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.

Designation: D7681 − 11 (Reapproved 2021)

Standard Test Method for Measuring Gradation of Glass Spheres Using a Flowing Stream Digital Image Analyzer[1]

This standard is issued under the fixed designation D7681; 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 determination of the gradation (size distribution) of glass spheres used in pavement marking systems using a Flowing Stream Digital Analyzer. Typical gradations for pavement marking systems are defined in ranges from Type 0 through 5 in AASHTO M247-08.

1.2 This test method provides for the presentation of the size data in a variety of formats to the requirements of the agency pavement marking material specification. For most specifications the standard format is to present the size data as "Percent Retained" or "Percent Passing" relative to a series of standard US sieve sizes.

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.*

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]

B215 Practices for Sampling Metal Powders

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

2.2 *AASHTO Standards:*[3]

AASHTO M247-08 Standard Glass Beads Used in Traffic Markings

3. Terminology

3.1 *Definitions:*

3.1.1 *flowing stream digital image analyzer, n*—a computer controlled particle size analyzer employing a high resolution digital imaging device and computer image processing software to do photo optical single particle counting and particle size analysis.

3.1.2 *gradation of glass beads, n*—the measurement of the size (diameter) of glass beads and their subsequent presentation in ranges between ASTM standard sieve sizes and/or micron designation starting with the largest to the smallest; the ranges are listed as "percent passing" and "percent retained."

4. Summary of Test Method

4.1 The glass particles are run through a flowing stream digital image analyzer, a measuring system for determining the gradation (size distribution) of dry, free flowing and harmless bulk products. The total recommended measuring range is between 100 µm and 2.36 mm. The operating test method uses photo optical single particle counting technology for the image processing. The measurement time depends on the quantity of material to be measured, the width of the metering feeder and the mean grain size. The quantity of material to be measured depends on the grain size and the width of the metering feeder. Typical measuring times are approximately 2 to 10 min.

5. Significance and Use

5.1 The gradation (size distribution) of glass beads has a significant influence on the retroreflective efficiency of a pavement marking system.

5.2 This test method is for the characterization of the gradation (size distribution) of glass beads for the purpose of

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

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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 American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

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compliance testing against standard specification for glass beads in pavement marking applications.

5.3 While there are potential industrial applications for this test method beyond the measurement of gradation (size distribution) of glass beads for pavement markings, those are beyond the scope of this standard.

6. Apparatus

6.1 *Typical Instrument Operating Conditions (Fig. 1):*

6.1.1 *Environmental Temperature*—10 °C...40 °C.

6.1.2 *Air Humidity*—80 % maximum relative humidity at temperatures up to 30 °C, linear decrease to 50 % maximum relative humidity at a temperature of 40 °C.

6.1.3 *Height of Installation and Operation*—Maximum 3000 m above sea level.

6.1.4 *Installation Location*—Place the particle analyzer on a firm, horizontal, vibration free surface.

6.1.5 *Light Conditions*—Avoid strong direct external light on the particle measurement shaft or on the cameras.

6.1.6 This test method is designed for indoor or outdoor use as prescribed by the manufacturer's design and instructions.

7. Hazards

7.1 *General Safety Information:*

7.1.1 Operate the instrument in accordance with the manufacturer's recommendations following all required safety precautions.

8. Sampling, Test Specimens, and Test Units

8.1 Obtain a minimum of approximately 50 ± 5 g specimen of the glass beads to be tested for gradation (size distribution). For larger sizes of glass spheres, such as Type III and larger, whose gradation is defined in AASHTO M247-08, 75 to 125 g samples shall be used.

8.2 In order to obtain representative samples when sampling from packaged containers, blenders or storage tanks, methods outlined in Practices B215 shall be followed.

9. Preparation of Apparatus

9.1 Follow the manufacturer's instructions for the particle analyzer being used.

10. Calibration and Standardization

10.1 The particle analyzer, in most cases, will be calibrated by the manufacturer prior to shipping. Re-calibration might become necessary occasionally, for example, after the transportation of the instrument or if required by quality management regulations. In this case, follow the calibration procedures as outlined in the manufacturer's instruction manual.

11. Conditioning

11.1 *Sample Preparation:*

11.1.1 Use a sample splitter, if necessary, to reduce the amount of sample to the appropriate size.

11.1.2 Pour entire glass bead sample into the glass beaker or suitable container.

11.1.3 Assure glass beads are moisture free and free flowing.

NOTE 1—Check with instrument manufacturer for suggestions on how to best set up any software that comes with their instrument. Setting up the instrument software properly will speed up any glass sphere gradation measurements and allow for meaningful reports.

12. Procedure

12.1 Load the sample into the feeder of the flowing stream digital image analyzer. The analyzer software allows the user to carry out his measurements quickly and without error. All measuring and analysis parameters are determined initially and set into the computer program. Different task files are created for different specifications.

NOTE 2—When assigning sieve ranges to be used in a task file, the user must use the ASTM mesh sieve choice, not the W. S. Tyler mesh. The quantity of the material to be measured has to be placed into the funnel of the metering feeder. The material handling mechanisms must not restrict or segregate product flow in any way that allows for a non-representative flow of product through the measurement zone.

