Designation: D7486 – 14

Standard Test Method for Measurement of Fines and Dust Particles on Plastic Pellets by Wet Analysis[1]

This standard is issued under the fixed designation D7486; 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 measures the amount of fine particles adhered on plastic pellets or granules in which they are commonly produced and supplied. The lower limit of this test method is restricted only by the porosity of the filter disc used to capture the particle size being quantified.

1.2 The wet analysis technique allows for separation and collection of statically charged particles by liquid wash and filtration methods. This must be performed under standard laboratory conditions.

1.3 The values stated in SI units are to be regarded as standard.

1.4 This test method describes an essential practice to check the quality of plastics once the production cycle is terminated and to evaluate the performance of pellet cleaning systems or of the special pneumatic conveying systems for the distinct size fractions below 500 micron only.

1.5 *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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D1921 Test Methods for Particle Size (Sieve Analysis) of Plastic Materials

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 *FEM Standards:*[3]

FEM 2482 Test Method to Determine Content of Fines and Streamers in Plastic Pellets.

3. Terminology

3.1 *Definitions*—For definitions of terms associated with plastic materials, see Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *fines*—fines (Dust) are small particles of plastic which have been torn away from the original pellet by contact with a rough surface and friction at high conveying velocities. They are defined as the particle fraction with a particle size up to 2000 µm.

3.2.2 *streamers*—strings of plastics of various lengths that are created when the resin travels through pneumatic conveying lines at high velocity. Streamers are also known as angel hair, foil, floss, film or snake skin.

4. Summary of Test Method

4.1 Fines or streamers, or both, are generated in polymer resins during pneumatic conveying. Fines and streamers are known to cause problems in plastic producing and processing plants by clogging the transporting and procession lines. On finished products, fines cause the formation of gel, contamination, surface imperfection and weak spots.

4.2 The streamer content for a particle size considerably larger than the pellet diameter are determined by dry screening according to Test Method D1921.

4.3 The fundamental principle used in this document is based upon the condition that statically charged fines normally adhering to plastic pellets be removed by rinsing the test sample with a washing liquid, causing fines in the sample to wash away into a container. These particles are then poured into a filter funnel to separate the fines from the washing liquid. The weight of the filter paper is accurately measured before and after the collection process, and used to calculate the PPM level for that sample.

[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.

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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.

[3] Available from European Federation of Materials Handling, Diamant Building, 80 Boulevard Auguste Reyers B, 1030 Brussels, Belgium, http://www.fem-eur.com.

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

TABLE 1 Comparison of Typical Particle Sizes Measured

Type	Lower Limit	Upper Limit	Test Method
D1921	500 µm	2000 µm	dry
D7486	1.6 µm[A]	500 µm	wet

[A]The lower limit is set by the porosity of the filter disk.

4.4 Wet analysis quantifies the amounts of dust with particle sizes typically from 1.6 to 500 microns.

5. Significance and Use

5.1 Molding and extruding plastic pellets require dust free, dry pellets to prevent processing problems. Plastic producers try to remove the dust and streamers with dust removal systems prior to packaging and loading. How to accurately measure dust and streamer content in plastic pellets is an important quality control issue.

5.2 Particle size analysis is used to determine a percentage of particle size distribution from a representative sample of the whole. In terms of size analysis concerning plastic pellets, sieving is used to determine the dust content in the range of 500 to 2000 micron. Test Method D1921, Test Method B, is used to determine this type of particle sizing.

5.3 After dry sieve analysis, particles smaller than 500 microns need to be analyzed by wet method. A fresh sample shall be used for wet analysis. This test method allows washing down the fines attached to the pellets by electrostatic forces.

5.4 The wet analysis provides accurate quantification of small to large amounts of fines, negating static effects, and eliminating detrimental effects of mechanical agitation. A wet analysis must be employed to accurately quantify lower PPM dust levels in plastic pellets.

6. Interferences

6.1 Filtration paper has to be made of glass microfiber material. Paper filter fibers swell, reducing filtration speed and increasing filtration time.

6.2 Glass microfiber filters are fragile. Care must be taken when handling the wet filter to keep tears from being produced.

