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Standard Test Methods for Specific Gravity of Pigments[1]

This standard is issued under the fixed designation D153; 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 These test methods cover three procedures for determining the specific gravity of pigments, as follows:

Test Method A—For Routine Testing of Several Samples Simultaneously.

Test Method B—For Tests Requiring Greater Accuracy than Test Method A.

Test Method C—For Rapid and Accurate Testing of Single Samples.

1.2 The specific gravity value obtained by these procedures may be used with the weight of a dry pigment to determine the volume occupied by the pigment in a coating formulation.

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

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.* For specific hazard statements, see Sections 5, 11, and 15.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

3. Purity of Reagents

3.1 *Purity of Water*—Reference to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

TEST METHOD A—FOR ROUTINE TESTING OF SEVERAL SAMPLES SIMULTANEOUSLY

4. Apparatus and Materials

4.1 *Pycnometer*—A pycnometer (Note 1) having a 50-mL capacity.

NOTE 1—The Weld type with the cap seal on the outside of the neck of the bottle is preferred because there is less danger of trapping air just under the capillary tube than with types having the ground glass seal on the inside of the neck.

4.2 *Water Bath,* maintained at 25 ± 0.5°C and equipped with a stirring device.

4.3 *Manometer,* open- or closed-tube (see Part *f* of the apparatus for Test Method C), made of glass tubing 6 mm in diameter, fitted with rubber pressure tubing attached to a T-joint leading to the desiccator and the pump. For the open-tube type 860 mm of mercury shall be used. The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, shall be considered the absolute pressure of the system in millimetres of mercury.

4.4 *Desiccator,* glass, constructed with heavy walls to withstand a vacuum of one atmosphere, and with an opening at the side.

4.5 *Vacuum Pumps*—A laboratory water vacuum-type pump (Note 2), to remove the greater portion of air in the desiccator, and an oil vacuum-type pump, motor-driven, and capable of reducing the absolute pressure of the system to 3 mm.

NOTE 2—The water vacuum pump may be omitted if the rate of evacuation with the oil pump can be controlled so as to avoid a rapid ebullition of entrapped air and possible loss of specimen.

4.6 *Thermometer,* having a range from 0 to 60°C, and graduated in 0.1°C divisions.

4.7 *Weighing Bottle,* wide-mouth cylindrical glass (about 30 mm in height and 70 mm in diameter), provided with a ground-glass stopper.

4.8 *Immersion Liquid*—Kerosine has been found to be a good wetting vehicle for most pigments, and shall be used generally as the immersion liquid. Refined, white kerosine of narrow evaporation and boiling range shall be used. With some

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are 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.

pigments that are not wetted well with kerosine, other immersion liquids such as glycerin, ethylene glycol, tetrahydronaphthalene, etc., may be substituted. The liquid must have a low evaporation rate and narrow boiling range, and the same procedure shall be followed as with kerosine. Water is not a preferred liquid because of the possibility of frothing.

5. Hazards

5.1 Before a desiccator is used for the first time, wrap it in a towel and test under an absolute pressure of under 3 mm. Exercise care in handling the desiccator when under vacuum, since a sudden jar may cause it to collapse.

6. Standardization of Pycnometer

6.1 Fill the pycnometer with freshly boiled water at 23 to 24°C, gradually bring to 25 ± 0.5°C, and then dry and weigh as specified in 7.6. Empty the pycnometer, and clean, dry, and reweigh it. Next fill the pycnometer with kerosine at 23 to 24°C, bring to 25 ± 0.5°C, dry, and weigh as before. Calculate the specific gravity, *S*, of the kerosine at 25/25°C as follows:

$$S = A/B \quad (1)$$

where:

A = weight of kerosine, g, and
B = weight of water, g.

7. Procedure

7.1 *Drying*—Dry the pigment, preferably in an electric oven, at 105 ± 2°C for 2 h.

7.2 *Weighing*—Transfer to a clean, dry, weighed pycnometer, sufficient sample to form a layer approximately 20 mm (¾ in.) deep. For black, blue, and lake pigments of low specific gravity, use about 1 g of sample; for inert crystalline pigments, about 4 g; for opaque white pigments, 7 to 10 g; and for red lead, from 15 to 20 g. Weigh pigments of a hydroscopic nature from the weighing bottle.

7.3 *Number of Specimens*—Run all samples at least in duplicate.

