Designation: F577 – 03 (Reapproved 2009)

Standard Test Method for Particle Size Measurement of Dry Toners[1]

This standard is issued under the fixed designation F577; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers aperture particle size analysis using an electronic sensing zone apparatus provided with a digital pulse processor. Dry inks, toners, and so forth, are covered. Particles as small as 1 µm and as large as 120 µm can be analyzed.

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

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

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *channel*—a size subgroup that has been obtained by dividing the range of the analysis into a certain number of size categories. The resolution of the analysis is increased when the number of channels is increased.

2.1.2 *dynamic range*—the ratio between the upper and lower limit of an analysis.

2.1.3 *number size distribution*—the number size distribution is measured and may be represented in a number percent curve as differential, cumulative larger than or cumulative smaller than. (Figs. 1-3).

2.1.4 *pulse (man height average) by sequence*—the max pulse height average is calculated from the pulses generated during the anaylsis (Fig. 4).

2.1.5 *median particle size*—the median size (50 % oversize or undersize) is a convenient value for the central tendency of a size distribution curve. For a distribution derived by number of particles, it is called the number median size.

2.1.6 *volume size distribution*—the volume size distribution is calculated by the instrument's software and may be represented in a volume percent curve as differential, cumulative larger than or, cumulative smaller than. (Figs. 5-7).

3. Summary of Test Method

3.1 This technique (**1**) [2] determines the number and size of particles suspended in an electrolyte by causing them to flow through a small orifice on both sides of which are immersed electrodes. Voltage pulses, whose amplitudes are proportional to the particle volumes, are generated by changes in resistance as the particles pass through the orifice. The signal generated is scanned, digitized and integrated in pulses. These pulses are processed yielding size and pulse distributions. The pulse data is saved and may be reprocessed at a later time for a different analysis range or resolution.

3.2 This test method covers the size range from 2 % to 60 % of the aperture diameter chosen as being appropriate to the expected particle size range.

Aperture Diameter, µm	Particle Size Range, µm
50	1 to 30
70	1.4 to 42
100	2.0 to 60
140	2.8 to 84
200	4 to 200

For broader size ranges two aperture tubes may be used and both results are combined by the instrument's software into a single size distribution.

4. Significance and Use

4.1 This test is useful in determining particle size characteristics of dry toners used in electrostatic imaging devices such as copiers and laser printers. It is a practiced method for use in quality control of toner particle size.

5. Apparatus

5.1 *Electrical Sensing Zone Instrumentation* **(2),** equipped with a minimum capability of 256 size channels, a digital pulse processor and 50, 70, 100, 140, or 200-µm aperture tubes.

5.2 *Software,* capable of processing the pulse data to yield size distribution graphs and statistics.

5.3 *Ultrasonic Dispersing Probe ,* or alternative equipment suitable for dispersing the dry toner in an aqueous electrolyte.

[1] This test method is under the jurisdiction of ASTM Committee F05 on Business Imaging Products and is the direct responsibility of Subcommittee F05.04 on Electrostatic Imaging Products.

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[2] The boldface numbers in parentheses refer to the list of references at the end of the test method.

