Designation: F2459 – 05

Standard Test Method for Extracting Residue from Metallic Medical Components and Quantifying via Gravimetric Analysis[1]

This standard is issued under the fixed designation F2459; 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 the quantitative assessment of the amount of residue obtained from metallic medical components when extracted with aqueous or organic solvents.

1.2 This test method does not advocate an acceptable level of cleanliness. It identifies one technique to quantify extractable residue on metallic medical components. In addition, it is recognized that this test method may not be the only method to determine and quantify extractables.

1.3 Although these methods may give the investigator a means to compare the relative levels of component cleanliness, it is recognized that some forms of component residue may not be accounted for by these methods.

1.4 The applicability of these general gravimetric methods have been demonstrated by many literature reports; however, the specific suitability for applications to all-metal medical components will be validated by an Interlaboratory Study (ILS) conducted according to Practice E691.

1.5 This test method is not intended to evaluate the residue level in medical components that have been cleaned for reuse. This test method is also not intended to extract residue for use in biocompatibility testing.[2]

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

1.7 *This standard may involve hazardous or environmentally-restricted materials, operations, and equipment. 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:*[3]

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

G121 Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents

G131 Practice for Cleaning of Materials and Components by Ultrasonic Techniques

G136 Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction

3. Terminology

3.1 *Definitions:*

3.1.1 *ionic compounds/water soluble residue*—residue that is soluble in water, including surfactants and salts.

3.1.2 *non-soluble debris*—residue including metals, organic solids, inorganic solids, and ceramics.

3.1.3 *non-water soluble residue*—residue soluble in solvents other than water. Inclusive in this are oils, greases, hydrocarbons, and low molecular weight polymers. Typical solvents used to dissolve these residues include chlorinated or fluorinated solvents, or low molecular weight hydrocarbons.

3.1.4 *reflux system*—an apparatus containing an extraction vessel and a solvent return system. It is designed to allow boiling of the solvent in the extraction vessel and to return any vaporized solvent to the extraction vessel.

3.1.5 *reuse*—the repeated or multiple use of any medical component (whether labeled SUD or reusable) with reprocessing (cleaning, disinfection, or sterilization, or combination thereof) between patient uses.

3.1.6 *single use component (SUD)*—a disposable component; intended to be used on one patient during a single procedure.

3.1.7 *surface area*—the projected surface area of a part. This area does not include the internal porosity of parts with cancellous, porous, or wire structure.

[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

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[2] For extraction of samples intended for the biological evaluation of devices or materials, refer to ISO 10993-12 Biological Evaluation—Sample Preparation and Reference Materials.

[3] 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.

3.2 *Symbols:*

m_1 = weight of extraction vessel and component before extraction
m_2 = weight of extraction vessel, component, and solvent after extraction
m_3 = mass of clean beaker used to hold removed aliquot of extracted solution
m_4 = mass of beaker and aliquot of solution before drying
m_5 = mass of beaker and residue after evaporating solvent
m_6 = mass of new filter
m_7 = mass of filter following filtration and drying
m_a = mass of residue in removed aliquot
c_r = concentration of residue in solution
c_b = concentration of residue in blank solutions
m_r = mass of soluble residue in the overall extract, corrected for the blank runs
m_i = weight of insoluble debris
m_t = mass of soluble and insoluble residue
E = extraction efficiency

4. Summary of Test Method

4.1 This test method describes the extraction and quantitative analysis procedures used to detect and quantify extractable residue from metallic medical components. The residues are grouped into three categories: *(1)* water-soluble extractables; *(2)* non-water soluble extractables; and *(3)* non-soluble debris.

5. Significance and Use

5.1 This test method is suitable for determination of the extractable residue in metallic medical components. Extractable residue includes aqueous and non-aqueous residue, as well as non-soluble residue.

5.2 This test method recommends the use of a sonication technique to extract residue from the medical component. Other techniques, such as solvent reflux extraction, could be used but have been shown to be less efficient in some tests, as discussed in X1.2.

5.3 This test method is not applicable for evaluating the extractable residue for the reuse of a single-use component (SUD).

6. Apparatus

6.1 *Ultrasonic Bath*, for extraction. The bath must be large enough to hold an extraction beaker containing the medical component. This apparatus is used with the technique described in 11.5. Alternatively, an ultrasonic probe can be used with a bath.

