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Standard Practice for Reporting Particle Size Characteristics of Pigments[1]

This standard is issued under the fixed designation D1366; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This practice for reporting the fineness characteristics of pigments is designed to apply in most cases where well-known methods for determining these particle size characteristics in the subsieve range are employed, such as microscopic, sedimentation, and turbidimetric methods; and partially to absorption and permeability methods.

1.2 Laminar, plate-like pigments and composite pigments having a definite bimodal distribution are not considered within the scope of this practice.

1.3 *Parameters*—The fineness characteristics are reported in the following three parameters:

1.3.1 *Particle Size Parameter.*

1.3.2 *Coarseness Parameter*—A parameter descriptive of the coarseness character of the pigment, making use of a limiting value in the subsieve range similar to that used in the sieve ranges.

1.3.3 *Dispersion Parameter*—A parameter descriptive of the uniformity of the particle size distribution.

1.4 *This standard does not purport to address the safety problems, 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. Referenced Documents

2.1 *ASTM Standards:*[2]

E20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 Micrometres by Optical Microscopy (Withdrawn 1994)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *particle size parameter* (specific surface diameter, *SSD*), *n*—diameter d_3 used elsewhere in ASTM test methods. This parameter is the same as that frequently reported as "Average Particle Size by Surface Mean," and "Specific Particle Size," and is defined as follows:

$$SSD = \sum d^3 f / \sum d^2 f \quad (1)$$

where:

SSD = specific surface diameter, µm,
d = mean class size, µm, and
f = frequency.

Therefore the *SSD* is the diameter of a sphere having the specific surface characteristic of the pigment. The true specific surface of all pigments involves a shape factor. Report *SSD* whether or not the effect of shape has been considered in the calculations. Presumably, as the effect of shape is better understood, it will figure more and more in calculations involving particle size, but in the meantime it will of necessity be ignored in many cases.

3.1.2 *coarseness parameter (CP), n*—that diameter, expressed in micrometres, below which 99.5 % of the pigment falls.

3.1.3 *dispersion parameter (DP), n*—the ratio of the micrometre size within which 50 % of the pigment lies, to the specific surface diameter, *SSD*. The larger the *DP* number, the greater the dispersion parameter and the lower the uniformity. Report the dispersion parameter in all cases when a distribution curve can be prepared from the original data. The dispersion parameter cannot be calculated from data obtained by absorption or permeability methods. Determine as follows:

3.1.3.1 Prepare a cumulative-size distribution curve on 3-phase log paper, using the vertical axis for the percent falling below the size indicated, and the horizontal axis (log scale) for diameter in micrometres. Use the upper class limit corresponding to the cumulative weight percentage. Subtract the micrometre size at 25 % cumulative weight from the micrometre size at 75 %, and multiply the difference by 100 ÷ *SSD*, as follows:

$$DP = (\mu m \text{ at } 75\,\% - \mu m \text{ at } 25\,\%)/(SSD) \times 100 \quad (2)$$

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[1] This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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TABLE 1 Example of Data Sheet for Microscopic Method

Class Limits, µm		Mean Class Size, *d*	Frequency, *f*	d^2f	d^3f	d^2f, %	Cumulative Weight, %
Lower	Upper						
0.25	0.75	0.5	71.4	17.8	8.9	0.007	0.007
0.75	1.25	1.0	50.9	50.9	50.9	0.040	0.047
1.25	1.75	1.5	57.9	130.2	195.4	0.154	0.191
1.75	2.25	2.0	36.9	147.5	295.0	0.233	0.424
2.25	2.75	2.5	41.1	256.8	641.9	0.506	0.93
2.75	3.25	3.0	34.5	310.9	932.7	0.736	1.69
3.25	3.75	3.5	34.5	423.2	1 481.2	1.17	2.86
3.75	4.25	4.0	21.0	336.1	1 344.5	1.06	3.92
4.25	4.75	4.5	42.0	850.8	3 828.8	3.02	6.94
4.75	5.25	5.0	81.2	2 030.8	10 154.0	8.02	14.96
5.25	5.75	5.5	283.8	8 586.3	47 225.0	37.25	52.21
5.75	6.25	6.0	155.9	5 613.4	33 680.0	26.60	78.81
6.25	6.75	6.5	71.0	2 998.1	19 487.9	15.38	94.19
6.75	7.25	7.0	7.9	388.9	2 722.4	2.15	96.34
7.25	7.75	7.5	5.1	288.8	2 166.3	1.71	98.05
7.75	8.25	8.0	4.2	268.9	2 151.4	1.71	99.76
3.25	8.75	8.5	0.5	33.7	286.8	0.23	99.99
Totals			999.8	22 733.1	126 653.1		