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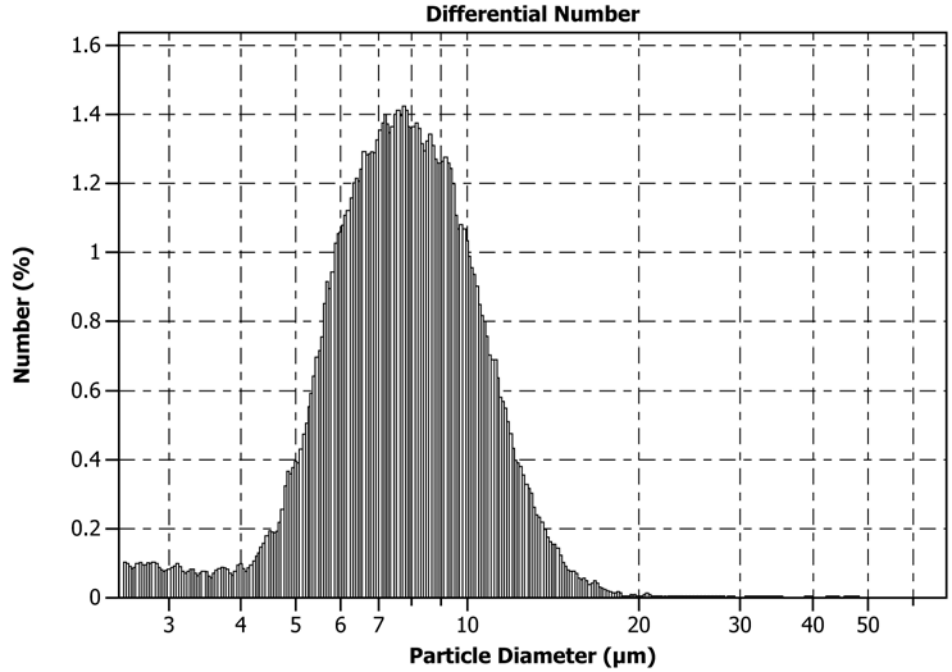

FIG. 1 Differential Number Size Distribution

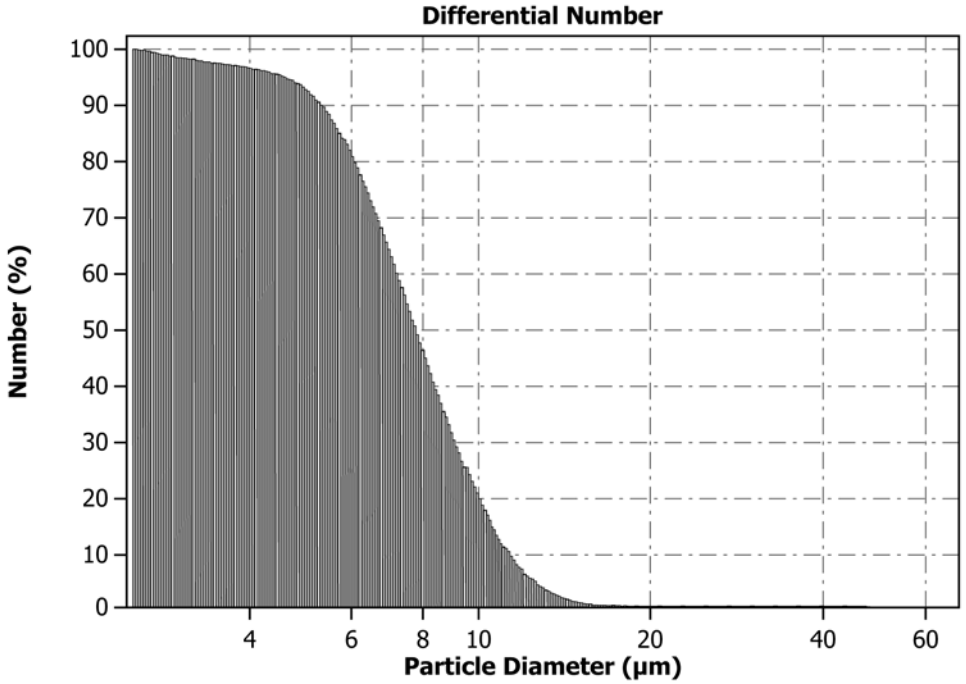

FIG. 2 Cumulative Number Size Distribution Larger Than

6. Reagents and Materials

6.1 *Electrolyte*—4 weight % aqueous sodium pyrophosphate or 1 weight % sodium chloride. The electrolyte shall be adequately filtered to remove almost all particle contaminants greater than 1 µm. Some aqueous electrolytes are commercially available.

6.2 *Surfactant*—a nonionic surface active agent suitable for keeping toner particles separated while in suspension.

6.3 Near monosized spherical particles standardized for the number % modal size as calibration standards.[3]

7. Sampling

7.1 Sample the powder when flowing (**1**).

[3] The standardized particles are usually available from the equipment manufacturer.

