Designation: D6906 – 12a (Reapproved 2016)

Standard Test Method for Determination of Titanium Treatment Weight on Metal Substrates by Wavelength Dispersive X-Ray Fluorescence[1]

This standard is issued under the fixed designation D6906; 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 wavelength dispersive X-ray fluorescence (WDXRF) techniques for determination of the coating weight of titanium treatments on metal substrates. These techniques are applicable for determination of the coating weight as titanium or total coating weight of a titanium containing treatment, or both, on a variety of metal substrates.

1.2 *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 Practice

3.1 *Excitation*—The measurement of titanium treatment coating weights by WDXRF methods is based on the combined interaction of the titanium coating and the substrate with an intense beam of primary radiation. Since each element fluoresces at an energy characteristic of the particular element, this interaction results in the generation of X-rays of defined energy. The primary radiation may be generated by an X-ray tube or derived from a radioisotope.

3.2 *Detection*—The secondary beam (fluorescent X-rays of the elements and scattered radiation) is read by a detector that can discriminate between the energy levels of fluorescing radiations in the secondary beam. The detection system includes the radiation detector with electronics for pulse amplification and pulse counting.

3.3 *Basic Principle:*

3.3.1 A relationship exists between the treatment coating weight and secondary radiation intensity. This relationship is usually linear within the desired coating weights of the titanium treatments on metal substrates. The measurements are based on primary standards of known coating weights and instrument calibration that correlates the secondary radiation intensity with the coating weight quantitatively.

3.3.2 The coating weight is determined by measurement of the fluorescent X-rays of the coating. The detection system is set to count the number of X-rays in an energy region that is characteristic of X-rays from the element of interest. The element of interest in this practice is titanium.

3.3.3 If a linear relationship exists, the coating weight and number of counts of X-rays of a titanium treatment on a particular substrate can be expressed by a conversion factor that represents the number of counts for a particular coating weight unit/unit area. This is usually expressed in mg/ft^2 or mg/m^2 of titanium or total coating weight.

3.3.4 The exact relationship between the measured number of counts and the corresponding coating weight must be established for each individual combination of substrate and titanium-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 specimens to be measured.

3.3.5 Some X-ray apparatuses have a 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. If such apparatus does not permit the entry of a conversion factor as described in 3.3.3, it is calibrated using a bare, untreated specimen and a minimum of three specimens with known coating weights of the treatment and substrate combination of interest. The coating weight to be measured must be within the range of these known coating weights. More than three known specimens must be used if the relationship of X-ray counts to coating weight is not linear over the range to

[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.

Current edition approved Dec. 1, 2016. Published December 2016. Originally approved in 2003. Last previous edition approved in 2012 as D6906 – 12a. DOI: 10.1520/D6906-12AR16.

[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.

be measured. The treatment supplier should be consulted for recommendations for establishing the curve in the instrument for the particular treatment and substrate combination of interest.

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 titanium treatments on metal substrates may be determined.

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

5. Apparatus and Materials

5.1 *Measuring Instrument,* which is capable of determining the coating weights of titanium-containing treatments on metal substrates by X-ray fluorescence is required. The treatment supplier should be consulted for the suitability of the instrumentation to be used

5.2 *Calibration Standard,* necessary to calibrate the instrument. The count value of this standard must be specified by the treatment supplier.

5.3 *Treated Coupon,* on which the coating weight is to be determined must be cut to the required size for the instrument from the treated substrate.

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

5.4.1 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 coupon will be taken.

6. Test Specimens

6.1 All test specimens must be flat in the area of measurement and free of burrs and distortions that would prevent proper seating in the specimen holder.

6.2 The treatment on the substrate must be uniform in the area of measurement.

6.3 The area of measurement must be maintained free of foreign materials. The specimen must be handled only by the edges that are outside of the area to be measured.

6.4 The coated area of the specimen must be larger than the measuring area.

7. Procedure

7.1 Operate the instrument in accordance with the manufacturer's instructions.

7.2 Set the instrument settings as follows:

Dial and arm	titanium position
Seconds indicator	pretreatment supplier
Multiplier switch	pretreatment supplier
Response switch	pretreatment supplier
Range	pretreatment supplier
Milliamps	adjust for calibration of output pretreatment supplier

7.3 All specimens must be seated firmly and securely over the measuring opening. The distance between the measuring apparatus and specimen must be maintained the same as that during the calibration. The blank and treated specimens must be placed in the holder so that the rolling direction of the metal is in the same orientation. Whenever a sample tray holder is a part of the apparatus, the same opening of the slide must be used for the blank and treated specimen unless the openings have been determined to produce equivalent results. If it is necessary to use a backer to hold the test specimen firmly against the window, make sure that the backer is of untreated coupons of the same metal as the specimen. The same backer must be used for each set of measurements.

