Designation: D480 – 88 (Reapproved 2020)

Standard Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes[1]

This standard is issued under the fixed designation D480; 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 procedures for sampling, qualitative analysis, and physical testing of flaked aluminum powders and pastes (leafing and nonleafing) for coatings.

1.2 These test methods apply equally to leafing and nonleafing flaked aluminum powders and pastes except where noted to the contrary.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 7.3.1.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D185 Test Methods for Coarse Particles in Pigments

D235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)

D329 Specification for Acetone

D962 Specification for Aluminum Powder and Paste Pigments for Paints

D3980 Practice for Interlaboratory Testing of Paint and Related Materials (Withdrawn 1998)[3]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E34 Test Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys (Withdrawn 2017)[3]

E607 Test Method for Atomic Emission Spectrometric Analysis Aluminum Alloys by the Point to Plane Technique Nitrogen Atmosphere (Withdrawn 2011)[3]

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Significance and Use

3.1 Flaked aluminum pigments are produced in a variety of forms. These test methods allow the user to determine the applicability of a given product to this use.

4. Sampling

4.1 Sampling is subject to mutual agreement between the seller and the purchaser. Place each sample in a clean, dry metal or glass container which shall be nearly filled, then close with a tight cover, seal, mark, and send to the laboratory for testing.

4.2 When requested, duplicate samples may be taken from the same container and delivered to the seller, and the inspector may take a third set of samples to hold for test in case of disagreement.

5. Qualitative Analysis

5.1 *Significance and Use*—This test method determines if there are chemical impurities, other than fatty and oily matter, as specified in Specification D962, to ensure the absence of fillers or extender pigments.

5.2 *Procedure:*

5.2.1 *Total Impurities*—Determine the total impurities by weight in accordance with Test Methods E34 and E607 or by atomic absorption.

[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.

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

5.2.2 *Total Aluminum*—Determine the total aluminum by weight difference with elemental impurities in accordance with 5.2.1.

6. Leafing Properties

6.1 The leafing test conditions specified are selected arbitrarily, and even though the numerical leafing value obtained by this test method appears to be low, the pigment may give substantially perfect leafing under the conditions of practical application in a paint.

6.2 *Significance and Use*—This leafing test method has been established to determine the percent of leafed aluminum flakes at the surface of a simulated paint formula to ensure a bright metallic luster. Minimum leafing characteristics must conform to Specification D962.

6.3 *Apparatus:*

6.3.1 *Leafing Spatula,* nonmagnetic, stainless steel spatula having the following dimensions:

Length of blade min, in. (mm)	5.5 (139.7)
Width of blade, in. (mm)	0.540 ± 0.002 (13.72 ± 0.05)
Thickness of blade, in. (mm):	
10 mm from tip	0.013 ± 0.003 (0.33 ± 0.08)
100 mm from tip	0.038 ± 0.004 (0.97 ± 0.10)
Shape of tip	rounded or straight

The surface of the spatula shall be polished with 3/0 emery or silicon carbide metallographic paper wet with mineral spirits; polishing shall be repeated for 1-min intervals until a consistent minimum leafing result is obtained in a typical leafing test.

6.3.2 *Test Tube,* 152 mm (6 in.) in length by 19.0 mm (0.75 in.) in diameter.

6.3.3 *Glass Cylinder,* 203 mm (8 in.) in length and 38.1 to 50.8 mm (1.5 to 2.0 in.) in inside diameter. A stopper shall be used and the spatula shall be attached to the stopper so as to hang vertically in the cylinder with the tip about 25 mm (1 in.) from the bottom.

6.4 *Reagents:*

6.4.1 *Coumarone-Idene Resin*—The form commercially known as "chipped" and having the following properties:

Softening point, °C	109 to 117
Specific gravity	1.12 to 1.16
Acid value, max	0.5
Cloud point, °C, max	−7

6.4.2 *Petroleum Spirits*—Conforming to Specification D235 and having the following additional properties:

Specific gravity at 16°C (60°F)	0.800 to 0.810
Surface tension at 21°C (70°F), N/m, min	24.5
Kauri butanol value	42 to 44
Aniline point, °C (°F)	40 to 45 (105 to 115)

6.5 *Preparation of Leaf-Testing Vehicle*—Prepare solution by dissolving 30.0 g of resin, in the form of small chips, in 100 mL of petroleum spirits, while heating gently at about 60°C (140°F). The specific gravity of the solution shall be between 0.877 and 0.881 at 16°C (60°F). Allow the solution to settle and retain the clear portion for use.

