Designation: F3203 – 19 (Reapproved 2023)

Standard Test Method for Determination of Gel Content of Crosslinked Polyethylene (PEX) Pipes and Tubing[1]

This standard is issued under the fixed designation F3203; 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 The gel content of pipe and tubing produced from crosslinked polyethylene plastics as described in Specification F876 and other pipe or tubing standards is determined by extracting with solvents such as xylenes. A test method for quantitative determination of gel content is described herein. The method is applicable to PEX pipe and tubing of all densities, including those containing fillers, and provides correction for the inert fillers present in some of those compounds.

1.2 Continuous extraction (see definition in Section 3) is used in this method to test the gel content of crosslinked polyethylene specimens. Continuous extraction when used for testing gel content has the advantages of decreased cost of testing, increased accuracy and consistency of results, and decreased test time. This is because extraction with a pure solvent is more efficient than extraction with a partially saturated solvent.

1.3 While extraction tests may be made on articles of any shape, this test method is applicable for determining the gel content of crosslinked polyethylene pipes and tubing.

1.4 This test method makes use of xylenes or alternative solvents. Alternative solvents either have lower toxicity than xylenes or allow decreased extraction times. The alternative solvents are also potentially beneficial from an economic and environmental viewpoint. **Xylenes are used for referee tests.**

1.5 The values stated in SI units are to be regarded as standard. The inch-pound units in brackets are for information only.

1.6 *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.*

1.7 *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

D1603 Test Method for Carbon Black Content in Olefin Plastics

D2765 Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics

D7567 Test Method for Determining Gel Content in Crosslinked Ethylene Plastics Using Pressurized Liquid Extraction (Withdrawn 2015)[3]

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

F876 Specification for Crosslinked Polyethylene (PEX) Tubing

2.2 *ISO Standard:*[4]

ISO 10147 Pipes and Fittings Made of Crosslinked Polyethylene (Pe-X) – Estimation of the Degree of Crosslinking by Determination of the Gel Content

3. Terminology

3.1 Terms as shown in Terminology D883 are applicable to this test method.

3.2 *Definitions of Terms Specific to This Standard:*

[1] This test method is under the jurisdiction of ASTM Committee F17 on Plastic Piping Systems and is the direct responsibility of Subcommittee F17.40 on Test Methods.

Current edition approved July 1, 2023. Published July 2023. Originally approved in 2018. Last previous edition approved in 2019 as F3202–19. DOI: 10.1520/F3203–19R23

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

***A Summary of Changes section appears at the end of this standard**

3.2.1 *continuous extractor, n*—test apparatus for performing a continuous extraction.

3.2.1.1 *Discussion*—Soxhlet, Knofler-Bohm, and Kumagawa extractors are examples of continuous extractors. A continuous extractor has three main components which are a boiling flask or vessel for the solvent, a condenser, and a siphon cup. There are various designs for the siphon cup; the Soxhlet, Knofler-Bohm, and Kumagawa designs are the most common.

3.2.2 *continuous extraction, n*—an extraction performed in a continuous extractor where a solvent (normally xylenes) is heated in a vessel and boils forming vapors, the vapors rise and condense on a condenser to form droplets, the droplets fall into a cup where one or more specimens are placed, there the solvent in the cup dissolves some of the un-crosslinked polyethylene in the specimens, and when the cup holding the specimens is filled with solvent, the solvent now containing some of the un-crosslinked polyethylene drains out through a siphon tube back into the vessel where it started. The solvent and the sample specimens in the cup are kept warm by solvent vapor rising toward the condenser. The extraction steps repeat automatically and continuously as long as heat is applied to the vessel holding the solvent. Because only the solvent evaporates and not the material dissolved in it, the concentration of un-crosslinked polyethylene in the boiling solvent increases, while the concentration of un-crosslinked polyethylene in the specimens continually decreases.

3.2.3 *gel content, n*—the percentage by mass of polymer insoluble in a specified solvent after extraction under the specified conditions.

4. Summary of Test Method

4.1 Specimens of the crosslinked ethylene plastic are prepared, weighed and then extracted using a heated extraction solvent in a continuous extractor for the time designated by the procedure. After extraction, the specimens are removed from the continuous extractor, dried, and weighed as directed. The gel content is calculated using the final and initial specimen weights as directed in the calculations section of this test method.

5. Significance and Use

5.1 Many important properties of crosslinked ethylene plastics vary with the gel content. Hence, determination of the gel content provides a basis for controlling production processes and a means of establishing the quality of finished products.

5.2 Extraction tests permit verification of the proper gel content of any given crosslinked ethylene plastic and they also permit comparison between different crosslinked ethylene plastics, including those containing fillers, provided that, for the latter, the following conditions are met:

5.2.1 The filler is not soluble in the solvent used in this method at the extraction temperature.

5.2.2 The amount of filler present in the compound either is known or can be determined.

5.2.3 Sufficient crosslinking has been achieved to prevent migration of filler during the extraction. It has been found that, at gel content above 30 %, the solvent remains clear and free of filler.

5.3 Since some oxidative degradation of the material and solvent may occur at the reflux temperature of the solvents, a suitable antioxidant is added to the solvent to inhibit such degradation.

5.4 This test method is normally used for specimens consisting of an equal representation of the entire cross section of the product, but may also be used to examine specific portions of a product for differences in extent of cross-linking when compared to either a product standard or another sample.

5.5 This test method is intended for testing crosslinked polyethylene compounds that are not hygroscopic. If compounds that are hygroscopic are tested using this method, specimen conditioning before and after extraction is required.

5.6 This test method differs from Test Methods D2765, ISO 10147 and Test Method D7567 which also describe procedures for determining the gel content of crosslinked polyethylene. It allows for the use of naphthenic hydrocarbon blend, isoparaffin solvent, or light aromatic solvent naptha as alternatives to xylenes. Xylenes are the only solvent allowed to be used for referee tests. The preferred method of sample preparation in this test method is to use a lathe to create thin ribbons of PEX material. This standard requires the use of a continuous extractor in order to provide consistent results and to allow for reliable solvent re-use. Specialized specimen holders are used to minimize variability resulting from loss of specimen particles.

NOTE 1—Pressurized extraction techniques have been found to yield useful results in a shorter time frame, however not all grades of PEX tolerate the elevated extraction temperatures without substantial degradation. For this reason pressurized extraction techniques are recommended for control tests only if it is possible to determine that the crosslinked matrix of the PEX does not break down at the temperature of extraction

6. Conditioning

6.1 *Conditioning*—Conditioning of the test specimens is not required, unless specified by the manufacturer of the material being sampled.

6.2 *Test Conditions*—Test conditions do not affect results, with the exception of atmospheric pressure. Conduct tests in a laboratory with atmospheric pressure greater than 80 kPa (less than 6000 ft above average sea level).

NOTE 2—The altitude above sea level of the lab will influence the atmospheric pressure, which in turn will affect the boiling temperature of the solvent used and the rate at which it will be able to extract un-crosslinked polyethylene from the matrix.

7. Apparatus

7.1 *Continuous Extractor,* of the following general type, as illustrated in Fig. 1. Further information regarding possible sources of components is included in the Appendix X3.

7.1.1 *Boiling Flask,* with a ground-glass joint. The flask may be either flat bottom or round bottom. For one or two