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Standard Test Methods for Specific Gravity of Coating Powders[1]

This standard is issued under the fixed designation D5965; 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 These test methods cover three procedures for determining the specific gravity (see definition) of coating powders, as follows:

TEST METHOD A—For Testing Coating Powders, Excluding Metallics
TEST METHOD B—For Tests Requiring Greater Precision than Test Method A, Including Metallics, Using Helium Pycnometry
TEST METHOD C—For Theoretical Calculation Based on Raw Material Specific Gravities

1.2 Test Method A can be used as a less expensive method with reduced accuracy for determining the specific gravity of coating powders, excluding metallics.

1.3 The ideal gas law forms the basis for all calculations used in the Test Method B determination of density of coating powders.

1.4 Test Method B includes procedures that provided acceptable results for samples analyzed during round robin testing.

1.5 Test Method B uses SI units as standard. State all numerical values in terms of SI units unless specific instrumentation software reports surface area using alternate units. Many instruments report density as g/cm^3, instead of using SI units (kg/m^3).

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

1.7 *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:*[2]
D3924 Specification for Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials
D5382 Guide to Evaluation of Optical Properties of Powder Coatings
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions 3.1.1 and 3.1.3 are from Guide D5382.

3.1.2 *coating powder, n*—finely divided particles of resin, either thermoplastic or thermosetting, generally incorporating pigments, fillers, and additives and remaining finely divided during storage under suitable conditions, which, after fusing and possibly curing, give a continuous film.

3.1.3 *meniscus, n*—curved upper surface of a liquid column that is concave when the containing walls are wetted by the liquid.

3.1.4 *powder coating, n*—coatings which are protective or decorative, or both, formed by the application of a coating powder to a substrate and fused into continuous films by the application of heat or radiant energy.

3.1.5 *pycnometer, n*—instrument designed to measure the volume of solid materials using Archimedes' principle of fluid displacement. The displaced fluid is a helium gas.

3.1.6 *specific gravity*—(*1*) strict definition: the density of a substance relative to that of water; (*2*) practical, as used in this test method—The numerical value of the density when the latter is expressed in grams per millilitre.

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[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 D01.51 on Powder Coatings.

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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.

4. Significance and Use

4.1 Test Method A is a less expensive method of determining specific gravity of coating powders, excluding metallics, that produced less precise results than Test Method B.

4.2 Test Method B provides better precision at higher cost and includes metallics, although different models produced different grand averages for each of the three samples tested.

4.3 Test Method C is commonly used by the powder coating industry to estimate the coverage of a powder coating at a given thickness, using the theoretical specific gravity calculated from those of the raw materials.

5. Reagents

5.1 *Purity*—Wetting vehicles should be of reagent grades.

5.2 *Helium*—Shall be understood to mean high purity of commercial grade.

6. Conditioning

6.1 These tests should be standardized at 23 ± 2°C (73.5 ± 3.5°F) and relative humidity of 50 ± 5 % for the two methods in compliance with Specification D3924.

TEST METHOD A—FOR TESTING POWDER COATINGS, EXCLUDING METALLICS

7. Apparatus and Materials

7.1 *Volumetric Flask*—Calibrated narrow-necked glass type, having a 50-mL capacity.

7.2 *Balance*—A calibrated laboratory balance having a ±0.001 g-accuracy. A less accurate balance can be used with a relative effect on the results.

7.3 *Coating Powder*—Weighed to 15 g, within a ±0.01 g-accuracy.

7.4 *Immersion Liquid*—Hexane was found to be a good wetting vehicle for the epoxy and polyester coatings used in the round robin for the testing of repeatability and reproducibility.

7.5 *Glass Funnel*—Designed to fit within the neck of the volumetric flask.

7.6 *Polished Round-Bottom Glass Rods*—For dispersing powder.

7.7 *Squeeze Bottle*—Suitable for containing and dispensing wetting vehicle.

8. Hazards

8.1 Exercise care in handling all wetting vehicles. Make sure that personal equipment includes protective gloves, glasses, and clothing. Perform test method using wetting vehicles in a solvent hood.

