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Standard Test Methods for Specific Gravity Density of Coating Powders¹

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 <u>standard</u> test methods cover three procedures for determining the <u>specific gravity (see definition)</u> <u>density</u> 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. A, for testing coating powders, excluding metallics, is a method that uses readily available laboratory equipment (for example, analytical balance, volumetric flask, etc.).

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.

1.3 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/cmrequires the use of a pycnometer.³, instead of using SI units (kg/m³).

1.4 Test Method C is a method that calculates the density of a powder based upon the formula ingredients and their amounts and densities.

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

1.6 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 safety, health, and healthenvironmental practices and determine the applicability of regulatory limitations prior to use.

<u>1.7 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.</u>

2. Referenced Documents

2.1 ASTM Standards:²

D3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials (Withdrawn 2016)³

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 The last approved version of this historical standard is referenced on www.astm.org.

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.

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3.1.1 *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.

3.1.2 density, n—the mass per unit volume of a material, usually expressed in g/cm³.

3.1.2.1 Discussion—

In this standard, a volumetric flask capacity is expressed—by convention—in mL. However, convention also states density in terms of mass per cm³. Since 1 mL = 1 cm³, terms will be interchanged—by convention—but will not affect any of the calculations. 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 <u>specific gravity—wetting liquid, n—(an1)</u> strict definition: the density of a substance relative to that of water; (<u>organic</u> <u>solvent</u> 2) practical, as used in this test method—The numerical value of the density when the latter is expressed in grams per millilitre used to wet-out the powder and displace the air that is trapped between the powder particles.

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.straight-forward method using readily available laboratory equipment and glassware. Test Method A may only be used with powder that does not contain metallic pigments.

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<u>may be</u> 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. when the formulation is known, and the density of each raw material is available.

5. Reagents

5.1 *Purity*—<u>Wetting liquid</u>—Wetting vehicles should be of reagent grades.<u>May be a reagent grade solvent or a solvent blend</u>, such as kerosene.

5.2 Helium-Shall be understood to mean high purity of Must be high purity, commercial grade.

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6. Conditioning

6.1 These tests should be standardized at $23 \pm 2^{\circ}C$ (73.5 $\pm 3.5^{\circ}F$) 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-at least 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.at least ± 0.01 g-accuracy.

7.3 Coating Powder-Weighed to approximately 15 g, within a-at least ±0.01 g-accuracy.

7.4 *Immersion*<u>Wetting</u> Liquid—Hexane was (reagent grade) or a solvent blend such as kerosene has been 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-powder coatings.

NOTE 1-Wetting liquids must not swell or dissolve the powder.

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

7.6 Polished Round-Bottom Glass Rods-For dispersing powder.mixing powder to displace the air.

7.7 Squeeze Bottle—Suitable for containing and dispensing wetting vehicle.liquid (for example, hexane, kerosene).

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:

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

9. Procedure

9.1 Weigh the volumetric flask to at least the nearest 0.01 g. Record this mass as F_{empty} .

9.2 Weigh the 50-mL volumetric flask. Record this weight as WF.Add approximately 15 g of powder to the elean, dry, weighed empty flask and accurately reweigh. Record this weightmass as $WFP.F_{with powder}$ Add enough wetting vehicle to cover the powder and gently swirl until the powder is completely wet.to at least the nearest 0.01 g.

9.3 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 eovered Add just enough wetting liquid to cover the powder. Gently swirl until the powder is completely wet by the wetting vehicle. Wash the rod with wetting vehicle, adding the washings to the flask without exceeding the 50-mL calibration mark.liquid.

9.3.1 Displacing all entrapped air is mandatory. Care should be taken to ensure wetting-out of the powder is complete. When necessary, carefully stir the powder with a polished round-bottom glass rod until the powder is completely covered by the wetting liquid. Rinse the rod with wetting vehicle, being sure that all material is washed into the flask *without exceeding the volume mark*.

9.4 Add additional wetting vehicle liquid up to the 50-mLmL mark. Make sure that the bottom of the meniscus is aligned at eye level with the line on the front and back of the volumetric 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.

9.5 Multiple volumetric flasks can be used in rotation to reduce cleaning and complete drying time: Reweigh the flask, which now contains powder and wetting liquid, and record this mass as $F_{powder+hexane}$.

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

9.5.2 Clean the flask thoroughly after each test.

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

10. Calculation

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

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WII = WI	- numerator	(\mathbf{n})
$DP = \frac{WFPL - WFP}{WFPL - WFP}$	- = numerator	(2)
50 mL $-\frac{mHE}{DL} =$ denominator		

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.

Density of Wetting Liquid:

10.1.1 When using reagent grade wetting liquid (for example, hexane), use the density value supplied by the manufacturer. 10.1.2 When using a wetting liquid that is a blend of solvents (for example, kerosene), the density must be measured.

10.1.2.1 To measure the density of the wetting liquid, weigh a volumetric flask with a capacity of at least 50 mL. Record this mass to the nearest 0.01 g as F_{empty} . Fill the flask to the mL mark and reweigh the flask. Record this mass as $F_{wetting liquid}$. The density of the wetting liquid is determined as follows:

Density _{wetting liquid} =	$\boldsymbol{F}_{\rm wetting\ liquid}-\boldsymbol{F}_{\rm empty}$
	Volume of flask

For example, if a 50 mL volumetric flask is used, and if

10.2 An example, using hexane, would be Calculate the density of the powder as follows: