Designation: F2853 – 10 (Reapproved 2015)

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.

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.

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

[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.

Current edition approved Oct. 1, 2015. Published October 2015. Originally approved in 2010. Last previous edition approved in 2010 as F2853-10ε1. DOI:10.1520/F2853–10R15.

[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 ±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 ($L\beta_1$) 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 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 (HNO_3),

8.2.3 Hexane, and

8.2.4 Deionized water (H_2O).

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

[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.

(see Section 17). If possible, the QC samples shall be representative of samples typically analyzed. The materials shall be stable under the anticipated storage conditions.

9. Hazards

9.1 Occupational Health and Safety standards for X-rays and ionizing radiation shall be observed. Guidelines for safe operating procedures are also given in current handbooks and publications from original equipment manufacturers. For more information see similar handbooks on radiation safety.

9.2 **Warning—**Appropriate precautions are recommended when working with Pb and Pb compounds when creating and handling Pb reference materials and quality control samples.

10. Sample Preparation

10.1 *Uncoated Materials:*

10.1.1 From the sample to be tested, locate a section that is large enough to cover the measurement area of the spectrometer (see 1.4). The selected measurement area should have no obvious voids. The FP method accounts for variations in sample thickness. The minimum recommended sample thickness is 50 µm. When analyzing thinner layers of materials, it is acceptable to stack layers of materials of the same composition to generate the recommended thickness. Ensure no air is trapped between the layers.

10.1.2 If necessary, samples may be cleaned prior to measurement by rinsing with appropriate solvents (see Note 4). The following cleaning agents may be used:

10.1.2.1 Isopropanol or ethanol for removal of non-polar contaminants/hydrophobic contaminants (for example, grease),

10.1.2.2 A solution of 5 % HNO_3 in deionized water for removal of polar/hydrophilic contaminants (for example, salts and most mold release agents), and

10.1.2.3 Hexane for cleaning of polyamide and polyester samples.

10.2 *Paint Layers:*

10.2.1 Two areas of the sample must be identified for this measurement as follows: (*1*) Identify an area of the sample that is coated by the target paint layer. (*2*) Identify an area of the sample that is not coated by the target paint layer and is representative of the substrate under the target paint layer. If such a portion of the substrate is not available, a small portion of the target paint layer must be removed without scraping the sample too vigorously as the substrate integrity may be affected, particularly in thin substrates. The minimum recommended substrate thickness is 50 µm. When analyzing coatings on a thinner substrate, it is acceptable to stack layers of uncoated substrate of the same composition to generate the recommended substrate thickness. Ensure no air is trapped between the layers.

10.2.2 If necessary, samples may be cleaned prior to measurement by removing any noticeable foreign substances. Do not use strong solvents that may affect the paint layer. Acceptable reagents are listed in 10.1.2.

10.2.3 Care shall be taken to handle samples in such a way that oils and salts from the skin do not contaminate the portions of the sample that will be placed in the X-ray beam path of the spectrometer. The use of disposable gloves is recommended.

NOTE 4—Cleaning of reference materials may invalidate the certification. The user is cautioned to consult the certificate of analysis or contact the provider of the reference material for instructions.

11. Preparation of Apparatus

11.1 Allow the apparatus to stabilize for operation according to the manufacturer's guidelines.

11.2 Set the X-ray tube conditions for each monochromatic beam in accordance with the manufacturer's recommendations or requirements.

11.3 Set the correct detector parameters and calibrate the energy scale of the detector in accordance with the manufacturer's recommendations or requirements. The energy scale of the detector can be determined by using a sample containing a low energy fluorescent peak (for example, chlorine (Cl) K-$L_{2,3}$ ($K\alpha_{1,2}$)) and a high energy fluorescent peak (for example, tin (Sn) K-$L_{2,3}$ ($K\alpha_{1,2}$)).

11.4 Calculate a minimum measurement time resulting in a maximum counting statistical error (% CSE) of less than 5 % for a specimen containing approximately 100 mg/kg of the analyte. The required counting time may be calculated by using

$$\%\ CSE = 100/\sqrt{(Rt)} \quad (1)$$

where:

R = net count rate (in counts per second), and
t = counting time in seconds.

