



Designation: F3078 – 15

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 $\mu\text{g}/\text{cm}^2$ to approximately 10 $\mu\text{g}/\text{cm}^2$ 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 $\mu\text{g}/\text{cm}^2$ 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 $\mu\text{g}/\text{cm}^2$.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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, 2008³

SSPC-PA2 Paint Application Standard No. 2, Measurement of Dry Coating Thickness with Magnetic Gauges⁴

NIST Special Publication 829 Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories⁵

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. Terminology

3.1 Definitions:

3.1.1 Definitions of terms applying to X-ray fluorescence (XRF) spectrometry, plastics and declarable substances appear

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>.

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

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 criterion⁶:

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

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.

4.3 The test sample should cover the measuring aperture of an analyzer.

NOTE 1—Increased quantitative error may result if the coated sample area does not cover the measuring aperture of the analyzer. Correction schemes may be available to adjust the measurements of such samples. These schemes have not been evaluated for this method. Refer to the analyzers manufacturer’s instructions for guidance.

4.4 The test sample is irradiated by an X-ray source, and the resulting characteristic X rays of Pb and other elements present

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.

in the sample are measured. A value of the Pb mass per unit area of the paint sample is calculated and compared to the specification limit against which the sample is being evaluated.

5. Significance and Use

5.1 This test method provides for analysis of Pb in applied paint using measurement times on the order of several minutes. It can be used to determine whether the sample of applied paint has an areal mass of Pb either substantially less than a specification limit, and therefore does not exceed it, or substantially above the specified limit, and therefore exceeds it.

5.2 If the value obtained with this test method falls close to a specification limit, a more precise test method may be required to positively determine whether Pb content does or does not exceed the specified limit.

6. Interferences

6.1 *Spectral Interferences*—Spectral interferences in XRF analysis manifest themselves as overlaps of spectral peaks representing lines of different X-ray energies. These overlaps are the result of limited energy resolution of detectors. For example, the As K α peak overlaps completely the Pb La peak. Interactions of photons with the detector and limitations of associated electronics give rise to additional peaks in a spectrum known as escape peaks and sum peaks. For example, high content of iron in a paint or substrate may produce a sum peak that will overlap with the Pb L β line. Fundamental Parameters equations require that the measured net count rates be free from line overlap effects. Some empirical approaches incorporate line overlap corrections in their equations. The software used for spectrum treatment must compensate for line overlaps. Manufacturers' software typically provides tools to compensate for peak overlaps, escape peaks and sum peaks in spectra.

6.2 *Matrix Interferences*—Interelement effects, also called matrix 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 the apparent sensitivity for the element it represents. 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.

6.3 *Substrate Interferences*—Elements in the substrate may interfere with determination of Pb in a layer of paint. For example, if both substrate and paint contain Pb, the composite Pb signal will include contributions from both sources and effectively may result in a significant positive bias of Pb mass per unit area. For example, a plastic substrate containing 100 mg/kg of Pb may produce apparent areal Pb concentration of 30 $\mu\text{g}/\text{cm}^2$, even if paint on such substrate does not contain Pb.

7. Apparatus

7.1 *EDXRF Spectrometer*, designed for X-ray fluorescence analysis of materials with energy dispersive selection of

radiation. Any EDXRF spectrometer can be used if its design incorporates the following features.

7.1.1 *A means of repeatable sample presentation for analysis*—For hand-held spectrometers this is usually a small, flat plane with round, oval or rectangular aperture that comes into direct contact with the sample and through which X rays can reach the sample under test. Laboratory embodiments of analyzer design may have specimen holders and a specimen chamber.

7.1.2 *Source of X-ray Excitation*, typically an X-ray tube, capable of exciting the Pb L₂-M₄ (L β ₁) line (secondary line: Pb L₃-M_{4,5} (L α _{1,2})).