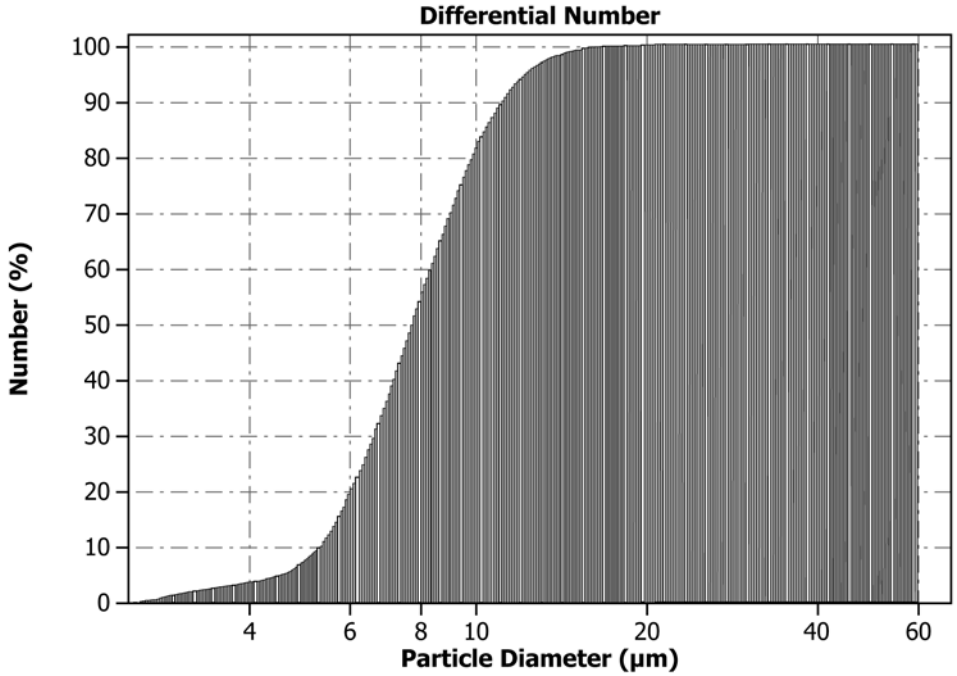

FIG. 3 Cumulative Number Size Distribution Small Than

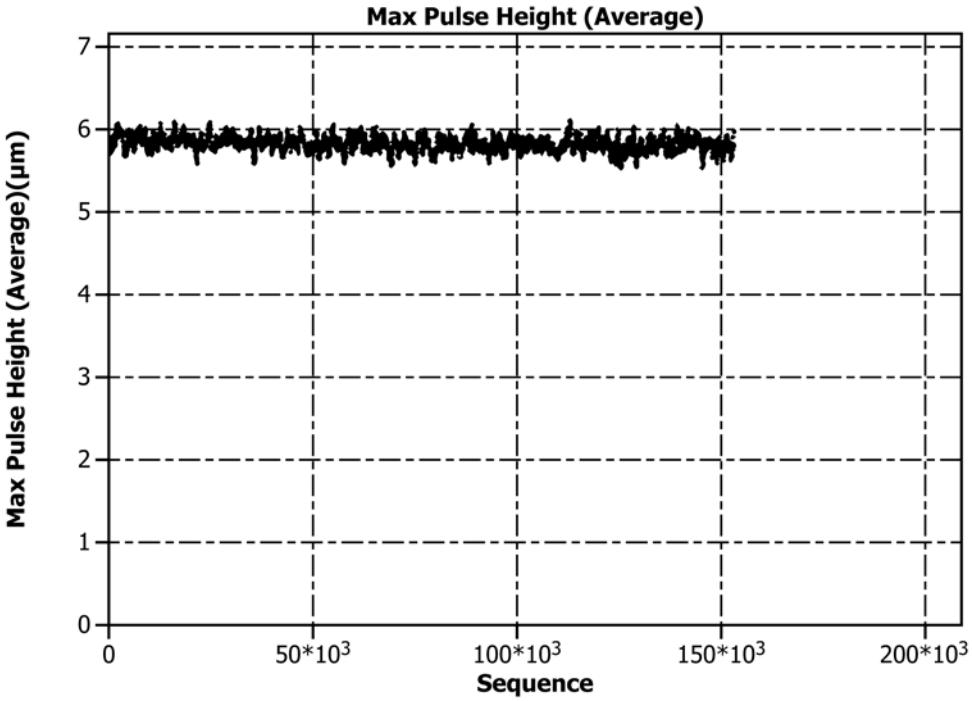

FIG. 4 Max Pulse Height Average by Sequence

7.2 Sample the entire powder flow over small intervals of time. This is preferable to a continuous withdrawal of a small fraction of the flow.

