithms supplied by the equipment manufacturer, establishing the relationship between zirconium intensity and zirconium treatment coating weight.

7.4 When using drift correction monitors, determine the intensity of the drift correction monitor sample(s).

7.5 Immediately after completing the calibration, determine the zirconium coating weight of one or more calibration check sample. Check samples can be stable, well-characterized materials. The differences between two measured values shall be within the repeatability of this test method. When this is not the case, the stability of the instrument and the repeatability of the sample preparation should be investigated and corrective measures taken. Calibration check samples should have the same substrate and same treatment as calibration standards and samples to be measured.

## 8. Preparation of Apparatus

8.1 *Instrument Setup*—Before using any XRF spectrometer, it is essential that the instrument is performing to the manufacturer's specifications. Consult with the manufacturer on how to perform spectrometer quality control checks.

## 9. Procedure

9.1 Following manufacturer's instructions, place the test specimen in the spectrometer for analysis or, in the case of handheld spectrometers, place the instrument's analysis head against the test specimen to measure. If the instrument does not operate with a sample spinner, ensure that test specimens are