TABLE 2 Example of Data Sheet for Sedimentation Method

Diameter Class Size Limits		Class Size Diameter, *d*, µm	Weight %, d^3f	d^2f	Cumulative Weight, %
Lower	Upper				
0.0	0.5	0.25	11.0	44.00	11.0
0.5	1.0	0.75	10.0	13.33	21.0
1.0	1.5	1.25	10.0	8.00	31.0
1.5	2.0	1.75	9.0	5.14	40.0
2.0	3.0	2.50	15.0	6.00	55.0
3.0	4.0	3.50	9.5	2.71	64.5
4.0	5.0	4.50	8.5	1.89	73.0
5.0	7.5	6.25	12.5	2.00	85.5
7.5	10.0	8.75	8.0	0.91	93.5
10.0	15.0	12.50	5.3	0.42	98.8
15.0	20.0	17.50	1.2	0.07	100.0
20.0	30.0	25.00	0.0	0.00	
Totals			100.0	84.47	

4. Significance and Use

4.1 This practice is of value (*1*) to the producer of fine particles as a means of reporting particle characteristics with respect to quality control and (*2*) to the buyer to assure that the particle size and particle size distribution meet his requirements.

5. Procedure

5.1 *Particle Size by Microscopical Methods*—Procedures for determining particle size by microscopical methods are described in Practice E20. When microscopical methods are employed, report the data in tabular form similar to that shown in Table 1.

5.2 *Particle Size by Sedimentation Methods*—Sedimentation methods provide data from which may be calculated the mean class size and percentage by weight for each of these class sizes. Table 2 presents in the second and third columns data normally obtained by sedimentation methods. Column 3 of Table 2, which gives the distribution by weight of the class sizes, is identical with the function d^3f. Therefore, the d^3f function in Column 4 equals d^3f/d. The fifth column, cumulative weight, percent, is obtained from the values in Column 3.

5.3 *Particle Size by Turbidimetric Methods*—Like the sedimentation method, the turbidimetric methods provide class sizes and a percentage by weight for each class size, and the calculation is the same as that in Table 2. However, frequently a weight distribution curve is obtained from data not directly convertible into the class-size distribution table. In such cases, reconstruct the size-weight distribution table from the distribution by weight curve.

5.4 *Particle Size by Absorption and Permeability Methods*—The absorption and permeability methods, and a few others, provide no means of making a distribution curve. Therefore, obtain specific surface either as square metres per cubic centimetre, or as square metres per gram of material. In the first case, where *s* equals square metres per cubic centimetre, the equation is as follows:

$$SSD = 6/s \quad (3)$$

In the second case, where *S* equals square metres per gram (Note), the equation is as follows:

$$SSD = 6/(\text{sp gr} \times s) \quad (4)$$

NOTE 1—*Example*—Channel black has a specific gravity of 2.0. If the specific surface in square metres per gram is reported as 94.0, then:

$$SSD = 6/(2 \times 94) = 0.032 \quad (5)$$

6. Calculation and Report

6.1 To determine the coarseness and dispersion parameters from the microscopical data of Table 1, draw Curve I of Fig. 1, using the cumulative weight percentage and the upper class size units of Table 1. From this curve, read the *CP* at the point where the curve crosses the 99.5 % line, that is, 8.7, and obtain the *DP* from the sizes where the curve crosses the 25 and 75 % lines; then calculate as follows:

