Designation: ~~D3596 – 13~~ D3596 – 14

Standard Practice for Determination of Gels (Fisheyes) In General-Purpose Poly(Vinyl Chloride) (PVC) Resins[1]

This standard is issued under the fixed designation D3596; 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 practice provides a quantitative measure of the gels remaining in a flexible vinyl compound processed from general-purpose poly(vinyl chloride) (PVC) resins under a prescribed set of working conditions.

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

1.3 *This standard does not purport to address all of the safety problems, 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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

3. Terminology

3.1 *General*—Definitions are in accordance with Terminology D883 and abbreviations with Terminology D1600, unless otherwise indicated.

4. Summary of Practice

4.1 A sample of PVC resin is mixed with calcium/zinc stabilizer, stearic acid, carbon black, and a plasticizer in a laboratory mixer. The dry blend is milled on a two-roll laboratory mill.

4.2 The milled sheet is press-polished and the gels counted using a bottom-lighted viewing box and a magnifying comparator.

5. Significance and Use

5.1 The gel (fisheye) in PVC resins is generally recognized as a hard particle of resin which will not fuse when the plastic mass is subjected to set conditions of hot processing. The number of unfused particles present is related to the conditions used. The presence of an excess of such particles is detrimental to many applications.

6. Apparatus

6.1 *Laboratory Mixer,* with stainless steel mixing bowl.

6.2 *Two-Roll Mill,*152.4 by ~~304.8 mm (6 by 12 in.), 263.5 mm (10.5 in.)~~ 304.8 mm (6 by 12 in.), 263.5 mm (10.5 in.) between guides, differential speed ratio 1.40 ± 0.04, fast roll speed 34 ± ~~2 rpm,~~ 2 rpm, with an adjustable temperature range of ~~120–205°C (250–400°F).~~120–205 °C (250–400 °F).

NOTE 2—If the mill available does not meet these criteria, the specifications of the mill used should be included in the report.

6.3 *Surface Pyrometer*

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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[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.

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

6.4 *Balance,* 0.1-g sensitivity.

6.5 *Hydraulic Press* having platens at least 203 by 203 mm (8 by 8 in.), rated at a minimum of 140 MPa (20 000 psi) ram pressure on a 100-mm (4-in.) ram, which has the capacity for heating to 163 ± ~~3°C~~3 °C (325 ± ~~5°F)~~5 °F) on the pressing surfaces and equipped with cooling water.

6.6 *Timer,* capable of being read in seconds.

6.7 *Cotton Gloves.*

6.8 *Mill Knife.*

6.9 *Plates or Mold,* two 203 by 203-mm (8 by 8-in.) chrome-plated ferro plates, or a chrome-plated mold with a 152 by 152 by 6.4-mm (6 by 6 by 0.25-in.) cavity.

6.10 *Backing Plates,* two 203 by 203 by 6.4 mm (8 by 8 by 0.250 in.).

6.11 *Brass Shims,* four, 0.51 by 13 by 203 mm (0.020 by 0.5 by 8.0 in.).

6.12 *Magnifying Comparator (optional),* equipped with a transparent reticule (scale) showing circles from 0.03 to ~~1.27 mm~~ 1.27 mm (0.001 to ~~0.050 in.)~~ 0.050 in.) in diameter.

6.13 *Bottom-Lighted Viewing Box.*

7. Materials

7.1 *PVC Resin*

7.2 *Plasticizer*

7.2.1 *Plasticizer Type*—The plasticizer selected shall represent the plasticizer type commonly used with the resin being tested. Less solvating plasticizers will yield a higher gel count.

7.2.2 *Plasticizer Level*—The plasticizer usage level shall yield a test compound with approximately the same shore hardness as the compound that will be based on the resin being tested.

7.3 *Stearic Acid*

7.4 *Liquid Calcium/Zinc Stabilizer*

7.5 *Carbon Black*

8. Procedure

8.1 Weigh 200 g of the sample resin, 4.0 g liquid calcium/zinc stabilizer, 0.5 g stearic acid, ~~0.6g~~0.6 g carbon black, and plasticizer into the stainless steel mixing bowl. Blend the ingredients in the laboratory mixer at ~~23 ± 2°C (73 ± 3.6°F)~~ 23.0 ± 2.0 °C (73.0 ± 3.6 °F) for 3 min.

NOTE 3—Other mixers capable of yielding a uniform dispersion can be employed.

8.2 Set the mill temperature so the dry blend fuses and bands on the front roll of the mill within 45–90 seconds and check to determine that the mill rolls and guides are clean.

8.2.1 Set the mill rolls to produce a milled sheet with a thickness of 0.64 ± 0.05 mm (0.025 ± 0.002 in.).

NOTE 4—If the mill cannot be set to give the specified thickness set the rolls as closely together as possible and report the sheet thickness with the data. If the mill opening between the rolls is in excess of ~~0.64 mm (0.025 in.)~~ 0.64 mm (0.025 in.) the reduced working of the stock can result in an apparent higher gel (fisheye) count for the sample.

8.2.2 Place a clean sheet of paper beneath the rolls. With the mill running, transfer ~~150 g~~ 150 g of the dry blend (produced in 8.1) to the mill and immediately start the timer. Work the stock continuously by making a cut from one side to within about ~~25 mm (1 in.)~~ 25 mm (1 in.) of the opposite side. Use the mill knife to peel off as much of the stock as possible without completely removing it, fold the strip over and feed it back into the mill. During the first 0.5 min, add the initial droppings back into the stock; discard subsequent droppings. Cut and fold the stock after ~~1.5 min~~ 1.5 min and every ~~0.5 min~~ 0.5 min thereafter. Mill for a total of 5 ± ~~0.1 min~~ 0.1 min from start of test.

NOTE 5—Due to differences between mills, it will be necessary to adjust the amount of dry blend used to that weight which will band on the roll and produce a 9.5-mm (0.375-in.) diameter (pencil size) rolling bank. Use a clean-up batch to determine the correct amount. Report the weight of dry blend used.

8.3 Cut a 152 by 152-mm (6 by 6-in.) section from the milled stock.

8.3.1 Place the section cut from the milled sheet between the two chrome-plated ferro plates, chrome side to the specimen, then put the chrome ferro plates and the specimen between the backing plates and insert in a press at 163 ± ~~3°C~~3 °C (325 ± ~~5°F).~~5 °F). Preheat for 0.5 min with the press closed at zero pressure. A chrome-plated mold can be used instead of the plates.

8.3.2 After preheating, apply ram pressure for 2 min. Use enough pressure to produce a sheet thickness of 0.5 ± 0.03 mm (0.020 ± 0.001 in.). At the end of the 2 min, turn the heat off and the cooling water on. Allow the press to cool to room temperature under pressure. Release the pressure; remove the plates from the press and carefully remove the pressed sheet specimen.