7.4 Insert the titanium calibration standard that has been recommended by the treatment supplier into the instrument, and obtain a count. Adjust the current with the control knob on the probe until the count value is within a single significant figure rounded approximation of ± 3 times the square root of the counts provided by the treatment supplier with each titanium calibration standard.

7.5 Obtain the counts of a blank.

7.6 Obtain the counts of the treated specimen.

7.7 Consult the instrument manufacturer's instruction manuals for calibrating and operating procedures if the X-ray apparatus has a data handling system for direct readout of coating weights.

8. Calculation

8.1 Use 8.2 – 8.5 for calculating the coating weight if an automated data handling system is not available.

8.2 The average of a minimum of three readings of both the blank and treated specimen is used to calculate the coating weight.

8.3 Calculate the delta (Δ) counts by subtracting the counts of the blank from the counts of the treated specimen.

8.4 The coating weight is calculated by dividing the Δ counts by the conversion factor that is supplied by the treatment supplier for the particular substrate and treatment combination under study.

$$\text{Coating weight (weight/unit area)} = \frac{\Delta \text{ counts}}{\text{conversion factor}} \quad (1)$$

Other methods as recommended by the treatment supplier may be used to calculate the coating weight.

8.5 The conversion factors supplied by the treatment supplier are valid only for the instrument calibration procedure recommended by the treatment supplier.

TABLE 1 Net Integrated Ti Signal Intensity (Counts)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	s_r	s_R	r	R
Al Bare A	455.42	60.37	71.08	169.04	199.03
Al Bare B	678.76	55.69	71.97	155.94	201.53
Al Bare C	863.99	44.30	83.50	124.03	233.81
Galvalume Bare A	443.09	85.97	109.86	240.71	307.62
Galvalume Bare B	651.77	93.14	128.89	260.78	360.90
Galvalume Bare C	836.00	92.81	133.00	259.86	372.40
HDG Bare A	198.34	89.19	109.30	249.73	306.03
HDG Bare B	381.63	125.40	137.87	351.12	386.03
HDG Bare C	318.95	93.91	105.65	262.95	295.83

[A] The average of the laboratories' calculated averages.

9. Precision and Bias

9.1 The precision of this test method is based on an interlaboratory study of D6906, Standard Test Method for Determination of Titanium Treatment Weight on Metal Substrates by Wavelength Dispersive X-Ray Fluorescence conducted in 2011. A total of eleven laboratories tested samples prepared on three different representative coil industry metal substrates, each substrate having been coated with three different target coating weights (low, intermediate, and high) of either of two different Ti-containing commercial coil dry-in-place metal pretreatments. Each laboratory reported test results from triplicate samples made for each substrate/pretreatment/coating weight variation in this study. Every test result was the average of triplicate measurements made to determine the net (Sample – Blank) WDXRF integrated Ti signal intensities of a particular sample, presumed to be proportional to its respective pretreatment coating weight. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D01-1167.[3]

9.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same pretreatment/substrate combination at the same intended applied coating weight, obtained by the same operator using the same equipment on the same day in the same laboratory.

9.1.1.1 Repeatability limits are listed in Table 1.

9.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same pretreatment/substrate combination at the same intended applied coating weight, obtained by different operators using different equipment in different laboratories.

9.1.2.1 Reproducibility limits are listed in Table 1.

9.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

9.1.4 Any judgment in accordance with statements 9.1.1 and 9.1.2 would have an approximate 95 % probability of being correct.

9.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

9.3 The precision statement was determined through statistical examination of 295 results, from a total of eleven laboratories, on three substrates, with three applied pretreatment target coating weights. The coating weight targets were designated in the study as:

A: Low target coating weight
B: Intermediate target coating weight
C: High target coating weight

10. Keywords

10.1 coating weight; non-chrome; titanium; treatment; X-ray fluorescence

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1167. Contact ASTM Customer Service at service@astm.org.