7.3 A further positive aspect is that electrostatic imaging requires material that produces uniform, stable, and acceptable image quality, one copy after the other. In general, the usage rate is in the range from 1 to 100 mg per copy, depending on the original document and the electrostatic conditions. Each copy, consequently, contains a small sampling of the bulk toner.

NOTE 1—Often the processes used to produce dry toner and the semicohesive, electrostatic nature of the fine material can make it prohibitively difficult to follow these important general rules for powder sampling.

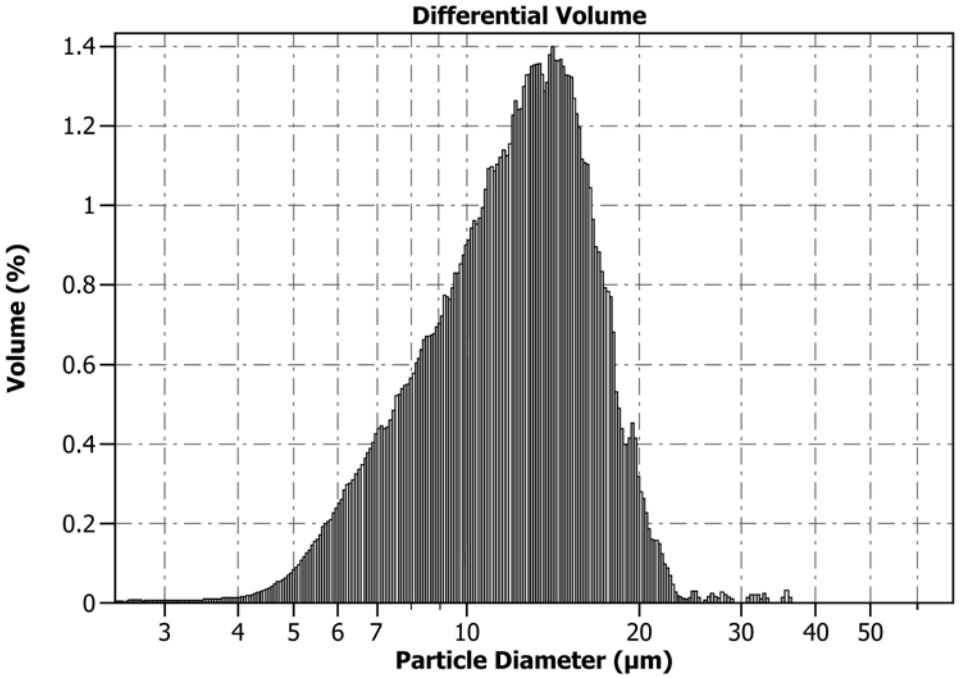

FIG. 5 Differential Volume Size Distribution

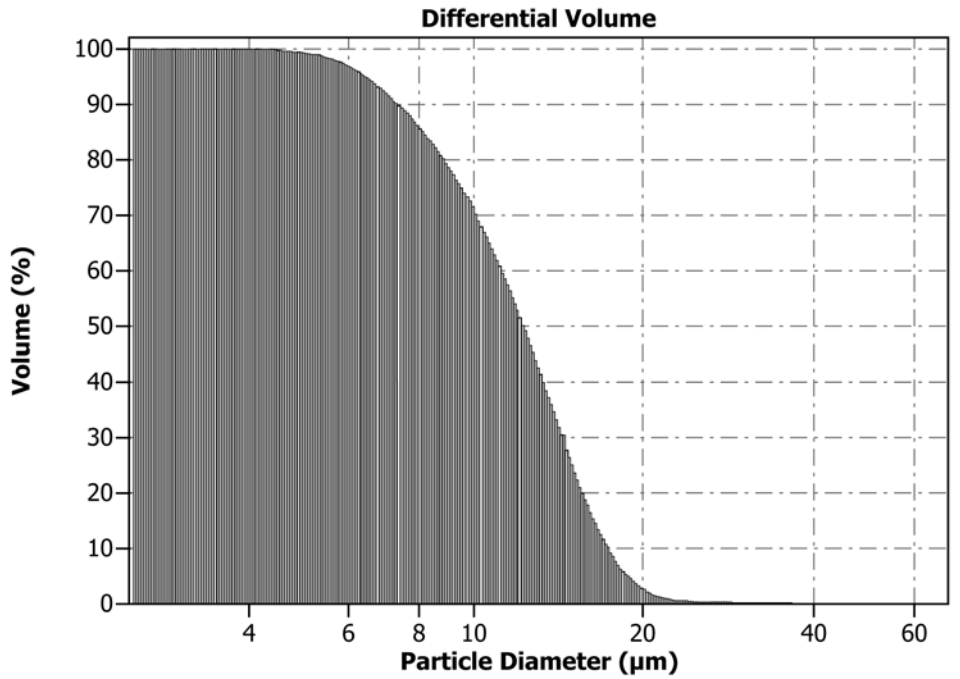

FIG. 6 Cumulative Volume Size Distribution Larger Than