6.2 *Solvent Reflux Extraction Assembly*, shown in Fig. 1. This assembly is composed of a vessel large enough to hold the medical component, and a water-cooled refluxing column. A heating manifold or hotplate stirrer capable of reaching the boiling point of the solvent is also included. This apparatus is

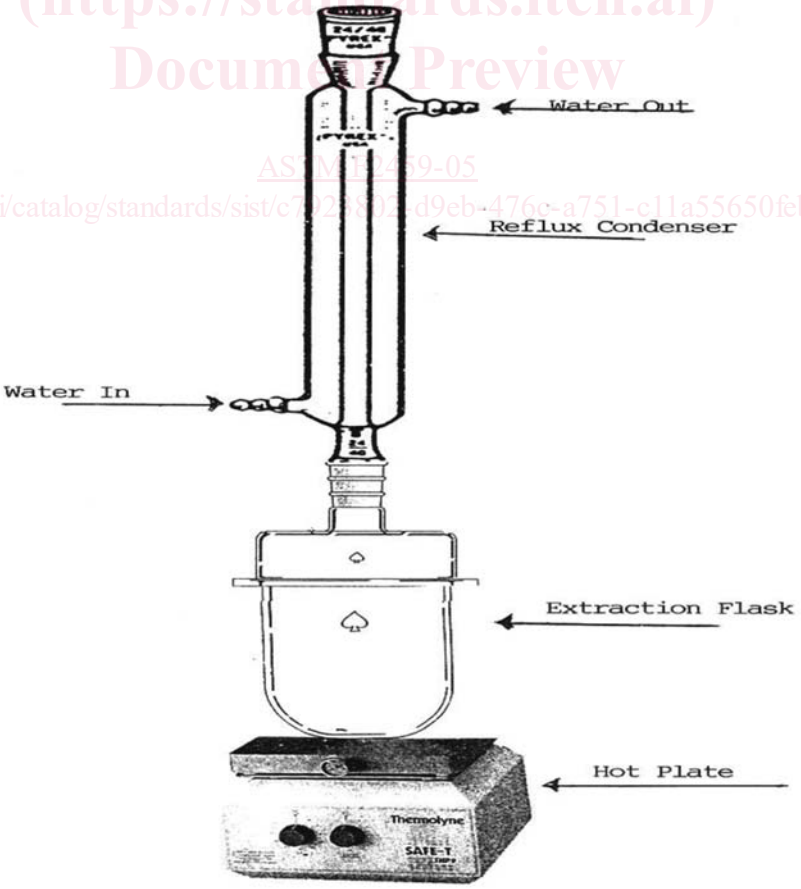

FIG. 1 Sample Solvent Reflux Extractor Assembly

used in the procedure described in 11.3. A Soxhlet extractor, as shown in Fig. 2, could be used as well using the procedure described in 11.3.

6.3 *Analytical Balance*, with 0.1 mg accuracy or better.

6.4 *Balance*, with accuracy of 10 mg of better and sufficient capacity to weigh the extraction beaker with the medical component and solvent combined.

6.5 *Glass Beaker and Extraction Vessel*, large enough to hold sufficient solvent to cover the medical component in the extraction vessel. Additionally, metal beakers could be used. Plastic beakers should not be used as low molecular weight residues could be extracted from the beakers.

6.6 *Desiccator*.

6.7 *Pipets*, for transferring liquid. Some solvents can leach extractable compounds from plastic pipets. Glass or metallic pipets are recommended for organic solvents.

6.8 *Aluminum Foil*, degreased in extraction solvent.

6.9 *Forceps, Tweezers, or Tongs*, cleaned with acetone or extraction solvent.

6.10 *Filtration Apparatus*, containing a removable 0.2 µm filter medium that is non-soluble in the extraction solvent.

7. Reagents and Materials

7.1 Each user needs to demonstrate solubility of all of their suspect sources of residue in the solvent(s) of choice. Several solvents may be required if more than one type of residue may be present on the component.

7.2 Spectroscopy or ACS-grade solvents should be used.

8. Hazards

8.1 Many organic solvents are toxic, flammable, or explosive and should be handled only with chemically protective laboratory gloves and used in a fume hood.

8.2 If sonication is used, the user should make sure that the solvent is not heated, directly or through sonication, to a temperature above the flash point of the solvent.

9. Sampling, Test Specimens, and Test Units

9.1 Metallic medical components should be taken in random groupings from different lots if available.

9.2 It is up to the user to determine the number of medical components that need to be used to establish known reproducibility.

9.3 It is up to the user to determine the number of test blanks that need to be used to establish known reproducibility.

9.4 Separate components should be tested for organic and aqueous extractions.

9.5 If a long medical component is cut, it is recommended that the original length and the cut lengths be recorded before the final cleaning operation for validation purposes. Individual cut lengths may be separately extracted and the results combined to provide a total residue value for the medical component. Cutting lubricants must be avoided in this procedure.

FIG. 2 Sample Soxhlet Extractor Assembly