**Designation: ~~E2776 – 18~~ E2776 – 20**

# Standard Guide for Correlation of Results of Solid Particle Size Measurement Instruments[1]

This standard is issued under the fixed designation E2776; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. ~~Scope~~ Scope*

1.1 This guide describes one methodology to correlate solid particle analysis results between solid particle analysis instruments for user specified products of user specified particle sizes and distributions in order to expand the capability of particle measurement throughout the manufacturing process and provide better control and efficiency. The guide is not limited to instrument type or product type.

1.2 **Warning—** ~~All~~ Not all instruments may ~~not~~ correlate to all other instruments for various user specified products and size ranges. Instruments may measure different particle features, and they may also measure the same particle features differently and thus correlating the results of any two may be possible for some products but not possible for others. It is also the case that certain materials can be altered by the instruments measuring them which would eliminate them from consideration under this guide if the instrument's results are determined based on measurements made after the instrument has altered the user specified product.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*



[1] This guide is under the jurisdiction of ASTM Committee E29 on Particle and Spray Characterization and is the direct responsibility of Subcommittee E29.02 on Non-Sieving Methods.

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

## 2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *bin, n*—a user specified division of the overall particle size range of a user specified product.

2.1.2 *correlation, n*—mathematical equation(s) relating one set of numerical values to another.

2.1.3 *particle analysis instrument, n*—any instrument of any type that can produce a particle size distribution measurement of a product. There is no restriction on technology or methodology used by the instrument to measure particles nor is there any restriction of particle characteristics used to report results of the measurement.

2.1.4 *user specified product, n*—indicates a product manufactured by the user to a specified size distribution, usually indicated by common sieve screen sizes.

## 3. Summary of Guide

3.1 This guide describes a method which can be used to correlate results between instruments which measure particle size, and distribution, of materials by the same or different parameters and principles.

3.2 The primary interest is the correlation of particle size measurements of user specified products.

3.3 This guide can be used for any two particle measuring instruments which output a user specified distribution and have the capacity to shift bin boundaries within the software. Therefore, a set of sieves cannot correlate to another instrument, but another instrument may correlate to the set of sieves. Ideally, the bin boundaries for the correlating instrument would match the bin boundaries of the primary instrument, or if correlating to sieves, match the range of each corresponding sieve, but if they do not, the method described in this ~~document~~guide could be used to adjust the individual bin boundaries used by the correlating instrument to make the volume percent detected for each bin closely match the percent retained by each corresponding bin of the primary instrument or set of sieves.



3.4 The guide is valid for any two instruments as long as it can be demonstrated that the correlation results are useful to the user.

## 4. Significance and Use



4.1 It is useful to be able to obtain particle size measurement results of a user specified product from multiple instruments and to be able to correlate the results of the measurements. This capability can be advantageous in expanding the use of different technologies to make a measurement or simply to correlate results between instruments of the same technology. An example might be comparing in-process particle size measurements to final inspection particle size measurements.

4.2 The viability of this guide will need to be tested on a case-by-case basis as various products may present measurement challenges for some instruments and not all results from all instruments may be able to be correlated to all other results from all other instruments. In addition, positive results should be confirmed and improved with continued data comparisons over time using process measurements from the instruments selected.

## 5. Procedure

NOTE 1—Depending on instrument type and features, this procedure might not exactly match the steps required to create a correlation on a particular instrument. The steps referenced herein are meant to guide the user toward creating a correlation by following similar logic appropriate to the instrument selected.

5.1 The first step is for the user to select a particular manufactured product which has a defined particle size range and distribution already determined by the user's sieves or other instrument.

NOTE 2—Ideally, distributions will be reported in ~~discrete gradations however~~discreet gradations; however, this guide can also be used where the determination of the distribution is a continuous curve.

5.2 The procedure requires the gathering of twenty-one representative samples of the user specified product which will be used to determine a correlation which can be used for future measurements.

5.3 The sample size (mass) can be determined by assessing the statistical particle count for the largest bin size that will yield a particular confidence level. The example in Annex A1 illustrates the exercise in a straight forward manner.

5.4 Using the gradations indicated in the user's product specification, determine the particle size distribution of each sample using the primary instrument of measure. Confirm that the instrument is in good calibration in accordance with the manufacturer's instructions or a standard procedure appropriate to the industry.

5.5 Run one sample through the correlating instrument three times and create a correlation by shifting the bin boundaries so the result closely matches the primary instrument result.

5.6 Calculate the variation and average repeatability of the correlating instrument based on these results. Ideally, the repeatability would be better than 5 % in the highly populated bins. The user will have to make a judgment on the applicability of any correlation based on the accuracy and repeatability of the correlating instrument involved and the precision needed to control the process.

5.7 If the results are acceptable, then verify the set up by running the remaining twenty samples. If adjustments are required, make them and then re verify. Determine the final accuracy and repeatability of the correlation and determine if it is adequate for measuring the process.

5.8 Once the correlation is confirmed, the software file of the correlating instrument must be saved as a unique file containing the fixed settings and correlation for the particular user specified product tested. If either the primary or correlating instrument are altered in a way that might affect measurement results, this entire procedure must be repeated.

