Designation: D8280 – 20

Standard Test Method for Determination of the Blooming of Brominated Flame Retardants onto the Surface of Plastic Materials by Ion Chromatography[1]

This standard is issued under the fixed designation D8280; 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 quantification of the blooming of brominated flame retardants onto the surface of plastic materials by an ion chromatographic method.

1.2 The test method involves recovery of the deposits on the surface of plastic materials, containing brominated flame retardants, on unaged specimens, on specimens aged at 70°C for 14 days and on specimens aged at 70°C for 35 days. These deposits are quantitatively analyzed to obtain the fraction of brominated flame retardant that has bloomed onto the surface.

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

1.4 *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.*

NOTE 1—There is no known ISO equivalent to this standard.

1.5 *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]

D573 Test Method for Rubber—Deterioration in an Air Oven

D883 Terminology Relating to Plastics

D3045 Practice for Heat Aging of Plastics Without Load

E176 Terminology of Fire Standards

2.2 *ISO Standards:*[3]

ISO 188 Rubber, vulcanized or thermoplastic—Accelerated ageing and heat resistance tests

3. Terminology

3.1 *Definitions of Terms:*

3.1.1 For definitions of terms used in this test method and associated with plastics refer to Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blooming, n*—the formation of a visible exudation or efflorescence on the surface of a plastic.

3.2.1.1 *Discussion*—Additives typically have a higher solubility in the polymer at processing temperature than at ambient temperature (since processing temperature is usually above ambient). Blooming occurs when, after cooling, a portion of the additive segregates out of the cooled polymer at ambient temperature and in some instances, migrates to the surface of the polymer.

3.2.2 *flame retardant, n*—a substance which, when added to a combustible material, inhibits flame spread of the resulting substance or material when exposed to flame impingement (Terminology E176).

3.2.2.1 *Discussion*—Flame retardants may be incorporated in plastics as additives (additive flame retardant) or as chemical groups in the base polymer by use of reactive intermediates in the polymerization process (reactive flame retardant).

4. Summary of Test Method

4.1 The test method presents a procedure for the quantification of the blooming of brominated flame retardants onto the surface of plastic materials.

4.2 The first stage involves the aging of two sets of samples of plastic materials at 70°C for 14 and 35 days, respectively, which are compared to a set of unaged materials.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

Current edition approved Jan. 1, 2020. Published January 2020. DOI: 10.1520/D8280-20.

[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.

NOTE 2—Test Method D573, Practice D3045, and ISO 188 provide guidance on heat aging of plastics or rubbers.

4.3 The second stage involves using a flag made from chromatography paper to sweep the deposits from the surface of both unaged and aged plastic materials.

4.4 The next stage is the analysis of the paper flags by the Schöniger[4] method. The Schöniger method involves the combustion of a sample in pure oxygen, followed by the absorption of the combustion products by a solution of sodium hydroxide.

NOTE 3—The Schöniger oxidation (also known as the Schöniger flask test or the oxygen flask method) is a method of elemental analysis developed by Wolfgang Schöniger in 1955. The test is typically conducted in an Erlenmeyer flask, or in a separatory funnel. It involves the combustion of a sample in pure oxygen, followed by the absorption of the combustion products by a solution of sodium hydroxide. It allows quantitative determination of halogens in a sample.

4.5 The next stage involves determining the concentration of bromide in the solution by means of ion chromatography.

4.6 The final stage is the calculation of the amount of bromine originating from the flame retardant that has bloomed onto the surface.

5. Significance and Use

5.1 This test method will allow the quantitative determination of the bromine originating from the flame retardant that has bloomed onto the surface after aging under specified conditions. Based on the known structure of the flame retardant used, the amount of the flame retardant that bloomed can also be calculated.

