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Optical Sizing and Roundness Determination of Glass Beads Used in Traffic Markings

DETAILS

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OPTICAL SIZING AND ROUNDNESS DETERMINATION OF GLASS BEADS USED IN TRAFFIC MARKINGS

This digest summarizes key findings from NCHRP Project 20-07, Task 243, "Development of a Test Method for Optical Sizing and Roundness Determination of Glass Beads Utilized in Traffic Markings," conducted by the AASHTO Materials Reference Laboratory (AMRL) and the National Institute of Standards and Technology (NIST), Gaithersburg, Maryland. The digest was prepared from the project final report authored by Haleh Azari (AMRL) and Edward Garboczi (NIST).

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INTRODUCTION

Glass beads are used to enhance the nighttime and wet visibility of traffic markings such as paints and thermoplastics. The size and shape (i.e., the roundness) of the beads are significant determinants of their ability to reflect the light they receive from a source. Perfectly round and well graded beads most effectively reflect light back to the source; this *retroreflectivity* is critical for visibility in low-light situations. Due to the great effect of the size and roundness of glass beads on their reflectivity, AASHTO M 247 specifies requirements for the size distribution and degree of roundness of glass beads used for traffic markings.

Measurement of bead size and roundness has traditionally been performed using sieves following ASTM Method D1214, the roundometer following ASTM D1155, and manual microscopy. Computerized optical methods have been used for quite some time for characterization of fine particles. Several manufacturers of computerized optical equipment have developed applications for measuring the size and shape of translucent glass beads. The main advantage of this approach is faster measurement of the glass bead properties, which is very time consuming if determined traditionally using the manual sieve and roundometer.

The majority of glass bead manufacturers and distributors and a number of state highway agency laboratories have purchased computerized optical equipment to expedite the quality assurance of glass beads used in traffic markings. Despite the increasing popularity of such equipment, no standard test method exists for their use with glass beads. This creates confusion when comparing the results of one laboratory with the results of another. In addition, the accuracy and precision of the data obtained with the computerized methods are not yet known. Finally, the results from computerized optical equipment have not been compared with those obtained using the traditional ASTM methods, and the correlation between mechanical and computerized methods is not known.

In NCHRP Project 20-07, Task 243, an interlaboratory study (ILS) was designed and conducted to (1) determine the precision and bias of both optical and traditional mechanical methods for different glass bead types (as defined in AASHTO M 247), (2) compare the precision and bias of various measurement methods, and (3) develop a

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practice in AASHTO standard format for the use of computerized optical methods and equipment to measure the size and roundness of glass beads used in traffic markings (see Appendix A).

This digest summarizes the results of NCHRP Project 20-07, Task 243, as presented in the full project final report titled "Optical Sizing and Roundness Determination of Glass Beads Used in Traffic Markings." The project final report is available as *NCHRP Web-Only Document 156* at http://www.trb.org and contains nine appendices: Appendix A, Instructions and Data Sheet for Interlaboratory Study; Appendix B, Results of Percent Retained by Mechanical Sieve; Appendix C, Results of Roundometer; Appendix D, Results of Percent Retained Using COM-A; Appendix E, Results of SPHT Roundness by COM-A; Appendix F, Results of b/l Roundness by COM-A; Appendix G, Results of Size Measurements by COM-B; Appendix H, Results of Roundness by COM-B; and Appendix I, Recommended Test Method for Measurement of Size Distribution and Roundness of Glass Beads Using Computerized Optical Equipment. The main text of the project final report also presents the results of a complementary study to evaluate the effectiveness of various computerized optical method size and shape parameters in discriminating real shape and size through an analysis of X-ray microtomography images of various glass bead types.

EXPERIMENTAL DESIGN

An ILS was conducted to evaluate the precision and accuracy of both the optical and traditional mechanical methods. Glass bead samples with specific size distribution and percent roundness were prepared and sent to participating laboratories for property measurements. The ILS was designed in accordance with ASTM E691-09, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method. ASTM E691 specifies that development of a precision statement requires participation of a minimum of 6 laboratories with a preferred number of 30.

ILS Sample Preparation

Commercial glass beads of Types 1 (fine gradation), 3 (medium gradation), and 5 (coarse gradation) as specified in AASHTO M 247 were obtained in 23 kg bags from two manufacturers. Three blends of fine, medium, and coarse glass bead samples were prepared. The gradations of the three blends were selected according to the Type 1, Type 3, and Type 5 gradings in AASHTO M 247. The roundness of the three blends was selected as 70%, 80%, and 90% by mass, respectively. Although the original glass beads in the 23 kg bags had the overall gradation that was required for the ILS samples, the test specimens were not sampled directly from the bags for two reasons: (1) the beads might have become segregated during transportation and (2) the test specimens were required to have specified roundness levels that could not be achieved by direct sampling.

Sets of glass bead samples each including three replicates of the three blends were prepared for each participant in the ILS. The first blend, referred to as Y, was prepared to meet the Type 1 gradation. Each size class in the Y samples was a blend of 70% round and 30% non-round glass beads, where the roundness level was determined from roundometer and spiral separator results. The second blend, referred to as P, was prepared according to the Type 3 gradation. Each size class in the P samples was prepared with 80% round and 20% non-round beads. The third blend, referred to as C, was prepared according to the Type 5 gradation. Each size class in the C samples was prepared with 90% round and 10% non-round glass beads. Table 1 provides information on the

Table 1 Percent passing of the three glass bead types

Sieve Opening µ	U.S. Sieve Size	Type 1 (Y)	Type 3 (P)	Type 5 (C)
2350	#8			
2000	#10			100%
1700	#12			95%
1400	#14		100%	40%
1180	#16	100%	95%	5%
1000	#18		40%	0%
850	#20	100%	5%	
710	#25		0%	
600	#30	95%		
500	#35			
425	#40			
300	#50	35%		
180	#80			
150	#100	0%		

Table 2 Percent retained of round and non-round glass beads for each sample type

Sieve Opening µ	U.S. Sieve Size	Type 1 (Y)		Type 3 (P)		Type 5 (C)	
		Round	Non-Round	Round	Non-Round	Round	Non-Round
2000	#10						
1700	#12					4.5%	0.5%
1400	#14					49.5%	5.5%
1180	#16			4.0%	1.0%	31.5%	3.5%
1000	#18			44.0%	11.0%	4.5%	0.5%
850	#20			28.0%	7.0%		
710	#25			4.0%	1.0%		
600	#30	3.5%	1.5%				
500	#50	42.0%	18.0%				
425	#100	24.5%	10.5%				

gradation (percent passing) of the three blends and Table 2 on their roundness, which includes percent retained round and non-round beads in each size class.

Specimen sets were sent to 30 participating laboratories for property measurements. Depending on the measurement capability of each laboratory or its willingness to conduct both mechanical and computerized methods, some laboratories received more than one specimen set.

Test Methods Evaluated in the ILS

Participating laboratories were asked to conduct traditional sieve and roundometer tests, computerized optical methods, or both. The traditional methods followed ASTM D1214, "Sieve Analysis of Glass Spheres" and ASTM D1155, "Roundness of Glass Sphere" test methods. Two types of computerized optical scanning instruments were used, denoted COM-A and COM-B. One set of Type 1 samples was also tested by a second type of COM-B device, which operates exactly in the same way as the other COM-B devices, so that their results in this report are both referred to as COM-B and were combined in the precision and bias analysis for the Type 1 samples.

Participating Laboratories

State department of transportation (DOT) laboratories and glass bead and equipment manufacturers and distributors were invited to participate in the ILS. Thirty laboratories responded to the invitation; 15 laboratories returned results using mechanical sieving, eight laboratories returned results using COM-A, and four laboratories returned results using COM-B.

ILS Instructions

Instructions for performing the tests and collecting data were provided to each participant. The laboratories conducting the mechanical measurements were requested to follow the instructions prepared according to ASTM D1214 and D1155 and report the measured retained weights and the corresponding percentages of round and non-round glass beads in specified size classes. The laboratories using the computerized optical method were requested to follow instructions prepared with the help of the COM-A and COM-B manufacturers. Participants using the computerized optical method equipment measured percent retained and percent round in each size class of each sample type in terms of the specific parameters of the COM-A and COM-B instruments as presented in Table 3 and Table 4. The ILS instructions are available in Appendix A of the project final report.

ILS TEST RESULTS AND ANALYSIS

Test Results

Of the 30 sets of glass beads that were distributed to the laboratories, 27 sets of results were received. Fifteen laboratories submitted full sets of size distribution data using sieve analysis. Ten laboratories

Table 3 COM-A parameters for size distribution and roundness measurements

COM-A Parameters	Description of Parameters
X_{cmin} (b)	Breadth, particle diameter, which is the shortest chord of the measured set of maximum chords of a particle. This is thought to be a good measure of the mechanical sieve opening.
X_{Femax} (l)	The particle diameter, which is the longest Feret diameter of the measured set of Feret diameters of a particle.
Feret diameter	The distance between two parallel tangents of the particle at an arbitrary angle.
b/l	Sphericity parameter $b/l = X_{cmin}/X_{Femax}$. For an ideal circle, b/l is 1, otherwise it is smaller than 1. The threshold value used for b/l was 0.83.
SPHT	Sphericity parameter $SPHT = 4\pi A/U^2$ U—measured circumference of a particle A—measured area covered by a particle. For an ideal circle, SPHT is 1, otherwise it is smaller than 1. The threshold value used for SPHT parameter was 0.9.

submitted roundness data using the roundometer. Eight laboratories submitted size distributions and roundness data using the COM-A device. Four laboratories submitted size distribution and roundness data using the COM-B instrument. The data were reviewed to identify possible outliers; confirmed outliers were eliminated from the subsequent analyses.

Method of Analysis

The ILS test results were analyzed for precision in accordance with ASTM E691. Prior to the analysis, any partial sets of data were eliminated by following the procedures described in E691 for determining repeatability (S_r) and reproducibility (S_R) estimates of precision. Data exceeding critical *h* and *k* values were eliminated as described in the method. Once identified for elimination, the same data were eliminated from any smaller subsets analyzed.

The data from different analysis methods were also analyzed for bias by comparing them with the target percent retained and roundness using t-statistics. The rejection probability of the computed t-statistics for a 5% level of significance would indicate which of the utilized methods most accurately measured the intended properties of the glass beads.