$$SSD = 126\ 653.1/22\ 733.1 = 5.57\ \mu m \quad (6)$$

$$p_1 = d_{75}\,\% - d_{25}\,\% = 0.70\ \mu m$$

$$DP = 100\,p_1/SSD = (100 \times 0.70)/5.57 = 12.6$$

$$CP = 8.7\ \mu m$$

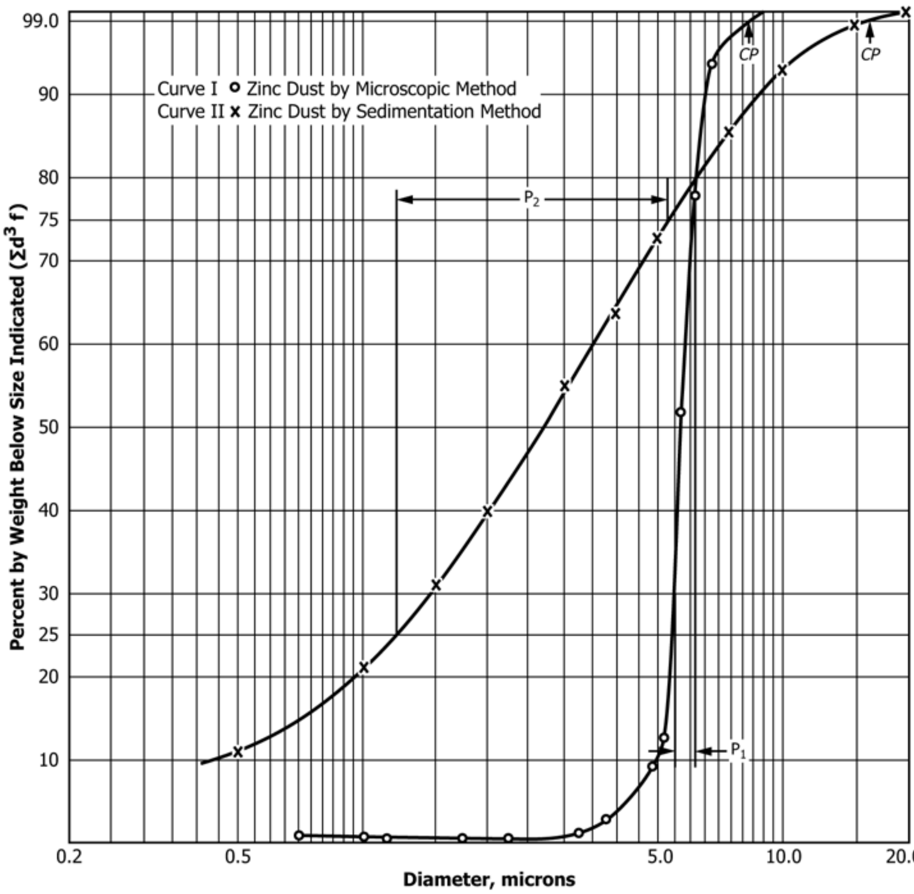

FIG. 1 Curves for Obtaining Coarseness and Dispersion Parameters

6.2 To determine the coarseness and dispersion parameters from the sedimentation data of Table 2, draw Curve II of Fig. 1, using the cumulative weight percentage and upper class size units of Table 2. From this curve, determine the coarseness and dispersion parameters as described in 5.1, as follows:

$$SSD = \sum d^3 f / \sum d^2 f = 100.0/84.47 = 1.183\ \mu\text{m} \quad (7)$$

$$DP = 17.0\ \mu\text{m}$$

$$p_2 = 5.30 - 1.18 = 4.12\ \mu\text{m}$$

$$DP = 100\, p_2 / SSD = 348.0$$

$$CP = 17.0\ \mu\text{m}$$

6.3 While theoretically the size of pigment particles is independent of the method of determination used, it is recognized that various methods of determining subsieve particle size distribution give somewhat different results, depending on the assumption made in the particle method. It is recommended, therefore, that the method of determination always be made a part of the report of the size distribution determined under this practice.

7. Keywords

7.1 microscopic; pigments; sedimentation; turbidimetric permeability absorption

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