7.4 *Addition of Kerosine*—Add enough kerosine to the pycnometer to form a clear layer approximately ¼ in. (6 mm) above the pigment. When necessary, stir the specimen with a polished round-bottom glass rod until completely covered by kerosine, adding more kerosine if necessary. Wash the rod with kerosine, adding the washings to the pycnometer.

7.5 *Removal of Occluded Air*—Place the pycnometer in the desiccator. Close the desiccator and attach to the water pump until the greater part of the air is removed from the system. Complete this procedure within a period of 5 to 10 min. Close the system with a pinchcock and attach the desiccator to the oil pump for the removal of the small amounts of air given off at the low pressures obtainable with the oil pump. Use the manometer to indicate whether the oil pump is giving the proper vacuum. When the manometer indicates that the absolute pressure is 3 mm and constant, cut off the oil pump for short periods, taking care that the vacuum does not change materially due to leakage. At first bubbles of air rise from the pigments very rapidly, then this action gradually decreases and finally stops. The time required for complete removal of air may vary from 30 min to 24 h, depending upon the nature of the pigment. When no more bubbles can be seen, it may be assumed that the occluded air has been removed and that the pigment is thoroughly wet with kerosine. Then slowly admit air to the desiccator by means of the pinchcock.

7.6 *Filling and Bringing to Temperature*—Remove the pycnometer from the desiccator, fill with kerosine at 24 to 25°C taking care to add a sufficient quantity to prevent air bubbles where the pycnometer is closed, and permit to come to constant temperature at 25 ± 0.5°C in the water bath. Carefully stopper the pycnometer and remove excess kerosine with lens paper. Take the pycnometer out of the bath, allow to come to room temperature, and weigh.

8. Calculation

8.1 Calculate the specific gravity, *S*, of the pigment as follows:

$$S = \frac{P_1}{W - \frac{K_1}{D}} \quad (2)$$

where:

P_1 = weight of pigment used, g,
W = weight of water to fill the pycnometer, g,
K_1 = weight of kerosine added to the pigment, g, and
D = specific gravity of the kerosine.

9. Precision

9.1 Duplicate determinations by this test method should not differ by more than 0.02.

TEST METHOD B—FOR TESTS REQUIRING GREATER ACCURACY THAN TEST METHOD A

10. Apparatus (see Fig. 1 and Fig. 2)

10.1 *Pycnometer, Water Bath, Manometer, Vacuum Pump, Thermometer, Weighing Bottle, and Immersion Liquid*—See Section 4; also Fig. 2 (*e*) and (*f*).

10.2 *Bell Jar,* glass, with a two-hole rubber stopper. Into one hole of the stopper shall be fitted a separatory funnel with a well-ground stopcock (Fig. 1 (*c*)), extending into the pycnometer. Into the other hole of the stopper shall be fitted a glass tube with a well-ground three-way stopcock (Fig. 2 (*d*)) and