Analysis of Results from Traditional Mechanical Methods

Sieve analysis and roundness measurements using mechanical sieves and the roundometer were conducted following the ASTM D1214 and ASTM D1155 test methods, respectively. The following sections provide the results of the precision and bias analysis of the measurements using the traditional methods.

Size Measurements Using Mechanical Sieve

The sieve openings and the corresponding sieve numbers for each glass bead type are provided in Table 1.

Table 4 COM-B parameters for size distribution and sphericity measurements

COM-B Parameters	Description of Parameters
Thickness (T)	Thickness of particle. This is used for sieve analysis of particles.
Length (L)	The largest length of the particle.
TL = T/L	Aspect ratio of thickness/length, which is 1 for a perfect circle. Otherwise it is smaller than 1.
NSP Ratio	Average Ratio of D_a/D_p of all particles analyzed, which is the same as $(SPHT)^{1/2}$ D_a = Diameter calculated of the imaged area of the particle, as an equivalent circle D_p = Diameter of perimeter calculated of the imaged circumference of the particle, as an equivalent circle Aspect ratio of D_a/D_p is 1 for a perfect circle. Otherwise it is smaller than 1.

Table 5 Statistics of percent retained of Type 1 samples using mechanical sieve shaker

Sieve Sizes	No. of Labs	Target % Retained	Measured % Retained, Average	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#30	13	5.0	5.0	0.2	3.7	0.4	7.5
#50	14	50.0	48.4	1.0	2.1	1.7	3.4
#100	14	45.0	46.4	1.1	2.4	1.6	3.5

Type 1 Samples. The results of mechanical sieve analysis of Type 1 (Y) samples were received from 15 laboratories. The precision estimates of the size distribution of Type 1 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 5. As indicated from this table, the measured percent retained agrees relatively well with the target percent retained for all sieve sizes, e.g., measured retained value of 48.4% for #50 sieve agrees well with the 50% target retained value. The variability of the data as indicated by the repeatability and reproducibility coefficients of variation is relatively low. The repeatability and reproducibility coefficients of variation corresponding to the #50 sieve size are lower than those corresponding to the #30 and #100 sieve sizes. This may result from the larger mass percentage of beads in the #50 size class than in other size classes.

Type 3 Samples. The results of mechanical sieve analysis of the Type 3 (P) samples were received from 14 laboratories. The precision estimates for the size distribution of the Type 3 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 6. The table shows that the measured mass percent retained values agree reasonably well with the target percent retained for all sieve sizes, e.g., the measured retained value of 58.8% for the #18 sieve is compared with the target retained value of 55%. As indicated in the table, the repeatability and reproducibility coefficients of variation are significantly larger for sieves #16 and #25 than for sieves #18 and #20. This is likely due to the smaller mass percentage of beads in the #16 and #25 size classes relative to the mass percentage of beads in the #18 and #20 size classes.

Type 5 Samples. The results of mechanical sieve analysis of the Type 5 (C) samples were received from 14 laboratories. The variability of the size distribution of the Type 5 specimens was determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 7. This table indicates that the measured percent retained agrees fairly well with the target percent retained for all sieve sizes, e.g., the measured percent retained value of 58.5% for the #14 sieve is compared with the target retained value of 55%. As shown in Table 7, the repeatability and reproducibility variability values of all except the #14 size class are rather large. This is likely due to the smaller amount of beads in those size classes compared to the amount of beads in the #14 size class.

Summary of Percent Retained by Mechanical Sieves. The analysis of the traditional mechanical sieve mass percent retained data suggests that in general

Table 6 Statistics of percent retained of Type 3 samples using mechanical sieve shaker

Sieve Sizes	No. of Labs	Target % Retained	Measured % Retained, Average	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#16	13	5.0	6.5	0.4	6.9	2.5	38.0
#18	13	55.0	58.8	1.1	1.9	3.4	5.7
#20	12	35.0	30.3	1.2	4.0	1.9	6.3
#25	14	5.0	4.3	0.8	18.2	1.1	26.5

Table 7 Statistics of percent retained of Type 5 samples using mechanical sieve shaker

Sieve Sizes	No. of Labs	Target % Retained	Measured % Retained, Average	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#12	14	5.0	5.2	0.6	12.4	1.0	18.9
#14	13	55.0	58.5	2.2	3.7	4.3	7.4
#16	14	35.0	31.4	2.8	9.0	4.1	13.2
#18	13	5.0	5.0	0.7	14.4	1.2	23.6

the measured percent retained in the most prevalent size classes provides the closest agreement with the target value. The measured values also yield the smallest repeatability and reproducibility coefficients of variation for the sieves with the largest number of beads. Furthermore, the traditional mechanical sieve measures mass percent retained of Type 1 beads more accurately and precisely than that of Type 3 and Type 5 beads.

Roundness Measurements Using Roundometer

Type 1 Samples. The results of roundness analysis of Type 1 samples were received from 11 laboratories. The variability of percent roundness of Type 1 specimens was determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 8. A review of the statistics in Table 8 indicates that the average roundness of the Type 1 samples was overestimated, e.g., the measured percent round of 74.2% for the #50 beads is larger than the target roundness of 70%. Similar to the observation from the size distribution statistics, both repeatability and reproducibility coefficients of variation of roundness measurement are significantly smaller for the #50 beads, which was the most prevalent size in the Type 1 samples.

Type 3 Samples. Nine out of the 14 laboratories that conducted mechanical sieve analysis on the Type 3 samples also returned results on the roundness of the samples. After removal of outliers, all remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 9. A review of the statistics in Table 9 indicates that there is a good agreement

Table 8 Roundness statistics of Type 1 samples using roundometer, target roundness of 70%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#30	9	72.2	2.3	3.2	5.7	7.9
#50	11	74.2	2.5	3.4	3.8	5.1
#100	10	77.7	4.8	6.2	4.6	5.8

Table 9 Roundness statistics of Type 3 samples using roundometer, target roundness of 80%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#16	6	75.1	1.7	2.3	4.1	5.5
#18	8	78.5	2.3	2.9	4.1	5.2
#20	7	73.9	1.6	2.1	4.7	6.4
#25	7	65.6	12.5	19.0	11.8	18.0

between measured and target roundness for the #18 size class, which is the most prevalent size class. It can also be observed from the table that the percent roundness was underestimated for all size classes, e.g., the measured percent round of 73.9% for the #20 beads is smaller than the target roundness of 80%. This might be due to the difference between separation methods of the roundometer and the spiral separator, which were both used in the preparation of ILS samples. Similar to the previous observations, the repeatability and reproducibility coefficients of variation of roundness measurements corresponding to the size class with the largest amount of beads (#18 sieve) are smaller than those of other size classes.

Type 5 Samples. Nine out of the 14 laboratories that conducted mechanical sieve analysis on the Type 5 samples also returned results on the roundness of the samples. The variability of percent round of the Type 5 samples was determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 10. A review of the statistics in the table indicates the roundness of the Type 5 beads was underestimated for all size classes, e.g., the measured percent round of 83.4% for the #14 beads is smaller than the target roundness of 90%. This again could be due to the difference between the methods of separating the beads using the roundometer and the spiral separator. The repeatability and reproducibility coefficients of variation of the Type 5 beads are relatively low for all size classes. This might indicate better control over the roundness determination of larger beads than of smaller beads.

Summary of Percent Round by the Roundometer. The analysis of the roundometer percent round data suggests that for Type 1 samples, the average measured round was larger than the target round. For Types 3 and 5 the average measured round was smaller than the target round value. The reason for this difference may be the different methods used for separating the glass beads. The spiral separator and roundometer were used in the preparation of the samples but only the roundometer was used for testing of the ILS samples. The measured values indicated that the smallest repeatability and reproducibility coefficients of variation correspond to the sieve with the largest number of beads.

Analysis of Results from COM-A Measurements

A total of eight laboratories returned measurements by COM-A. The measurements included size distribution and percent roundness. The roundness measurement was made using two parameters, b/l and SPHT, as explained in Table 3. The measurements using COM-A were conducted following the instructions provided by the COM-A manufacturer. The data were analyzed to evaluate the precision and bias of each measured property and to compare these with the precision and bias of the properties measured using the traditional methods and COM-B computerized optical method.

Size Measurements

The size distributions of the three sample types were measured using two-dimensional (2-D) analysis of the images of the glass beads passing through the COM-A measurement unit.

Type 1 Samples. The precision estimates for size distribution of Type 1 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics

Table 10 Roundness statistics of Type 5 samples using roundometer, target roundness of 90%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#12	7	86.1	3.1	3.6	5.0	5.8
#14	8	83.4	1.8	2.1	4.5	5.3
#16	7	87.0	2.1	2.4	4.1	4.7
#18	7	87.3	2.9	3.4	4.8	5.4

Table 11 Percent retained statistics of Type 1 samples using COM-A

Sieve Sizes	No. of Labs	Target % Retained	Measured % Retained, Average	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#30	6	5	4.5	0.3	5.6	0.3	7.1
#50	8	50	46.4	1.4	3.0	3.0	6.5
#100	8	45	48.6	1.6	3.3	3.3	6.7

shown in Table 11. As indicated in Table 11, the measured mass percents retained are in relatively good agreement with the target values. For example, the average retained of 46.4% for the #50 sieve size is compared with the target retained of 50%. Similar to the mechanical sieve analysis of Type 1 samples, the repeatability and reproducibility coefficients of variation of all class sizes are relatively small.

Type 3 Samples. The precision estimates for size distribution of Type 3 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 12. A comparison of the measured and target percent retained in Table 12 indicates a good agreement between the measured percent retained and the target percent retained in the #18 size class, which has the largest number of beads. The measured retained value of 57.2% compares relatively well with the target retained value of 55%. The repeatability and reproducibility coefficients of variation for the #18 sieve are smaller than those for the other sieves.

Type 5 Samples. The precision estimates for size distribution of the Type 5 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 13. A very good agreement between the measured and target percent retained is observed for the #14 size class, which has the largest number of beads. The measured retained value of 55.5% compares very well with the target retained value of 55%. It is also indicated from the table that the smallest repeatability and reproducibility coefficients of variation for the percent retained correspond to #14 sieve.

