Designation: F2853 – 10ᵉ¹

Standard Test Method for
Determination of Lead in Paint Layers and Similar Coatings or in Substrates and Homogenous Materials by Energy Dispersive X-Ray Fluorescence Spectrometry Using Multiple Monochromatic Excitation Beams[1]

This standard is issued under the fixed designation F2853; 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.

ε¹ NOTE—Section 7.1.3 was changed editorially from "...≥ than 250 eV..." to correctly read "...equal to or better than 250 eV..." in July 2011.

1. Scope

1.1 This test method uses energy dispersive X-ray fluorescence (EDXRF) spectrometry for detection and quantification of lead (Pb) in paint layers, similar coatings, or substrates and homogenous materials. The following material types were tested in the interlaboratory study for this standard test method: ABS plastic, polyethylene, polypropylene, PVC, glass, zinc alloy, wood, and fabric.

1.2 This technique may also be commonly referred to as High Definition X-ray Fluorescence (HDXRF) or Multiple Monochromatic Beam EDXRF (MMB-EDXRF).

1.3 This test method is applicable for the products and materials described in 1.1 for a Pb mass fraction range of 14 to 1200 mg/kg for uncoated samples and 30 to 450 mg/kg for coated samples, as specified in Table 1 and determined by an interlaboratory study using representative samples

1.4 Ensure that the analysis area of the sample is visually uniform in appearance and at least as large as the X-ray excitation beam at the point of sample excitation.

1.5 For coating analysis, this test method is limited to paint and similar coatings. Metallic coatings are not covered by this test method.

1.6 *X-ray Nomenclature*—This standard names X-ray lines using the IUPAC convention with the Siegbahn convention in parentheses.

1.7 There are no known ISO equivalent methods to this standard.

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

1.9 *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]

D883 Terminology Relating to Plastics

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

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

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

F2576 Terminology Relating to Declarable Substances in Materials

3. Terminology

3.1 *Definitions*—Definitions of terms applying to XRF, plastics and declarable substances appear in Terminology D883, E135, and F2576.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Compton scattering*—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 scattering.

3.2.2 *fundamental parameters (FP) model*—a model for calibration of X-ray fluorescence response, including the correction of matrix effects, based on the theory describing the physical processes of the interactions of X-rays with matter.

TABLE 1 Mass Fraction Ranges for Various Sample Types

Sample Type	Homogenous Material or Substrate Type	Lead (Pb) Mass Fraction Range, mg/kg
Uncoated	Non-PVC Plastic, Glass or Ceramic	14–1200
Uncoated	Metal	66–600
Uncoated	PVC	376–1150
Paint Layer	Plastic or Metal	30–450
Paint Layer	Fabric	79–200
Paint Layer	Wood	58

3.2.3 *homogenous material*—materials are considered homogenous when the elemental composition as determined by the technique in this test method is independent with respect to the measured location on the specimen and among separate specimens prepared from the same material.

3.2.4 *low energy monochromatic beam*—a focused monochromatic beam having its selected photon energy between 3 and 9 keV.

3.2.5 *medium energy monochromatic beam*—a focused monochromatic beam having its selected photon energy between 15 and 23 keV.

3.2.6 *monochromatic beam*—an incident monochromatic beam on a sample having a selected photon energy with a narrow energy bandwidth relative to the selected energy. Method precision is achieved with a monochromatic beam having an energy bandwidth (Full Width Half Maximum) less than ±1.5 % relative to the selected energy and containing more than 98 % flux of the spectrum of the excitation beam which is incident on the sample.

3.2.7 *multiple monochromatic excitation beams*—two or more monochromatic beams.

3.2.8 *paint layer*—a single paint layer or other similar surface-coating material on a substrate.

3.2.9 *Rayleigh scattering*—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 scattering.

3.2.10 *substrate*—the material beneath a paint layer. The substrate may or may not be homogenous.

3.3 *Acronyms:*

3.3.1 *EDXRF*—energy dispersive X-ray fluorescence

3.3.2 *FP*—fundamental parameters

3.3.3 *HDXRF*—high definition X-ray fluorescence

3.3.4 *MMB*—multiple monochromatic beams

4. Summary of Test Method

4.1 The relevant samples include paint layers, their substrates, and homogenous materials.

4.2 This technique uses one or more monochromatic excitation beams to separately quantify the Pb mass fractions in paint layers and substrates, and homogenous materials. The area of the sample to be analyzed is placed against an X-ray

aperture. Depending on the data acquisition mode (see 13.1 and 13.2), one or more monochromatic X-ray beams are focused on the sample. The appropriate region of the fluorescence spectrum is processed by an FP method to obtain the analysis result, that is, the Pb mass fraction in the sample.

4.3 The apparatus is calibrated for each data acquisition mode. The calibration may be performed by the manufacturer or by the user.

5. Significance and Use

5.1 This test method may be used for quantitative determinations of Pb in painted and unpainted articles such as toys, children's products, and other consumer products. Typical test time for quantification of Pb in homogenous samples is 1 to 3 min; and typical test time for quantification of Pb in paint is 4 to 8 min.

6. Interferences

6.1 *Spectral Interference*—Spectral interferences result from spectral overlaps among the X-ray lines that remain unresolved due to the limited energy resolution of the detector. For instance, the arsenic (As) K-L$_{2,3}$ (K$\alpha_{1,2}$) peak directly overlaps the Pb L$_3$-M$_{4,5}$ (L$\alpha_{1,2}$) peak. The arsenic-Pb interference may be minimized by a de-convolution algorithm, but the precision of the Pb analysis may be affected. If the presence of arsenic is suspected, the user may further investigate the arsenic interference. Interactions of photons and electrons inside the detector result in additional peaks in the spectrum known as escape peaks and sum peaks. These peaks can overlap with X-ray lines of interest, for example, the sum peak of iron (Fe) K-L$_{2,3}$ (K$\alpha_{1,2}$) can overlap with the Pb L$_2$-M$_4$ (Lβ_1) peak.

6.2 *Substrate Interference*—The presence of Pb in a substrate can interfere with the determination of the Pb mass fraction of the paint layer. If the Pb signal of the paint layer and substrate composite is dominated by the contribution from the substrate, the uncertainty of the FP analysis can be significant and the Pb measurement for the paint layer will exhibit a positive bias and may not meet the precision statement of this test method. See Note 8 in Section 16.

6.3 *Matrix Effect*—Matrix effects, also called interelement effects, exist among all elements as the result of absorption of

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