



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 Beams¹

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.

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 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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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.

3.2.3 *homogenous material*—materials are considered homogenous when the elemental composition as determined by

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.

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

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 $\pm 1.5\%$ 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 L₃-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 L₂-M₄ ($L\beta_1$) 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 fluorescent X-rays (secondary X-rays) by atoms in the specimen. Absorption reduces the apparent sensitivity for the element. In contrast, the atom that absorbs the X-rays may in turn emit a fluorescent X-ray, increasing apparent sensitivity for the second element. Mathematical methods may be used to compensate for matrix effects. A number of mathematical correction procedures are commonly utilized including full FP treatments and mathematical models based on influence coefficient algorithms.

7. Apparatus

7.1 *EDXRF Spectrometer*³—designed for X-ray fluorescence analysis using multiple monochromatic excitation beams with an energy dispersive detector. Any EDXRF spectrometer may be used if it is capable of meeting method precision and its design incorporates the following features:

7.1.1 *Source of X-ray Excitation*—typically an X-ray tube, capable of exciting the Pb L lines in a sample. For instance, an X-ray tube with a zirconium, molybdenum, rhodium, palladium, or silver target can be used.

7.1.2 *X-ray Optics*—X-ray optical elements capable of accepting X-rays from a tube and directing monochromatic beams on the sample. Two or more X-ray optical elements are necessary to provide multiple monochromatic beams. At least one optical element provides a low energy monochromatic beam, and at least one optical element provides a medium energy monochromatic beam.

7.1.3 *X-Ray Detector*—with energy resolution equal to or better than 250 eV full width at half maximum of the manganese (Mn) K-L_{2,3} (K $\alpha_{1,2}$) line.

7.1.4 *Digital Pulse Processor and Multi-channel Analyzer*—a digital pulse processor for pulse shaping and conditioning, and a multi-channel analyzer for binning the pulses according to X-ray energy.

7.1.5 *Detector Aperture*—an aperture in the beam path between the sample and the detector to limit the field of view of the detector.

7.2 The following spectrometer features and accessories are optional:

7.2.1 *Beam Shutter*—used to select a monochromatic beam or select a combination of monochromatic beams.

7.2.2 *Drift Correction Monitors*—due to instability of the measurement system, the sensitivity and background of the spectrometer may drift with time. Drift correction monitors may be used to compensate for this drift. The optimum drift correction monitor samples are permanent materials that are stable with repeated exposure to X-rays.

7.3 *Discussion*—the data acquisition has two modes, one for homogenous materials and one for paint layers. The uncoated mode only requires one monochromatic beam for the excitation of the Pb L shell. The paint layer mode requires a second and lower energy monochromatic beam with less penetration of the sample to determine paint surface information and the Pb mass fraction in the paint layer.

8. Reagents and Materials

8.1 *Purity of Reagents*⁴—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

³ The sole source of supply of the apparatus known to the committee at this time is X-Ray Optical Systems, Inc., 15 Tech Valley Drive, East Greenbush, NY 12061. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annular Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeia Convention, Inc. (USPC), Rockville, MD.

all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents used include materials used for cleaning of samples.

8.2 Reagents:

8.2.1 Isopropanol or ethanol,

8.2.2 Nitric acid (HNO₃),

8.2.3 Hexane, and

8.2.4 Deionized water (H₂O).

8.3 *Gloves*—Disposable gloves are recommended for handling reference materials and other samples to minimize contamination.

8.4 Appropriate personal protective equipment for the handling of reagents.

8.5 *Uncoated Mode Calibration Standards*—At least two standards are required for calibration, one a scattering standard, and the other a Pb-containing homogenous material (see [Note 3](#)). The scattering standard shall have a known density, thickness, and composition. The other standard shall be a Pb containing homogenous standard with a known Pb mass fraction. Refer to manufacturer's recommendations.

NOTE 1—Better performance is expected if the Pb mass fraction of the Pb containing calibration standard is within the upper half of the scope range (see [Section 1](#)).

8.6 *Paint Layer Mode Calibration Standards*—A minimum of four standards are needed for calibration (see [Note 3](#)). Two standards shall be scattering standards, and the other two shall be Pb containing paint layer-on-substrate standards. Refer to manufacturer's recommendations.

8.6.1 *Scattering Standards*—At least two scattering standards are necessary due to the overlap of Compton and Rayleigh scattering of the low energy beam. One scattering standard shall have a known density, thickness, and composition. The other scattering standard shall be a thin paint layer, with a known mass per unit area, mounted on a thin polyester film. An example of the polyester film is film used for liquid cells having a thickness of 3.7 μm or similar.

8.6.2 *Paint Layer Standards*—At least two Pb-containing paint layer standards each with differing paint layer thicknesses and known Pb mass fraction are required.

NOTE 2—Better performance is expected if the Pb mass fraction of the two Pb containing paint layer standards are within the upper half of the scope range (see [Section 1](#)).

NOTE 3—Additional calibration standards may be used for improved accuracy.

8.7 Reference Materials:

8.7.1 Homogeneous reference materials are available from commercial sources. At the time of this publication, there are no commercially available paint layer reference materials.

8.8 Quality Control Samples:

8.8.1 To ensure the quality of the results, quality control (QC) samples are used for establishing and monitoring the stability and precision of an analytical measurement system