NOTE 2—The above considerations tend to permit a practical assessment of quality by the measurement of a number of small samples taken from various sections of nonmoving powder beds and containers. These samples may be obtained by probes, also known as "thieves," for which many designs exist. In fact, this method is often preferable to more elaborate techniques, like sample splitters, which have moving parts. Such devices are difficult to maintain, and may have places where the thermally sensitive powder is fused by shear to form large, undesirable aggregates.

8. Calibration and Standardization

8.1 The electrical sensing zone equipment should be calibrated with monosized latex polymer microspheres (**3**, **4**) which have been standardized for the number % modal size. Calibration should be regularly verified to ensure the accuracy

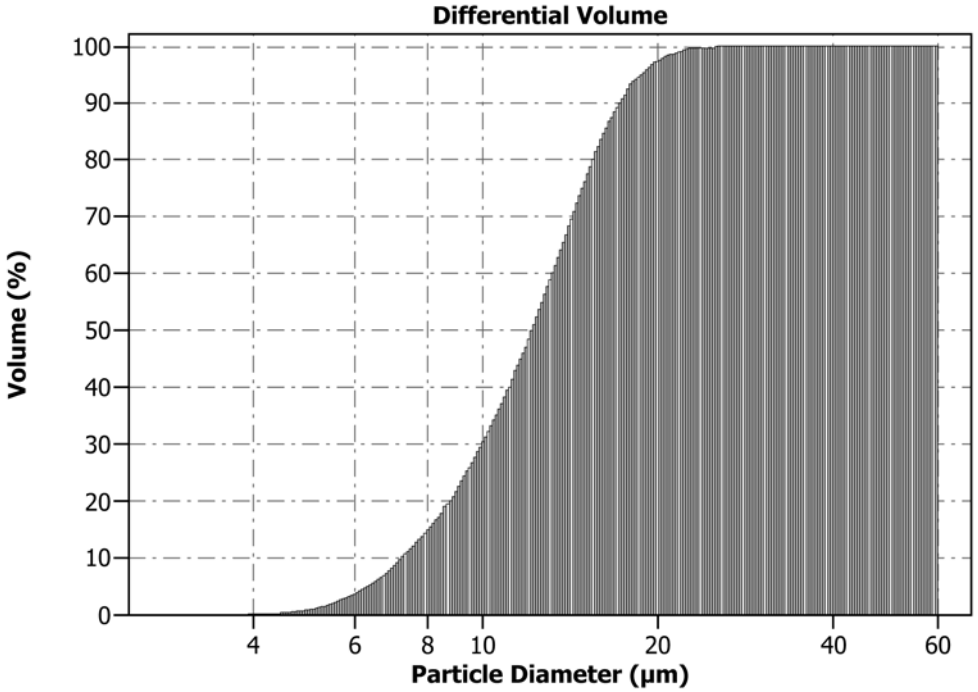

FIG. 7 Cumulative Volume Size Distribution Smaller Than

of calibration. For calibration and verification follow the manufacturer's recommended procedure.

