Designation: D7639 – 10

StandardTest Method for Determination of Zirconium Treatment Weight or Thickness on Metal Substrates by X-Ray Fluorescence[1]

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 (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) 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 regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 mg/m^2 or mg/ft^2 (or μm or 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 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

[1] 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] 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.