Designation: F2617 − 15 (Reapproved 2023)

Standard Test Method for Identification and Quantification of Chromium, Bromine, Cadmium, Mercury, and Lead in Polymeric Material Using Energy Dispersive X-ray Spectrometry[1]

This standard is issued under the fixed designation F2617; 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) spectrometric procedure for identification and quantification of chromium, bromine, cadmium, mercury, and lead in polymeric materials.

1.2 This test method is not applicable to determine total concentrations of polybrominated biphenyls (PBB), polybrominated diphenyl ethers (PBDE) or hexavalent chromium. This test method cannot be used to determine the valence states of atoms or ions.

1.3 This test method is applicable for a range from 20 mg/kg to approximately 1 wt % for chromium, bromine, cadmium, mercury, and lead in polymeric materials.

1.4 This test method is applicable for homogeneous polymeric material.

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

1.6 This test method is not applicable to quantitative determinations for specimens with one or more surface coatings present on the analyzed surface; however, qualitative information may be obtained. In addition, specimens less than infinitely thick for the measured X rays, must not be coated on the reverse side or mounted on a substrate.

1.7 *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.*

1.8 *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]

D883 Terminology Relating to Plastics

D3641 Practice for Injection Molding Test Specimens of Thermoplastic Molding and Extrusion Materials

D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

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

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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

E1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis

F2576 Terminology Relating to Declarable Substances in Materials

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions of terms applying to XRF, plastics and declarable substances appear in Terminology E135, Terminology D883 and Terminology F2576, respectively.

3.1.2 *Compton scatter*—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.

3.1.3 *Rayleigh scatter*—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.

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[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.

3.1.3.1 *Discussion*—The measured count rate of Compton and Rayleigh scattered radiation varies depending upon specimen composition and may thus be used to compensate for matrix effects. One option is to use the measured count rate of the Compton scatter in the same manner as the measured count rate of an internal standard element. Alternatively, the measured count rate of the Compton scatter or the Compton/Rayleigh scatter ratio may be used indirectly for estimating the effective mass absorption coefficient of the specimen, which is used to compensate for matrix effects. The concept of corrections based on the Compton scatter effect is discussed as an optional part of several calibration choices in this standard.

3.1.4 *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.

3.1.5 *homogeneous polymeric material*—polymeric material is considered homogeneous for XRF when the elemental composition is independent with respect to the measured location on the specimen and among separate specimens prepared from the same polymeric material.

3.1.6 *infinite thickness (or critical thickness)*— the thickness of specimen which, if increased, yields no increase in intensity of secondary X rays, due to absorption by the polymer matrix.

3.1.6.1 *Discussion*—This thickness varies with secondary X-ray energy, or wavelength.

3.2 *Abbreviations:*

3.2.1 *EDXRF*—energy dispersive X-ray fluorescence

3.2.2 *FP*—fundamental parameters

3.2.3 *PBB*—polybrominated biphenyl

3.2.4 *PBDE*—polybrominated diphenyl ether

4. Summary of Test Method

4.1 The optimum test sample is a smooth plaque or disk large enough to cover the viewed area of the spectrometer. Suitable specimens may be die-cut from extruded sheets, or molded from resin pellets, from powders or from granules.

4.2 The specimen is placed in the X-ray beam, and the appropriate region of its spectrum is measured to give the count rates or fluorescent intensities of lead, mercury, cadmium, chromium and bromine.

4.3 The EDXRF spectrometer is calibrated by one of several approaches including fundamental parameters and empirical, classical curve construction, with either empirical or theoretical influence coefficients, from measured polymer reference materials. The calibration may be performed by the manufacturer or by the user.

4.4 Choices of appropriate characteristic X-ray lines and spectrometer test conditions may vary according to each element and with factors such as detector response, concentration range and other elements present in the polymer matrix.

5. Significance and Use

5.1 This test method is intended for the determination of chromium, bromine, cadmium, mercury, and lead, in homogeneous polymeric materials. The test method may be used to ascertain the conformance of the product under test to manufacturing specifications. Typical time for a measurement is 5 to 10 min per specimen, depending on the specimen matrix and the capabilities of the EDXRF spectrometer.

