



# Standard Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments<sup>1</sup>

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 ( $\epsilon$ ) 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 (talca), 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<sup>2,3,4,5</sup> are relatively inaccurate for pigment particles smaller than about 1  $\mu\text{m}$  e.s.d., and centrifugal procedures may be required for the much finer ranges. Nevertheless, the data obtained above the 1- $\mu\text{m}$  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  $\mu\text{m}$ , but have a total particle-size range of at least two decades.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

Current edition approved May 10, 1996. Published July 1996. Originally published as D3360 – 74. Last previous edition D 3360 – 80 (1989).

<sup>2</sup> Norton, F. H., and Speil, S., *Journal of the American Ceramic Society*, Vol 21, 1938, p. 89.

<sup>3</sup> Casagrande, A., "Hydrometer Method for Determination of Fineness Distribution of Soils," Julius Springer, Berlin, 1934.

<sup>4</sup> Andreasen, A. H. M., "The Evaluation of Ground Materials," *Kolloid Beihfte*, Vol 27, 1928, p. 349.

<sup>5</sup> 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.

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<sup>6</sup>
- D 422 Test Method for Particle-Size Analysis of Soils<sup>7</sup>
- D 1193 Specification for Reagent Water<sup>8</sup>
- E 100 Specification for ASTM Hydrometers<sup>9</sup>
- E 300 Practice for Sampling Industrial Chemicals<sup>10</sup>

## 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,<sup>4</sup> 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

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>7</sup> *Annual Book of ASTM Standards*, Vol 04.08.

<sup>8</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>9</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>10</sup> *Annual Book of ASTM Standards*, Vol 15.05.