12.2 After the task file has been defined only a minimal number of operative steps are required for carrying out a measurement. They are: *(a)* filling a quantity of the material to be measured into the funnel to the metering feeder, *(b)* calling the measurement and choosing the task file, *(c)* confirming the suggested comments or entering new comments, *(d)* starting the measurement, and *(e)* reading the result or printing a record. The measured result is available a few moments after

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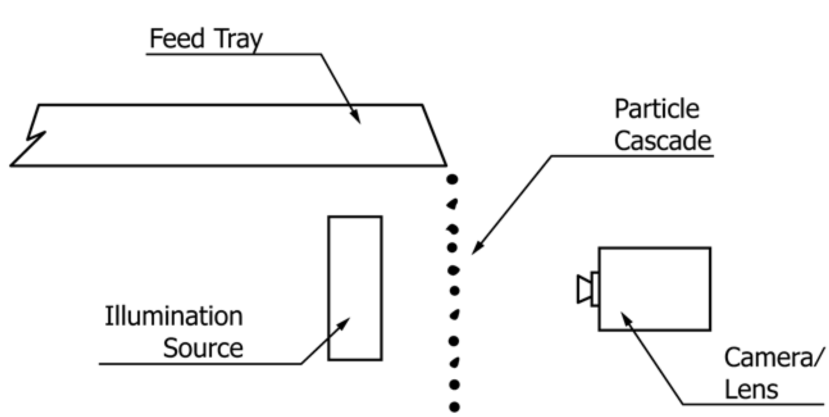

FIG. 1 Typical Apparatus

the measurement is completed and can be displayed in many forms, and be printed and saved with the help of the PC.

13. Report

13.1 Report the percentage of particles in each size classification.

14. Precision and Bias[4]

14.1 *Precision*—A round-robin study has been used to generate a precision statement.

14.1.1 The precision of this test method is based on an interlaboratory study of D7681, Standard Test Method for Measuring Gradation of Glass Spheres Using a Flowing Stream Digital Image Analyzer, conducted in 2008. A total of six laboratories participated in this study. Each of the labs was asked to report duplicate test results for three different materials. Every "test result" reported represents an individual determination. Except for instances where laboratories reported fewer replicates or materials, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D01-1157.

14.1.2 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.1.2.1 Repeatability limits are listed in Tables 1-12.

14.1.3 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

14.1.3.1 Reproducibility limits are listed in Tables 1-12.

14.1.4 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

14.1.5 Any judgment in accordance with statements 14.1.2 and 14.1.3 would normally have an approximate 95 % probability of being correct, however the precision statics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the listed precision limits as general guides, and the associated probability of 95 % only as a rough indicator of what can be expected.

14.2 *Bias*—At the time of the study, there was no reference made for bias for this test method.

15. Keywords

15.1 glass beads; gradation; particle size analyzer; sieve size

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1157. Contact ASTM Customer Service at service@astm.org.

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TABLE 1 Sieve Size 20 – Average Grams Retained

Material	Average[A] Grams Retained	Standard Deviation	Repeatability Standard Deviation	Repeatability Limit	Reproducibility Standard Deviation	Reproducibility Limit
	$\bar{x}$	$S_{\bar{x}}$	S_r	r	S_R	R
Type I	0.190	0.044	0.020	0.046	0.057	0.129
Type III	0.291	0.258	0.104	0.269	0.291	0.752
Type V	0.624	0.298	0.057	0.301	0.161	0.843

[A] The average of the laboratories' calculated average.

TABLE 2 Sieve Size 20 – Average Grams Passing

Material	Average[A] Grams Passing $\bar{x}$	Standard Deviation $S\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r	Reproducibility Standard Deviation S_R	Reproducibility Limit R
Type I	99.810	0.044	0.020	0.046	0.057	0.129
Type III	99.709	0.258	0.104	0.269	0.291	0.752
Type V	99.376	0.298	0.057	0.301	0.161	0.843

[A] The average of the laboratories' calculated average.

TABLE 3 Sieve Size 30 – Average Grams Retained

Material	Average[A] Grams Retained $\bar{x}$	Standard Deviation $S\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r	Reproducibility Standard Deviation S_R	Reproducibility Limit R
Type I	5.917	1.169	0.268	1.184	0.749	3.317
Type III	23.987	7.750	8.235	9.694	23.057	27.143
Type V	8.728	1.071	0.119	1.074	0.333	3.008

[A] The average of the laboratories' calculated average.

TABLE 4 Sieve Size 30 – Average Grams Passing

Material	Average[A] Grams Passing $\bar{x}$	Standard Deviation $S\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r	Reproducibility Standard Deviation S_R	Reproducibility Limit R
Type I	93.893	1.209	0.271	1.224	0.759	3.428
Type III	81.851	9.625	10.318	12.078	28.890	33.817
Type V	90.648	1.360	0.113	1.362	0.318	3.814

[A] The average of the laboratories' calculated average.

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TABLE 5 Sieve Size 40 – Average Grams Retained

Material	Average[A] Grams Retained $\bar{x}$	Standard Deviation $S\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r	Reproducibility Standard Deviation S_R	Reproducibility Limit R
Type I	28.287	1.790	1.650	2.136	4.621	28.287
Type III	51.778	8.190	6.150	9.273	17.221	25.965
Type V	34.642	9.067	0.398	9.071	1.114	25.400

[A] The average of the laboratories' calculated average.

TABLE 6 Sieve Size 40 – Average Grams Passing

Material	Average[A] Grams Passing $\bar{x}$	Standard Deviation $S\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r	Reproducibility Standard Deviation S_R	Reproducibility Limit R
Type I	65.608	2.067	1.742	2.406	4.879	6.737
Type III	81.851	9.625	10.318	12.078	28.890	33.817
Type V	56.006	10.402	0.433	10.406	1.212	29.138

[A] The average of the laboratories' calculated average.