6.3 Some plastics contain certain additives that dissolve in washing liquids. This creates a particulate cloud of the washing liquid, clogs the filter paper, and gives inaccurate weight results.

6.4 When analyzing material that reacts with heat (low melting temperature), a lower drying temperature is required.

6.5 Too much material on a sieve causes mass blinding. Pellet bed depth has to be kept to no more than two pellets deep.

6.6 Replace the water and clean supply hoses on a regular basis. Algae or slime can develop and skew test results.

6.7 Discard sieves if the wire-cloth is damaged or no longer taut, as they no longer conform to Specification E11.

6.8 Use a non-stick, high temperature surface for the oven racks. The glass microfiber filter discs stick to the metal oven rack during the drying process.

7. Apparatus

7.1 The function of the apparatus (see Fig. 1) is to separate all fines below 500 micron from the pellets of the product sample to be tested. The apparatus runs with a washing liquid in recirculation mode.

7.2 The apparatus consists of a wash beaker with spray hose and nozzle, a filter funnel, a water tank reservoir with drain, a pump with throttle valve and flow meter, and connecting piping. The entire set-up consists of the following equipment.[4]

7.2.1 *Balance,* with a capacity of 500 g or higher, sensitive to 0.1 mg.

7.2.2 *Scale,* with a capacity of 1200 g or higher, sensitive to 0.1 g.

7.2.3 *Oven,* gravity convection, controlled up to 100 ± 5°C.

7.2.4 *Wire-Cloth Sieve,* 8-in. or 200-mm, full height, conforming to the requirements of Specification E11, 500 micron.

7.2.5 *Filtration Disc,* glass microfiber, diameter 90 mm, porosity 0.7 to 2.7 micron, thickness , 260 to 420 micron, tensile range, 5.5 to 8.9 N/15 mm.

7.2.6 *Filter Funnel Assembly,* 3-piece, borosilicate glass, able to hold a 90-mm filter disc.

7.2.7 *Filtering Flask,* 1000-mL, borosilicate glass, to connect to filter funnel.

7.2.8 *Beaker,* 1500-mL, borosilicate glass, to hold washed test sample.

7.2.9 *Funnel,* 260-mm. top O.D., to hold sieve during test sample washing.

7.2.10 *Nozzle,* flat fan spray, for washing test sample at line pressures below 2.8 Bar.

7.2.11 *Tweezers or Forceps,* blunt nose, for handling filtration disc.

7.2.12 *Plastic Sheet,* non-stick surface, polytetrafluoroethylene (PTFE), 1-mm thick, to keep filtration discs from sticking to oven rack during drying process.

7.2.13 *Aspirator,* to allow flushing of sample and support return flow of water back to the storage tank.

7.2.14 *Water Storage Tank,* with drain valve and bottom outlet for recirculation loop. All inlet and outlet openings must be of sufficient size to enable the necessary liquid flow rates to be achieved.

7.2.15 *Wash Bottle,* with safety label, to rinse 1500-mL beaker and filter funnel assembly.

8. Test Specimen

8.1 Three test specimens of 100 g each have to be taken from a sample size of approximately 50 litres.

NOTE 2—The preferred sample size is 100 g but can be up to 200 g if the fines are low.

8.2 For samples taken during any part of the conveyance process, a larger sample size (test unit) can be obtained in order

[4] The apparatus uses standard laboratory equipment. However, the apparatus is also available commercially. The sole source of supply of the apparatus, FineAnalyzer, known to the committee at this time is by Pelletron Corporation, 499 Running Pump Rd., Lancaster, PA 17601, http://www.pelletroncorp.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