6.6 *Aliquot*—Unless otherwise specified, the aliquot weight shall be as follows:

Classification	ASTM Designation	Class	Aliquot Weight, g
Flaked aluminum powder	D962, Type 1	A	1.0
		B	1.0
		C	2.0
Aluminum paste	D962, Type 2	A	1.5
		B	1.5
		C	3.0

6.7 *Procedure*—Perform the test at a room temperature of 25 ± 2°C. Place approximately 5 mL of the leaf-testing vehicle in the glass cylinder and cover with a watch glass. Transfer 25.0 ± 1.0 mL of the vehicle to a clean container. Weigh the required aliquot of aluminum powder or paste to the nearest 0.01 g and transfer to a small dish. Add about 2.5 mL of the 25-mL quantity of vehicle and mix to a stiff paste with a spatula or small stiff brush. Add approximately 5 mL more of the vehicle and stir to a smooth mixture, then add the remainder of the vehicle and continue stirring to obtain complete dispersion. Transfer the mixture to the test tube to give a depth of about 110 mm when the specified spatula is immersed. Avoid formation of bubbles. Dip the spatula to the bottom of the mixture and rotate it gently through an arc of about 90° for 10 s. Withdraw the spatula at a uniform rate of about 50 mm (2 in.)/s. The spatula should not touch the wall of the test tube above the liquid level. Immediately suspend the spatula vertically in the glass cylinder with leafing liquid in it and allow to remain for 3 min. Measure the height of immersion and the height of complete leafing on both sides of the spatula, measuring to the bottom of the meniscus in each case. Clean the spatula by rinsing it in petroleum spirits and wipe dry with a clean cloth. Stir (do not shake) the mixture in the test tube and repeat the determination.

6.8 *Calculation*—Calculate the leafing value *L* as follows:

$$L = A/B \times 100 = A \times 100/B$$

where:

A = height of complete leafing, in. (mm), and
B = height of immersion, in. (mm).

7. Coarse Particles

7.1 This section provides two test methods for determining the percent residue retained on a sieve by wet-screening techniques.

7.2 *Significance and Use*—These procedures determine the percent of coarse particles present in aluminum pigments to ensure a smooth appearance, as required in Specification D962.

7.3 *Procedure A:*

7.3.1 *Aluminum Powder*—Fill two "straight wall" containers not less than 305 mm (12 in.) in diameter to a depth of not less than 102 mm (4 in.) with petroleum spirits, and fill a third similar container to a depth of not less than 76 mm (3 in.) with acetone. The petroleum spirits used for the test shall conform to Specification D235, and the acetone shall conform to Specification D329. Weigh 5 g of the aliquot, transfer to a 250-mL beaker, and add in small portions a total of about 150 mL of petroleum spirits, mixing thoroughly to a uniform consistency after each addition. Clamp a standard sieve 203 mm (8 in.) in diameter conforming to Specification E11,

just above the level of the petroleum spirits in the first container and pour the specimen on the sieve, using a 45-µm (No. 325) sieve for Classes A, B, and C powder and in addition a 150-µm (No. 100) sieve for Class C powder. Rinse the beaker with clear petroleum spirits and transfer the rinsings to the sieve. Holding the sieve at a slight angle to prevent air lock, shake it backward and forward working the sieve alternately just under and just above the level of the liquid with rotation, avoiding spillage from the screen, at a rate of approximately 60 to 100 cpm. It is important that the material be thoroughly sieved in each container. After the bulk of the aliquot has passed through the sieve (usually about 2 min) repeat the procedure in the second container for about 2 min. When it is evident that practically none of the residue on the sieve is passing through it, repeat the procedure in the acetone container for about 2 to 3 min until nothing more passes through the sieve. Rinse the sides of the sieve with a small stream of acetone, and collect the residue at one side of the sieve. Transfer the residue to a dried and weighed aluminum cup or small evaporating dish with a small stream of acetone, using not less than 15 to 25 mL. Place the aluminum cup or dish in an oven, evaporate to dryness at 105 to 100°C, and continue heating at this temperature for 15 min. (**Warning—**Do not close the door of the oven while evaporating the acetone, as there is danger of explosion or flash of acetone vapor.)