9. Standardization

9.1 Weigh the empty, clean volumetric flask. Record this weight as *WF*.

9.2 The density of the wetting vehicle, recorded as *DL*, can be determined by adding exactly 50 mL of wetting vehicle to the previously weighed flask and reweighing. Record this weight as *WFL*. Calculate the density of the wetting vehicle (DL) as follows:

$$DL = \frac{(WFL - WF)}{50\ \text{mL}} \quad (1)$$

10. Procedure

10.1 Weigh the 50-mL volumetric flask. Record this weight as *WF*. Add 15 g of powder to the clean, dry, weighed flask and accurately reweigh. Record this weight as *WFP*. Add enough wetting vehicle to cover the powder and gently swirl until the powder is completely wet.

10.2 The removal of entrapped air has a significant effect on the accuracy of the results. Care should be taken to insure wetting out of the powder is complete. When necessary, stir the powder with a polished round-bottom glass rod until completely covered by the wetting vehicle. Wash the rod with wetting vehicle, adding the washings to the flask without exceeding the 50-mL calibration mark.

10.3 Add additional wetting vehicle up to the 50-mL mark. Make sure that the bottom of the meniscus is aligned at eye level with the line on the front and back of the flask neck. This addition of wetting vehicle can be done with a squeeze bottle in a manner to wash any residual powder from the neck of the flask. Reweigh and record this weight as *WFPL*.

10.4 Multiple volumetric flasks can be used in rotation to reduce cleaning and complete drying time.

10.5 **Immediately** clean the flask after each test to increase the ease with which this is accomplished. Each flask shall be completely clean and dry before proceeding to the next test.

11. Calculation

11.1 Calculate the density of the powder (*DP*) as follows:

$$DP = \frac{WFP - WF}{50\ \text{mL} - \dfrac{WFPL - WFP}{DL} = \text{denominator}} = \text{numerator} \quad (2)$$

where:

WFP = weight of flask and powder,
WF = weight of flask,
WFPL = weight of flask, powder, and wetting vehicle,
DL = density of wetting vehicle, and
DP = specific gravity of powder.

11.2 An example, using hexane, would be as follows:

$$DP = \frac{50.545\ \text{g} - 36.581\ \text{g}}{50\ \text{mL} - \dfrac{77.200\ \text{g} - 50.545\ \text{g}}{0.663\ \text{g/mL}}} = \frac{13.964}{9.796}$$

$$= 1.42\ \text{specific gravity} \quad (3)$$

where:

WFP = 50.545 g,
WF = 36.581 g,
WFPL = 77.200 g,
DL = 0.663 g/mL, and
DP = unknown.

12. Report

12.1 Report the following information:

12.1.1 Use duplicate determinations with the average reported to two significant figures to the right of the decimal.

12.1.2 Report the complete sample identification and the wetting vehicle used to determine the specific gravity.

13. Precision and Bias[3]

13.1 *Precision*—The average of duplicate determinations by this test method should not differ by more than 0.025 using a balance with 0.0001 significant figures or 0.04 using a balance with 0.001 significant figures.

13.2 *Bias*—Bias has not been determined.

TEST METHOD B—FOR TESTS REQUIRING GREATER PRECISION THAN TEST METHOD A, INCLUDING METALLICS, USING HELIUM PYCNOMETRY

14. Apparatus and Materials

14.1 *Commercial Pycnometer Instruments,* available from several manufacturers for the measurement of skeletal volume by gas displacement. Some instruments perform calculations of volume or density, or both, upon completion of the analysis. Others require manual calculation of skeletal volume and density.

14.2 *Analytical Balance,* having a ±0.0001-g accuracy.

15. Sampling

15.1 It is important that the sample being analyzed represent the larger bulk from which it is taken. The bulk sample should be homogeneous before any sampling takes place.

16. Calibration and Standardization

16.1 Follow manufacturer's instructions for calibration and operational verification of the pycnometer and analytical balance.

