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Standard Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments[1]

This standard is issued under the fixed designation D 3360; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the particle-size distribution in the sub-sieve size range of the common extender pigments such as aluminum silicate (kaolin clay), magnesium silicate (talc), calcium carbonate (calcite or dolomite or precipitated calcium carbonate), and mica pigments, and may also be extended to the denser prime pigments such as the white titanium pigments (rutile or anatase) and similar mineral pigments when and if such information is of concern. Particle-size distribution has significance in the evaluation of rheological and pigmentary properties of pigments in paint and also may sometimes be used to characterize the identity or grade of pigments.

1.2 Sedimentation methods having as their basis Stoke's law have found general acceptance for this purpose. Results are expressed in terms of equivalent spherical diameter (e.s.d.), the diameter of a sphere having the same specific gravity as the particle in question and which settles at the same rate. Most mineral pigment particles are more or less asymmetrical, but despite differences in the relationship between equivalent spherical diameter and actual dimensions, the results of a sedimentation particle-size analysis can be correlated readily with many pigment properties.

1.3 Procedures limited to gravitational sedimentation[2,3,4,5] are relatively inaccurate for pigment particles smaller than about 1 µm e.s.d., and centrifugal procedures may be required for the much finer ranges. Nevertheless, the data obtained above the 1-µm limitation provide useful information. This test method is particularly applicable to pigments if a major fraction of the particles fall in the range from about 15 to 1.5 µm, but have a total particle-size range of at least two decades.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments[6]
D 422 Test Method for Particle-Size Analysis of Soils[7]
D 1193 Specification for Reagent Water[8]
E 100 Specification for ASTM Hydrometers[9]
E 300 Practice for Sampling Industrial Chemicals[10]

3. Summary of Test Method

3.1 For the determination of particle-size distribution by the application of Stokes' law to the sedimentation of particulate material out of an initially homogeneous suspension, any systematic set of measurements which permit the determination of the suspension density (that is, the "percent solids") at some defined distance beneath the surface of the suspension at some appropriately selected series of sedimentation time durations can be converted to a particle-size distribution. In this procedure, the suspension density is estimated at the effective distance beneath the suspension surface of the center of gravity of a floating hydrometer observed at a series of convenient time intervals selected to increase roughly exponentially.

NOTE 1—Any alternative system that provides an equivalent set of measurements (for example, the Andreasen Pipet Method,[4] or any of the optical sampling methods based on change in turbidity, light scattering, and light or X-ray absorption, etc.) will also yield a particle-size distribution. Methods based on optical measurements, however, are much less generally applicable because of certain technological limitations too complex to be dealt with here.

4. Apparatus

4.1 *Stirring Apparatus*, commonly known as a "malted milk mixer," with a vertical high-speed shaft (approximately 10 000

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[2] Norton, F. H., and Speil, S., *Journal of the American Ceramic Society*, Vol 21, 1938, p. 89.

[3] Casagrande, A., "Hydrometer Method for Determination of Fineness Distribution of Soils," Julius Springer, Berlin, 1934.

[4] Andreasen, A. H. M., "The Evaluation of Ground Materials," *Kolloid Biehefte*, Vol 27, 1928, p. 349.

[5] Lane, Marvin K. "Improved Method for Measuring Particle Size Distributions by Gravity Sedimentation with a Hydrometer," A Work Manual by Lane, M. K., 4147 West Byron St., Chicago, IL 60641.

[6] *Annual Book of ASTM Standards*, Vol 06.03.
[7] *Annual Book of ASTM Standards*, Vol 04.08.
[8] *Annual Book of ASTM Standards*, Vol 11.01.
[9] *Annual Book of ASTM Standards*, Vol 14.03.
[10] *Annual Book of ASTM Standards*, Vol 15.05.