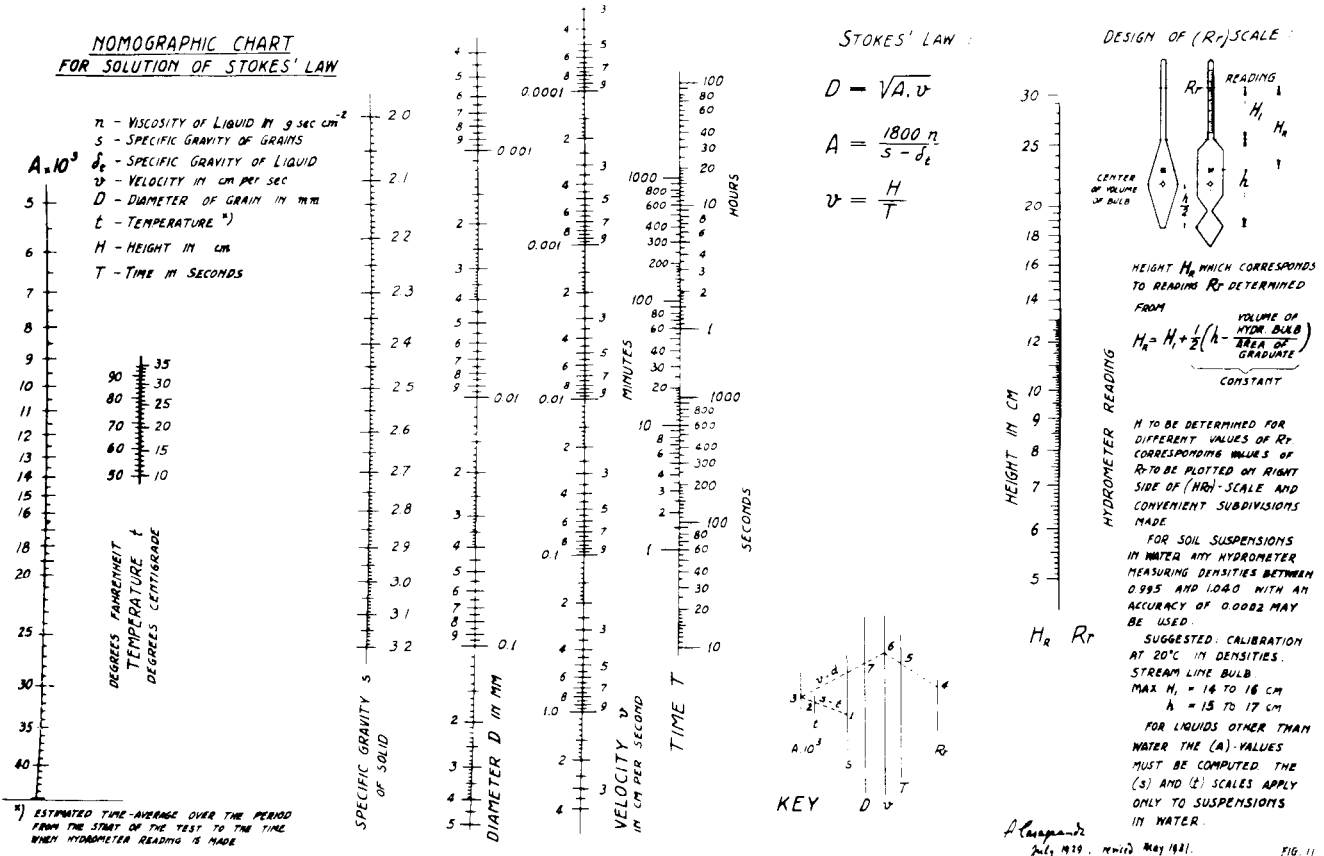


FIG. 1 Casagrande Nomographic Chart

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"<sup>5</sup> 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.<sup>5</sup>

4.4 *Thermometer*, accurate to  $\pm 0.1^{\circ}C$  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  $\pm 0.1^{\circ}C$  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  $\pm 2^{\circ}C$  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*—( $NaPO_3$ )<sub>6</sub>.

5.2.3 *Calgon T*.<sup>11</sup>

5.2.4 *Daxad 30* (25 % active dispersant).<sup>12</sup>

5.2.5 *Dispex N-40* (40 % active dispersant).<sup>13</sup>

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

<sup>11</sup> Calgon T, Calgon Corp., P. O. Box 1346, Pittsburgh, PA 15230.

<sup>12</sup> Daxad 30, W. R. Grace, 3 Hanover Square, New York, NY 10004.

<sup>13</sup> Dispex N-40, Allied Colloids Inc., 1 Robinson Lane, Ridgewood, NJ.

## 6. Test Specimens and Sample

6.1 Sampling procedures should conform to the general practice outlined in Practice E 300.

6.2 *Specimen Size*—At least 50 g of well-pulverized pigment is required for each particle-size distribution determination.

## 7. Procedure

7.1 Determine the moisture content of the pigment by drying a 10-g sample to constant weight at 110°C essentially as described in Test Methods D 280.

7.2 Weigh out the equivalent of  $30 \pm 0.05$  g of dry (moisture-free) pigment. (In the special case of predispersed aluminum silicate pigments, weigh out  $30.10 \pm 0.05$  g of dry sample. For denser pigments such as TiO<sub>2</sub> (sp gr 4.0), the sample size should be reduced to about 20 g.)

7.3 *Preparation of the Pigment Dispersion*—Place 150 to 200 mL of distilled water in the mixing cup, start the mixer, add the pigment sample, and stir for 5 min. Then add the amount and type of dispersant (see Note 3) as suggested in Table 1, and mix for an additional 5 min. Loose agglomerates or flocs must be broken up into individual particles, but it is equally important to avoid mechanical attrition of the particles, especially in coarser pigments. For most common pigments, a 10-mm mixing period is sufficient.

7.4 Transfer the suspension into one of the graduated sedimentation cylinders, rinse out the mixing cup several times to ensure complete transfer of the sample, and finally dilute to the 1205-mL mark with distilled water adjusted to the same temperature as the water bath. If needed (but only if needed) add a drop of the antifoaming agent. Close the mouth of the cylinder with a rubber stopper to fit (No. 13) or with the palm of the hand, and mix the suspension in the cylinder by inverting several times with shaking. Place the cylinder in the constant temperature bath (see Note 4) and stir the suspension occasionally with a glass rod to prevent sedimentation. Make a reference (blank) solution by filling the second cylinder to the mark with distilled water containing the same amount and type of dispersant as used with the pigment sample, and place along side in the bath. When both cylinders have attained the temperature of the bath, remove the cylinder with the suspension and remix by inversion and shaking as before.

variable degree to form flocs which descend as loosely associated groups or clusters of particles. When this occurs the apparent test results are much too “coarse.” Signs of flocculation prior to or during the sedimentation test may be any or all of the following:

(a) Large clusters or pigment flocs suddenly become visibly apparent in the sedimenting suspension.

(b) An ill-defined, loosely mobile bed of sediment flocs may form at the bottom of the cylinder. If the pigment has been properly and stably dispersed, the sediment bed will be dense, solidly and tightly packed, well-defined, and may appear to be graded in size if the particle size is widely distributed. Such a dispersed sediment bed is extremely difficult to resuspend by simple shaking and mixing.

