



Standard Practice for Determination of Gels (Fisheyes) In General-Purpose Poly(Vinyl Chloride) (PVC) Resins¹

This standard is issued under the fixed designation D 3596; 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 are no ISO standards covering the primary subject of this practice.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1249 Specification for Octyl Ortho-Phthalate Ester Plasticizers²

D 1600 Terminology for Abbreviated Terms Relating to Plastics²

3. Terminology

3.1 *General*—Definitions are in accordance with Terminology D 883 and abbreviations with Terminology D 1600, unless otherwise indicated.

4. Summary of Practice

4.1 A sample of PVC resin is mixed with calcium stearate and di-2-ethylhexyl phthalate plasticizer (DOP) in a laboratory mixer. After mixing, the dry blend is milled on a two-roll laboratory mill; carbon black dispersion is added during the milling.

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*,³ with stainless steel mixing bowl.

6.2 *Two-Roll Mill*, 152.4 by 304.8 mm (6 by 12 in.), 263.55 mm (10.5 in.) between guides, differential speed ratio 1.40 ± 0.04 , fast roll speed 34 ± 2 rpm, suitably heated to maintain roll temperature of $154 \pm 3^\circ\text{C}$ ($310 \pm 5^\circ\text{F}$).

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* calibrated in the range of 154°C (310°F) and a leaf pyrometer calibrated in the range of 163°C (325°F).

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 may be heated to $163 \pm 3^\circ\text{C}$ ($325 \pm 5^\circ\text{F}$) 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*,⁴ equipped with a transparent reticule (scale) showing circles from 0.03 to 1.27 mm (0.001 to 0.050 in.) in diameter.

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. Current edition approved October 15, 1992. Published December 1992. Originally published as D 3596 – 77. Last previous edition D 3596 – 88.

² *Annual Book of ASTM Standards*, Vol 08.01.

³ The Hobart Model N-50 is satisfactory.

⁴ A Comparator supplied by Finescale Co. of Los Angeles, CA, equipped with a No. 128 reticule has been found suitable for this application.

6.13 Bottom-Lighted Viewing Box.⁵

7. Materials

- 7.1 *PVC Resin.*
- 7.2 *DOP, Specification D 1249, Type 1.*
- 7.3 *Calcium Stearate.*⁶
- 7.4 *Carbon Black Dispersion.*⁷

8. Procedure

8.1 Weigh 300 g of the sample resin, 9 g of calcium stearate, and 150 g of DOP into the stainless steel mixing bowl. Blend the ingredients in the laboratory mixer at $23 \pm 2^\circ\text{C}$ ($73 \pm 3.6^\circ\text{F}$) for 1 min at the No. 1 speed setting and at the No. 2 speed for 2 min.

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

8.2 Adjust the mill temperature to $154 \pm 3^\circ\text{C}$ ($310 \pm 5^\circ\text{F}$) 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.) the reduced working of the stock can result in an apparent higher gel (fisheye) count for the sample.

8.2.2 Weigh 0.5 g of carbon black dispersion onto a piece of weighing paper.

8.2.3 Place a clean sheet of paper beneath the rolls. With the mill running, transfer 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.) 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. After 1 min add the carbon black dispersion across the surface of the stock with a spatula. Cut and fold the stock after 1.5 min and every 0.5 min thereafter. Mill for a total of 5 ± 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 $163 \pm 3^\circ\text{C}$ ($325 \pm 5^\circ\text{F}$). 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.

8.4 Place the 152 by 152-mm (6 by 6-in.) press polished sheet on the view box and count the gel particles using the magnifying comparator. Classify the gels (clear unpigmented areas) as follows:

8.4.1 *Gel Size A*, gels greater than or equal to 0.5-mm (0.020-in.) diameter.

8.4.2 *Gel Size B*, gels greater than or equal to 0.4-mm (0.015-in.) diameter.

8.4.3 *Gel Size C*, gels greater than or equal to 0.3-mm (0.01-in.) diameter and less than 0.4-mm (0.015-in.) diameter.

9. Report

9.1 *A Gels*, count/232 cm² (count/36 in.²). *B Gels*, count/232 cm² (count/36 in.²). *C Gels*, count/232 cm² (count/36 in.²),

9.2 Mill specifications if different from the specifications listed in the procedure,

9.3 Thickness of milled sheet if other than specified in 8.2.1,

9.4 Amount of dry blend used if other than 150 g, and

9.5 Thickness of pressed sheet if other than specified in 8.3.2.

10. Precision and Bias ⁸

10.1 Interlaboratory evaluation by five laboratories yielded seven sets of determinations. Analysis indicated the following values of precision of count at a probability level of $P = 0.95$.

10.1.1 *Gel Size A*, ± 1 .

10.1.2 *Gel Size B*, ± 5 .

10.1.3 *Gel Size C*, ± 12 .

10.2 Since no absolute method of gel count is available, no statement of bias can be prepared.

11. Keywords

11.1 fisheyes; gels; poly(vinyl chloride) resin

⁵ Instruments for Research and Industry of Cheltenham, PA, glow box Model 11-17, or equivalent.

⁶ Calcium stearate (Synpro 24-46), supplied by Synthetic Products Co., Cleveland, OH, or equivalent.

⁷ Stan-Tone 90-PC-01 Furnace Medium Jet Black, Harwick Chemical Corp., Akron, OH.

⁸ Supporting data are available from ASTM Headquarters. Request RR:D20-1106.

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