5.9 It is recommended that the correlation be tested near the specification boundaries. An example is given in Appendix X3.



## 6. Report

6.1 The particle distribution produced by the correlating instrument should be reported using the gradations identified for the user specified product.

NOTE 3—Changing the bin boundaries will not change the name of the bin. For example, a 325 mesh bin might have its boundary shifted from 44 µm to 35 µm, but it is still identified as the 325 mesh bin.



6.2 Report the correlating instrument model and software version as well as the file name and version containing the correlation. Include a statement indicating the instrument is reporting results correlated to another instrument ~~per~~ in accordance with this guide. Indicate the primary instrument and reference this ~~document.~~guide. It may also be informative to include the correlation results in a format similar to the tables presented in Section X2.1. An example report format follows:

Correlation Report

Primary Instrument: (include identifying information; model #, serial number etc....)
Correlating Instrument: (include identifying information; model, serial number etc....)
Sample: (include sample product name, lot, date run and any other identifying codes as appropriate)
Results: (report size distribution of sample in tabular or graphical form)

## 7. Keywords

7.1 bin; correlation; distribution; particle size

## ANNEX

**(Mandatory Information)**

## A1. DETERMINATION OF SAMPLE SIZE

A1.1 As indicated in 5.3, it is important to select a sample size that will yield a high enough number of particles for statistical significance. A straight forward way to do this is detailed in this annex by means of an example.

### A1.2 Assumptions

A1.2.1 Particle range: 1 µm – 100 µm.

A1.2.2 Five gradations (bins) as follows with 20 % by mass in each: pan, 20 µm, 40 µm, 60 µm, and 80 µm. Fewest number of particles will reside in the 80-µm bin, so using that as the limiting factor of the analysis, compute the number, and then mass, of sample required.

### A1.3 Method



A1.3.1 Select a standard error (Sn) allowable. The standard error is the measured standard deviation of a sample. For this example, 5 % will be used.

A1.3.2 Calculate the number of particles required in the largest bin. Assume all particles at the median bin size, in this case 90 microns.



$$\begin{aligned} Sn &= 1/(n^{1/2}) \\ n &= \text{number of particles} \\ n &= (1/0.05)^2 \\ n &= 400 \text{ particles required} \end{aligned} \tag{A1.1}$$

A1.3.3 With the number of particles now calculated for the top bin, a minimum sample mass can be projected for the entire sample by converting the 20 % bin mass to 100 %. Assume the product density is 1 g/cm$^3$.

$$\text{Sample Mass} = 400 \times (100/20) \times [(\text{Pi}/6) \times (90 \times 10^{-4})^3 \text{ cm}^3] \times 1 \text{g/cm}^3 = 0.00076 \text{ g} \tag{A1.2}$$

One must still ensure that the correlating instrument actually measures the minimum number of particles ($n$).

# APPENDIXES

(Nonmandatory Information)

## X1. SAMPLE CORRELATION

### X1.1 Sample Description

X1.1.1 The sample used in this illustration of the correlation is silica frac sand. Correlation is between a sieve stack and an imaging system.

### X1.2 User Specified Distribution

X1.2.1 Sieves that ~~makeup~~ make up the distribution of this sand are as follows:

*(1)* 20 USS,
*(2)* 30 USS,
*(3)* 35 USS,
*(4)* 40 USS,
*(5)* 45 USS,
*(6)* 50 USS,
*(7)* 70 USS, and
*(8)* PAN.



X1.2.2 Product distribution is defined as follows:

*(1)* 90 % passing 30 screen and retained above 50 screen,
*(2)* No more than 0.1 % on 20 screen, and
*(3)* No more than 1 % on 70 screen.



### X1.3 Analysis of Sample 1

X1.3.1 Sample 1 is taken from production inventory at a sand manufacturing facility. Table X1.1 shows the percentage in each bin as sieved (on the left) and the raw correlating instrument results after three runs (on the right) and the correlated values in bold type in Column 4 of Table X1.1. Correlation is within 1.5 % for each bin.

**TABLE X1.1 Sample 1**

| Sample 1 Sieve Results | | | | | Sample 1 Uncalibrated | | Instrument Results | | |
|---|---|---|---|---|---|---|---|---|---|
| Bin | Grams | % | Recal Run | | Run 1 | Run 2 | Run 3 | Std Dev | *r* (%) |
| 20 USS | 0.05 | 0.05 | 0 | 20 USS | 1.09 | 0.74 | 0.78 | 0.19 | 42.94 |
| 30 USS | 2.67 | 2.72 | 2.43 | 30 USS | 21.03 | 19.48 | 17.49 | 1.77 | 17.97 |
| 35 USS | 16.69 | 17.03 | 16.07 | 35 USS | 39.98 | 39.51 | 38.00 | 1.03 | 5.17 |
| 40 USS | 51.10 | 52.13 | 51.25 | 40 USS | 30.41 | 31.65 | 33.75 | 1.69 | 10.37 |
| 45 USS | 22.70 | 23.16 | 24.64 | 45 USS | 6.79 | 7.56 | 8.75 | 0.99 | 25.20 |
| 50 USS | 4.21 | 4.29 | 4.92 | 50 USS | 0.51 | 0.76 | 0.88 | 0.19 | 50.88 |
| 70 USS | 0.56 | 0.57 | 0.61 | 70 USS | 0.16 | 0.25 | 0.28 | 0.06 | 52.65 |
| Pan | 0.05 | 0.05 | 0.06 | Pan | 0.03 | 0.05 | 0.06 | 0.02 | 68.87 |
| Total | 98.03 | 100.00 | | | | | | | |