11.4.1 The product of R and t equals the area under the peak in EDXRF measurements. This time corresponds to a measuring time which results in collection of more than 400 counts (net). Overall measurement time shall not exceed 20 min per specimen.

11.5 Ensure that the spectral processing operates correctly according to the manufacturer's guidelines. In general, this involves removal of escape peaks, sum peaks, pulse pileup tails, and background. Ensure the correct fitting of Compton and Rayleigh scattering for low Z matrices. The manufacturer's software may have provisions for this.

12. Calibration and Standardization

12.1 An FP model is used to determine Pb mass fraction in paint layers, substrates, and homogenous materials. Compton and Rayleigh scattering are used in FP modeling and calculation. The FP method accounts for matrix effects, and paint layer and substrate interactions, based on the interactions between X-ray photons and matter.

12.2 *Uncoated Mode Calibration*—Prepare or obtain scattering standard(s) and Pb standard(s) for calibration (see 8.5). See Note 5.

12.2.1 Place the scattering standard in the X-ray beam path and select the uncoated mode. Measure the net counting rate of the Compton and Rayleigh scattering peaks. Determine the sensitivity factors for Compton and Rayleigh scattering.

12.2.2 In accordance with the manufacturer's instructions, place a Pb containing homogenous standard in the X-ray beam path and select the uncoated mode. Measure the net Pb counting rate. Repeat for additional calibration standards. The total net Pb counts for each calibration standard should be

>10 000 counts to minimize the counting error. Determine the calibration coefficient (sensitivity) for the FP model being used according to the manufacturer's instructions.

12.3 *Paint Layer Mode Calibration*—Prepare or obtain scattering standards for the low energy monochromatic beam and paint layer Pb standards (see 8.6). See Note 5.

12.3.1 Place the scattering standards, one at a time, in the X-ray beam path and select the paint layer mode. Measure the net counting rates of the Compton and Rayleigh scattering peaks. Determine the sensitivity factors for Compton and Rayleigh scattering according to the manufacturer's instructions.

12.3.2 Place the paint layer standards in the X-ray beam path and select the paint layer mode. Measure the net Pb counting rate. The total net Pb counts should be >1000 counts to minimize the counting uncertainty. Determine the calibration coefficient (sensitivity) for the FP model being used according to the manufacturer's instructions.

12.4 *Verification of Calibration*—Verify the calibration by analyzing one or more reference samples (see Note 2) immediately after completing an FP calibration.

12.4.1 When using a method calibrated by an outside source, verify the calibration by analyzing one or more reference materials. For each material, the confidence interval of the determined mass fraction must overlap the confidence interval of the reference value. See Note 6.

NOTE 5—The FP model may account for various types of substrates such as plastic, wood, fabric, glass, and metals. The use of calibration standards and reference materials of various matrices will improve accuracy of calibration and cover the variety of materials analyzed by this method.

NOTE 6—To further verify proper calibration, ensure that a calibration is performed by the manufacturer or the manufacturer's authorized representative, at least on an annual basis.

13. Procedure

13.1 *Measurement of Unknown Uncoated Sample:*

13.1.1 Prepare the sample according to Section 10, and prepare the instrument according to Section 11. Place the sample in the X-ray beam path and perform the measurement selecting the uncoated mode.

13.1.2 Process the spectrum using the same procedure chosen in Sections 11 and 12, including the same process for handling escape peaks, sum peaks, background modeling, and spectral overlaps.

13.2 *Measurement of Unknown Paint Layer Sample:*

13.2.1 Prepare the sample according to Section 10, and prepare the instrument according to Section 11. Place the paint layer sample in the X-ray beam path and perform the measurement selecting the paint layer mode.

13.2.2 Acquire spectra from the paint layer and substrate composite using the low energy and medium energy monochromatic beams or others.

13.2.3 Acquire spectra from the substrate only using low energy and medium energy monochromatic beams or others. This may require scraping the paint layer off as described in Section 10.

13.2.4 Process the spectrum using the same procedure chosen in Sections 11 and 12, including the same process for handling escape peaks, sum peaks, background modeling, and spectral overlaps.