6. Interferences

6.1 *Spectral Interferences*—Spectral interferences result from the behavior of the detector subsystem of the spectrometer and from scattering of X rays by the specimen, by a secondary target or by a monochromator, if the spectrometer is so equipped. Overlaps among the X-ray lines from elements in the specimen are caused by the limited resolution of the detection subsystem. Depending upon the resolution of the detector system, the peaks from Zn, Br, Hg and Pb may overlap with one another. Peaks from Cd may overlap with peaks from Ca, Sn, or other elements. Interactions of photons and electrons inside the detector give rise to additional peaks in a spectrum known as escape peaks and sum peaks. 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. Manufacturers' software may provide tools to compensate for overlapped peaks, escape peaks, and sum peaks in spectra. The degree of line overlap and the best method to account or correct for it must be ascertained on an individual basis and must be considered when calibrating the instrument.

6.2 *Interelement Effects*—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 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. The influence coefficients may be calculated either from first principles or from the empirical data, or some combination of the two approaches. See Guide E1361 for examples of these approaches. Also, consult the software manual for the spectrometer for information on the approaches provided with the spectrometer. Any of these that will achieve the necessary analytical accuracy is acceptable. Examples of common interelement effects are listed in Table 1.

7. Apparatus

7.1 *EDXRF Spectrometer*—Designed for X-ray fluorescence analysis with energy dispersive selection of radiation. The spectrometer is equipped with specimen holders and a specimen chamber. Any EDXRF spectrometer may be used if its design incorporates the following features.

7.1.1 *Source of X-ray Excitation* , capable of exciting the recommended lines listed in Table 2, typically an X-ray tube.

7.1.2 *X-ray Detector,* with sufficient energy resolution to resolve the recommended lines listed in Table 2. An energy resolution of better than 250 eV at Mn K-$L_{2,3}$ (Kα) has been found suitable.

TABLE 1 Common Interelement Effects in Formulated Plastics

Cause	Effect
Absorption by Cl in PVC	Reduced sensitivity for all analytes as compared to when they are occurring at the same concentration level in polyolefins
Polymers of similar composition but differences in the relative concentrations of H and C	Differences in C/H among calibrants and samples may result in biases of a few percent (relative).
Unmeasured elements B, N, O, and F present in the matrix of the polymer, for example, amide, fluorinated, and terephthalate compounds.	If concentrations differ from the calibrants, substantial concentrations of these elements may cause significant changes in both apparent sensitivity and background count rates.
Absorption by elements present in flame-retardant compounds such as PBBs, PBDEs, and Sb_2O_3	Reduction of apparent sensitivity for most analytes
Absorption by Na, P, S, Ca, Ti, Zn, Mo, Sn, Ba, and other elements included in a formulation as fillers or performance additives	Reduction of apparent sensitivity for most analytes

TABLE 2 Recommended X-ray Lines for Individual Analytes

NOTE 1—Other choices may provide adequate performance.

Analyte	Preferred Line	Secondary Line
Chromium, Cr	$K\text{-}L_{2,3}$ ($K\alpha_{1,2}$)	
Bromine, Br	$K\text{-}L_{2,3}$ ($K\alpha_{1,2}$)	$K\text{-}M_{2,3}$ ($K\beta_{1,3}$)
Cadmium, Cd	$K\text{-}L_{2,3}$ ($K\alpha_{1,2}$)	$K\text{-}M_{2,3}$ ($K\beta_{1,3}$)
Mercury, Hg	$L_3\text{-}M_{4,5}$ ($L\alpha_{1,2}$)	
Lead, Pb	$L_2\text{-}M_4$ ($L\beta_1$)	$L_3\text{-}M_{4,5}$ ($L\alpha_{1,2}$)

7.1.3 *Signal Conditioning and Data Handling Electronics* that include the functions of X-ray counting and peak processing.

7.2 The following spectrometer features and accessories are optional:

7.2.1 *Beam Filters*—Used to make the excitation more selective and 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 *Vacuum Pump*—For improved sensitivity of atomic numbers 20 (Ca) or lower, the X-ray optical path may be evacuated using a mechanical pump.

7.2.5 *Helium Flush*—For improved sensitivity of atomic numbers 20 (Ca) or lower, the X-ray optical path may be flushed with helium.

7.3 *Drift Correction Monitor(s)*—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 correct for this drift. The optimum drift correction monitor specimens are permanent materials that are stable with time and repeated exposure to X rays [Note 1].

NOTE 1—Suitable drift correction monitors may be fused bead specimens containing the relevant elements (Cr, Br, Cd, Hg, and Pb) or elements that have fluorescence with the same energies as the elements of interest.

8. Reagents and Materials

8.1 *Purity of Reagents*[3]—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) 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.