(c) A very clear layer of solution may suddenly appear at the top of the cylinder, and this clear layer gradually descends with a definite demarcation between clear solution and “flocculated” sediment. If the pigment has been properly and stably dispersed, there will always be a slight residual turbidity extending to the surface of the suspension due to ultra-fine particles which remain in stable suspension even to the end of the test.

(d) Slight or partial flocculation (that is, when only a few flocs form intermittently during the settling period) is more difficult to detect. Evidence for this occurrence is also the clear zone of solution at the surface of the suspension as well as a qualitatively “looser” sediment bed. Slight or partial flocculation is particularly difficult to detect when the size distribution of the pigment is very narrow.

(e) One may be sure with confidence that flocculation is not a source of error in the test results by running a series of sedimentation tests on the same pigment sample using different dispersing agents over an appropriate range of dispersant concentrations. That sedimentation test that yields the finest distribution is the one least likely to contain flocs and most likely to represent the “actual” size-distribution. Dispersion requirements so determined then become “standard for that particular pigment type.”

NOTE 4—The bath may be held at any constant temperature between 18 and 30°C, preferably only a few degrees above the normal maximum temperature of the laboratory.

7.5 Immediately after this final mixing, replace the sample cylinder in the bath, insert the hydrometer into it, and simultaneously start the timer. Immerse the hydrometer only slightly beyond the point where it floats freely.

7.6 Take hydrometer readings at such intervals that the total accumulative elapsed times form an approximate geometrical progression. A convenient series of time intervals is 1, 2, 2, 5, 10, 10, and 30 min and 1, 2, 3, 7, and 10 h, which together yield an elapsed time series of 1, 3, 5, 10, 20, and 30 min and 1, 2, 4, 7, 14, and 24 h. Read the hydrometer at the top of the meniscus, to the nearest estimated 0.001 units of specific gravity. After the first two or three time intervals, it is good

**TABLE 1 Examples of Recommended Dispersants and Suggested Dispersant Concentrations**

Pigment	Specific Gravity	Recommended Dispersant	Concentration, or Amount per 30-g Sample
Aluminum silicate	2.58	TSPP (NaPO <sub>3</sub> ) <sub>6</sub>	6 mL of 5 % solution 0.15 to 0.25 g
Aluminum silicate (predispersed)	2.58	TSPP	3 mL of 5 % solution
Magnesium silicate	2.85	TSPP	6 mL of 5 % solution
Calcium carbonate	2.71	Daxad 30 Calgon T	0.5 to 0.8 % “active” dispersant based on weight of dry pigment as above
Mica pigment	2.82	Calgon T	as above
TiO <sub>2</sub> Pigment (rutile, anatase)	4.00	Calgon T TSPP	as above 4 mL of 5 % solution/20 g sample

NOTE 3—The accuracy of the test depends entirely on obtaining complete and stable deflocculation of the pigment so that each of its particles settles individually. The quantity and type of dispersing agent noted in Table 1 is usually satisfactory for the pigments listed when received in the usual form. Flocculation may occur due to attractive charges between the particles at which time the individual particles cease to fall independently. The pigment particles may come together to a

practice to remove the hydrometer from the suspension, rinse it off, and float it in the reference (blank) solution. Great care must be taken to minimize any disturbance of the suspension by inserting and removing the hydrometer very slowly and evenly.

7.7 For each reading, record the elapsed sedimentation time,

the temperature of the pigment suspension (or water bath), and the hydrometer readings for both the pigment suspension and the reference (blank) solution. See Table 2, which provides an example of a convenient data and computing form.

**8. Calculation**

8.1 *Percent of Pigment in Suspension*—Calculate for each reading the percent of dry pigment remaining in suspension at the level which the hydrometer measures the density of the suspension by the following equation:

$$P = (100 - 000/C) \times [G/(G - B)] \times (R_r - B)$$

where:

- P* = %pigment remaining in suspension,
- C* = original concentration of suspension in grams of pigment per litre, (for a sample of 30-g dry weight in 1205 mL of suspension, *C* = 24.9 g/L),
- R<sub>r</sub>* = hydrometer reading in the pigment suspension,
- B* = hydrometer reading in the blank solution, and
- G* = specific gravity of pigment particles. (For example, in the absence of an actual determination of the specific gravity, a value of 2.6 may be taken for aluminum silicate pigment.)

8.1.1 Take the value of (*R<sub>r</sub>* - *B*) for the initial 1-min sedimentation reading to be a measure of the starting concentration. The percent remaining in suspension for each subsequent reading then becomes simply the ratio of its (*R<sub>r</sub>* - *B*) value to the initial (*R<sub>r</sub>* - *B*) value multiplied by 100.

