Designation: D 1199 – 86 (Reapproved 2003)

# Standard Specification for Calcium Carbonate Pigments[1]

This standard is issued under the fixed designation D 1199; 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 specification covers two types of high-content calcium carbonate pigments, as follows:

1.1.1 *Type PC*—Calcium carbonate precipitate, prepared either by complete solution or by carbonation of lime.

1.1.2 *Type GC*—Ground mineral product.

1.2 Six grades of pigments, based on particle size (see 3.3) are covered.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

## 2. Referenced Documents

2.1 *ASTM Standards:*

C 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime[2]

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments[3]

D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-out[4]

D 718 Test Methods for Analysis of Aluminum Silicate Pigment[3]

D 1366 Practice for Reporting Particle Size Characteristics of Pigments[3]

D 3360 Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments[3]

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry[5]

## 3. Composition and Properties

3.1 The pigment may be prepared by chemical precipitation or by the fine grinding of natural calcium carbonate containing minerals. If additional agents are used or any surface treatment is given, their purpose shall be indicated; acceptance shall be as agreed upon by the purchaser and the seller.

3.2 *Composition*—The pigment shall conform to the requirements for composition prescribed in Table 1.

3.3 *Fineness*—The pigment shall conform to the following general requirements for fineness for the grade specified:

3.3.1 *Grade I* (*Fine Paint Grade*)—This grade possesses substantial amounts of material in the fine sizes, and is in general essentially below 15 to 20 µm maximum size. Coarse particles retained on the No. 325 (45-µm) sieve shall be less than 0.05 %. The maximum Specific Surface Diameter (SSD) shall be 2.5 µm.

3.3.2 *Grade II* (*Coarse Paint Grade*)—This grade is characterized by substantial amounts in the 5- to 45-µm range, and is lower in pigment value than Grade I. Coarse particles retained on the No. 325 (45-µm) sieve shall be less than 0.5 %. The maximum SSD shall be 6 µm.

3.3.3 *Grade III* (*Filler Grade*)—This grade is characterized by substantial amounts in the 10- to 45-µm range but with the coarse particles retained on No. 325 (45-µm) sieve less than 25 % and a maximum SSD of 9 µm.

3.3.4 *Grade IV* (*Putty Powder Grade*)—This grade possesses less fines, and have substantial amounts of coarse particles. The coarse particles, however, shall not exceed 30 % retained on the No. 200 (75-µm) sieve. The maximum SSD shall be 12 µm.

3.3.5 *Grade V* (*Superfine Grade*)—This grade is a superfine ground natural limestone and is characterized by major amounts less than 5 µm and a weight median particle size in the range of 1 µm. The SSD is finer than 1 µm.

3.3.5.1 Particle size methods for Grade V that are applicable include transmission electron microscopy, scanning electron microscopy, and the Sedigraph.[6] Specific Surface Diameter can be determined by BET nitrogen absorption. The method of measurement produces different values, therefore, the method of measurement shall be agreed upon by the purchaser and the seller.

3.3.6 *Grade VI* (*Ultrafine Grade*)—This grade is an ultrafine precipitated calcium carbonate and is characterized by major amounts less than 2 µm and a median particle diameter determined by electron microscopy in the range of 0.05 µm.

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[1] This specification 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] *Annual Book of ASTM Standards*, Vol 04.01.

[3] *Annual Book of ASTM Standards*, Vol 06.03.

[4] *Annual Book of ASTM Standards*, Vol 06.01.

[5] Discontinued; See *1992 Annual Book of ASTM Standards*, Vol 06.01.

[6] The sole source of supply of the sedigraph known to the committee at this time is Micromeritics, 5680 Goshen Springs Rd., Norcross, GA 30093. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

**TABLE 1 Composition of Calcium Carbonate Pigments**

| | Type PC | Type GC |
|---|---|---|
| Moisture and other volatile matter, max, % | 0.7 | 0.2 |
| Calcium reported as carbonate, moisture-free, min, % | 96.5[A] | ... |
| Total calcium and magnesium reported as carbonates, moisture-free, min, % | ... | 94 |
| Magnesium as carbonate, max, % | ... | ... |

[A]Does not apply to specialty calcium carbonate.

3.3.6.1 Particle size methods for Grade VI that are applicable include transmission electron microscopy and scanning electron microscopy. The Sedigraph will give weight median particle size values aproximately 10 times greater (~0.5 µm) than by microscopy. Since the method of measurement produces different values, the method of measurement shall be agreed upon by the purchaser and the seller.

3.3.7 When closer control within a grade is required, the fineness requirements shall be as agreed upon by the purchaser and the seller.

3.4 *Dry Brightness or Dispersed Color*— The dry brightness or dispersed color shall be equal, within agreed upon tolerances, to that of a reference sample agreed upon by the purchaser and the seller.

3.5 *Oil Absorption*—Oil absorption values shall be as agreed upon by the purchaser and the seller.

## 4. Sampling

4.1 Two samples, each more than 1 lb (0.45 kg) shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5000 kg (10 000 lb), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending, in equal quantities, the samples from the same production unit forming a composite sample. Before testing, each of the samples shall be split, and one half of each may be sealed for referee testing.

## 5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be agreed upon by the purchaser and the seller.

5.2 *Calcium and Magnesium Reported as Carbonate*—Test Methods C 25.

5.3 *Moisture and Other Volatile Matter*— Method A of Test Methods D 280.

5.4 *Oil Absorption*—Test Method D 281.

5.5 *Coarse Particles*—Test Methods D 718.

5.6 *Dispersed Color*—Test Methods D 718, substituting the reference and test samples of calcium carbonate pigment for the standard extender pigment and sample respectively.

5.7 *Specific Surface Diameter*—Practice D 1366.[7]

5.8 *Dry Brightness*—The test sample and reference sample shall be prepared into suitable smooth, dry, packed surfaces in accordance with accepted practice and tested for reflectance using the green filter in accordance with Test Method E 97.

5.9 *Particle Size*—Test Method D 3360 is applicable only to Grades I, II, III, and IV.

NOTE 1—See 3.3.5 for other acceptable methods.

## 6. Keywords

6.1 calcium carbonate

[7] The sole source of supply of the sub-sieve sizer known to the committee at this time is Fisher Manufacturer. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.