7.1.3 *X-ray Detector*, with energy resolution sufficient to resolve the recommended Pb L₂-M₄ (L β ₁) line from X-ray lines of other elements present in sample. An energy resolution of better than 250 eV at the energy of Mn K-L_{2,3} (K α) has been found suitable for the purpose of Pb analysis.

7.1.4 *Signal conditioning and data handling electronics*, that include the functions of X-ray counting and peak/spectrum processing.

7.1.5 *Data Processing Software*, for calculating elemental composition of sample from measured X-ray intensities using one of calibration methods.

7.2 The following spectrometer features and accessories are optional.

7.2.1 *Beam Filters*—Used to make the excitation more selective and to reduce background count rates.

7.2.2 *Secondary Targets*—Used to produce semi-monochromatic radiation enhancing sensitivity for selected X-ray lines and to reduce spectral background for improved detection limits. The use of monochromatic radiation also allows the simplification of FP calculations.

7.2.3 *Specimen Spinner*—Used to reduce the effect of surface irregularities of the specimen.

7.2.4 *Built-in Camera*—Used to capture and record an image of the tested area/object.

7.3 *Drift Correction Monitors*—Due to potential instability of the measurement system, the sensitivity and background of the spectrometer may drift with time. Drift correction monitors may be used to correct for this drift. The optimum drift correction monitor specimens are permanent materials that remain stable with time and repeated exposure to X rays. Drift correction monitors may be permanently installed inside the spectrometer and exposed only for diagnostic measurement when necessary.

8. Reagents and Materials

8.1 *Purity of Reagents*⁷—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 (ACS)

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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 all materials used for the preparation of reference materials and for cleaning of specimens and parts of the analyzer which come in direct contact with tested samples.

8.2 Reference Materials:

8.2.1 The user of this test method shall obtain applicable reference materials available from sources such as the National Institute of Standards and Technology or from reputable commercial vendors.

8.2.2 Reference materials can be prepared by adding known amounts of pure compounds or additives (or both), to an appropriate base paint material, mixing, and depositing the homogenized mixture uniformly on a flat substrate.

8.2.2.1 Thorough mixing of ingredients is required for optimum homogeneity.

8.2.2.2 Element concentrations can be calculated from the concentrations and molecular formulae of the compounds and additives used.

8.2.2.3 The elemental compositions of user-prepared reference materials must be confirmed by one or more independent analytical methods.

8.2.2.4 The preferred form of a reference material is a standard paint film, that is paint film deposited as a layer of uniform thickness onto a polyester foil so that the whole assembly may be placed for test over any substrate.

8.2.2.5 The Pb levels in the standard paint films (CRMs) shall be based on the appropriate specifications against which the samples of lead containing paint are anticipated to be measured. Such paint films may be used for instrument calibrations or method validation. Preferably, for the purpose of this test method, the paint films shall be as shown in [Table 1](#).

8.2.2.6 In addition, the layer thickness, density of paint layer and mass fraction of Pb in the paint layer shall be known for each paint film. The thickness of paint films should be in the range 25 μm to 75 μm (1 mil to 3 mil), and density should be in the range 1.1 g/cm^3 to 1.8 g/cm^3 . Specifically, at least one paint film standard shall have thickness between 35 and 45 μm and Pb mass fraction between 80 mg/kg and 110 mg/kg. Mass fractions of Pb in remaining paint films should be in the range of 50 mg/kg to 1200 mg/kg.

NOTE 2—A preferable form of standard paint film is paint film deposited on polyester foil so that it may be placed for measurement over any substrate. Polyester foil is made of polyethylene terephthalate (PET) resin. Such foil has been found to be mechanically strong and durable and yet thin enough to not interfere with XRF analysis. A 50 μm thick polyester foil absorbs less than 1 % of Pb $L\beta$ X rays and less than 2 % of

Pb $L\alpha$ X rays. Other film materials of equivalent or better than PET's properties may be used as substrates for standard paint films.