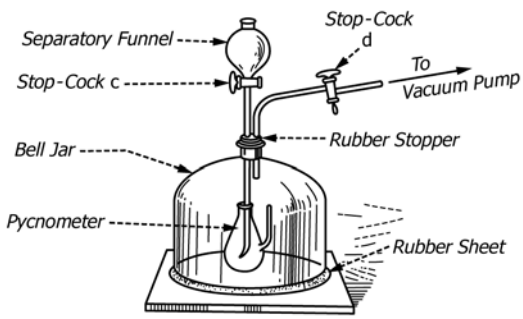


FIG. 1 Apparatus for Test Method B

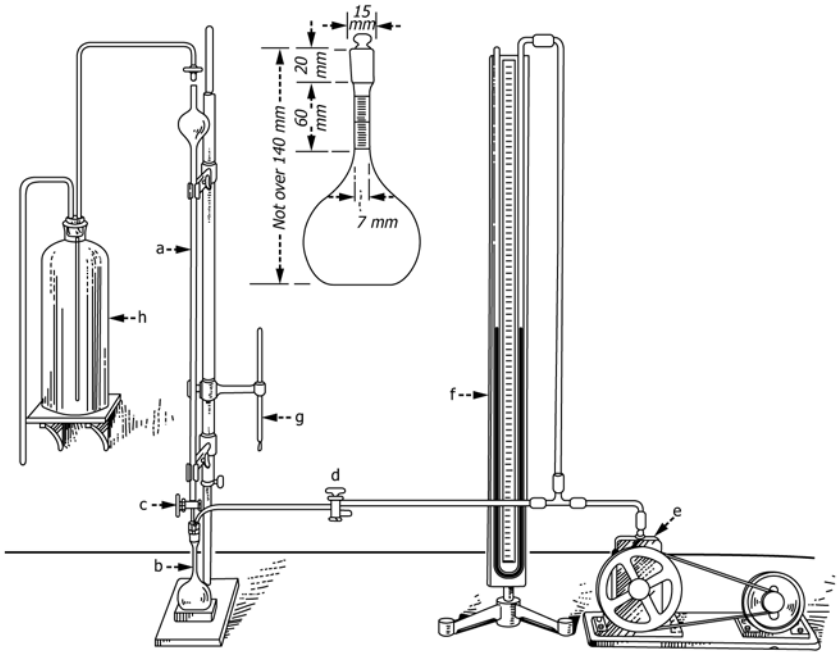


FIG. 2 Apparatus for Test Method C

connected with the vacuum pump (Fig. 2 (*e*)). The bell jar shall rest on a sheet of rubber, cemented or vulcanized to a glass or iron plate. With stopcock *c* closed and stopcock *d* open to the pump, the system shall maintain an absolute pressure of at most 3 mm. A desiccator may be used instead of a bell jar.

10.3 *Bottle,* storage, (Fig. 2 (*h*)) for kerosine or other wetting liquid.

11. Hazards

11.1 Before a bell jar (or desiccator) is used for the first time, test under a vacuum as described in Section 5.

11.2 Use a buret stopcock (Fig. 2 (*c*)) that is well ground and lubricated with silicone lubricants or use a PTFE-coated stopcock.

12. Procedure

12.1 Place the pycnometer containing the weighed, dried pigment under the bell jar. Close stopcocks *c* and *d*, start the vacuum pump, and then gradually open stopcock *d* to the pump. When an absolute pressure of 3 mm has been attained and can be maintained, fill the separatory funnel with kerosine, close stopcock *d*, and gradually open stopcock *c*, adding sufficient kerosine to cover the pigment. Then stop the pump and release the suction at stopcock *d*. Finally, fill the pycnometer with kerosine, and complete the test as described in 7.6 and Section 8, under Test Method A.

13. Precision

13.1 Duplicate determinations by this test method should not differ by more than 0.01.

TEST METHOD C—FOR RAPID AND ACCURATE TESTING OF A SINGLE SPECIMEN

14. Apparatus (see Fig. 2 and Fig. 3)

14.1 *Buret,* 100-mL, with a 75-mL bulb in the upper part, and with the lower part (25 mL) graduated in 0.05-mL divisions (see Fig. 3).

14.2 *Flask*—A special 100-mL graduated flask (Fig. 2 (*b*)) with ground-glass stopper. The flask shall be thick enough to withstand an absolute pressure of 1 mm, and shall weigh between 50 and 60 g. The neck of the flask shall be graduated in 0.05-mL divisions between the 99 and 100-mL marks. The dimensions of the flask shall be as shown in Fig. 2.

14.3 *Stopcocks*—A tightly ground stopcock (Fig. 2 (*c*)) as part of buret, *a*, and a three-way stopcock (Fig. 2 (*d*)) connecting with the vacuum pump, *e*. To prevent leakage of kerosine use a buret stopcock (Fig. 2 (*c*)) that is well ground and lubricated with silicone lubricant or use a PTFE-coated stopcock.

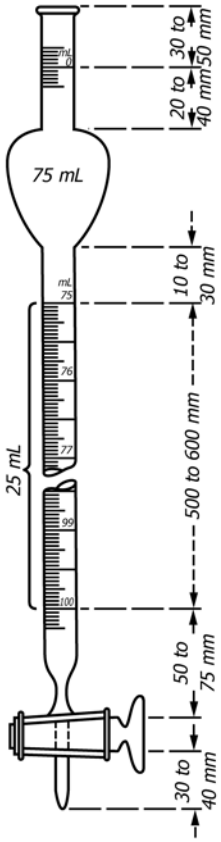
14.4 *Vacuum Pump*—See 4.5; also Fig. 2 (*e*). In this procedure the oil vacuum pump shall be capable of reducing the absolute pressure of the system to 1 mm.

14.5 *Manometer, Thermometer, Weighing Bottle, and Immersion Liquid*—See Section 4; also Fig. 2 (*f*).