NOTE 5—In the case of pigments reasonably free from coarse particles (larger than about 15 μm), the preceding calculations for the initial hydrometer readings should give values of 100 % for the percent remaining in suspension. Variation from this figure may be due to inaccurate measurements of the test specimen or volume of the suspension.

NOTE 6—Readings taken during the first few minutes of sedimentation are more subject to possible error from several causes than are subsequent

readings. These causes include settling of coarse particles on the hydrometer bulb, failure to attain complete temperature equilibrium, and the possible presence of entrained air in the suspension. Of these, the last named is perhaps the most frequent source of error. Treatment of the suspension before mixing with a small amount of an antifoam agent helps to prevent this.

8.2 *Equivalent Spherical Diameter of the Particles:*

8.2.1 Calculate the equivalent spherical particle diameter corresponding to the percent indicated by each hydrometer reading in accordance with Stokes' law. A particle of this equivalent spherical diameter is assumed to be at the surface of the suspension at the beginning of sedimentation and to settle during the accumulated time to the level at which the hydrometer measures the suspension density.

8.2.2 Casagrande's nomographic solution of Stokes' law<sup>2,5</sup> shown in Fig. 1 offers a convenient means for calculating equivalent spherical diameter. Its use requires calibration of the hydrometer in terms of the actual depth of its center of gravity below the surface of the suspension (*H<sub>r</sub>*) for each hydrometer reading, *R<sub>r</sub>*. The formula for calibration of hydrometers graduated in specific gravity units is shown in the upper right-hand corner of the chart. Calculated values of *H<sub>r</sub>* for convenient intervals of *R<sub>r</sub>* must be plotted on the *H<sub>r</sub>* - *R<sub>r</sub>* scale for each individual hydrometer. Note that the hydrometer is calibrated for actual depth below the surface of the liquid, but that hydrometer readings of the density of the pigment suspension are made at the top of the meniscus, which is slightly raised above the surface of the suspension. For this reason a meniscus correction must be made. With the recommended hydrometer, it has been found that the addition of 0.0003 to the specific gravity reading made at the top of the meniscus gives values which correspond closely to the specific gravity reading at the surface level.

8.2.3 The use of the chart is explained in the key. A line through the value of *S*, the specific gravity of the sedimenting

**TABLE 2 Particle Size Distribution-Hydrometer Method—Calculation and Data Sheet**

				Temperature _____					
				Density _____					
				A × 10 <sup>3</sup> _____					
Sample No: _____		Operator: _____							
Date	Time	Time Interval	Total Time	"R"	"D"	V	D(μ)	(R - d) × 10 <sup>3</sup>	% Finer

"R"—Hydrometer reading on suspension.  
 "D"—Hydrometer reading on blank.  
 D(μ)—Equivalent spherical diameter in micrometres

Use Casagrande Nomographic Chart to Determine  
 1. A × 10<sup>3</sup> from temperature and density.  
 2. V from "R" and time.  
 3. D(μ), from V and A × 10<sup>3</sup>

pigment particles and  $t$ , the temperature, gives the value for  $A \times 10^3$ , which is constant for a given temperature (of the bath or constant-temperature room). A line through  $H_r$  and  $T$ , the accumulated time, gives a value for  $v$ , the sedimentation velocity in centimetres per second; and finally, a line through  $A \times 10^3$  and  $v$  gives the corresponding value for the equivalent spherical diameters,  $D$ , in millimetres.

**9. Report**

9.1 The report shall consist of a graph made by plotting for each hydrometer reading the percent remaining in suspension (“percent finer than”) against the equivalent spherical diameter, using semilogarithmic graph paper with the particle size values on the logarithmic scale (see Fig. 2). A value for the percent finer than 2  $\mu\text{m}$  may be read from the graph and reported separately.

9.2 Although the entire cumulative particle size distribution curve may be of general interest, the particle size distribution criteria customarily used for extender pigments is either the weight percent less than 2  $\mu\text{m}$  e.s.d. or the weight median particle size, that is, the “50 % finer than” point on the distribution curve, or both. At either extreme of the particle size distribution curve, that is, the largest 10 % and the smallest

10 %, the test precision and accuracy tend to be considerably less than in the remaining 80 % mid-range of the distribution.

**10. Precision**

10.1 Based on interlaboratory studies of this test method in which operators in 6 laboratories measured the particle size distribution of three extender pigments: aluminum silicate (kaolin), magnesium silicate (talc), and calcium carbonate, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

10.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 1  $\mu\text{m}$  at the weight median (“50 % finer than”) point, or 3 % at the percent less than the 2- $\mu\text{m}$  point on the cumulative distribution curve.