Table 12 Percent retained statistics of Type 3 samples using COM-A

Sieve Sizes	No. of Labs	Target % Retained	Measured % Retained, Average	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#16	7	5	10.3	0.4	4.0	3.4	32.6
#18	8	55	57.2	0.7	1.2	2.1	3.7
#20	8	35	26.8	0.8	3.0	1.8	6.7
#25	8	5	4.6	0.7	15.4	0.9	19.6

Table 13 Percent retained statistics of Type 5 samples using COM-A

Sieve Sizes	No. of Labs	Target % Retained	Measured % Retained, Average	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#12	8	5	6.9	0.7	10.2	1.4	19.6
#14	7	55	55.5	0.9	1.5	1.2	2.1
#16	7	35	30.1	1.7	5.7	2.1	6.9
#18	7	5	6.5	0.3	4.2	0.7	10.3

Table 14 Statistics of percent round of Type 1 samples using SPHT parameter of COM-A, target 70%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#30	4	80.8	1.9	2.3	5.8	7.2
#50	3	84.5	0.7	0.8	1.5	1.8
#100	4	84.8	2.5	2.9	2.2	2.6

Summary of Percent Retained by COM-A. The analysis of COM-A mass percent retained data suggests that the measured percent retained in the size class with the largest number of beads provides closest agreement with the target value. Looking at the percent retained in the size classes with the largest number of beads in the Type 1, Type 3, and Type 5 samples indicates that both accuracy and precision of measurements improved with the coarseness of the glass bead types.

Roundness Measurements Using the SPHT Parameter

A total of four laboratories reported the percent roundness of the three sample types measured by the SPHT parameter (see Table 3) using the COM-A device. A threshold value of 0.9 was used for roundness determination using the SPHT parameter, meaning that any particle with an SPHT value of 0.9 and above was considered to be round.

Type 1 Samples. The precision estimates for the mass percent roundness of the Type 1 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 14. Table 14 indicates that the roundness of all size classes are overestimated by the SPHT parameter, e.g., the measured percent round of 84.5% for the #50 beads is significantly larger than the target roundness of 70%. This suggests that the SPHT threshold value may be unsuitable because it allows non-round beads to be classified as round. Despite the inaccuracy of the SPHT parameter, both the repeatability and reproducibility coefficients of variation of the SPHT parameter are relatively small for all size classes.

Type 3 Samples. The precision estimates for the mass percent roundness of the Type 3 specimens were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 15. A review of the statistics in Table 15 indicates that the percent round of the size classes with the most number of beads (#18 and #20) was overestimated by the SPHT parameter. The measured percent round of 86.3% for the #18 beads is compared with the target roundness of 80%. This might indicate that the SPHT threshold value is too low, which allows non-round beads to be classified as round. The repeatability and reproducibility coefficients of variation of the roundness data obtained based on the SPHT parameter are relatively small for all size classes.

Type 5 Samples. Based on outlier analysis, no data were eliminated from the determination of the precision estimates for the mass percent roundness of the

Table 15 Statistics of percent round of Type 3 samples using SPHT parameter of COM-A, target 80%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#16	4	77.7	2.6	3.4	2.9	3.7
#18	4	86.3	0.9	1.0	1.0	1.2
#20	3	92.1	0.4	0.4	0.8	0.8
#25	4	85.1	3.4	4.0	4.1	4.9

Table 16 Statistics of percent round of Type 5 samples using SPHT parameter of COM-A, target 90%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#12	4	88.5	1.0	1.1	2.6	2.9
#14	4	92.1	0.6	0.7	1.2	1.4
#16	4	91.2	1.7	1.8	5.7	6.2
#18	4	92.1	0.7	0.7	0.7	0.7

Type 5 specimens. The computed repeatability and reproducibility statistics of the Type 5 sample roundness are shown in Table 16. A review of the statistics in Table 16 indicates that the roundness of all size classes was measured reasonably correctly by the SPHT parameter. The measured percent round of 92.1% for the #14 beads agrees well with the target roundness of 90%. In addition, a review of the variability values in Table 16 indicates that both the repeatability and reproducibility coefficients of variation of the SPHT parameter are very small for all size classes. This suggests that (1) the threshold value of 0.9 is more appropriate for larger beads than for smaller beads and (2) a higher threshold value is needed for more accurate measurements of the roundness of smaller glass beads.

Summary of Percent Round by COM-A SPHT. The analysis of mass percent round using the COM-A SPHT parameter indicates that the measured percent round in the most prevalent size classes provided closest agreement with the target value. However, the level of agreement between measured and target percent round differs for the Type 1, Type 3, and Type 5 samples. This suggests that the threshold value for all glass bead types is not the same and would need adjustment according to the glass bead type. Specifically, a threshold value of 0.9 appears acceptable for Type 5 beads, but it did not measure the intended roundness of Type 1 and Type 3 beads accurately.

Roundness Measurements Using b/l Parameter

Eight laboratories reported the percent roundness of the three sample types measured by the COM-A b/l parameter. The threshold value for measuring percent round by the b/l parameter was 0.83, meaning that any particle with b/l ≥ 0.83 was considered to be round.

Type 1 Samples. The variability of percent round of Type 1 specimens was determined after eliminating the outlier data. All remaining data were reanalyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 17. A review of the statistics in Table 17 indicates that the roundness of all size classes of the Type 1 samples is overestimated by the b/l parameter. The measured percent round of 78.6% for the #50 beads is larger than the target roundness of 70%. This difference may result from an artifact of the 2-D image analyses. A single or even several 2-D projections of a non-spherical object cannot fully capture its 3-D shape, which would tend to bias the percent round results. This is also an indication that

Table 17 Statistics of percent round of Type 1 samples using b/l parameter of COM-A, target 70%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#30	8	76.8	2.5	3.2	6.1	8.0
#50	6	78.6	1.2	1.6	1.7	2.1
#100	7	80.9	3.1	3.8	3.5	4.3

Table 18 Statistics of percent round of Type 3 samples using b/l parameter of COM-A, target 80%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#16	8	66.5	2.2	3.3	3.4	5.0
#18	8	80.1	0.9	1.1	1.1	1.4
#20	7	84.3	0.9	1.0	1.1	1.3
#25	8	76.6	4.7	6.1	9.5	12.4

the threshold value for the b/l parameter is not large enough to eliminate all particles considered to be non-round by the roundometer. Similar to the observations made on the roundometer data, both the repeatability and reproducibility coefficients of variation of the b/l parameter are significantly smaller for the #50 beads, which are the most prevalent size in the Type 1 samples.

Type 3 Samples. The variability of percent round of Type 3 specimens was determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 18. A review of the statistics in Table 18 indicates that the measured percent round of the #18 glass beads (80.1%), which is the most prevalent size, agrees very well with the target roundness of 80%. The percent round of the #20 sieve is slightly overestimated, which may also be due to 2-D image analysis artifacts as noted in the previous section. The results in Table 18 further indicate that the repeatability and reproducibility coefficients of variation of percent round according to the b/l parameter for the #18 and #20 beads, which are the most prevalent sizes, are very small. This indicates that b/l is a reliable parameter for measuring the roundness of the most prevalent size classes of Type 3 glass beads.

Type 5 Samples. The variability of percent round of Type 5 specimens was determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 19. A review of the statistics in Table 19 indicates that the measured roundness of the #14 and #16 glass beads, which are the most prevalent sizes, agrees very well with the target roundness of 90%. The repeatability and reproducibility coefficients of variation corresponding to the #14 and #16 size classes are also very small. This indicates that b/l is a reliable parameter for measuring the roundness of the most prevalent size classes of Type 5 glass beads.

Summary of Percent Round by COM-A b/l. The analysis of mass percent round of COM-A b/l data indicates that the b/l parameter captured the roundness of Type 3 and Type 5 glass beads very well but overestimated the roundness of Type 1 beads. This indicates that the threshold value for b/l should not be the same for all glass bead types. While the threshold value of 0.83 seems adequate for Type 3 and Type 5 glass beads, it may need to be increased for Type 1 beads.

Table 19 Statistics of percent round of Type 5 samples using b/l parameter of COM-A, target 90%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#12	8	80.8	1.2	1.5	3.0	3.7
#14	8	89.2	1.1	1.2	1.5	1.7
#16	7	91.4	1.9	2.1	2.3	2.6
#18	8	89.8	1.1	1.2	1.4	1.5

D10, D50, and D90 Measurements

The accuracy of COM-A in measuring the size distribution of glass beads was evaluated. For this purpose, the three diameters where 10%, 50%, and 90% of the particles, by mass, are smaller than these diameters (D10, D50, D90) were compared with the sieve sizes and the mass percent passing that were used to prepare the bead samples. Tables 20, 21, and 22 provide the measured diameters corresponding to 10%, 50%, and 90% of particles having a diameter less than the given diameter. These tables also provide the sieve sizes and the percent passing of Types 1, 3, and 5 glass beads prepared in this study.

Type 1 Samples. Column 4 of Table 20 shows the values of D10, D50, and D90 of Type 1 samples according to COM-A data. The results in Column 4 are averages of COM-A measurements received from seven laboratories. The comparison of the measured and target values of particle size with respect to percent smaller and percent passing values indicates that the COM-A instrument has measured the size distribution of the Type 1 samples reasonably well. For example, bead samples were prepared to have 95% passing a 0.6 mm sieve opening (#30 sieve) and the COM-A data measured that 90% of the beads are smaller than 0.53 mm. The same logic is applied to other sieve sizes of Type 1 samples, which indicates that the COM-A device, using the width (b or X_{cmin}) parameter, has measured the size distribution of Type 1 beads reasonably well.

Type 3 Samples. Column 4 of Table 21 provides the values of D10, D50, and D90, in terms of the parameter X_{cmin}, for the Type 3 samples; Columns 2 and 3 provide the sieve openings and the corresponding percent passing used in preparing the Type 3 samples. The comparison of the measured and target values of particle size with respect to percent smaller and percent passing values indicates that the COM-A instrument has measured the size distribution of the Type 3 samples reasonably well. The percent smaller for D90 has an expected value of 95% compared to a measured value of 90%, but the percent smaller than D10 and D50 are reasonable. For example, 40% of the beads were prepared to pass through a 1.0 mm opening (#18 sieve) and the COM-A predicted that 50% of the beads have diameter smaller than 1.07 mm, which is reasonable agreement.