9. Procedure for Toner Samples within 1:30 Dynamic Size Range

9.1 Select the appropriate aperture from 3.2 according to the size range of the sample.

9.2 Set up the electrical sensing zone apparatus in accordance with the manufacturer's instruction manual.

9.3 Measure approximately 5 to 10 mg of toner into a 50-mL borosilicate beaker filled with filtered electrolyte, and add 1 or 2 drops of filtered nonionic surfactant(**5**) . The amount of toner used in an analysis is very important if the material has a high percentage of finer particles.

9.4 Fully disperse the toner in the electrolyte-surfactant mixture using an ultrasonic probe at approximately 6 W power. Care should be taken in sonication to avoid the fracturing of particles from the beaker. Thirty seconds maximum sonication at the wattage setting recommended has been found acceptable.

9.5 Transfer the dispersed sample suspension to a larger borosilicate round-bottom beaker and dilute to 150 to 200 mL volume with filtered electrolyte. Sample concentrations should not exceed the level recommended by the manufacturer.

9.6 Place the sample on the sensor stand and mechanically stir the toner suspension for approximately 1 min. Take care here to avoid cavitation and air bubbles. Erroneous large particle counts can cause a shift in the volume size distribution if this procedure is not carefully followed.

9.7 Measure at least three samplings of the particle suspension. It is advisable to accumulate at least 50 000 counts in each analysis to ensure good statistical precision.

9.8 Overlay the max high average pulse distribution graphs for the three samples and verify that all of them were stable through the analysis. If the sample was not stable in any of the analyses, that is, formation of aglomerates, repeat the analysis.

9.9 Using the instrument's software, average the three results and report the resulting number as the parameter being measured.

9.10 Clean all glassware and thoroughly rinse the orifice tube externally with clean electrolyte between measurements. This will prevent contamination between analyses and keep noise level at an acceptably low level.

9.11 Repeat 9.3 through 9.11 using a second sampling of the toner.

10. Procedure for Toner Samples with Larger than 1:30 Dynamic Size Range

10.1 Select two apertures from 3.2 to cover the size range of the sample.

10.2 Set up the electrical sensing zone apparatus in accordance with the manufacturer's instruction manual for the larger of the two apertures selected in 10.1.

10.3 Measure approximately 5 to 10 mg of toner into a 50–mL borosilicate beaker filled with filtered electrolyte, and add 1 or 2 drops of filtered nonionic surfactant (**5**)**.** The amount of toner used in an analysis is very important if the material has a high percentage of finer particles.

10.4 Fully disperse the toner in the electrolyte-surfactant mixture using an ultrasonic probe at approximately 6 W power. Care should be taken in sonication to avoid the fracturing of particles from the beaker. Thirty seconds maximum sonication at the wattage setting recommended has been found acceptable.

10.5 Transfer the dispersed sample suspension to a larger borosilicate round-bottom beaker and dilute to 150 to 200 mL volume with filtered electrolyte. Sample concentrations should not exceed the level recommended by the manufacturer.

10.6 With the larger of the two selected apertures installed in the instrument, place the sample on the sensor stand and mechanically stir the toner suspension for approximately 1 min. Use caution to avoid cavitation and air bubbles. Erroneous large particle counts can cause a shift in the volume size distribution if this procedure is not carefully followed. Measure at least three samplings of the particle suspension. It is advisable to accumulate at least 50 000 counts in each analysis to ensure good statistical precision.

10.7 Overlay the max high average pulse distribution graphs for the three samples and verify that all of them were stable through the analysis. If the sample was not stable in any of the analyses, that is, formation of agglomerates, repeat the analysis.