8.3 Quality Control Samples:

8.3.1 To ensure the quality of results, analyze quality control (QC) samples at the beginning and at the end of each batch of specimens or after a fixed number of specimens, but at least once each day of operation. Each QC sample shall be a homogeneous layer of paint deposited on polyester film with a minimum thickness of 50 μm (2 mil). The areal mass of Pb, mass fraction of Pb in paint, thickness of paint layer as well as its density must be known and consistent with the requirements in [8.2.2.5](#) for each QC sample. The QC samples must be stable under the anticipated storage and use conditions. The QC samples must be handled with care. The surface of a QC sample must not be scratched or contaminated by foreign substances. They should be stored at room temperature, away from direct exposure to UV radiation.

9. Hazards

9.1 Occupational Health and Safety standards for X rays and ionizing radiation shall be observed. It is recommended that proper practices be followed as presented by most manufacturers' documentation. Guidelines for safe operating procedures are also given in current handbooks and publications from original equipment manufacturers, and the National Institute of Standards and Technology (NIST). For more information, see ANSI-NIST Handbook 114 or similar handbooks on radiation safety.

9.2 *Warning*—Appropriate precautions are recommended when working with the element and compounds of Pb.

10. Sample Preparation

10.1 The user of this test method must define the sample using documented work instructions. These instructions should at minimum address the following items:

10.1.1 Ensure that the tested sample is within the measurement aperture of the analyzer. In addition, the measurement aperture must not include adjacent materials which may be of different compositions than the measured sample.

10.1.2 The sample within the measurement aperture must be uniform.

10.1.3 The sample must be clean and free of foreign elements such as stickers or markings.

10.1.4 If feasible, the sample should be tested in multiple locations of similar composition.

11. Preparation of Apparatus

11.1 Turn on the analyzer and allow it to warm up and stabilize in accordance with the manufacturer's recommendation.

11.2 Follow the manufacturer's recommendation to set the optimum current and voltage for analysis of Pb-bearing paint or select the appropriate manufacturer supplied or laboratory prepared calibration.

11.3 Determine a minimum measurement time resulting in a maximum counting statistical error (CSE) at one sigma of 10 %

TABLE 1 Recommended Lead Contents in Paint Calibrants

Paint Film	Pb ($\mu\text{g}/\text{cm}^2$) (see the note below)
Film 1	< 0.4
Film 2	0.4 to 1.0
Film 3	1.4 to 2.6
Film 4	5.0 to 10.0
Film 5	>10.0

relative for a specimen containing Pb at a level close to the specification limit. This shall be performed for each anticipated substrate type.

11.3.1 The required measurement time can be calculated by using Eq 2:

$$t \geq \left(\frac{100}{CSE\%} \right)^2 \cdot \frac{1}{R} + \left(\frac{100}{CSE\%} \right)^2 \cdot \frac{BGD}{R^2} \quad (2)$$

where:

R = net count rate of Pb X rays in counts per second (cps) measured for time, t ,

t = counting time in seconds, s ,

BGD = count rate of background under the Pb peak in cps, measured for time, t , and

CSE = relative error of counting statistics, (%).

11.3.1.1 When background count rate, BGD , is much less than the net count rate, R , the second term in Eq 2 may be omitted, then the product of R and t equals the total number of net counts accumulated under the Pb peak in EDXRF measurements. This time corresponds to a measuring time resulting in collection of > 100 counts after accounting for background.

11.3.2 In cases of instruments pre-calibrated by the manufacturer, measure specimens containing Pb at levels close to the specification limit for as long as it takes the measurement error reported by the instrument at one sigma level to be < 10 % relative to the value measured. The measurement time thus determined shall be used for subsequent tests.

11.4 Verify the limit of detection. The limit of detection (L_D) shall be estimated for each combination of sample presentation, substrate and measurement conditions by the use of Eq 3:

$$L_D = 3 \cdot s \quad (3)$$

where:

s = the standard deviation of a set of at least seven measurements of a Pb-free paint film presented on a substrate.

11.4.1 For optimum results the L_D should be less than 30 % of the specification limit or of the laboratory's action limit, whichever is less.