14.6 *Bottle*—See 10.3.

15. Hazards

15.1 The variations that occur under normal conditions in a room do not materially affect the specific gravity of a pigment. However, take care that the temperature of the liquid after transferring to the flask is approximately the same as it was when in the buret.



Buret: Geissler, straight; glass stopcock, ground accurately.

Total capacity	100 mL
Capacity of bulb	0 to 75 mL
Graduated	75 to 100 mL
Subdivisions	0.05 mL
Rate of outflow	about 2 min
Permissible variations:	
Capacity, total	±0.10 mL
Capacity graduated portion	±0.03 mL

Markings on graduations shall be in conformity with the National Bureau of Standards *Circular No. 9*.[3]

FIG. 3 Buret in Apparatus

15.2 Since in determining both K_2 and V the tip of the buret and bore of the stopcock plug are empty, no correction is to be made; but stopcock *c* must be so well ground that under an absolute pressure of 1 mm for 30 min no leakage of kerosine shall take place. The usual sources of error are failure to remove all the air from the pigment, and leaks in the system. Use a minimum amount of rubber tubing in the system and, wherever it is used, coat the joints between rubber and glass with a melted mixture of beeswax and rosin.

15.3 In cleaning the flask of kerosine only, a rinsing two or three times with ether, followed by dry air (dried over sulfuric acid and calcium chloride), is considered sufficient. When pigment is also present, remove both pigment and kerosine and follow with ether rinses until no more pigment remains. Add some filter pulp (macerated filter paper) and water (with or without glass beads), and shake vigorously. Repeat if necessary. Rinse the flask with reagent water, and either dry in an oven, or rinse with alcohol and ether followed by dry air.

16. Standardization of Apparatus

16.1 Connect the flask to the buret and the pump by means of a two-holed rubber stopper. Evacuate the system with the buret stopcock (Fig. 2 (*c*)) closed until the pump maintains an absolute pressure of 1 mm in the flask. Close the three-way stopcock, *d*, for 30 s, and again open to the pump. There shall be no appreciable change in the mercury levels in the manometer, indicating that the system beyond stopcock *d* is tight. With the vacuum still maintained, fill the buret from the top with kerosine, adjusting the level to the zero mark with a piece of capillary tubing. Now close stopcock *d*, and carefully open stopcock *c*, admitting about 75 mL of kerosine into the flask. Open stopcock *d* to the air, thus releasing the vacuum in the flask, and fill the flask with kerosine to a definite mark on the neck. Read the buret, calling this reading K_2 (the volume of the flask).[3]

17. Procedure

17.1 Clean the flask dry, and weigh. Transfer a quantity of the dry pigment to be tested to the flask by means of a clean, dry, glass funnel with the stem reaching to the bottom of the bulb. A piece of stiff nickel wire is convenient to push the powder down the stem. Nearly fill the bulb of the flask with the pigment, which, however, shall occupy a volume of less than 25 mL after all air is expelled. Greater accuracy may be obtained with a large specimen than with a small one. Wipe the inside stem as well as the entire outside of the flask with a clean piece of dry, lintless cloth. Weigh the flask and pigment, and calculate the weight of pigment by deducting the weight of the empty flask. With the buret clean and dry, attach the flask to the evacuating system as shown in Fig. 2. After closing stopcocks *c* and *d*, start the pump and carefully open stopcock *d* to the pump. Continue evacuation until the pump maintains an absolute pressure of 1 mm in the flask, or until all the air is removed from the system. Then fill the buret from the top as described in Section 16, close stopcock *d*, gradually open stopcock *c*, and add kerosine until the pigment is covered. Tap the flask gently to dislodge any air bubbles. Stop the pump, open stopcock *d* to the air, and fill the flask up to the same mark as was obtained in determining its volume. Designate the volume of kerosine required as *V*. Read the height of the liquid in the buret to the nearest estimated 0.01 mL.

18. Calculation

18.1 Calculate the specific gravity, *SG*, of the pigment as follows:

$$SG = P_2/(K_2 - V)$$

where:

P_2 = weight of pigment used, g,
K_2 = volume of kerosine required to fill the flask when empty, mL, and
V = volume of kerosine required to fill the flask when the pigment is present, mL.

[3] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

19. Precision

19.1 Duplicate determinations by this test method should not differ by more than 0.01.

20. Keywords

20.1 pigments; specific gravity