5.2 Section 14 contains some safety recommendations.

6. Test Equipment

6.1 An Ion Chromatograph equipped with a conductivity detector and an autosampler.[5]

6.2 An analytical column sized 250 × 0.46 mm and protected by a guard column sized 50 × 0.46 mm.[6]

6.3 An integrator or data station.

6.4 An apparatus that consists of a heavy-walled conical, deeply lipped, cupped 500 mL flask, fitted with a ground glass stopper to which is fused a test specimen carrier consisting of a heavy-gauge platinum wire and a piece of welded platinum gauze measuring approximately 1.5 × 2 cm.

6.5 An oven, capable of being controlled at 70ºC.

6.6 Ion chromatography 5 mL plastic vials.

6.7 Centrifuge 50 mL plastic vials.

6.8 Platinum specimen carrier for Schöniger Combustion.

6.9 Standard laboratory glassware.

6.10 Protective gloves

7. Reagents

7.1 *Sodium Carbonate,* analytical grade.

7.2 *Potassium Bromide,* analytical grade.

7.3 *Sodium Hydroxide,* analytical grade.

7.4 *Deionized Water,* 18.2 MΩ (Mili-Q).

7.5 *Cellulose chromatography paper,* (smooth surface, 0.18 mm thick, with a linear flow rate (water) of 13 mm per 3 minutes, in rolls of 3.0 cm × 100 m (Whatman grade No. 1 chromatography paper or equivalent).

7.6 *Oxygen Gas,* analytical grade.

8. Aging Procedures

8.1 The test specimens shall be plastic bars 125 ± 5 mm long by 13.0 ± 0.5 mm wide, and provided in the minimum thickness of 3.0 (−0.0 +0.2) mm. The edges shall be smooth, and the radius on the corners shall not to exceed 1.3 mm. Protective gloves shall be worn during the aging procedure and the gloves shall be replaced after every test.

8.2 A total of 15 test specimens shall be prepared for each material to be tested. The blooming is measured immediately after the preparation of the test specimens (time 0, first set of 5 test specimens) then on test specimens aged at 70ºC for 14 days (time 14 days; second set of 5 test specimens) and then on test specimens aged at 70ºC for 35 days (time 35 days; third set of 5 test specimens).

NOTE 4—The plastic materials to be tested are intended to be various formulations containing brominated flame retardants.

8.3 Hold the chromatography paper firmly against the top and bottom surface of the test bar and wipe the surface forward and backward. Repeat this action three more times, for a total of four sweeps, to remove the bloomed flame retardant from the surface of the unheated plastic bars immediately after the preparation of the specimen (time 0, first set). At each time point (0, 14 days and 35 days), two plastic bar test specimens (plastic bars) of each plastic material shall be analyzed to assess the amount of blooming. The procedure requires two test specimens for each set and three other test specimens need to be available for each set, in case the analysis needs to be repeated.

8.4 Use the chromatography paper described in Section 7to prepare 15 folded paper flags, each with approximate dimensions of 30 x 30 mm, with a flag stick of approximately 30 mm as shown in Fig. 1a.

8.5 Place the plastic test specimens into the folded paper flags as shown in Fig. 1b.

8.6 Sweep 4 times (twice upwards and twice downwards) the bloomed flame retardant from the surface of unheated plastic bars, immediately after the preparation of the specimen (time 0, first set).

8.7 Fold the chromatography paper with the bloomed material as shown in Fig. 1c through Fig. 1e.

8.8 Repeat the procedures in 8.6 and 8.7 on the same test bar, using a new paper flag. This results in the use of two paper

[4] Schöniger W, "Eine Mikroanalytische Schnellbestimmug von Halogenen in organischen Substanzen", *Mikrochemica Acta*43 (1), 123-129 (1955).

[5] A Dionex Ion Chromatograph (IC) 2100 series equipped with an AS-DV autosampler AS-DV is suitable for this purpose.

[6] An IonPac AS-9HC analytical column protected by an IonPac AG-9HC guard column is suitable for this purpose.