Designation: D5723 – 95 (Reapproved 2015)

Standard Practice for Determination of Chromium Treatment Weight on Metal Substrates by X-Ray Fluorescence[1]

This standard is issued under the fixed designation D5723; 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 practice covers the use of X-ray fluorescence (XRF) techniques for determination of the coating weight of chromium treatments on metal substrates. These techniques are applicable for determination of the coating weight as chromium or total coating weight of a chromium-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. Summary of Practice

2.1 *Excitation*—The measurement of chromium treatment coating weights by XRF methods is based on the combined interaction of the chromium coating and 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 derive from a radioisotope.

2.2 *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.

2.3 *Basic Principle:*

2.3.1 A relationship exists between the treatment coating weight and secondary radiation intensity. This relationship is usually linear within the desired coating weights of the chromium 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.

2.3.2 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 chromium.

2.3.3 If a linear relationship exists, the coating weight and number of counts of X rays of a chromium 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 chromium or total coating weight.

2.3.4 The exact relationship between the measured number of counts and corresponding coating weight must be established for each individual combination of substrate and chromium-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.

2.3.5 Some X-ray apparatus 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 2.3.3, 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 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.

3. Significance and Use

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

[1] This practice 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 July 1, 2015. Published July 2015. Originally approved in 1995. Last previous edition approved in 2010 as D5723 – 95 (2010). DOI: 10.1520/D5723-95R15.

3.2 This procedure is applicable for determination of the total coating weight and the chromium coating weight of a chromium-containing treatment.

4. Apparatus and Materials

4.1 *Measuring Instrument,*[2] which is capable of determining the coating weights of chromium-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.

4.2 *Calibration Standard,*[3] necessary to calibrate the instrument. The count value of this standard must be specified by the treatment supplier.

4.3 *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.

4.4 *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 with a small abrasive pad.[4]

4.4.1 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.

5. Test Specimens

5.1 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.

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

5.3 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.

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

6. Procedure

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

6.2 Set the instrument settings as follows:

Dial and Arm	Chromium Position
Seconds indicator	per treatment supplier
Multiplier switch	per treatment supplier
Response switch	per treatment supplier
Range	per treatment supplier
Milliamps	adjust for calibration of output per treatment supplier

6.3 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.

6.4 Insert the chromium instrument 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 ±0.50 % of the counts provided by the treatment supplier with each chromium calibration standard.

6.5 Obtain the counts of a blank.

6.6 Obtain the counts of the treated specimen.

6.7 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.

7. Calculation

7.1 Use 7.2 – 7.5 for calculating the coating weight if an automated data handling system is not available.

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

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

7.4 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.

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

[2] A measuring instrument such as a Portaspec, a registered trademark of Cianflone Scientific Instruments Corp., or equivalent, available from Cianflone Scientific, 228 RIDC Park West Drive, Pittsburgh, PA 15275, has been found suitable for this purpose.

[3] The calibration standard may be a coupon of 316 stainless steel or other chromium standard provided by the treatment supplier, Standard Reference Material 1155 (AISI 316 stainless steel) from National Institute of Standards and Technology (NIST), Office Standard Reference Materials, Gaithersburg, MD 20899, or other chromium calibration standard as agreed upon between the purchaser and the seller.

[4] An abrasive pad such as Scotchbrite, a registered trademark of 3M company, No. 7447 General Purpose Hand Pad or No. 96 General Purpose Commercial Scouring Pad, available from 3M, St. Paul, Minnesota, or equivalent, has been found suitable for this purpose.

8. Keywords

8.1 chromium; coating weight; treatment; X-ray fluorescence