NOTE 3—Longer measurement time may be necessary for some instruments to achieve performance stipulated in 11.3 and 11.4. Relative error of measurement in EDXRF decreases twofold for each fourfold extension of measurement time. Therefore, the reduction of error obtained at 200 s measurement time by a factor of two would require a measurement time of 800 s, which would significantly reduce the number of samples that could be measured.

12. Calibration

12.1 *Empirical Calibration*—Obtain a set of calibration standards that cover the range of areal mass of Pb prepared in the matrix typical of natural paints. Standard paint films recommended in 8.2 may be used for instrumental calibration. It is important to have available several standards when using an empirical calibration. For Pb areal mass up to 100 $\mu\text{g}/\text{cm}^2$ the relation between count rate of Pb X-rays and Pb mass per unit area is linear; therefore, a small number of standards (at least three) may be sufficient to determine the slope and the intercept of the calibration curve.

12.1.1 Place each standard specimen on the appropriate substrate and then into the X-ray beam of analyzer and measure the count rate of Pb using the measurement conditions chosen in Section 11. Measure each standard at least twice.

12.1.2 Follow the manufacturer's instructions to obtain net count rates of Pb X rays and to perform a regression of net count rate of Pb X rays versus Pb mass per unit area.

12.1.3 As an option, the net count rates may first be divided by the Compton scatter count rate for the specimen.

12.1.4 If the spectrum processing options of the instrument do not include corrections for peak overlaps, corrections must be included in the regression model.

12.1.5 Repeat the calibration procedure for each typical substrate expected to be analyzed.

NOTE 4—With some instruments it may be possible to generate a single (global) calibration curve which will be valid for more than one type of substrate.

12.2 *FP Calibration and Manufacturer Pre-calibrated Instruments*—Matrix correction procedures by FP are based on mathematical descriptions of physical interactions between X-ray photons and matter. Calibration with FP can be accomplished using very few standards, and depending on the mathematical formalism chosen, with multielement or pure, single element ones. This is because the corrections for interelement effects (such as absorption and enhancement) are done entirely from theory. For instruments that are pre-calibrated at the factory either using an FP approach, or using procedures specific to the analytical software employed in a given instrument, follow exactly the calibration procedure supplied by the manufacturer.

12.2.1 If applicable, follow the manufacturer's instructions to perform a regression of net count rate of Pb versus Pb mass per unit area.

12.2.2 If the spectrum processing options do not include corrections for peak overlaps, corrections must be included in the regression model. FP approaches are predicated on the assumption that the count rate data has already been processed to remove background and spectral interferences.

12.2.3 As an option, the inclusion of the count rate of Compton scattered radiation (or the ratio of Compton and Rayleigh scattered radiation) in the FP algorithm may be used to compensate for matrix effects caused by sample elements that cannot be measured directly.

12.2.4 Unless specifically instructed otherwise, repeat the calibration procedure for each type of substrate expected to be analyzed.

12.3 Verification of Calibration:

12.3.1 Verify the calibration by analyzing one or more reference materials. Measure the reference materials immediately after completing calibration of the instrument.

12.3.2 When using a pre-calibrated system for which user calibration is not available, verify the calibration by running the reference materials before measuring unknown samples for the first time.

12.3.3 Measure areal mass of Pb in one or more reference materials. The areal masses of Pb from these measurements must be in agreement with the known (certified) values for Pb in the measured reference materials samples to within agreed

precision and bias of this test method, inclusive of uncertainty reported for known (certified) values for Pb in these samples. All measurements must be performed on samples placed over appropriate substrates. If a bias is detected, an investigation must be carried out to find the root cause.

12.4 Drift Monitors and Quality Control Samples:

12.4.1 When using drift correction, measure the count rates of the drift correction monitors in the same manner as the calibrants with the exception of counting times. The monitors' compositions and the count time for measurement of a monitor shall be optimised to achieve a minimum of 2,500 counts for each element for CSE = 2 %.