Table 21 Comparison of measured and target particle sizes of Type 3 beads for 10%, 50%, and 90% passing

No. of Labs	Sieve Size (mm)	Target Percent Passing	Particle Diameter–X_{cmin} (mm)	Percent Smaller
7	0.85	5	0.89	10
7	1.00	40	1.07	50
7	1.18	95	1.18	90

Type 5 Samples. Column 4 of Table 22 provides the sizes in millimeters for D10, D50, and D90, as determined from the COM-A results. The second and third columns provide the sieve openings and the corresponding percent passing used in preparing the Type 5 bead samples. The comparison of the measured and target values of particle size with respect to the percent smaller and percent passing indicates that the COM-A device has measured the size distribution of the Type 5 samples reasonably well. For example, 40% of the beads were prepared to pass through a 1.40 mm opening (#14 sieve); the COM-A result is that 50% of the beads by mass have diameters smaller than 1.49 mm.

Summary of D10, D50, and D90 Measurements by COM-A. The analysis of D10, D50, and D90 data measured by COM-A indicated that COM-A measured the size distribution of the three types of glass bead samples reasonably well. Only one out of nine

Table 20 Comparison of measured and target particle sizes of Type 1 beads for 10%, 50%, and 90% passing

No. of Labs	Sieve Size (mm)	Target Percent Passing	Particle Diameter–X_{cmin} (mm)	Percent Smaller
7	0.15	0	0.20	10
7	0.30	45	0.31	50
7	0.60	95	0.53	90

Table 22 Comparison of measured and target particle sizes of Type 5 beads for 10%, 50%, and 90% passing

No. of Labs	Sieve Size (mm)	Target Percent Passing	Particle Diameter–X_{cmin} (mm)	Percent Smaller
7	1.18	5	1.21	10
7	1.40	40	1.49	50
7	1.70	95	1.66	90

Table 23 Percent retained statistics of Type 1 samples using COM-B

Sieve Sizes	No. of Labs	Measured % Retained, Average	Target % Retained	Repeatability		Reproducibility	
				STD, %	CV, %	STD, %	CV, %
#30	3	5.1	5.0	0.3	5.5	0.5	10.8
#50	4	46.7	50.0	2.1	4.6	3.5	7.4
#100	3	46.8	45.0	1.2	2.5	2.0	4.2

measurements (D90 of Type 3) did not compare well with the target sieve opening and its corresponding percent passing.

Analysis of Results from COM-B Measurements

Four computerized optical systems from the COM-B manufacturer were available for measuring the size and roundness of the glass bead samples. These included three of one type of system and one of another type that uses the same measuring system and software as does the other COM-B equipment but is exclusively built for use with fine particles and powders. Therefore, only properties of the Type 1 samples were measured using this second instrument. Although the COM-B device has the capability of measuring properties of large particles (Type 3 and Type 5 samples), two of the three COM-B systems used in the study were not calibrated for use with large particles and therefore were not used for the Type 3 and Type 5 samples. As a result, the properties of the Type 1 samples were reported by four laboratories but the properties of the Type 3 and Type 5 samples were measured by only one laboratory. For convenience, the data measured using the two different kinds of COM-B equipment were combined and are jointly referred to as COM-B data.

The COM-B measurements were conducted following the instructions provided by the manufacturer. The data were analyzed to evaluate the precision and bias for the COM-B method and to compare its precision and bias with those of the traditional methods and the COM-A method. The size distribution and roundness of the three sample types were measured by analysis of 2-D images of the glass beads. Multiple images of single particles were taken from different angles as they tumbled through the measuring unit of the equipment.

Size Measurements

With the COM-B devices, the size of the glass beads was determined from the thickness (T) of the glass beads as described in Table 4.

Type 1 Samples. The percent retained in various size classes of the Type 1 samples was reported by four laboratories. The precision estimates for the percent retained were determined after eliminating the outlier data. All remaining data were re-analyzed according to the E691 method to determine the repeatability and reproducibility statistics shown in Table 23. As indicated in the table, the measured and target percent retained values agree relatively well with the target retained values. The repeatability and reproducibility coefficients of variation corresponding to these size classes are relatively small.

Type 3 Samples. As previously noted, only one laboratory equipped with COM-B equipment had the capability of measuring the properties of the large glass beads. As shown in Table 24, there is reasonable

Table 24 Percent retained statistics of Type 3 samples using COM-B

Sieve Sizes	No. of Labs	Measured % Retained, Average	Target % Retained	Standard Deviation, %	Coefficient of Variation, %
#16	1	13.1	5.0	1.3	10.0
#18	1	52.5	55.0	0.8	1.5
#20	1	25.6	35.0	2.1	8.2
#25	1	4.1	5.0	0.8	19.5

Table 25 Percent retained statistics of Type 5 samples using COM-B

Sieve Sizes	No. of Labs	Measured % Retained, Average	Target % Retained	Standard Deviation, %	Coefficient of Variation, %
#12	1	9.9	5.0	0.3	2.8
#14	1	51.9	55.0	2.6	4.9
#16	1	29.4	35.0	2.4	8.3
#18	1	4.8	5.0	0.4	7.9

agreement between the measured and target percent retained in the size class with the largest mass percentage of the beads. The measured retained value of 52.5% for the #18 size class is relatively close to the target retained value of 55%. This size class also provided the smallest coefficient of variation.

Type 5 Samples. The percent retained data on various size classes of the Type 5 samples were reported by one laboratory. As shown in Table 25, there is a fair agreement between the measured and target percent retained. The measured retained value of 51.9% for the #14 size class agreed fairly well with the target retained value of 55%. The coefficient of variation indicated by this size class, which has the highest mass percentage of the beads, was also relatively small.

Summary of Percent Retained by COM-B. Despite the small number of laboratories reporting COM-B results, the measured percent retained in the most prevalent size classes provided reasonable agreement with the target values. The measurements in the most prevalent size classes also indicated small repeatability and reproducibility coefficients of variation for Type 1 samples and small coefficient of variation for the Type 3 and Type 5 samples.

Roundness Measurements

The roundness of the glass beads measured by COM-B was determined based on the T/L parameter described in Table 4. COM-B also measures roundness using the NSP parameter defined in Table 4; however, the NSP results of roundness measurement were not complete and, therefore, were not included in the statistical analysis.

Type 1 Samples. Based on outlier analysis, no data were eliminated from the determination of the precision estimates for the mass percent round of the Type 1 specimens. Therefore, all the reported data were included in determining the repeatability and reproducibility statistics shown in Table 26 according to the E691 method. Table 26 indicates that the roundness of the most prevalent size class is overestimated by the T/L parameter. The measured percent round of 77.2% for the #50 beads is larger than the target percent round of 70%. The reason for this difference may be the 2-D image analysis artifact previously discussed or a wrong cutoff value for the T/L parameter. The repeatability and reproducibility coefficients of variation for the most prevalent size class of the Type 1 beads are relatively large.

Type 3 Samples. The percent round data in various size classes of the Type 3 samples were reported by one laboratory. The data are summarized in Table 27, which shows that the roundness of the P samples is significantly overestimated. The percent round of 86.8% in the #18 size class, which has the highest mass percentage of the beads, is compared with the target percent round of 80%. Despite the

Table 26 Percent round of Type 1 samples using T/L parameter of COM-B, target round of 70%

Sieve Sizes	No. of Labs	Measured % Round, Average	Repeatability		Reproducibility	
			STD, %	CV, %	STD, %	CV, %
#30	4	69.0	2.4	3.5	7.2	10.5
#50	4	77.2	4.3	5.6	7.0	9.1
#100	4	78.2	3.8	4.8	5.8	7.4

Table 27 Percent round of Type 3 samples using T/L parameter of COM-B, target round of 80%

Sieve Sizes	No. of Labs	Measured % Round, Average	Standard Deviation, %	Coefficient of Variation, %
#16	1	79.9	3.2	4.0
#18	1	86.8	2.7	3.1
#20	1	84.6	9.0	10.7
#25	1	85.2	2.2	2.6

Table 28 Percent round of Type 5 samples using T/L parameter of COM-B, target round of 90%

Sieve Sizes	No. of Labs	Measured % Round, Average	Standard Deviation, %	Coefficient of Variation, %
#12	1	84.2	3.6	4.3
#14	1	91.0	0.3	0.3
#16	1	91.9	0.6	0.6
#18	1	91.0	2.8	3.1

significant bias, the coefficient of variation corresponding to the #18 size class is relatively small.

Type 5 Samples. The percent round of the Type 5 samples is summarized in Table 28, which shows a very good agreement between the measured and target roundness of the Type 5 samples. The percent round of 91.0% in the #14 size class, which has the greatest mass percentage of beads, is compared with the target of 90% percent round. The coefficient of variation corresponding to this size class is also the smallest.

Summary of Percent Round by COM-B. The analysis of mass percent round with the COM-B T/L parameter indicates that the threshold value for the parameter is not the same for all glass bead types. While the threshold value of 0.83 seems adequate for Type 5 glass beads, it did not correctly determine the percent round of Type 1 and Type 3 beads.

D10, D50, and D90 Measurements

The accuracy of the COM-B method in measuring the size distribution of the beads was evaluated. For this purpose, the values of D10, D50, and D90 were computed and compared with the sieve sizes and their percent passing used in making the bead samples. Tables 29, 30, and 31 present the measured values of D10, D50, and D90, and the target diameters and their corresponding percent passing for the three glass bead types.

Type 1 Samples. The D10, D50, and D90 values of the Type 1 samples were received from two COM-B instruments. Column 4 of Table 29 shows the average D10, D50, and D90 values from the two COM-B devices; the second and third columns provide the sieve openings and the corresponding target percent passing for the samples. The comparison of the measured and target values of particle size with respect to percent smaller and percent passing values indicates that the COM-B instrument has measured the size distribution of the Type 1 samples reasonably well. As shown in Table 29, 95% of Type 1 particles would pass a 0.6 mm sieve opening (#30 sieve) and the COM-B device measured D90 to be 0.52 mm, meaning that 90% of the beads are smaller than 0.52 mm. This is reasonable, since fewer particles would pass through an opening smaller than 0.6 mm.