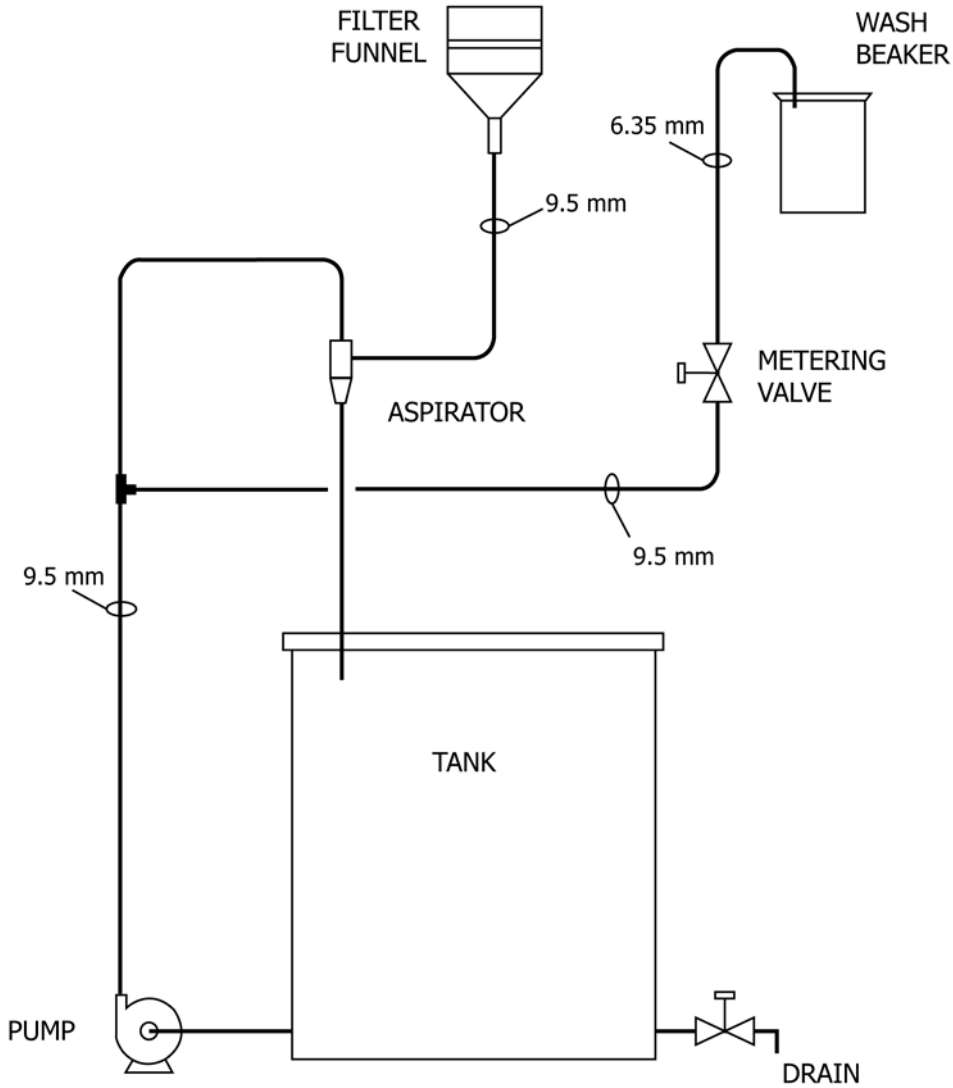

FIG. 1 Test Equipment for Determining Fines Content in Plastic Pellets

to collect the test specimen. When ready to perform the test, use a small scoop or cup to retrieve the test specimen from the approximate center of the test unit. With the beaker, funnel and sieve on the scale, pour the desired amount of material into the sieve. Any contaminate that remains inside the cup, or on the inner surfaces of the scoop, has to be brushed into the sieve pan, because it belongs to the test specimen.

8.3 For samples taken from the end process (packaged or containerized material), use the following procedure.

8.4 Measure out specimen into a small, resealable, plastic bag. Any contaminate that remains inside the cup, or on the inner surfaces of the scoop, has to be brushed into the bag, because it belongs to the test specimen.

8.5 For large containers of material, take test specimens at various levels of the container to measure the contaminate distribution.

9. Calibration

9.1 Calibrate instruments using procedures recommended by the equipment manufacturer.

9.2 Standard lab practices have to be observed. Balances and scales have to be periodically calibrated to maintain consistent results.

9.3 Sieves have to be periodically tested or verified for accuracy or replaced as necessary in order to maintain accurate results.

