Designation: C1606 – 10 (Reapproved 2014)

Standard Test Method for Sampling Protocol for TCLP Testing of Container Glassware[1]

This standard is issued under the fixed designation C1606; 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 defines the way in which container glassware should be prepared before performing the Toxicity Characteristic Leaching Procedure (TCLP). The method covers the homogenization of the sample, and the selection of a representative portion of the sample to test and get reproducible results.

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

1.3 *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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Summary of Test Method

3.1 Container glass articles decorated with ceramic glass decorations are broken to fit the requirements of the TCLP protocol by using a disc mill or roll mill apparatus, and then the samples are homogenized using a riffle box. A representative sample of the glass articles can then be put through the TCLP test with reproducible results.

4. Significance and Use

4.1 Sampling of decorated glass containers for the TCLP can vary greatly, resulting from the size and shape of the article relative to the amount of ceramic decoration on the ware. Breaking the glass can cause some of the pieces to have no decoration on them, and others to be heavily decorated and more likely to leach lead and cadmium under the TCLP test. This method provides an effective tool to homogenize the glass containers so that reproducible results can be attained from the TCLP test.

5. Apparatus

5.1 Pre-breaking apparatus, consisting of:

5.1.1 Two L (or larger) polythene bucket with a tightly sealed lid,

5.1.2 One or two clean 35 to 40 mm diameter alumina grinding balls,

5.2 Disc mill or roll mill,

5.3 Sieves, as needed,

5.4 Riffle box, and

5.5 Roller or tumbler apparatus.

6. Reagents and Materials

6.1 Glassware to be tested, in two sets of 10 units each.

7. Hazards

7.1 When working with glassware, always be aware and careful of the potential of getting cut by the glass. Personal protective equipment, such as a face shield or goggles, leather gloves, and a protective apron should be worn when working with glass.

Take precautions when working with the samples and apparatus to avoid the possibilities of getting hurt.

8. Sampling, Test Specimens, and Test Units

8.1 Since the amount of lead and cadmium that leaches from a decoration can be affected by the relative size of the decoration, it is important to start with two sets of 10 units each.

[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Productsand is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns.

Current edition approved Dec. 1, 2014. Published December 2014. Originally approved in 2004. Last previous edition approved in 2010 as C1606 – 10. DOI: 10.1520/C1606-10R14.

[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.

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9. Procedure

9.1 To minimize the variation of sampling, this testing shall be done in duplicate, and therefore, take two sets of 10 units of the glassware to be tested.

9.2 Pre-break the glass prior to milling, using the following procedure:

9.2.1 Place the first set of glassware into the 2 L bucket, along with one or two clean 35 to 40 mm diameter alumina grinding balls.

9.2.2 After tightly sealing the lid, shake the vessel hard enough to smash the glass to create pieces that can be fed into the roll or disc mill.

9.3 Clean both the mill and riffle box, prior to use.

9.4 Feed the pre-broken glass into the disc mill or roll mill as feedstock.

9.4.1 Grind the glass as two separate lots, with the selected mill apertures set between 2.5 mm and 3.5 mm. These aperature settings tend to produce crushed glass with a maximum particle size of 5 mm.

9.5 Any oversized material must be removed by sieving or hand picking, and be broken up prior to blending back into the original sample.

9.6 Homogenize the sample using a roller or tumbler apparatus.

9.7 Immediately pass sample through the riffle box collecting two sub-samples of 100g each.

9.8 Perform TCLP extraction according to EPA specifications.

10. Precision and Bias

10.1 The precision of this test method is based on an interlaboratory study of C1606-04 Standard Test Method for Sampling Protocol for TCLP Testing of Container Glassware, conducted in 2008. Four laboratories participated in this study. Each of the labs was asked to report two replicate test results for three different materials, however not every lab reported replicate data for each material. Every "test result" that was reported represents an individual determination. Except for the use of only four laboratories, and inconsistent reporting of replicates, Practice E691 was followed for the design and analysis of the data; the details are given in RR:C21-1006.[3]

10.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.1.1 Repeatability limits are listed in Table 1 and Table 2.

10.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

10.1.2.1 Reproducibility limits are listed in Table 1 and Table 2.

10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.1.4 Any judgment in accordance with statements 10.1.1 and S1012 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. The repeatability limit and the reproducibility limit should be considered as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

10.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

10.3 The precision statement was determined through statistical examination of 32 results, from four laboratories, on three materials.

To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

11. Keywords

11.1 TCLP; ceramic glass enamels; ceramic glass decorations

TABLE 1 Pb (*ppm*)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
Pochet	0.47	0.08	0.44	0.22	1.23
FB	2.17	2.12	2.96	5.95	8.28
Green	0.00	0.00	0.00	0.00	0.00

[A] The average of the laboratories' calculated averages.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C21-1006.

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