Table 29 Comparison of measured and target particle size of Type 1 samples for 10%, 50%, and 90% passing

No. of Labs	Sieve Size (mm)	Target Percent Passing	Particle Diameter-T (mm)	Percent Smaller
2	0.15	0	0.20	10
2	0.30	45	0.32	50
2	0.60	95	0.52	90

Table 30 Comparison of measured and target particle size of Type 3 samples for 10%, 50%, and 90% passing

No. of Labs	Sieve Size (mm)	Target Percent Passing	Particle Diameter-X_{cm} (mm)	Percent Smaller
1	0.85	5	0.91	10
1	1.00	40	1.09	50
1	1.18	95	1.43	90

The same trend is observed from the other sieve classes for the Type 1 samples.

Type 3 Samples. The D10, D50, and D90 values of the Type 3 samples were received from one COM-B instrument. Column 4 of Table 30 shows the D10, D50, and D90 values; the second and third columns provide the sieve openings and the corresponding target percent passing for the Type 3 samples. The comparison of the measured and target values of particle size with respect to percent smaller and percent passing indicates that the COM-B instrument has correctly measured D10 and D50 but not D90. As shown in Table 30, 95% of the Type 3 particles, by mass, should be smaller than the 1.18 mm sieve opening (#18 sieve); however, COM-B measured 90% of particles smaller than 1.43 mm.

Type 5 Samples. The D10, D50, and D90 values of the Type 5 samples were available from one COM-B instrument. Column 4 of Table 31 shows the values of D10, D50, and D90; the second and third columns provide the sieve openings and the corresponding target percent passing for Type 5 samples. The comparison of the measured values of D10, D50, and D90 with the target sieve sizes and their corresponding percent passing indicates that COM-B has correctly measured D10 and D50 for the Type 5 samples but not D90. As shown in Table 31, 95% of the Type 5 particles should be smaller than a 1.70 mm sieve opening (#14 sieve), while COM-B measured 90% smaller than 1.84 mm.

Table 31 Comparison of measured and target particle size of Type 5 samples for 10%, 50%, and 90% passing

No. of Labs	Sieve Size (mm)	Target Percent Passing	Particle Diameter-T (mm)	Percent Smaller
1	1.18	5	1.22	10
1	1.40	40	1.54	50
1	1.70	95	1.84	90

Summary of D10, D50, and D90 Measurements by COM-B. Since an insufficient number of laboratories submitted D10, D50, and D90 data, a firm conclusion regarding the accuracy of COM-B for measuring size distribution of glass bead samples cannot be made. However, analysis of the limited datasets in this study indicated that for all size classes of Type 1 samples and for four out of six size classes of Type 3 and Type 5 samples, COM-B correctly predicted the D10, D50, and D90 values of the glass beads.

COMPARISON OF PRECISION ESTIMATES OF VARIOUS MEASUREMENT METHODS

Comparing the precision estimates for size and roundness measurements by the various traditional mechanical and computerized optical methods will indicate which method provided the most precise measurements. This comparison was conducted on the size class of each glass bead type that contained the largest mass percentage of the beads—the #50 size class for the Type 1 samples, the #18 size class for the Type 3 samples, and the #14 size class for the Type 5 samples.

Size Measurements

Type 1 Samples

The precision estimates for measuring the mass percent retained in the #50 size class for the Type 1 samples by various methods of measurement are provided in Table 32. The precision estimates are based on the size distribution of three 50-g Type 1 glass bead replicates measured by participating laboratories. The results in Table 32 indicate that for Type 1 beads the mechanical sieve provided the smallest and COM-B provided the largest within-laboratory (repeatability) and between-laboratory (reproducibility) precisions. The better precision in size measurement of Type 1 beads using the mechanical sieve may result from agglomeration of the small beads due to electrostatic forces. Mechanical shaking would overcome these forces and separate the beads, while they would likely stay clustered passing through computerized optical equipment.

Type 3 Samples

The precision estimates for measuring the percent retained in the #18 size class of the Type 3 samples by

Table 32 Precision estimates for measuring percent retained for Type 1 samples by various methods

Method of Measurement—Sample Type	No. of Labs	Repeatability STD, %		Reproducibility STD, %	
		1s	d2s	1s	d2s
Mechanical Sieve—Type 1	14	1.0	2.8	1.7	4.6
COM-A—Type 1	8	1.3	3.8	3.0	8.3
COM-B—Type 1	4	2.1	5.9	3.5	9.7

Table 33 Precision estimates for measuring percent retained for Type 3 samples by various methods

Method of Measurement—Sample Type	No. of Labs	Repeatability STD, %		Reproducibility STD, %	
		1s	d2s	1s	d2s
Mechanical Sieve—Type 3	13	1.1	3.1	3.4	9.4
COM-A—Type 3	8	0.7	1.9	2.1	5.9

the mechanical sieve and by COM-A are provided in Table 33. The comparison does not include COM-B results because only one set of measurements on Type 3 samples was available by COM-B. The precision estimates are based on the size distribution of three 100-g Type 3 glass bead replicates measured by participating laboratories. As shown in Table 33, the COM-A data provided significantly smaller repeatability and reproducibility standard deviations of Type 3 glass beads than did the mechanical sieve.

Type 5 Samples

The precision estimates for the percent retained in the #14 size class of the Type 5 samples by the mechanical sieve and COM-A are provided in Table 34. There are no precision estimates available for COM-B measurements since only one set of data was provided on Type 5 glass beads by COM-B. The precision estimates are based on the size distribution of three 200-g Type 5 glass bead replicates measured by the participating laboratories. As shown in Table 34, the COM-A data provided significantly smaller repeatability and reproducibility standard deviations of Type 5 glass beads than did the mechanical sieve.

Summary of Precision in Size Measurement

Comparison of the precision estimates for measuring the percent retained in the most prevalent size classes of Type 1, Type 3, and Type 5 glass beads revealed important information about the methods of measurement. For Type 1 samples, the mechanical sieve provided the least within and between variability. However, for the Type 3 and Type 5 glass beads, COM-A provided significantly lower variability than the mechanical sieve. The reason for this observed difference may lie in the tendency of fine glass bead particles to agglomerate through electrostatic attraction. Mechanical sieving can break down the agglomerated glass beads, but this will not occur when agglomerations pass through computerized optical method units. A definite conclusion about the variability of the COM-B results cannot be made at this point since only a small number of laboratories reported size measurement of

Table 34 Precision estimates for measuring percent retained for Type 5 samples by various methods

Method of Measurement—Sample Type	No. of Labs	Repeatability STD, %		Reproducibility STD, %	
		1s	d2s	1s	d2s
Mechanical Sieve—Type 5	13	2.2	6.0	4.3	12.0
COM-A—Type 5	7	0.9	2.4	1.2	3.3

Table 35 Precision estimates for measuring percent round in Type 1 samples by various methods

Method of Measurement—Sample Type	No. of Labs	Repeatability STD, %		Reproducibility STD, %	
		1s	d2s	1s	d2s
Roundometer—Type 1	11	2.5	7.1	3.8	10.6
COM-A b/l—Type 1	6	0.7	2.0	1.5	4.2
COM-A SPHT—Type 1	3	1.2	3.4	1.7	4.7
COM-B—Type 1	4	4.3	12.1	7.0	19.7

Type 1 glass beads and only one laboratory reported size measurements of Type 3 and Type 5 glass beads.

Roundness Measurements

Type 1 Samples

Precision estimates of the percent roundness in the #50 size class for the Type 1 samples for various methods of measurement are provided in Table 35. The precision estimates are based on the roundness of three 50-g Type 1 glass bead replicates measured by participating laboratories. As indicated by Table 35, the COM-A b/l parameter provided the least within- and between-laboratory variability among the four methods of measurement.

Type 3 Samples

Precision estimates of the percent roundness in the #18 size class for the Type 3 samples for various methods of measurement are provided in Table 36. The precision estimates are based on the mass percent round of three 100-g Type 3 glass bead replicates measured by participating laboratories. Since precisions for the Type 3 glass beads as measured by the COM-B device could not be developed with the one available dataset, the comparison of the precision estimates was made between the mechanical roundometer and the COM-A SPHT and b/l parameters. As shown in Table 36, the COM-A b/l and SPHT parameters provided comparable repeatability and reproducibility precisions and these precisions were both significantly smaller than those for the roundometer.

Type 5 Samples

Precision estimates of the percent round in the #14 size class of Type 5 samples for various methods of measurement are provided in Table 37. The precision estimates are based on the percent round of three 200-g Type 5 glass bead replicates measured by the participating laboratories. Because the precision for the Type 5 glass beads as measured by the single COM-B instrument could not be developed with one

Table 36 Precision estimates for measuring percent round in Type 3 samples by various methods

Method of Measurement—Sample Type	No. of Labs	Repeatability STD, %		Reproducibility STD, %	
		1s	d2s	1s	d2s
Roundometer—Type 3	8	2.3	6.4	4.1	11.5
COM-A b/l—Type 3	8	0.9	2.5	1.0	2.9
COM-A SPHT—Type 3	4	0.9	2.4	1.1	3.1

Table 37 Precision estimates for measuring percent round in Type 5 samples by various methods

Method of Measurement—Sample Type	No. of Labs	Repeatability STD, %		Reproducibility STD, %	
		1s	d2s	1s	d2s
Roundometer—Type 5	8	1.8	4.9	4.5	12.5
COM-A b/l—Type 5	8	0.6	1.7	1.2	3.5
COM-A SPHT—Type 5	4	1.1	3.0	1.5	4.2

available dataset, the comparison of the precision estimates was made between the mechanical roundometer and the COM-A SPHT and b/l parameters. As shown in Table 37, both the COM-A SPHT and b/l parameters provided lower variability than did the roundometer; the b/l parameter provided better repeatability and reproducibility precision than did the SPHT parameter for measuring the roundness of the Type 5 glass beads.

Summary of Precision in Roundness Measurement

The COM-A b/l parameter consistently provided the smallest repeatability and reproducibility standard deviations for roundness of the three types of glass bead samples examined in this study. Although the SPHT precisions surpassed the roundometer precisions for all three sample types, the SPHT precisions were consistently lower than the b/l precisions. A definite conclusion about the variability of the COM-B results cannot be made at this point because only one set of results on the roundness of each Type 3 and Type 5 was provided. In addition, the number of datasets reported for Type 1 glass beads was much smaller than the number of results reported by the roundometer and the COM-A device.