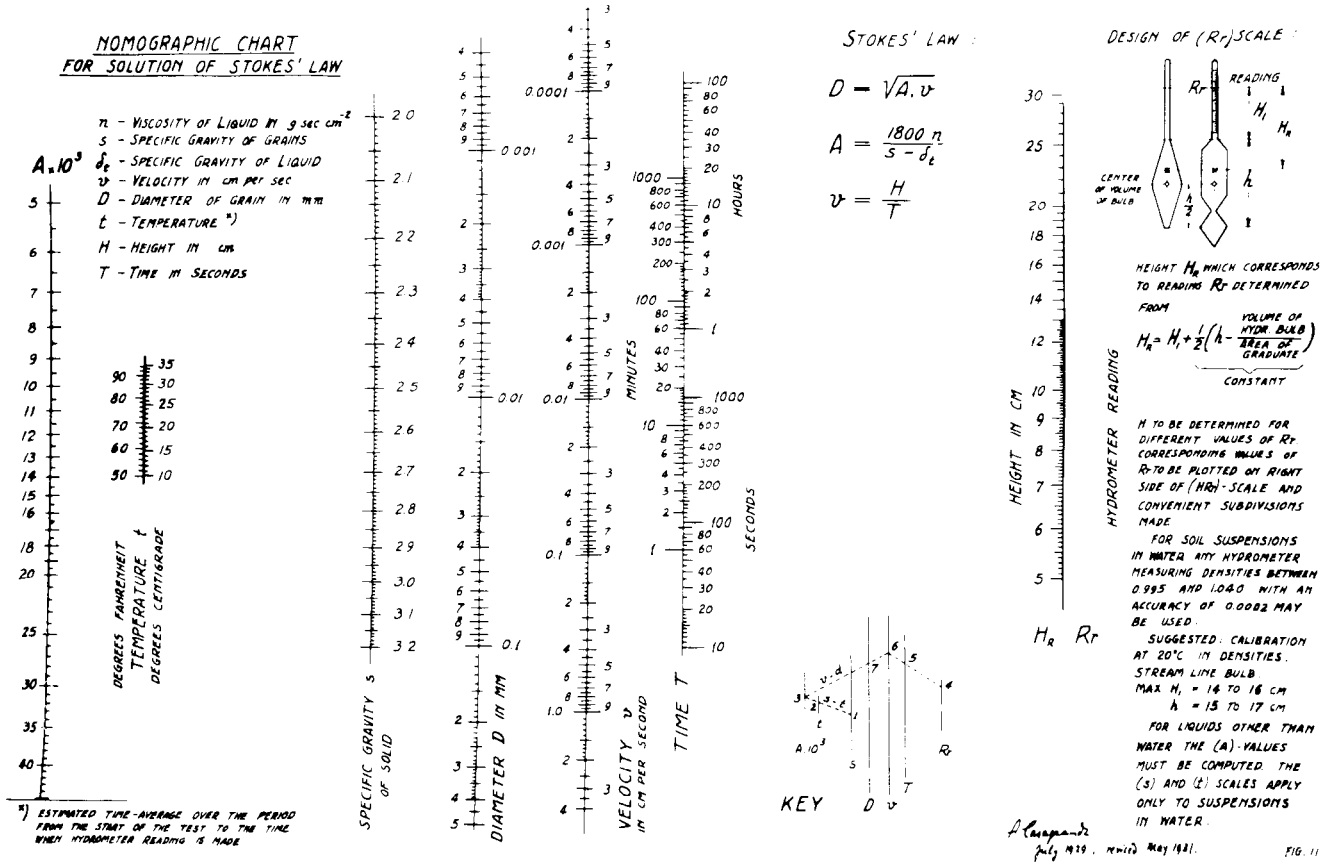

FIG. 1 Casagrande Nomographic Chart

r/min) tipped with a 25-mm diameter sine-wave impeller. The preferred mixing cup is a stainless steel cup about 180-mm deep and slightly tapered from an outside diameter at the top of about 100 mm to about 70 mm at the bottom. Some very fine particle pigments may require additional shear for complete dispersion. In some circumstances, dispersion in a blender cup at maximum r/min may be considered instead of preparation with a "malted-milk mixer." (See 7.3.)

4.2 *Hydrometer*, certified with a minimum of 3 points of certification, graduated in units of specific gravity, and having a range from 0.995 to 1.038. The approximate dimensions are as follows: bulb length, 139 mm, bulb diameter, 31 mm, and overall length 280 mm. Such a hydrometer is identified as a "Soil Hydrometer"[5] in Specification E 100, and is also described in Test Method D 422.

4.3 *Sedimentation Cylinders*, glass, (two or more), having an inside diameter of about 65 mm and an overall height of 450 mm with a calibration mark to hold 1205 mL.[5]

4.4 *Thermometer*, accurate to ±0.1°C over a range from 15 to 35°C.

4.5 *Water Bath*, large enough to accommodate two or more of the sedimentation cylinders immersed to slightly above the 1205-mL graduation mark, and having circulating water and means for keeping its temperature throughout the bath to within ±0.1°C over a range from 18 to 30°C.

NOTE 2—In place of the water bath, the procedure may be carried out in a constant-temperature room controlled to the same precision as noted in 4.5.

4.6 *Time*—A stopwatch, or the equivalent, and an ordinary watch or clock.

4.7 *Balance*, sensitive to 0.01 g.

4.8 *Drying Oven*, with accurate thermostatic control to ±2°C at 110°C.

4.9 *Wash Bottle*, containing reagent water.

4.10 *Casagrande Nomographic Chart* (see Fig. 1).

5. Reagents

5.1 *Purity of Water*—Unless otherwise indicated references to water shall be understood to mean Type II reagent water conforming to Specification D 1193.

5.2 *Dispersing Agents*—The different pigments, depending on their specific surface properties, may require differing dispersant systems to effect optimum stable dispersions. Among the various common dispersing agents which have been found useful are:

5.2.1 *Tetrasodium Pyrophosphate*, TSPP ($Na_4P_2O_7$)—A freshly prepared 5 % solution of TSPP in water.

5.2.2 *Sodium Hexametaphosphate*—$(NaPO_3)_6$.

5.2.3 *Calgon T.*[11]

5.2.4 *Daxad 30* (25 % active dispersant).[12]

5.2.5 *Dispex N-40* (40 % active dispersant).[13]

5.3 *Antifoaming Agent*—Pine oil, or capryl alcohol, or the equivalent.

[11] Calgon T, Calgon Corp., P. O. Box 1346, Pittsburgh, PA 15230.
[12] Daxad 30, W. R. Grace, 3 Hanover Square, New York, NY 10004.
[13] Dispex N-40, Allied Colloids Inc., 1 Robinson Lane, Ridgewood, NJ.