12.4.2 In many contemporary instruments, drift correction is accomplished with monitors which are integral parts of the analyzer (external or internal). In such a case, follow manufacturer-provided procedures, and monitor for drift correction.

12.4.3 When employing quality control charts, measure the control samples in the same manner as the calibrants. Measure each QC sample used in the QC process at least seven times. Construct control charts using this data. Analysis of result(s) from these specimens must be carried out following Practice D6299 or laboratory-specific control procedures. When the QC sample result indicates the laboratory is in an out-of-control situation, such as exceeding the laboratory's control limits, drift correction or instrument calibration may be required.

NOTE 5—Procedures for testing for bias between measured results and assigned (certified) values are beyond the scope of this standard. Information and examples can be found in NIST Special Publication 829. Discussion and procedures for interpretation of uncertainty estimates for assigned values can be found in the certificate of analysis of the reference material and in the ISO Guide to the Expression of Uncertainty in Measurement⁸.

13. Procedure

13.1 Allow the instrument to stabilize as per manufacturer's recommendations.

13.2 Measure the unknown test sample prepared according to the work instructions in Section 10 using the analyzer as calibrated, prepared and verified in Sections 11 and 12.

13.3 Measure the sample for at least the time calculated in 11.3.

13.4 If applicable, measure also an uncoated area of sample to verify that the substrate does not contain Pb.

14. Calculation

14.1 Allow the analyzer to calculate the areal mass of Pb in $\mu\text{g}/\text{cm}^2$.

14.2 Record the result.

14.3 Some combinations of extremely thin layer of paint and substrate may result in measurements which produce results less than the limit of detection, L_D , of the instrument. In

such instances, always report the actual limit of detection reported or applicable for the specific test, not the symbol ND.

14.4 Approaches to interpretation of results or decisions based on them are discussed in Annex A1.

15. Report

15.1 Report the following information:

15.1.1 A unique sample identification.

15.1.2 The date and time of the test.

15.1.3 Numerical results of the test, inclusive of less than L_D results, to the second decimal place (that is to the nearest $0.01 \mu\text{g}/\text{cm}^2$).

15.1.4 Reference to this standard test method (F40, F3078).

15.1.5 Identification of the substrate on which the paint film was measured.

15.1.6 Information on sample preparation (if any).

15.1.7 Any deviations from this standard or sample preparation guideline.

16. Precision and Bias

16.1 The precision of this test method is based on an interlaboratory study for WK21957, New Standard Test Method for Identification and Quantification of Lead in Paint and Similar Coating Materials Using Energy Dispersive X-ray Spectrometry (EDXRF), conducted in 2010. Five different makes of commercially available handheld XRF analyzers and one type of bench top analyzer were represented in the study. It is noted here that all instruments used a silicon drift detector although this is not a requirement of the standard. It is further noted that ten participants in the study were either instrument manufacturers or their direct affiliates which may imply that the participants were exceptionally qualified to perform the tests. Each of ten participants in the study was asked to report the Pb concentration of six paints of thicknesses between 26 and 42 μm on 16 different substrates. Different precision and bias values may be found for paint thicknesses outside of this range. Every "test result" represents an individual determination, and all participants were instructed to report three to six test results for each paint/substrate combination. Practice E691 was followed for the design of the ILS and analysis of the data; the details are given in an ASTM Research Report.⁹

16.1.1 *Repeatability limit (r)*—The value below which the absolute difference between two individual test results obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time, may be expected to occur with a probability of approximately 0.95 (95 %).

16.1.1.1 Repeatability limits are listed in Tables 2-13.

16.1.2 *Reproducibility limit (R)*—The value below which the absolute difference between two test results obtained with the same method on identical test items in different laboratories with different operators using different equipment may be expected to occur with a probability of approximately 0.95 (95 %).

⁸ ISO GUM: Guide to the Expression of Uncertainty in Measurement; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization, Geneva, Switzerland, 1993.

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F40-1004. Contact ASTM Customer Service at service@astm.org.