10.8 Using the instrument's software, average the three results and save the resultant size distribution.

10.9 Remove the sample beaker and the larger aperture. Install the smaller aperture and set up the electrical sensing zone apparatus for the smaller aperture in accordance with the manufacturer's instruction manual. Transfer the sample suspension to a clean second beaker passing all of it through an appropriate large particle-scalping screen to remove particles larger than the upper size range for the smaller aperture but leaving particles overlapping part of the range for both apertures. Flush the original sample beaker with clean electrolyte. Transfer the entire flushing electrolyte to the second beaker passing it through the scalping screen. Further flush down the scalping screen into the second beaker to get as many sample particles through the screen as possible. Place the second sample beaker in the instrument and using the small aperture repeat 10.6 through 10.8.

10.10 Open the files for the averaged size distribution from the tow apertures; merge both in a single size distribution using the instrument's software.

10.11 Repeat 10.3 through 10.10 using a second sampling of the toner.

11. Calculation

11.1 No manual calculations are necessary; all measured parameters are automatically calculated by the instrument's software.

12. Interpretation of Results

12.1 The most common ways to display particle size distributions are number % and volume %; the values for the mean, meidan, mode, and so forth, in many cases will be totally different. A number size distribution reflects the percentage of the particle population in different size categories. In general, most powder grinds have more fines than large and because of this, the graphs for the number size distribution have a tendency to shift towards the lower size of the distribution (Fig. 1). A volume % size distribution reflects the percentage of the particle volume in different size categories. Sinc ethe larger particles have the most volume displacement, the graph will be shifted to the larger sizes (Fig. 5). This graph is very similar to the results obtained from running a sample through a sieve set. If the relative number of fine particles in a powder will affect the quality of a product, it will be advisable to report the results of size analyses as a number distribution.

13. Precision and Bias

13.1 The precision and bias of the electrical sensing zone method **(4, 6, 7)** have been reported in the referenced scientific literature.

13.2 An example of the precision obtained for dry toner based on triplicate measurements of the mean value of the size distribution is shown in Table 1. Analyses were done on five different units of the same model Instruments A, B and C were in a different location from instruments D and E. The unit of measurement was microns (μm).

13.3 It is not difficult to obtain this level of precision if the procedure is carefully followed.

14. Keywords

14.1 dry toners; particle size

TABLE 1 Particle Size Measurements from Five Instruments at Two Locations

Test No.	Instrument				
	A	B	C	D	E
1	11.19	11.18	11.20	11.21	11.20
2	11.19	11.23	11.28	11.28	11.06
3	11.18	11.06	11.20	11.18	11.16
4	11.24	11.23	11.12	11.23	11.16
5	11.26	11.25	11.24	11.20	11.18
6	11.16	11.24	11.14	11.28	11.13
7	11.16	11.25	11.10	11.18	11.12
8	11.16	11.22	11.18	11.12	11.11
9	11.12	11.24	11.25	11.20	11.08
10	11.22	11.16	11.26	11.15	11.12
11	11.17	11.09	11.16	11.16	11.10
12	11.16	11.21	11.19	11.10	11.12
13	11.18	11.19	11.17	11.17	11.13
14	11.15	11.22	11.17	11.12	11.09
15	11.05	11.18	11.10	11.15	11.13
16	11.08	11.20	11.15	11.24	11.23
17	11.20	11.22	11.21	11.22	11.14
18	11.16	11.15	11.18	11.17	11.13
19	11.16	11.15	11.18	11.26	11.14
20	11.17	11.24	11.12	11.25	11.12
21	11.15	11.21	11.19	11.14	11.13
22	11.24	11.32	11.14	11.12	11.07
23	11.15	11.19	11.20	11.13	11.17
24	11.17	11.28	11.24	11.10	11.09
25	11.19	11.19	11.19	11.15	11.15
26	11.17	11.21	11.26	11.12	11.06
27	11.19	11.18	11.14	11.19	11.14
28	11.16	11.19	11.20	11.13	11.09
29	11.16	11.24	11.20	11.18	11.19
30	11.14	11.29	11.16	11.18	11.17
Mean	11.17	11.21	11.18	11.18	11.13
SD	0.04	0.05	0.05	0.05	0.04
CV	0.38	0.47	0.42	0.46	0.37

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