17. Outgassing

17.1 Weigh the clean, empty sample holder to the nearest 0.1 mg. Record the empty holder weight.

17.2 Add representative sample to the empty sample holder. The sample quantity should be sufficient to satisfy the minimum skeletal volume as required by the manufacturer. Weigh and record the weight of the sample and sample holder.

NOTE 1—Move to the Procedure Section if the sample is to be outgassed in the pycnometer at the time of analysis.

17.3 Place prepared sample holder in outgassing device.

17.4 Program outgassing device for initial outgassing temperature. Increase temperature as appropriate for the sample. Allow sample to continue to outgas until prescribed vacuum level is achieved or prescribed outgassing time, or both.

17.5 Reduce the temperature of the outgassing device to ambient. Remove the sample holder.

17.6 Weigh the sample holder to the nearest milligram to obtain the sample and holder weight. Subtract the empty sample holder weight determined in 16.1 to obtain the outgassed sample weight. Record the calculated weight.

18. Procedure

18.1 Place the filled sample holder in the pycnometer and close the sample chamber.

18.2 *Automated Instruments Only*—Select, or input, the desired analysis and report parameters. Include the outgassing parameters if the sample preparation is performed as a part of the sample analysis. If necessary, input the outgassing sample weight. The final weight should be determined and entered after the analysis. Determine the skeletal volume a minimum of five times.

18.3 *Manually Operated Instruments*—Collect three to five sets of analysis data according to the manufacturer's recommended procedure for maximum accuracy and precision.

18.4 When the analysis has finished, remove the sample holder. Weigh the holder to the nearest 0.1 mg. Record the final holder and sample weight. Subtract the empty holder weight recorded in 16.1 to obtain the final sample weight.

18.5 *Automated Instruments Only*—Input the final sample weight. Generate the final sample report.

19. Calculations

19.1 *Automated Instruments Only*—Have software that automatically calculates the results for the chosen reports using the final weight input in 18.5.

19.2 *Manually Operated Instruments*—Calculate the skeletal volume using collected data according to the manufacturer's instructions. Use the final sample weight from 16.4 to calculate skeletal densities. Calculate the average and standard deviation for skeletal volume and density in accordance with Practice E691.

20. Report

20.1 Report the following information:

20.1.1 Complete sample identification and measured skeletal volumes, statistics, and density determined. Note any units used other than standard.

20.1.2 Analysis gas type used.

20.1.3 Sampling outgassing method, including total time and outgassing temperature(s).

TEST METHOD C—FOR THEORETICAL CALCULATION BASED ON RAW MATERIAL SPECIFIC GRAVITIES

21. Calculations

21.1 *To Calculate the Theoretical Specific Gravity of a Coating Powder When the Formula is Known*—Divide the amount of each raw material (*RM*) by its specific gravity. Add the raw material amounts together and divide by the sum of the

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1100.

resulting values for all of the raw materials in the subject powder. The product of this calculation shall be the theoretical specific gravity of the coating powder, as follows:

$$\text{Theoretical specific gravity} = \frac{\text{grand total of amounts } (RM1 \text{ through } RM6)}{\text{sum of resulting values } (RM1 \text{ through } RM6)} \quad (4)$$

where:

RM1 amount divided by specific gravity	=	$RM1$ resulting value
RM2 amount divided by specific gravity	=	$RM2$ resulting value
RM3 amount divided by specific gravity	=	$RM3$ resulting value
RM4 amount divided by specific gravity	=	$RM4$ resulting value
RM5 amount divided by specific gravity	=	$RM5$ resulting value
RM6 amount divided by specific gravity	=	$RM6$ resulting value
Grand total		Sum of resulting values

21.2 Report the powder specific gravity.

22. Precision and Bias[3]

22.1 Precision and bias of the procedures in Test Methods A and B for measuring the specific gravity of coating powders has not been determined because the minimum number of laboratories required by Practice E691 was not met. An interlaboratory study was conducted by four laboratories to determine the specific gravity of two coating powders using Test Method A and three coating powders using Test Method B.

23. Keywords

23.1 coating powders; density; metallics; powder coatings; pycnometer; specific gravity