10. Procedure

10.1 The selection of a suitable washing liquid is dependent on the characteristics of the test product. Unless otherwise indicated, use filtered, deionized, demineralized, or distilled water. In the case that water interacts with the plastic to be tested, use other washing solution instead. In case another washing liquid is used, it has to be ensured that the chosen liquid does not leave any residue after drying the filters, nor change the characteristics of the product, by decomposing or dissolving. Known problems concerning this point are the combination of waxy products or XLDPE with ethanol as a solvent. The washing liquid must not affect any parts of the measuring device with which it is in contact. The use of any

other liquids, as a substitute for water, has to be thoroughly researched to define associated hazards. Modifications to the equipment, to allow the use of a different medium, must meet applicable Occupational Safety and Health Administration (OSHA) safety requirements.

10.2 Fill the water tank with clean water. If not previously done, clean the tank so that NO particle remains. Ensure all components are properly connected. Assemble the filter funnel assembly. For initial use of the apparatus, flush the system by circulating water through the system with a filter in place. To complete this, position the rinse tube over the filter collection unit and turn the pump on. Adjust the level control so that the water level in the filter collection sleeve is maintained at approximately half full and circulate water for 10 to 20 minutes at a flow rate of 250 ± 50 mL/min. After this initial flush, discard all of the water and the filter and refill the system. Turn on the oven and obtain a stable inside temperature of about 100°C.

10.3 Weigh a dry disc on the balance. Place the filter in the filter funnel assembly.

10.4 Place the sieve into the funnel, and place this assembly on top of the 1500-mL beaker. Add the test specimen to the sieve, ensuring all the fines in the specimen container are rinsed into the sieve.

10.5 Rinse the sample with a strong spray of liquid at 500 ± 50 mL/min. Make sure to evenly agitate and wash all the pellets. When the beaker is almost filled, pour the liquid into the filter funnel to start the filtration process.

10.6 Using another beaker, continue the washing process one-quarter beaker at a time. Check each quarter beaker for visible fines or particles. When no more are observed, the rinse process is complete. When emptying the beaker contents, rinse the beaker into the filter funnel to ensure capture of all fines. When all the liquid has been filtered, rinse the sides of the filter funnel to ensure all are on the filter disc. To remove the filter from the filter funnel, remove the clamps and the reservoir. If any fines are on the reservoir body, use the wash bottle to rinse the fines onto the filter disc, ensuring no over spillage. Use the blunt nose forceps to carefully fold the filter disc in half, and then in half again. Place the removed filter disc into the oven and dry the disc for at least one hour. After one hour, remove the filter disc from the oven and place on the balance for weight calculation. Allow the balance to stabilize and record the weight.

TABLE 2 Repeatability of Polystyrene

	Fines	
	Average, PPM	Coefficient of Variation, %
Polystyrene, as is	802	1.5
Polystyrene, de-dusted	20	5.6

NOTE 3—One hour has been found adequate for most samples. Use other time if necessary.

11. Calculation or Interpretation of Results

11.1 Calculate the weight of the collected fines on the filter disc(s) by subtracting the tare weight from the final filter weight. Divide the contaminate weight by the test specimen weight.

11.2 Multiply the calculated number by 1,000,000 to obtain the PPM of the sample. For Example: Test Specimen is 100.0 g, and Fine Weight is 0.0045 g. Calculation: (0.0045 g/100.0 g) × 1,000,000 = 45 PPM.

11.3 To express the results in terms of percent passing, use the following calculation: Test Specimen is 100.0 g, Contaminate Weight is 0.0045 g, and Sieve Size Used is 500-micron. Calculation: (0.0045 g/100.0 g) × 100 = 0.0045 % passing.

11.4 Take the average of three specimens to determine the final result.

12. Report

12.1 A report indicates material test method and standard used, test specimen size, sieve size, and calculated result.

12.2 Optional description of the material type, trade name, manufacturer, bulk density, pellet size, pellet shape, and an electronic photo of the test sample.

12.3 Number of specimens tested.

12.4 Date of test and person performing the test.

13. Precision and Bias

13.1 Repeatability study was conducted on a polystyrene sample. The sample was tested as is and tested again after being passed through a de-duster (see Table 2).

14. Keywords

14.1 dust particles; fines; plastics; wet analysis

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D7486 – 08(2013)$^{\epsilon 1}$) that may impact the use of this standard. (August 1, 2014)

(1) Changed English units to SI units in Fig. 1.