8.2 *Reagents:*

8.2.1 Isopropanol or ethanol.

8.2.2 Nitric acid (HNO_3).

8.2.3 Hexane.

8.2.4 Deionized water (H_2O).

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

8.4 Appropriate personal protective equipment for the handling of reagents.

8.5 *Reference Materials:*

8.5.1 Polymer reference materials are available from both metrology institutes and commercial sources. Some are provided in disk form, and some are available as granules or extruded pellets.

8.5.2 Reference materials may be prepared by adding known amounts of pure compounds or additives (or both), to an appropriate polymeric base material. It is recommended to make reference materials using the same base polymer as the unknown samples.

8.5.2.1 Thorough mixing of ingredients is required for optimum homogeneity. Options may include grinding, melt-blending, repeated extrusion, and solvent dissolution.

8.5.2.2 Elemental concentrations may be calculated from the concentrations and molecular formulae of the compounds and additives used.

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

8.6 *Quality Control Samples:*

8.6.1 To ensure the quality of the 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. If possible, the QC sample shall be representative of samples typically analyzed. The material shall be homogeneous and stable under the anticipated

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[3] *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 *Annular Standards for Laboratory Chemicals*, BDH Ltd. Poole, Dorset, UK, and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeia Convention, Inc. (USPC), Rockville, MD.

storage conditions. An ample supply of QC sample material shall be available for the intended period of use.

9. Hazards

9.1 Occupational Health and Safety standards for X rays and ionizing radiation shall be observed. It is also 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. For more information see similar handbooks on radiation safety.

9.2 **Warning—**Appropriate precautions are recommended when working with the elements and compounds of chromium, bromine, cadmium, mercury, and lead in creating polymer mixtures and fused beads.

10. Specimen Preparation

10.1 From the polymer to be tested, obtain a flat, smooth piece that is large enough to cover the viewed area of the spectrometer [Note 2]. Specimens shall have no obvious voids within the measured area. It is preferable that the test specimen be either of infinite thickness [Note 3] or the same thickness as the reference materials. The minimum recommended thickness is 2 mm (0.08 in.) [Note 4].

10.1.1 Specimens taken directly from molded components of a product may be analyzed without modification provided their geometry and surface characteristics are suitable. Excessive curvature or rough surface texture will affect the results of the analysis.

10.1.2 *Compression or Injection Molding* —Specimens may be compression molded from pellets, granules or powder. General guidelines for molding may be found in Practice D4703 for compression molding and Practice D3641 for injection molding. Specific conditions for molding specimens may be obtained from the appropriate material specification, if one is available, material supplier's recommendation, or past experience. Select conditions to produce a smooth, plane surface without voids. Since the specimens will not be used for mechanical testing, cooling and heating rates specified for some materials, are not critical.

10.1.3 Specimens may be obtained from sheets of material by cutting, punching, or die cutting. When analyzing thin films and foils, it is acceptable to stack layers of films of the same composition to generate the required thickness. Care must be taken to ensure that the layers are in full contact across the viewed area in the spectrometer and that no air is trapped between layers.

10.1.4 Specimens shall not have surface coatings, nor shall they be attached to a substrate, if the specimen is less than infinitely thick with respect to the X rays of the primary beam, that is, from the X-ray source.

10.2 Prior to measurement, samples of polymers must be cleaned by rinsing with appropriate solvents [Note 5]. In general, non-solvents for the polymer under investigation are appropriate. The following cleaning agents may be used.

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

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

10.2.3 Hexane for cleaning of polyamide and polyester specimens.

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

NOTE 2—Refer to Appendix X1 for alternative specimen handling techniques.

NOTE 3—Materials with a matrix of low atomic number elements, such as polymeric materials, exhibit relatively low X-ray absorption. This leads to a requirement that the specimens must be thick, generally in excess of several millimeters, depending on the X-ray energies to be measured and the composition of the matrix. A minor contribution to the effect comes from the geometry of the instrument used. A specimen thickness of 2 mm is commonly used; however, some laboratories employ lesser or greater thickness (for example, 6 mm to more closely approach infinite thickness). The convenience of making discs of the same thickness for all specimens, instead of infinite thickness, may be a factor for user consideration. In general, more accurate and precise results may be obtained when the reference materials and the unknowns are of infinite thickness or of the same thickness.

NOTE 4—Variations of 10 % relative in thickness at a level of 2 mm have been observed to result in count rate differences of 5 to 10 %. In some cases the effects on the results caused by the variations in thickness may be corrected for by the instrument manufacturer's software or by the calibration, or both.

NOTE 5—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 XRF instrument to stabilize for operation according to the manufacturer's guidelines.