COMPARISON OF BIAS OF VARIOUS MEASUREMENT METHODS

The *bias of an estimator,* i.e., the statistical comparison of the average measured properties with the target values, is an indication of the accuracy of a test method. In this research, the one-sample t-test was used to test the significance of the difference between the measured and target properties and indicate which of the methods evaluated provided the most accurate measurements of size and roundness. This analysis of bias included a t-test on the size and roundness measurements of the most prevalent size class of each glass bead type. The prevalent size classes are the #50 size class for Type 1 samples, the #18 size class for Type 3 samples, and the #14 size class for Type 5 samples.

Size Measurements

Tables 38, 39, and 40 summarize the percent retained statistics for the three sample types. The computed and critical t-values for a 5% level of significance were used to compute the rejection probabilities provided in the last column of the tables. A rejection probability smaller than 0.05 would indicate that measured and target retained values are significantly different.

Type 1 Samples

Table 38 compares the percent retained measurements on the #50 sieve from the various measuring methods. As the results indicate, the mechanical sieve and COM-A did not provide accurate measurements of the size distribution of Type 1 samples. The p values of 0.001 and 0.007 show that the mechanical sieve and COM-A were statistically different from the target percent retained value of 50%. The COM-B measurement, on the other hand, was not significantly different from the target sieve size as indicated by the rejection probability of 0.111.

Type 3 Samples

Table 39 compares the percent retained measurements on the #18 sieve for the Type 3 samples by the various measuring methods. The COM-B measurements could not be compared statistically since only one set of size measurements of the Type 3 samples was available. The computed t-values exceed the critical t-statistics for both the COM-A and the mechanical sieve methods, indicating that both measurements were statistically different from the target value. However, the COM-A measurements were

Table 38 Results of t-test for comparison of measured and target percent retained on #50 sieve of Type 1 samples

Method of Measurement—Sample Type	No. of Labs	Average% Retained, Measured	% Retained, Target	S_x	Computed t	Critical t	Decision	Rejection Probability (p)
Mechanical Sieve—Type 1	14	48.4	50.0	1.350	4.435	2.160	Reject	0.001
COM-A—Type 1	8	46.4	50.0	2.697	3.775	2.365	Reject	0.007
COM-B—Type 1	4	46.7	50.0	2.945	2.241	3.182	Accept	0.111

Table 39 Results of t-test for comparison of measured and target percent retained on #18 sieve of Type 3 samples

Method of Measurement—Sample Type	No. of Labs	Average% Round, Measured	% Retained, Target	S_x	Computed t	Critical t	Decision	Rejection Probability (p)
Mechanical Sieve—Type 3	13	58.8	55.0	3.197	4.286	2.179	Reject	0.001
COM-A—Type 3	8	57.2	55.0	2.026	3.071	2.365	Reject	0.018
COM-B—Type 3	1	52.5	55.0	—	—	—	—	—

closer than the mechanical sieve measurements to the target value, as indicated by the larger rejection probability for the COM-A data (0.018) when compared with the rejection probability for the mechanical sieve data (0.001).

Type 5 Samples

Table 40 compares the percent retained measurements on the #14 sieve for the Type 5 samples by the various measurement methods. The COM-A percent retained measurements were in very good agreement with the target value of 55% (rejection probability of 0.167) while the mechanical sieve significantly overestimated the percent retained (rejection probability of 0.006).

Summary of Bias in Size Measurement

The t-test results for the size measurement of the glass bead samples revealed that computerized optical methods in general provided more accurate measurements of size than the mechanical sieve. For Type 1 beads, COM-B provided more accurate measurement than did the mechanical sieve; for the larger beads, COM-A was more accurate than the mechanical sieves. It was also indicated that the level of accuracy and precision of COM-A measurements increased as the size of the beads increased. This suggests that computerized optical equipment is especially suitable for measuring the size distribution of the larger-sized glass beads.

Roundness Measurement

Tables 41, 42, and 43 summarize the percent round statistics of the three sample types. The computed and critical t-values for a 5% level of significance are utilized to compute the rejection probabilities. A rejection probability greater than 0.05 indicates that the measured and target roundness of a sample are the same.

Type 1 Samples

Table 41 compares the percent round measurements with the target percent round on the #50 sieve for the Type 1 samples for various measuring methods and parameters. Except for the rejection probability for the COM-B measurements, the rejection probabilities are smaller than 0.05, indicating that only COM-B measured the roundness of Type 1 samples correctly. Despite its accuracy, COM-B provided the most variable measurements as indicated by the standard deviation of the laboratory means ($S_x = 5.95$). The least variable measurement was provided by the COM-A b/l parameter.

Table 40 Results of t-test for comparison of measured and target percent retained on #14 sieve of Type 5 samples

Method of Measurement—Sample Type	No. of Labs	Average % Round, Measured	% Retained, Target	S_x	Computed t	Critical t	Decision	Rejection Probability (p)
Mechanical Sieve—Type 5	13	58.5	55.0	3.771	3.313	2.201	Reject	0.006
COM-A—Type 5	7	55.5	55.0	0.876	1.571	2.447	Accept	0.167
COM-B—Type 5	1	51.9	55.0	—	—	—	—	—

Table 41 Results of t-test for comparison of measured and target percent round on #50 sieve of Type 1 samples

Method of Measurement—Sample Type	No. of Labs	Average % Round, Measured	% Round, Target	S_x	Computed t	Critical t	Decision	Rejection Probability (p)
Roundometer—Type 1	11	74.2	70.0	2.89	4.776	2.228	Reject	0.001
COM-A-b/l—Type 1	6	78.6	70.0	1.26	16.833	2.571	Reject	0.000
COM-A-SPHT—Type 1	3	84.5	70.0	1.38	18.231	4.303	Reject	0.003
COM-B-NSP—Type 1	4	77.3	70.0	5.95	2.435	3.182	Accept	0.093

Type 3 Samples

Table 42 compares the percent round measurements with the target percent round on the #18 sieve for the Type 3 samples. No t-statistics were calculated for the COM-B measurements since only one set of roundness data was available for Type 3 samples. The most accurate roundness measurement was provided by the COM-A b/l parameter (p = 0.616). The roundometer also measured the roundness of the Type 3 samples correctly (p = 0.257), although with the highest variability (S_x = 3.52). The lowest rejection probability (p = 0.000) in Table 42 corresponds to the SPHT parameter, indicating that the measured roundness as judged by this parameter was significantly different from the target roundness.

Type 5 Samples

Table 43 compares the percent round measurements with the target percent round on the #14 sieve of the Type 5 samples. No t-statistics were calculated for the COM-B measurements because only one set of Type 5 roundness data was available. The roundness measurement using the b/l parameter agreed very well with the target roundness of 90% (p = 0.08). The b/l parameter also had a very small standard deviation compared to that of the roundometer (S_x = 1.13 vs. S_x = 4.14). Although both SPHT and roundometer measurements were significantly different from the target roundness, the results from the SPHT parameter were closer to the target value than those from the roundometer (p = 0.036 and 0.003). The SPHT measurements were also less variable than the roundometer measurements (S_x = 1.13 and 4.14).

Summary of Bias in Roundness Measurement

The one-sample t-test results on the glass bead roundness measurements indicated that the computerized optical methods provided more accurate measurements than did the mechanical roundometer. For Type 1 beads, the COM-B device provided the most accurate roundness measurement and for Types 3 and

Table 42 Results of t-test for comparison of measured and target percent round on #18 sieve of Type 3 samples

Method of Measurement—Sample Type	No. of Labs	Average % Round, Measured	% Round, Target	S_x	Computed t	Critical t	Decision	Rejection Probability (p)
Roundometer—Type 3	8	78.5	80.0	3.52	1.233	2.365	Accept	0.257
COM-A-b/l—Type 3	8	80.1	80.0	0.77	0.525	2.365	Accept	0.616
COM-A-SPHT—Type 3	4	86.3	80.0	0.70	17.898	3.182	Reject	0.000
COM-B-NSP—	1	86.8	80.0	—	—	—	—	—

Table 43 Results of t-test for comparison of measured and target percent round on #14 sieve of Type 5 samples

Method of Measurement—Sample Type	No. of Labs	Average % Round, Measured	% Round, Target	S_x	Computed t	Critical t	Decision	Rejection Probability (p)
Roundometer—Type 5	8	83.4	90.0	4.14	4.534	2.365	Reject	0.003
COM-A-b/l—Type 5	8	89.2	90.0	1.13	2.049	2.365	Accept	0.080
COM-A-SPHT—Type 5	4	92.1	90.0	1.13	3.621	3.182	Reject	0.036
COM-B-NSP—Type 5	1	91.0	90.0	—	—	—	—	—

5 beads, the COM-A b/l parameter provided a significantly more accurate roundness measurement than did the mechanical roundometer. However, the SPHT parameter of COM-A failed to measure the roundness of any of the glass bead types correctly. This may result from shortcomings of the parameter or a poor choice of threshold value.

CONCLUSIONS

Three methods for size and roundness measurement were evaluated in this research: two computerized optical methods (COM-A and COM-B) and the traditional mechanical methods—sieving and roundometer following ASTM D1214 and ASTM D1155, respectively. The largest number of datasets were obtained for mechanical sieving and the roundometer: 14 datasets of size distribution and 11 of roundness data. COM-A users provided eight datasets, which included both size and roundness data. COM-B datasets were received from four laboratories for small size glass beads (Type 1) and from one laboratory for larger glass beads (Types 3 and 5). The samples upon which these tests were run were carefully prepared via mechanical sieving and mechanical roundness measurement, so accuracy in the ILS for the roundometer, COM-A, and COM-B results is defined as how close these measurement methods came to the original mechanical sieving and roundness measurements, allowing for the error introduced in the sample preparation process.

The ILS data received from participating laboratories were statistically analyzed for precision. The significance of the bias for each method between measured and target values was evaluated separately for each sieve size of each glass bead type using the one-sample t-test. Following the ASTM E691 methodology, both within- and between-laboratory variability of the computerized and mechanical methods was also determined for each sieve size of each glass bead type.

The computed t-values and the computed within- and between-laboratory standard deviations for each glass bead type were compared to determine the accuracy and precision of the different methods in measuring the properties of the glass beads. For each glass bead type, comparison was made between the statistics corresponding to the sieve sizes retaining the highest mass percentage of the beads. This was done because these sieves provided the most precise measurements of the glass bead type.