**TABLE X1.2 Imaging – Sieve Comparison of a 30/50 Frac Sand Sample**

| US Sieve Size | Sieve % Retained | Imaging % Retained | Correlated % Retained | Shifted Bin Low End | Shifted Bin High End |
|---|---|---|---|---|---|
| 20 | 0 | 0.1 | 0.08 | 850 | 850+ |
| 30 | 4.1 | 7.8 | 4.2 | 634.9 | –850 |
| 50 | 95 | 91.4 | 95 | 310.9 | –634.9 |
| 70 | 0.8 | 0.6 | 0.7 | 224.4 | –310.9 |
| Pan | 0.2 | 0.1 | 0.02 | 0 | –224.4 |

**TABLE ~~X1.2~~X1.3 Sample 2**

| Bin | Mass (g) | % | Correlating Instrument |
|---|---|---|---|
| 20 USS | 0.05 | 0.05 | 0 |
| 30 USS | 2.26 | 2.39 | 3.81 |
| 35 USS | 20.46 | 21.60 | 18.82 |
| 40 USS | 49.69 | 52.46 | 51.1 |
| 45 USS | 19.28 | 20.35 | 22.3 |
| 50 USS | 2.78 | 2.93 | 3.73 |
| 70 USS | 0.20 | 0.21 | 0.22 |
| Pan | 0.00 | 0.00 | 0.01 |
| Total | 94.72 | 100.00 | |

X1.3.2 In this example, the boundaries of the bins used by the correlating instrument were adjusted so that the total percent mass of each bin matched closely with the corresponding gradation of the primary instrument, in this case a sieve. The requirements stated in Section X1.2 were met and so this correlation would be acceptable (pending completion of testing in accordance with Section 5 of this guide). The data captured in the sample runs was used by the software to compare one gradation at a time to its corresponding sieve. A sieve was selected to match and the instrument boundaries corresponding to that sieve were adjusted to include a revised number of particles to better match the percent retained on the corresponding sieve. The remaining gradations were then adjusted to match the sieves they correspond to. In this particular case, the boundary changes were made by the ~~software, however~~software; however, manual changes could also have been made. Changes may need to occur on an iterative basis.



## X1.4 Bin Shift Example



X1.4.1 An example of boundary shifts for a frac sand test are shown in Table X1.2.

## X1.5 Analysis of Sample 2

X1.5.1 Sample 2 is the same type of sand as in Sample ~~1, however~~1; however, it was taken from a different production lot several weeks later. The same software correlation is used to run this sample through the correlating instrument. Column 3 of Table ~~X1.2~~X1.3 shows the sieve percent in each bin and Column 4 of Table ~~X1.2~~X1.3 shows the correlating instrument percent for each bin.

## X2. MULTI SAMPLE DATA

### X2.1 Correlated Sieve Results from Multiple Samples

X2.1.1 Another 30/50 silica frac sand was used for testing. Twenty samples from a lot of approved sand was analyzed using the correlation determined in Appendix X1. See Tables X2.1-X2.20.

### X2.2 Correlation of Abrasive Sample

X2.2.1 A silicon carbide material was correlated to a sieve measurement and then a fresh sample was tested. The specification and result are shown in Table X2.21 and Table X2.22, respectively.



**TABLE X2.1 Sample 1**

| | | | Instrument % | | | | |
|---|---|---|---|---|---|---|---|
| | Mass in Grams | Sieve % | Run 1 | Run 2 | Run 3 | Std Dev | *r* |
| 20 | 0.02 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30 | 2.68 | 2.33 | 1.93 | 1.78 | 1.81 | 0.08 | 0.22 |
| 35 | 19.76 | 17.20 | 16.06 | 14.96 | 14.65 | 0.74 | 2.08 |
| 40 | 59.95 | 52.18 | 52.55 | 51.34 | 51.89 | 0.16 | 1.70 |
| 45 | 26.18 | 22.79 | 24.08 | 25.82 | 25.27 | 0.89 | 2.49 |
| 50 | 5.14 | 4.47 | 4.58 | 5.20 | 5.44 | 0.45 | 1.25 |
| 70 | 0.8 | 0.70 | 0.58 | 0.71 | 0.74 | 0.09 | 0.25 |
| Pan | 0.37 | 0.32 | 0.23 | 0.20 | 0.19 | 0.02 | 0.06 |
| Total | 114.9 | 100.00 | | | | | |