11.2 Follow the manufacturer's recommendations [Note 6] and set up measurement conditions (X-ray tube excitation voltage, tube current, filters, etc.) to measure the count rates of the preferred lines of chromium, bromine, cadmium, mercury, and lead as suggested in Table 2.

11.3 If applicable, measure the Compton scatter radiation resulting from scatter of X-ray tube characteristic lines from the samples [Note 7].

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 Eq 1:

$$\%\mathrm{CSE} = 100/\sqrt{(R \cdot t)} \tag{1}$$

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 [Note 8].

11.5 Ensure the software removes escape peaks and sum peaks from the spectrum.

11.6 Ensure the software subtracts the background of the spectrum. For low atomic number materials, background subtraction is necessary to compensate for varying matrices. Measurement strategies that determine count rates using library spectra or deconvolution may include background subtraction in which case no separate background subtraction is required.

NOTE 6—Many spectrometers use measurement conditions configured by the manufacturer. The user is cautioned to confirm that pre-configured instruments conform to this standard.

NOTE 7—A background region from 23.5 to 23.7 keV may be used as an alternative for the Compton scatter radiation. Depending on the anode material of the X-ray tube (or the secondary target), Compton scatter radiation may be observable. For example, tube anodes consisting of Mo, Ag, Rh exhibit clear Compton scattered K-series radiation; while the Compton scatter when using tubes with anodes such as Cr or Ti is of little or no use for the purpose of matrix correction. In these cases, the use of the background region is suggested as an alternative. A tube with an anode of Mo, Rh, or Ag must be operated at a voltage in excess of 28 kV to produce K-series lines that result in a strong Compton scatter peak.

NOTE 8—Polymer materials are subject to damage by ionizing radiation. Susceptibility to damage varies greatly among common polymers. The user is cautioned to keep measurement times as short as practical and to avoid the repeated measurement of a single specimen.

12. Calibration

12.1 *Calibration*—When calibrating, use one of the described calibration methods: an empirical calibration, or a FP calibration. Both methods rely on the use of a set of known standards or certified reference materials, or both (see also 8.5) [Note 9].

12.2 *Empirical Calibration*—Prepare or obtain a set of calibration standards that cover the concentration range of interest of each analyte prepared in the matrix of interest including the relevant interferences described in Section 6. Standards that contain multiple analytes are preferred. It is important to have standards with concentrations that vary independently from one another and span the range of concentrations expected in the unknown samples. To the extent it is practical, avoid having correlations by ensuring that the concentrations of the different analytes do not vary in proportion to one another in the reference materials. Ensure that the low concentration of one analyte is combined with a high concentration of another analyte. It is important to have available several standards for each analyte when using an empirical calibration to provide enough degrees of freedom to determine empirically the influence coefficients as well as the slope and intercept of the calibration curve. In an empirical approach, the influence coefficients may be determined from theory (using FP), which may reduce the number of required standards.

12.2.1 Place each standard specimen in the X-ray beam and measure the net count rate of each element using the measurement conditions chosen in Section 11.

12.2.2 Measure each standard at least twice, preferably with two separately prepared specimens.

12.2.3 For each analyte, follow the manufacturer's instructions to perform a regression of net count rate versus concentration.

12.2.3.1 As an option, the net count rates may first be divided by the Compton scatter count rate for the specimen (or the background count rate, if Compton scatter cannot be measured).

12.2.4 Include significant interelement effects (see 6.2) in the regression model by using influence coefficients.

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

12.3 *FP Calibration:*

12.3.1 Matrix correction procedures by FP are based on mathematical descriptions of the most important interactions between X-ray photons and matter. Calibration with FP may be done using very few standards because the only parameters to be determined are the slope and intercept of the calibration curve. At least one standard for each analyte must be available. Corrections for interelement effects are done entirely from theory.

12.3.2 Place each standard specimen in the X-ray beam and measure the net count rate of each element using the measurement conditions chosen in Section 11.

12.3.3 Measure each standard at least twice preferably with two separately prepared specimens.

12.3.4 For each analyte, follow the manufacturer's instructions to perform a regression of net count rate versus concentration.

12.3.5 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.

12.3.6 FP methods often require the sum of calculated constituents of a sample to be 100 %. Typically, results are improved when the matrix of the specimen is modeled as a compound of H, C, N, and O approximating the actual polymer composition.

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

12.4 *Verification of Calibration :*

12.4.1 Verify the calibration by analyzing one or more reference materials, preferably of the same polymers as the materials on which analyses will be performed. Measure the reference materials immediately after completing an empirical calibration or a Fundamental Parameter calibration. When using pre-calibrated systems, run the reference materials before measuring unknowns for the first time. The determined concentrations from these measurements must be in agreement with the known (certified) values [Note 9].

12.5 *Drift Monitors and Quality Control Samples:*

12.5.1 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 optimized to achieve a minimum of 2500 counts for each element for %CSE = 2.

12.5.2 After the initial verification of the calibration, the user may implement a control chart using one or more quality control samples. When using quality control charts, measure the control samples in the same manner as the calibrants. At this point in time, measure each QC sample at least seven times. Construct control charts using this data. The repeatability data of the QC sample shall be checked against the precision statement in 16.1.1 to ensure that the performance of the laboratory is comparable to the intralaboratory repeatability established during validation of this standard test method. Analysis of results from these specimens must be carried out following Practice D6299 or laboratory-specific control procedures. When a QC sample result indicates the laboratory is in an out-of-control situation, such as exceeding the laboratory's control limits, corrective action may be required.

NOTE 9—Many spectrometers are calibrated by the manufacturer. The user is cautioned to confirm that pre-calibrated instruments conform to this standard.

13. Procedure

13.1 *Conditioning:*

13.1.1 It is recommended to store the specimens in the same conditions as the reference materials to avoid large differences in environmental parameters such as temperature, humidity, and moisture content. Polyamides, for instance, are prone to adsorb relatively high amounts of moisture from their environment and differences in moisture content between unknowns and reference materials may cause biased results. The magnitude of this effect has not been studied and is as yet unknown.

13.2 *Measurement of Unknown Specimens :*

13.2.1 Allow the instrument to stabilize according the manufacturer's guidelines.

13.2.2 Place the specimen in the instrument and perform the measurement using the conditions chosen in Section 12.

13.2.3 Process the spectrum using the same procedure chosen in Sections 11 and 13, including the same processes for handling escape peaks, sum peaks, background modeling and subtraction, and spectral overlaps.

13.3 *Quality Control Samples:*

13.3.1 When using quality control (QC) samples, measure them before measuring any unknowns [Note 10].

13.3.2 To ensure the quality of the results, analyze additional reference material at the beginning and at the end of a batch of specimens or after a fixed number of specimens but at least once each day of operation.

13.3.3 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.

13.4 *Drift Correction:*

13.4.1 When using drift correction, measure the drift correction monitors prior to analyzing samples. By comparing the current count rate of the drift correction monitors to the count rate at the time of the calibration it is possible to calculate correction factors, which are then used to correct for any drift in sensitivity. The use of the instrument manufacturer's drift correction procedure is recommended.

13.4.2 Drift correction is usually implemented automatically in the manufacturer's instrument software, although the calculation may readily be done manually. For X-ray instruments that are highly stable, the magnitude of the drift correction factor may not differ significantly from unity.

NOTE 10—Verification of system control through the use of QC samples and control charting is highly recommended.

14. Calculation

14.1 Using the net count rates for a specimen and the calibration created in Section 12, calculate the results in units of mg/kg (ppm). Typically, the calculations may be done using the instrument software.

15. Report

15.1 Report the following information:

15.1.1 Unique identification of the sample. This may vary according to company guidelines and test purposes.

15.1.2 The date and time of the test.

15.1.3 The results of this test expressed to the nearest 0.1 mg/kg for concentrations < 50 mg/kg and to the nearest 1 mg/kg for higher concentrations. Follow the relevant procedures in Practice E29.

15.1.4 A reference to this standard method of test (F2617).

15.1.5 The origin of the sample.

15.1.6 A description of the specimen type: for example, disk, granulate, etc.

15.1.7 A description of the specimen preparation.

15.1.8 Deviations from this standard, if any.

16. Precision and Bias[4]

16.1 The precision of this test method is based on an interlaboratory study of ASTM F2617, Standard Test Method for Identification and Quantification of Chromium, Bromine, Cadmium, Mercury, and Lead in Polymeric Material Using Energy Dispersive X-ray Spectrometry, conducted in 2012. Fifteen laboratories participated in the study, testing up to four different polymers in both disk and granular form. Participants reported elemental concentrations obtained using one or more of the following analyzer technologies: bench top, handheld; or polarized floor standing. Due to a limited number of participants, statistical data for polarized floor standing XRF instrumentation is presented for information only in Appendix X2. Every analyst was instructed to report five replicate test results for each material in this study. Practice E691 was followed for the study design; the details are given in ASTM Research Report F40–1005.[4]

16.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F40-1005.