Because only one laboratory provided size and roundness measurements of Type 3 and 5 glass beads using the COM-B instrument, the discussion of precision and bias for measurements of the properties of Type 3 and Type 5 glass beads does not apply to the COM-B results. The results of the comparison are summarized as follows:

- Mechanical sieving, the COM-A device, and the COM-B instrument were used to measure the size of Type 1 glass beads. Analysis of the mass percent retained in the largest size class of the Type 1 samples indicated that of the three methods of measurement, the COM-B device provided the most accurate measurement of the size of Type 1 glass beads. With respect to variability, the mechanical sieve provided the smallest within-laboratory standard deviation for measuring the size of the small beads. However, the between-laboratory precisions of the three methods were very similar.

- Data from the mechanical sieve and the COM-A device were used to develop precision and bias estimates for size measurement of the Type 3 and Type 5 beads. Between the two methods, the COM-A instrument measured the size of the Type 3 and Type 5 glass beads with more accuracy than the mechanical sieves. In addition, the COM-A device provided smaller within- and between-laboratory variability than did the mechanical sieves.
- Four combinations of methods and parameters were used for measuring the roundness of Type 1 glass beads: mechanical roundometer, COM-A-b/l, COM-A-SPHT, and COM-B-T/L. Among the four combinations, the COM-B-T/L provided the most accurate measurement of roundness of small glass beads. However, COM-A-b/l provided the most precise within- and between-laboratory measurements. The mechanical roundometer did not provide equivalent accuracy and precision for measuring the roundness of Type 1 glass beads.
- Three combinations of methods and parameters were used for measuring the roundness of Type 3 and Type 5 glass beads: the mechanical roundometer, COM-A-b/l, and COM-A-SPHT. Among the three combinations, COM-A-b/l provided the most accurate and precise measurement of the roundness of Type 3 and Type 5 glass beads. The mechanical roundometer did not provide equivalent accuracy and precision in measuring the mass percent round of Type 3 and Type 5 glass beads.

Taken together, these results suggest that computerized optical methods are preferable to the traditional mechanical methods for measuring the size and roundness of glass beads. The improved statistics of the b/l parameter for the larger glass beads indicated the advantage of COM-A over the roundometer for roundness measurement of the larger glass beads. Although a smaller number of laboratories provided data using the COM-B device, both size and roundness of the Type 1 glass beads were correctly measured by the COM-B instrument.

A draft practice in AASHTO standard format for determining the size and roundness of glass beads utilized in traffic markings using the computerized optical method is provided in Appendix A. This draft practice includes a precision and bias statement for the computerized optical method measurements based on the results of this research.

APPENDIX A: RECOMMENDED TEST METHOD FOR MEASUREMENT OF SIZE DISTRIBUTION AND ROUNDNESS OF GLASS BEADS USING COMPUTERIZED OPTICAL EQUIPMENT

PROPOSED STANDARD PRACTICE FOR

Determination of Size and Roundness of Glass Beads Utilized in Traffic Markings Using a Computerized Optical Method

NCHRP 20-07: PP XX (to come)

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1. SCOPE

1.1 This practice describes measuring size and roundness of translucent glass beads used in traffic markings with computerized optical equipment. This practice is intended for glass beads from 0.15 mm to 2.35 mm in diameter.

1.2 *This standard may involve hazardous materials, operations, and equipment, This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to its use.*

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2. REFERENCED DOCUMENTS

2.1 *AASHTO Standard*
- M 247 Standard Specification for Glass Beads Used in Pavement Markings

2.2 *ASTM Standards*
- D1214-04 Standard Test Method for Sieve Analysis of Glass Spheres
- D1155-03 Standard Test Method for Roundness of Glass Spheres
- B215-08 Standard Practices for Sampling Metal Powders

2.3 *ISO Standards*
- ISO 13322-2 International Standard for Dynamic Image Analysis Method

- ISO 1448 International Standard for Particulate Materials– Sampling and Sample Splitting for the Determination of Particulate Properties

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3. TERMINOLOGY

3.1 *Definitions:*

3.1.1 *Dosage Funnel*—For feeding the glass beads to the device

3.1.2 *Dosage Feeder*—Vibration unit for control of particle delivery

3.1.3 *Guide plate*—For orienting the fine particles

3.1.4 *Measurement Shaft*—Volume through which particles fall and their images are captured.

3.1.5 *Image capture device*—Minimum of two digital cameras

3.1.6 *Particle illumination unit*—Light source for continuous illumination for image capture device

3.1.7 *Sample collection container*—For collecting the glass beads at the end of the test

3.1.8 *Particle size analyzer*—A general term for computerized optical equipment

3.2 *Description of Terms (See Figure 1)*:

3.2.1 X_{cmin} *(particle width) or b*—The shortest chord of the measured set of maximum chords of a particle projection (for close correlation to sieving).

3.2.2 *T*—Thickness of the particles

3.2.3 *Chord*—A chord is a line segment joining two points on a surface of a particle

3.2.4 X_{Fe} *Feret diameter*—Distance between two tangents placed perpendicular to the measuring direction. For a convex particle the mean Feret diameter (mean value of all directions) is equal to the diameter of a circle with the same circumference.

3.2.5 X_{Femax} *or L*—The longest Feret diameter out of the measured set of Feret diameters.

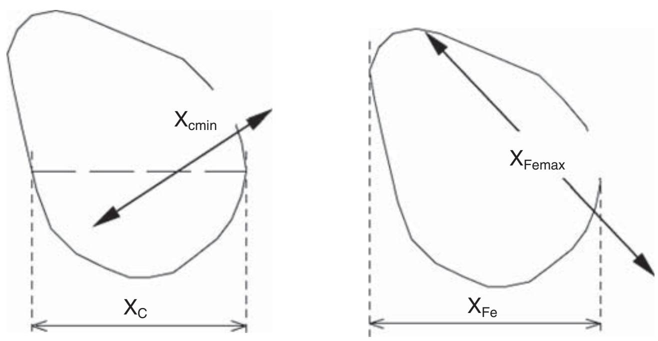

Figure 1 Scheme of X_{cmin} and X_{Femax}

3.3 *X_{cmin} / X_{Femax} or b/l*—Measure of roundness. For an ideal circle, *b/l* is 1, otherwise it is smaller than 1. The threshold value used for measuring percent round using b/l is approximately 0.85.

3.4 *SPHT*—Roundness parameter = $4\pi A/P^2$. For an ideal circle, SPHT is 1, otherwise it is smaller than 1. The threshold value used for measuring percent round using SPHT is approximately 0.93. A is the measured area, and P is the measured perimeter.

3.5 *NSP*—Roundness parameter, $(SPHT)^{1/2}$. For an ideal circle, NSP is 1, otherwise it is smaller than 1. The threshold value used for measuring percent round using NSP is the same as SPHT which is approximately 0.93.

3.6 *T/L ratio*—Measure of roundness, for an ideal circle *T/L* is 1, otherwise it is smaller than 1. The threshold value used for measuring percent round using T/L is 0.82.

Note 1—Based on analysis of X-ray tomography images of various glass bead types it was found that the threshold value of a roundness parameter is not the same for different glass bead types. Therefore, there are uncertainties associated with using a single cutoff threshold for all glass bead types. The proposed threshold values for each roundness parameter have been computed as the median over each range of threshold values corresponding to Types 1, 3, and 5 glass beads.

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4. SUMMARY OF PRACTICE

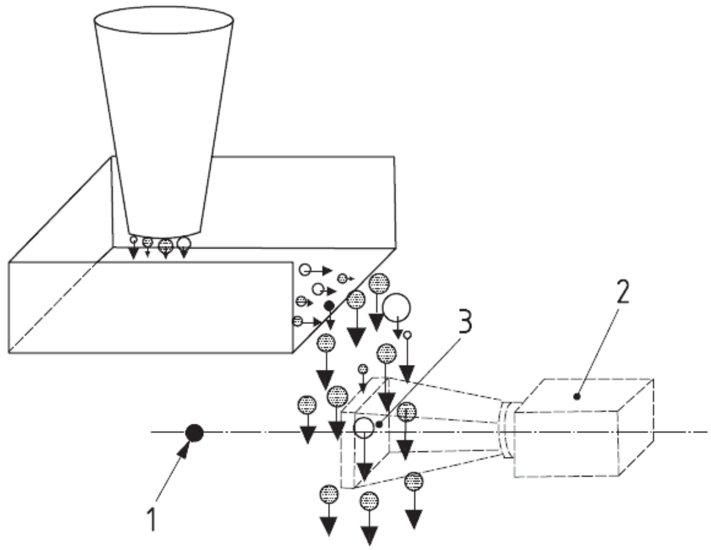
4.1 This practice describes the sample preparation and measuring size and roundness of translucent glass beads by computerized optical equipment. The glass particles are run through a flowing stream digital image analyzer and images of the free-falling particles are taken at a minimum rate of 60 images/s from different directions. The images are analyzed by image analysis software to measure the various properties of the glass beads such as size, roundness, and total number. The measurement time depends on the quantity of material to be measured, the width of the metering feeder, and the mean grain size. Typical measuring times are approximately 2 min to 10 min for the amount of glass beads specified (see Table 1).

5. SIGNIFICANCE AND USE

5.1 The size and roundness of glass beads affect the retroreflectivity of pavement markings. The purpose of this test method is to measure the size and roundness of glass bead types in compliance with AASHTO M 247 specifications. This test method replaces mechanical sieve analysis (ASTM D1214) and mechanical roundness measurement (ASTM D1155).

6. APPARATUS

6.1 *Computerized Optical Equipment*—An optical-electric instrument for the measurement and analysis of size, shape, and count of free-flowing glass beads. Figure 2 provides a schematic diagram of the measurement components of the system. The equipment is structured into a dosage funnel, a vibrating dosage feeder, guide plate, measurements volume, an illumination unit, image capturing device, image analysis software, and sample collection container. The instrument is capable of acquiring images of free-falling glass particles at a minimum speed of 60 frames/s using a minimum of two image capture devices.