10.1.2 *Reproducibility*—Two results, each the mean of duplicate measurements obtained by operators in different laboratories, should not be considered suspect unless they differ by more than 2  $\mu\text{m}$  at the weight median (“50 % finer than”) point, or 5 % at the percent less than the 2- $\mu\text{m}$  point on the cumulative distribution curve.

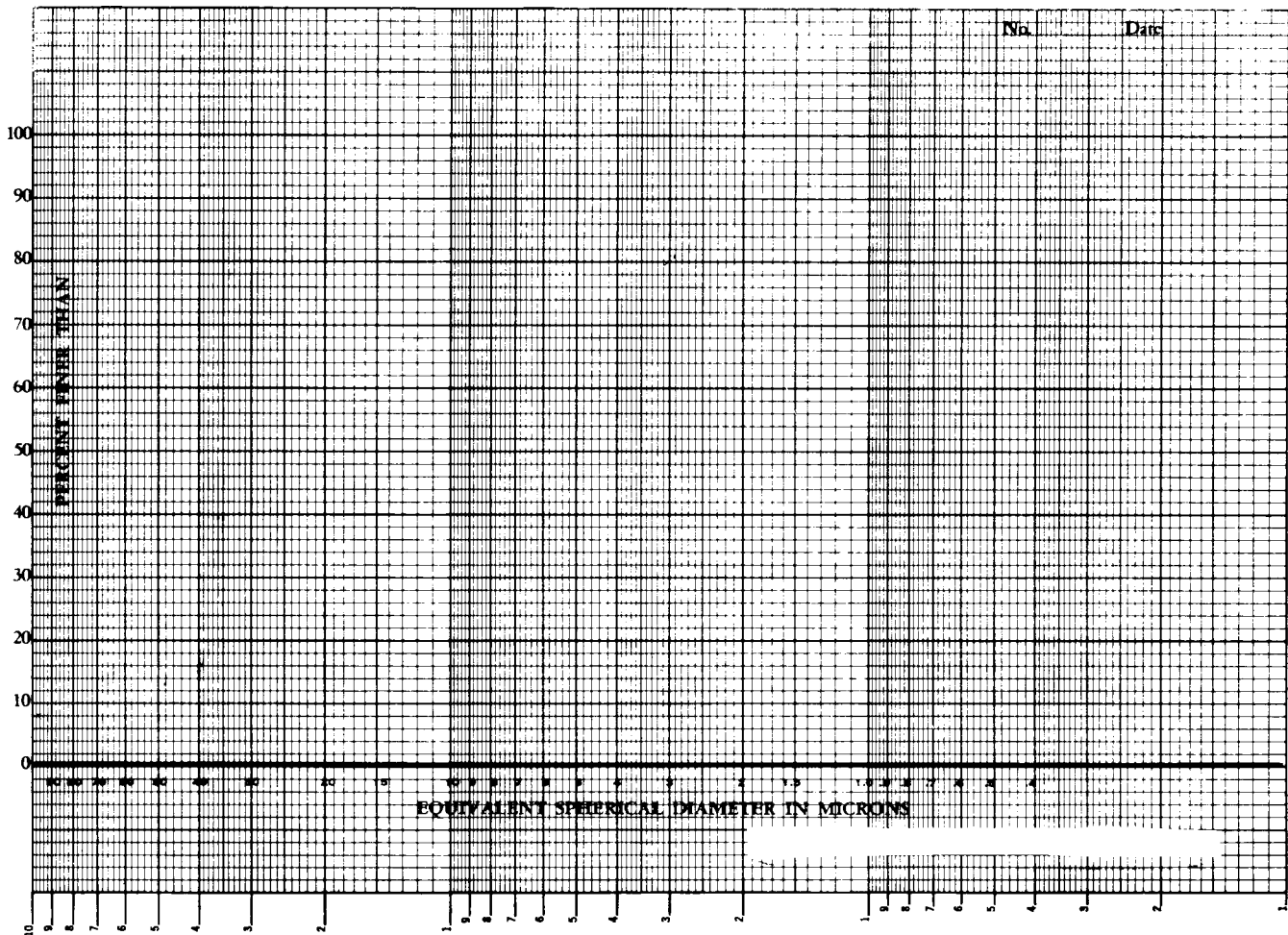



FIG. 2 Graph for Hydrometer Reading

 **D 3360**

*The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.*