Designation: F3078 – 15 (Reapproved 2023)

Standard Test Method for Identification and Quantification of Lead in Paint and Similar Coating Materials using Energy Dispersive X-ray Fluorescence Spectrometry (EDXRF)[1]

This standard is issued under the fixed designation F3078; 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 describes an energy dispersive X-ray fluorescence (EDXRF) procedure for determining the areal mass of Pb in mass per unit area in paint and similar coatings on common substrates of toys and consumer products, such as plastic, wood, steel, aluminum, zinc alloys or fabric.

1.2 This test method is applicable for homogeneous, single layer paint or similar coatings. The method does not apply to metallic coatings.

1.3 This test method is applicable for a range of Pb mass per unit area from 0.36 μg/cm^2 to approximately 10 μg/cm^2 for Pb in paint and similar coatings applied on common substrates. The lower limit of this test method is between 0.36 and 0.75 μg/cm^2 depending on the nature of the substrate. Based on the results obtained during the interlaboratory study (ASTM Report F40-1004), it is estimated that the applicable range of this method can be extended up to 50 μg/cm^2.

1.4 The values stated in SI units are to be regarded as standard. Values given in parentheses are for information only.

1.5 *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.*

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D16 Terminology for Paint, Related Coatings, Materials, and Applications

D883 Terminology Relating to Plastics

D1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers

D6132 Test Method for Nondestructive Measurement of Dry Film Thickness of Applied Organic Coatings Using an Ultrasonic Coating Thickness Gage

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D7091 Practice for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to Ferrous Metals and Nonmagnetic, Nonconductive Coatings Applied to Non-Ferrous Metals

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

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

C693 Test Method for Density of Glass by Buoyancy

F2576 Terminology Relating to Declarable Substances in Materials

2.2 *Other Standards:*

Consumer Products Safety Improvement Act of 2008 (CPSIA), Public Law 110-314, August 14, 2008[3]

SSPC-PA2 Paint Application Standard No. 2, Measurement of Dry Coating Thickness with Magnetic Gauges[4]

[1] This test method is under the jurisdiction of ASTM Committee F40 on Declarable Substances in Materials and is the direct responsibility of Subcommittee F40.01 on Test Methods.

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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.

[3] Full text is available on the Consumer Products Safety Commission website: http://www.cpsc.gov//PageFiles/113865/cpsia.pdf.

[4] Available from Society for Protective Coatings (SSPC), 40 24th St., 6th Floor, Pittsburgh, PA 15222, http://www.sspc.org.

NIST Special Publication 829 Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories[5]

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions of terms applying to X-ray fluorescence (XRF) spectrometry, plastics and declarable substances appear in Terminology E135, Terminology D883 and Terminology F2576, respectively. Definitions of terms applying to Paint appear in Terminology D16.

3.1.2 *areal mass (or mass per unit area), n*—mass of substance (element) contained in a unit area of surface over which substance (element) is uniformly spread.

3.1.2.1 *Discussion*—This way of expressing the mass of a substance is typical and useful when material is present in a form of thin layer rather than bulk volume. The term is used not only in XRF analysis but also in a variety of coating industry applications. Areal mass is related to mass fraction through the thickness and density of the layer (see X1 for an example).

3.1.3 *Compton scatter, n*—the inelastic scattering of an X-ray photon through its interaction with the bound electrons of an atom; this process is also referred to as incoherent scatter.

3.1.4 *empirical method, n*—a method for calibration of X-ray fluorescence response of an analyzer using well characterized, representative samples (calibrants).

3.1.5 *fundamental parameters (FP) method, n*—a method for calibration of X-ray fluorescence response of an analyzer, which includes the correction of matrix effects based on the theory describing the physical processes of the interactions of X rays with matter.

3.1.6 *homogeneous coating, n*—the coatings such as paints or similar types are considered homogeneous for purposes of XRF analysis when their elemental composition is independent with respect to the measured location on the specimen and among separate specimens obtained from the same material.

3.1.7 *infinite thickness, n*—the thickness of a specimen above which no measurable count rate increase is observed for any analyte is referred to as 'infinite thickness'.

3.1.7.1 *Discussion*—Bulk materials with a matrix of low atomic number elements, such as polymers or wood, exhibit relatively low X-ray absorption. This leads to a requirement that for the best quantitative analysis the specimens must be thick, generally in excess of several millimeters, depending on the X-ray energies to be measured and the actual composition of the matrix. In general, more accurate and precise results can be obtained when the reference materials and the unknown samples are of infinite thickness or if thicknesses of the reference materials and unknown samples are at least within 10 % relative of each other. Typical substrates on which paint is applied may often be considered to be of infinite thickness for the purpose of XRF analysis.

3.1.8 *Rayleigh scatter, n*—the elastic scattering of an X-ray photon through its interaction with the bound electrons of an atom; this process is also referred to as coherent scatter.

3.1.8.1 *Discussion*—The measured count rate of Compton and Rayleigh scattered radiation varies depending upon specimen composition. The measured count rate of the Compton and Rayleigh scattered radiation or the ratio of Compton/Rayleigh scatter may be used to compensate for matrix effects specific to XRF analysis.

3.1.9 *screening, n*—screening is an analytical test procedure to determine the presence or absence of a substance (such as Pb) or compound in the representative part or section of a product, relative to the value or values accepted as the criterion for such decision.

3.1.9.1 *Discussion*—The value or values accepted as the criterion for decision shall be within the applicable range and above the limit of detection of the method. If the screening test produces values that are not conclusive, then additional analysis or other follow-up actions may be necessary to make a final presence/absence decision.

3.1.10 *thin sample, n*—applied paints and similar coatings represent a type of sample which is markedly different from a bulk sample of infinite thickness. The absorption and enhancement phenomena typical of XRF analysis of bulk materials are minimized by the fact that layer of paint is "thin". A layer of paint is considered "thin" for XRF purposes if it fulfills the following criterion[6]:

$$m \cdot \mu \leq 0.1 \quad (1)$$

where:

μ = a mass absorption coefficient of the sample for exciting radiation and characteristic X radiation of excited element in cm^2/g, and

m = mass per unit area of the sample (areal mass) in g/cm^2.

3.2 *Acronyms:*

3.2.1 *EDXRF*—energy dispersive X-ray fluorescence

3.2.2 *FP*—fundamental parameters

4. Summary of Test Method

4.1 An EDXRF analyzer that has been calibrated using either a fundamental parameters approach or an empirical approach is used to directly measure the areal mass of Pb in paint applied on any of the common substrates described in 1.1 by placing the painted surface of the object to be tested over the measuring aperture (window) of the analyzer and initiating the measurement. Alternatively, when using a handheld XRF analyzer, its measuring aperture (window) should be placed flush against the painted area of the object. The analyzer can be calibrated either by the manufacturer or by the user.

4.2 The test sample for this method should be a single, homogenous layer of dry, solid paint or similar coating applied over substrate material.

[5] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

[6] Rhodes J.R., Stout J.A., Schindler S.S. and Piorek S., "Portable X-ray Survey Meters for In-Situ Trace Element Monitoring of Air Particulates," in *Toxic Materials in The Atmosphere: Sampling and Analysis, ASTM STP 786*, ASTM International, 1981, pp. 70–82.