13.3 *Quality Control Samples:*

13.3.1 When using quality control (QC) samples, measure them before measuring any unknowns (see Note 7).

13.3.2 Analysis of result(s) from these samples must be carried out following Practice D6299 or laboratory-specific control procedures (see Section 17). When the QC sample results exceed the laboratory's control limits, drift correction or instrument calibration may be required.

13.4 *Drift Correction:*

13.4.1 Drift is corrected by normalizing or standardizing the new measured count rates to make them comparable to count rates obtained at the time of calibration. Use the manufacturer's tools, if provided, or another suitable procedure.

NOTE 7—Verification of system control through the use of QC samples and control charting is highly recommended.

14. Interpretation of Results

14.1 Using the net count rates for a sample and the calibration from Section 12, the result is calculated by the instrument software in units of mg/kg or in mass per unit area Pb in units of mg/cm^2.

15. Report

15.1 Report Pb content of the test sample calculated from Section 14 using units of mg/kg, rounded to the nearest 0.1 mg/kg for mass fractions ≤100 mg/kg, and rounded to the nearest 1 mg/kg for mass fractions >100 mg/kg. Include a description of the sample type, and indicate that the test results were obtained using Test Method F2853.

16. Precision and Bias

16.1 The precision of this test method is based on an interlaboratory study (ILS) conducted in 2009.[5] Nine laboratories analyzed 13 homogenous and 21 paint-layer-on-substrate samples to determine method precision. Each of the analyzers used in this study had a 1 mm analysis spot size. The homogenous non-PVC plastic, glass and ceramic precision statement is based on one glass (sample ID#: S1-A), one non-PVC unknown plastic (S2-A), and four polyethylene (S1-B, S1-C, S2-B, S3-A), samples. The homogenous metal precision statement is based on four zinc alloy (S1-D, S2-C, S2-D, S2-E) samples. The homogenous PVC precision statement is based on two PVC (S3-B, S3-D) samples. The 12 to 100 mg/kg precision statement for coated plastic and metal is based on one ABS plastic (S1-H), one polypropylene (S3-G), five PVC (S1-J, S1-K, S2-J, S2-K, S3-E), and five zinc alloy (S1-E, S1-F, S2-F, S3-I, S3-J) samples; and the 100 to 450 mg/kg precision statement for coated plastic and metal is based on three PVC (S1-I, S1-K, S2-L), two ABS plastic (S2-I, S3-F), and two zinc alloy (S3-H, S3-K) samples. The precision

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F40-1001.

statement for coated fabric is based on two cotton fabric (S1-G, S2-H) samples, and the precision statement for coated wood is based on one pine (S2-G) sample. The analyzers used in this study were calibrated by the instrument manufacturer, and all participants in the ILS used validation samples supplied by the instrument manufacturer. Precision statements were generated according to Practice E691. Substrates for coated samples used in this ILS contained non-detectable amounts of Pb.

16.2 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operation conditions on identical test materials would, in the long run in the normal and correct operation of the test method, exceed the values in Table 2 and Table 3 only in one case in 20. See Note 8.

16.3 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test materials would, in the long run in the normal and correct operation of the test method exceed the values in Table 2 and Table 3 only in one case in 20. See Note 8.

16.4 *Bias*—No statistically significant bias was observed for this test method using NIST SRM 612; CRMs ERM-EC 680K and ERM-EC 681K from European Reference Materials; CRMs PE-High, PVC-Low, and PVC-High from Modern Analytical Techniques; CRMs 41X ZSC1, 41X ZSC3, 41X ZSC5, and 41X ZSC6 from MBH Analytical LTD; and PAINT-TiO2 from Analytical Services, Inc. The expanded uncertainty (95 % confidence) was calculated, and the SRM/CRMs and this test method's confidence intervals overlap; hence, no statistically observable bias from this dataset is observed. The statistical examination of the ILS results for the purposes of this test method was reviewed by ASTM's Interlaboratory Study Program.

NOTE 8—Based on a preliminary laboratory analysis of a 40 µm thick paint layer with a Pb mass fraction of 150 mg/kg on a plastic substrate containing 100 mg/kg Pb mass fraction the apparent bias of 17 measurements is 325 mg/kg. When this same paint layer was applied to a plastic substrate containing a 13.6 mg/kg Pb mass fraction, the apparent bias of ten measurements is 18 mg/kg which is within the precision of this test method.