Key

1 light source
2 camera
3 measurement volume

Figure 2 Schematic diagram of components of the Digital Particle Analyzer [Courtesy of ISO13322-2]

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7. HAZARDS

7.1 *General Safety Information*—These devices are suitable for measuring free-flowing dry and non-toxic material. Please make sure that all information contained in the material safety data sheets of the analyzed materials is observed. If used in compliance with the operating instructions, the instrument can be operated safely and efficiently.

7.2 *Personal Safety*—The following safety rules should be followed to prevent any personal injury caused by improper use:

7.2.1 Every person working with the Particle Analyzer should read and understand the manufacturer's safety regulations and operating instructions, and be familiar with the safe and intended use of the instrument.

7.2.2 Every person working with the Particle Analyzer should have access to the instruction manual for this instrument.

7.3 *Material Safety*—All safety regulations for the material to be analyzed should be observed. Use standard safety precautions when handling glass beads. Spilling glass beads on the floor will result in a slippery walking surface.

7.4 *Device Safety*—Repair of the equipment should not be carried out by the user. The equipment supplier should be contacted when repair is needed.

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8. OPERATING CONDITIONS

8.1 Environmental temperature: 10ºC … 40ºC.

8.2 Air humidity: 80% maximum relative humidity at temperatures up to 30ºC, linear decrease to 50% maximum relative humidity at a temperature of 40ºC.

8.3 Height of installation and operation: maximum 3000 m above sea level.

8.4 Installation location: place the Particle Analyzer on a firm, horizontal, vibration-free surface.

8.5 Light conditions: avoid strong direct external light on the particle measurement shaft or on the cameras.

8.6 This Test Method is intended for indoor use only. Deviation from this should be conducted with advice from the manufacturer.

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9. STANDARDIZATION

9.1 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. Equipment associated with this practice requires periodic calibration. Refer to the pertinent section of the manual documents for information concerning calibration.

9.2 Calibration has to be done for the first start-up of the program together with the customer, or each time the camera has been moved, or if the instrument has been moved to another location.

10. CLEANING

10.1 Occasionally, all parts that are in contact with the sample material, like the dosage funnel, dosage feeder, guide plate, measurement shaft, and sample collection container should be cleaned, especially if the material contains a high proportion of dust or if the sample type is changed. The cleaning may be performed with compressed air and with a soft, dry brush. The cover glass of the illumination unit and the protective glass coverings on the front of the camera unit can be cleaned with ethyl alcohol.

11. MEASUREMENT OF GLASS BEAD PROPERTIES

11.1 *Test Specimen Preparation*

11.1.1 Prepare at least two test specimens for each glass bead type. The sample size is dependent on the particle size range. Table 1 provides the appropriate mass of each glass bead type for use with the computerized optical equipment.

Note 2—A reasonable mass tolerance for test specimens is ± 0.5 g.

Table 1 Appropriate mass for various size glass bead types specified in AASHTO M 247

AASHTO Type	Range (μm)	Range of U.S. Sieve Sizes	Specimen Weight
Type 0	600–180	#30–#80	50 g
Type 1	1180–150	#16–#100	50 g
Type 2	1400–150	#14–#100	70 g
Type 3	1700–710	#14–#25	100 g
Type 4	2000–850	#10–#20	150 g
Type 5	2350–1000	#8–#18	200 g

11.1.2 Measure the mass of the glass beads from a sample reduced by a sample splitter following the sampling procedures recommended in ASTM B215-08 or ISO 1448.

11.1.3 Pour entire glass bead sample into a glass beaker or suitable container.

11.1.4 Place the beaker in a 110°C ± 5°C oven for 1 hr to dry out the glass beads to assure they are free flowing.

11.1.5 Remove the beads from the oven and allow them to cool to room temperature for about 15 min prior to testing.

11.1.6 Record the mass of each test specimen.

11.2 *Computerized Optical Equipment Preparation*

11.2.1 All measuring and analysis parameters should be determined initially and saved into the pre-defined files referred to as task files or method files.

Note 3—Check with instrument manufacturer for suggestions on how to best set up any software that comes with the instrument. Setting up the instrument software properly will allow for meaningful reports.

Note 4—For optimal future operation and measurements it is sensible to prepare different "task" files for the different materials, because the particle characteristics, size classes, and the optimum parameters for feeder control will usually be different for different materials.

11.2.2 Include the following information in the task file:

11.2.2.1 Insert the approximate maximum size of the particles.

11.2.2.2 Insert the width of the feeder.

11.2.2.3 Insert the height of the dosage funnel which is determined based on the size of the largest aggregate. The recommendations for the gap between funnel and vibration feeder is 2 times the size of the largest beads.

11.2.2.4 Adjust the vibration amplitude of the feeder plate.

11.2.2.5 Mark the use of guide plate when measuring very fine glass beads. This will ensure that the orientation of the particles during the free-fall phase is aligned.

11.2.2.6 Set the opening of the guide plate slightly larger than the largest particle diameter in the sample to prevent blocking of the guide plate during measurement. However, the distance should be as small as possible. The right gap for the guide plate is 1.5

times the diameter of the biggest beads or "1 mm fixed for all beads between 0 mm and 0.6 mm" and "3 mm fixed for all beads between 0.4 mm and 2.5 mm."

11.2.2.7 Activate the use of air flow if testing fine particles.

11.2.2.8 Enter the sieve classifications. Use the sieve sizes based on the sample types. Table 2 provides the sieve sizes of each glass bead type specified in AASHTO M 247.

Table 2 Sieve sizes in micrometers to be selected for various glass bead types specified in AASHTO M 247

Type 0	Type 1	Type 2	Type 3	Type 4	Type 5
600	1180	1400	1700	2000	2350
425	850	1000	1400	1700	2000
300	600	710	1180	1400	1700
180	300	500	1000	1180	1400
150	150	300	850	1000	1180
–	–	150	710	850	1000

11.2.2.9 Choose X_{cmin} (b) or T parameter for sizing. Choose percent passing and percent retained.

11.2.2.10 Select X_{cmin}/X_{Femax} (b/l) or T/L for roundness measurement; use a threshold of 0.85 for b/l and threshold value of 0.83 for T/L.

11.2.2.11 Select SPHT or NSP for roundness measurement; use a threshold value of 0.93.

11.2.2.12 Choose percent round in each class size based on X_{cmin}/X_{Femax} (b/l) or T/L.

11.2.2.13 Choose percent round in each class size based on SPHT or NSP.

11.2.2.14 Select weighted average percent round in each sample using X_{cmin}/X_{Femax} (b/l) or T/L.

11.2.2.15 Select weighted average percent round in each sample using SPHT or NSP.

11.2.2.16 Select D10, D50, and D90 for measuring the diameters at which 10%, 50%, and 90% of the mass of a glass bead sample is finer, respectively.

11.2.3 Save task file in order to save the created method.

11.2.4 Load the sample into the dosage funnel feeder of the equipment.

11.2.5 Choose the created task file and start the measurement.

11.2.6 The measured results are available a few moments after the measurements are completed.

11.2.7 After the measurements are completed, save the results.

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12. DATA ANALYSIS

12.1 Analysis of the data is done automatically using the computerized optical equipment software.

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13. REPORT

13.1 The report of the analysis should include the following information:

13.1.1 Percent retained and passing of particles in each class size

13.1.2 Percent of round by X_{cmin}/X_{Femax} (b/l) or T/L in each class size

13.1.3 Percent of round by SPHT or NSP parameter in each class size

13.1.4 Value of X_{cmin}/X_{Femax} (b/l) or T/L for each size classification and the weighted average value for the whole sample

13.1.5 Value of SPHT or NSP for each class size and the weighted average value for the whole sample

13.1.6 Values of D10, D50, and D90

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14. PRECISION AND BIAS

14.1 *Precision*—Criteria for judging the acceptability of percent retained and percent round results obtained by this computerized optical method are given in Table 3.

Single-Operator Precision (Repeatability)—The figures in Column 2 of Table 3 are the within standard deviations that have been found to be appropriate for the conditions of tests described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results exceeds the values given in Table 3, Column 3.

14.1.1 *Multilaboratory Precision (Reproducibility)*—The figures in Column 4 of Table 3 are the between standard deviations that have been found to be appropriate for the conditions of tests described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results exceeds the values given in Table 3, Column 5.

Table 3 Precision estimates for percent retained and percent round of Type 1, Type 3, and Type 5 glass beads

Type Index and Test Property	Standard Deviation (1s)[a]	Acceptable Range of Two Test Results (d2s)[a]	Standard Deviation (1s)[a]	Acceptable Range of Two Test Results (d2s)[a]
	Single-Operator Precision		Multilaboratory Precision	
Percent Retained				
Type 1	1.34	3.8	2.98	8.3
Type 3	0.67	1.9	2.12	5.9
Type 5	0.85	2.4	1.18	3.3
Percent Round				
Type 1	1.01	2.8	1.59	4.5
Type 3	0.88	2.5	1.08	3.0
Type 5	0.86	2.4	1.38	3.9

[a] These values represent the 1s and d2s limits described in ASTM Practice C670

Note—The precision estimates given in Table 3 are based on the analysis of test results from an AMRL interlaboratory study (ILS). The ILS data consisted of size and roundness results from eight laboratories testing three replicates of three sets of glass bead samples using computerized optical equipment. The materials included Type 1, Type 3, and Type 5 glass beads described in AASHTO M 247. The average mass percent retained of the predominant size class of Type 1 samples was 50% and the average mass percent retained in the predominant size class of Type 3 and Type 5 samples was 55%. The average mass percent round was 70%, 80%, and 90% for Type 1, Type 3, and Type 5, respectively. The details of this analysis are in *NCHRP Research Results Digest 346*

14.2 *Bias*—No information can be presented on the bias of the procedure because no comparison with the material having an accepted reference value was conducted.

15. KEYWORDS

15.1 Sieve size; roundness; glass beads; particle size analyzer

16. REFERENCES

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16.2 Anta Tec As, Norway

16.3 Wolfram Mathematica, http://mathworld.wolfram.com

16.4 *NCHRP Research Results Digest 346*

16.5 *NCHRP Web-Only Document 156*

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These digests are issued in order to increase awareness of research results emanating from projects in the Cooperative Research Programs (CRP). Persons wanting to pursue the project subject matter in greater depth should contact the CRP Staff, Transportation Research Board of the National Academies, 500 Fifth Street, NW, Washington, DC 20001.