7.3.2 *Aluminum Paste*—Determine the percent of coarse particles in accordance with the procedure described in 7.3.1 using a 45-µm (No. 325) sieve for Classes A, B, and C paste and in addition a 150-µm (No. 100) sieve for Class C paste.

7.3.3 *Calculation*—Calculate the weight percent of the residue retained on the sieve *R* from the increase in weight as follows:

$$R = [(A/W) \times CF] \times 100$$

where:

A = weight of dried residue, g,
CF = correction factor (1.0 for NIST sieves), and
W = aliquot weight, g.

NOTE 1—A correction factor shall be applied in the case of sieves used in the test that have not been certified by the National Institute of Standards and Technology. Sieves having a correction factor above 1.2 or below 0.8 should not be used. The correction factor may be calculated as follows: Determine the coarse particles of an aliquot on a master sieve certified by the National Institute of Standards and Technology following the procedure described in 7.3.1. Sieve the same aliquot on the unknown sieve by the same procedure. Calculate the correction factor, *CF*, as follows:

$$CF = X/Y$$

where:

X = weight of residue on the NIST sieve, g, and
Y = weight of residue on the unknown screen, g.

7.4 *Procedure B*—Test in accordance with Test Methods D185 to determine conformance of flaked aluminum powders and pastes to Specification D235.

8. Easily Extracted Fatty and Oily Matter

8.1 Easily extracted fatty and oily matter is determined by the following arbitrary test method. Details of the test procedure *shall* be strictly followed.

8.2 *Significance and Use*—This test method determines the percent of easily extracted fatty and oily matter in aluminum pigments in accordance with Specification D962.

8.3 *Procedure:*

8.3.1 *Aluminum Powder*—Weigh accurately (to within 0.01 g) approximately 2 g of the aliquot. Transfer to a 200-mL volumetric flask and add about 50 mL of redistilled acetone conforming to Specification D329. Heat the mixture to boiling while occasionally agitating. Cool, fill to the 200-mL mark with acetone, and mix. Allow the mixture to settle. Draw off approximately one half of the supernatant liquid and filter through dry paper. Discard the first 10 mL of the filtrate. Transfer 100 mL of the clear filtrate to a weighed dish. Evaporate the acetone at a temperature not above 75°C. Heat the dish for 15 min in an oven at 105 to 110°C. Cool and weigh the amount of fatty oily matter.

8.3.2 *Aluminum Paste*—Determine the percent of easily extracted fatty and oily matter in aluminum paste in accordance with the procedure described in 8.3.1.

8.4 *Calculation*—Calculate the weight percent of fatty and oily matter *F* as follows:

$$F = [2 \times (A/W)] \times 100$$

where:

A = weight of residue in 100 mL of acetone, g, and
W = weight of aliquot, g.

9. Nonvolatile Matter

9.1 Determine the percent of nonvolatile matter in aluminum paste consisting of aluminum, fatty acids, additives, and their inter-reaction products.

9.2 *Significance and Use*—This test method determines the combined percent of aluminum and other nonvolatile matter, such as fatty acids and their inter-reaction products, in aluminum pigments and their conformance with Specification D962.

9.3 *Procedure—Aluminum Paste and Powder*—Weigh into an evaporating dish 5 g of paste or powder to within 0.01 g. Evaporate in an oven at a temperature of 105 to 110°C for 3 h. Cool to room temperature and weigh.

9.4 *Calculation*—Calculate the weight percent of nonvolatile matter *NV* as follows:

$$NV = A/W \times 100$$

where:

A = weight of residue, g, and
W = weight of aliquot, g.

10. Precision and Bias

10.1 *Precision and Bias*—Determine in accordance with Practices D3980 and E691.

11. Keywords

11.1 aluminum powders and pastes; flaked powders; leafing properties