Designation: F2980 – 13 (Reapproved 2017)

# Standard Test Method for Analysis of Heavy Metals in Glass by Field Portable X-Ray Fluorescence (XRF)[1]

This standard is issued under the fixed designation F2980; 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 covers field portable X-ray fluorescence (XRF) spectrometric procedures for analyses of arsenic and lead in glass compositions using field portable energy dispersive XRF spectrometers.

1.2 The mass fraction range of arsenic within which this test method is quantitative is given in Table 1. Scope limits were determined from the interlaboratory study results using the approach given in Practice E1601.

1.3 The mass fraction range for which lead was tested is given in Table 1. However, lead results cannot be considered quantitative on the basis of single-sample results because the precision performance is not good enough to allow laboratories to compare results in a quantitative manner.

NOTE 1—The performance of this test method was evaluated using results based on single-sample determinations from specimens composed of glass beads. One laboratory has determined that performance can be significantly improved by basing reported results on the mean of determinations from multiple samples to overcome inherent heterogeneity of elements in glass beads, especially the element lead. Additional information is provided in Section 17 on Precision and Bias.

1.3.1 To obtain quantitative performance, lead results must consist of the average of four or more determinations.

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Some specific hazards statements are given in Section 7 on Hazards.

1.6 *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]

D75/D75M Practice for Sampling Aggregates

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

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1621 Guide for Elemental Analysis by Wavelength Dispersive X-Ray Fluorescence Spectrometry

F2576 Terminology Relating to Declarable Substances in Materials

2.2 *ANSI Standard:*[3]

N43.2 Radiation Safety for X-Ray Diffraction and Fluorescence Analysis Equipment

2.3 *AASHTO Standard:*[4]

TP-97-11 Test Method for Glass Beads used in Pavement Markings

[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 Nov. 1, 2017. Published November 2017. Originally approved in 2013. Last previous edition approved in 2013 as F29180-13. DOI: 10.1520/F2980-13R17.

[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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

**TABLE 1 Scope Ranges for Quantitative Results**

| Element | Scope Lower Limit (mg/kg) | Scope Upper Limit (mg/kg) |
|---|---|---|
| Arsenic | 240 | 2000 |
| Lead | 120 | 500 |

## 3. Terminology

3.1 *Definitions*—Definitions of terms applying to X-ray fluorescence (XRF) and declarable substances appear in Terminologies E135 and F2576, respectively.

3.2 *Compton-matrix correction, n*—measured intensity of Compton or incoherent scattered radiation may be used directly to compensate for matrix effects or indirectly for the determination of the effective mass absorption coefficient to correct for matrix effects.[5]

3.2.1 *Discussion*—The compensation for matrix effects is based on a combination of sample preparation and experimental intensity data.

3.3 *Compton scatter, n*—inelastic scattering of an X-ray photon through its interaction with the bound electrons of an atom.

3.3.1 *Discussion*—This process is also referred to as incoherent scatter.

3.4 *fundamental parameters, FP, model, n*—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.[6]

3.5 *Acronyms:*

3.5.1 *EDXRF*—Energy dispersive X-ray fluorescence

3.5.2 *QC*—Quality control

3.5.3 *XRF*—X-ray fluorescence

## 4. Summary of Test Method

4.1 Portable handheld instruments are used to measure glass spheres, ground glass, cullet, fiberglass, and sheet glass for their contents of arsenic and lead. Samples of sheet glass can be measured directly. Samples that are not in sheet form are measured as is or after pulverizing to an appropriate particle size.

4.2 The samples of glass spheres or powders may be placed into disposable cups with a polymer film supporting the glass. The filled cup is measured from below through the polymer film.

4.3 The glass specimen may be analyzed in situ by using a handheld spectrometer positioned in contact with sheet glass or the contents of a larger container, for example, a bulk shipping container.

4.4 The handheld XRF may be used while the operator is holding the unit or by being mounted in a stand for safer, more convenient laboratory use. The two measurement options are discussed throughout this test method.

## 5. Significance and Use

5.1 Waste glass is currently recycled into various consumer products. This test method has been developed as a tool for evaluation of heavy metals in glass to satisfy reporting requirements for maximum allowable content for some applications.

5.2 The ranges within which this test method is quantitative are given in Table 1.

5.3 For amounts of the analyte elements outside the ranges in Table 1, this test method provides screening results. That is, it provides an unambiguous indication that each element can be described as present in an amount greater than the scope upper limit or that the amount of the element can be described as less than the scope lower limit with a high degree of confidence.

NOTE 2—In general, when a quantitative result is obtained, the analyst can make a clear decision as to whether a material is suitable for the intended purpose. When the contents of elements of interest are outside the quantitative range, the analyst can still make a decision whether the amount is too high or whether additional analyses are required.

5.4 These methods can be applied to glass beads, plate glass, float glass, fiber glass, or ground glass. This test method has been validated for the ranges of matrix compositions that are summarized in Table 2.

5.5 Detection limits, sensitivity, and element ranges will vary with matrices, detector type, and other instrument conditions and parameters.

5.6 All analytes are determined as the element and reported as such. These include all elements listed in Table 1. This test method may be applicable to other glass matrices, additional elements, and wider concentration ranges provided the laboratory is able to validate the broadened scope of this test method.

## 6. Interferences

6.1 *Spectral Interferences*—These can occur for some elements as a result of partial or total line overlaps. These line overlaps can result from scattered characteristic lines from the target of the X-ray tube or by X-ray fluorescence from atoms in the specimen. Spectral interference can also be the result of escape peaks from the solid-state detector. See Guide E1621 for a full discussion of models used to correct for these effects. In this particular case, the most obvious line overlap is the overlap of As K-$L_{2,3}$(As K$\alpha_{1,2}$; 10.53 keV) on Pb $L_3$-$M_5$ (Pb L$\alpha_1$; 10.55 keV) and vice versa. The energy difference between these two lines is about 0.02 keV, which cannot be resolved with the detectors used. The emission lines of these two elements will appear as a single peak. However, both As and Pb have alternative lines that can be used for analysis. For Pb,

**TABLE 2 Matrix Components and Ranges**

| Oxide | Scope Lower Limit, % | Scope Upper Limit, % |
|---|---|---|
| $SiO_2$ | 58 | 80 |
| $Al_2O_3$ | 1 | 10 |
| $Na_2O$ | 3 | 15 |
| CaO | 6 | 20 |
| MgO | 1 | 5 |

[5] Andermann, G. and Kemp, J. W., "Scattered X-rays as Internal Standards in X-Ray Spectroscopy," *Analytical Chemistry*, Vol 20, No. 8, 1958.

[6] The algorithm used for the procedure is usually implemented in the instrument manufacturer's software. Third-party software is available and may be used.