17. Quality Control

17.1 Refer to Practice D6299 for suggested quality assurance/quality control (QA/QC) activities that can be used as a part of this test method.

17.2 Confirm the satisfactory performance of the instrument and the test procedure by analyzing a QC sample (see Section 13) at least once each day the analyzer is used.

17.3 When QA/QC protocols are already established in the testing facility, they can be used, provided they include procedures to monitor the reliability of the test results.

18. Keywords

18.1 children's products; EDXRF; HDXRF; lead; lead in children's products; lead in furniture; lead in paint; lead in toys; MMB-EDXRF; Multiple Monochromatic Beam EDXRF; Pb; toys

TABLE 2 Precision Calculations for Various Sample Types

Sample Type	Homogenous Material or Substrate Type	Lead (Pb) Mass Fraction Range, mg/kg	Repeatability (r)[A]	Reproducibility (R)[A]
Uncoated	Non-PVC Plastic, Glass or Ceramic	14 - 1200	$0.5502 \cdot X^{0.6046}$	$0.4201 \cdot X^{0.873}$
Uncoated	Metal	66 - 600	$2.3646 \cdot X^{0.5699}$	$7.3793 \cdot X^{0.3978}$
Uncoated	PVC	376 - 1150	$42.1092 \cdot X^{0.2238}$	$1.3742 \cdot X^{0.8186}$
Paint Layer	Plastic or Metal	30 - 100	$1.4511 \cdot X^{0.6385}$	$2.8932 \cdot X^{0.5565}$
Paint Layer	Fabric	79 - 200	$10.4728 \cdot X^{0.3227}$	$5.0386 \cdot X^{0.5791}$
Paint Layer	Plastic or Metal	>100 - 450	$1.1272 \cdot X^{0.7282}$	$0.5536 \cdot X^{0.9217}$

[A] Where *X* is the independent test result in mg/kg obtained under repeatability or reproducibility conditions.

TABLE 3 Precision Statistics for Various Sample Types

Sample Type	Homogenous Material or Substrate Type	X(bar), mg/kg	$s_{X(bar)}$	s_r	s_R	r	R
Paint Layer	Wood[A]	58.93	21.08	9.42	22.44	26.36	62.82

[A]Only one sample of wood was available for testing in the 2009 ILS, therefore a precision statement cannot be determined. Further testing in a later ILS is planned.

TABLE 4 Repeatability (*r*) Values for Various Sample Types

NOTE 1—The repeatability (*r*) values quoted in Table 4 were predicted using equations in Table 2, which were obtained by fitting the power function to the experimental data from ILS (ASTM Report # RR-F40:1001). Therefore, the expected repeatability achievable when using this test method may vary as much as 20 % relative, at the level of one standard deviation, from the values quoted in Table 4.

Pb, mg/kg	Sample Type				
	Uncoated Non-PVC Plastic or Glass r, mg/kg	Uncoated Metal r, mg/kg	Uncoated PVC r, mg/kg	Paint Layer on Plastic or Metal r, mg/kg	Paint Layer on Fabric r, mg/kg
30	4	. . .	. . .	13	. . .
50	6	. . .	. . .	18	. . .
90	8	31	. . .	26	45
100	9	33	. . .	27	46
300	17	61	. . .	72	. . .
400	21	72	161	88	. . .

TABLE 5 Reproducibility (*R*) Values for Various Sample Types

NOTE 1—The reproducibility (*R*) values quoted in Table 5 were predicted using equations in Table 2, which were obtained by fitting the power function to the experimental data from ILS (ASTM Report # RR-F40:1001). Therefore, the expected reproducibility achievable when using this test method may vary as much as 20 % relative, at the level of one standard deviation, from the values quoted in Table 5.

Pb, mg/kg	Sample Type				
	Uncoated Non-PVC Plastic or Glass R, mg/kg	Uncoated Metal R, mg/kg	Uncoated PVC R, mg/kg	Paint Layer on Plastic or Metal R, mg/kg	Paint Layer on Fabric R, mg/kg
30	8	. . .	. . .	19	. . .
50	13	. . .	. . .	26	. . .
90	21	44	. . .	35	68
100	23	46	. . .	38	73
300	61	71	. . .	106	. . .
400	79	80	183	139	. . .