



Designation: D 480 – 88 (Reapproved 2003)

Standard Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes¹

This standard is issued under the fixed designation D 480; 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 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 non-leafing 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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 7.3.1.

2. Referenced Documents

2.1 ASTM Standards:

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Drycleaning Solvent)³

D 329 Specification for Acetone³

D 962 Specification for Aluminum Powder and Paste Pigments for Paints²

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials⁴

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

E 34 Test Methods for Chemical Analysis of Aluminum and Aluminum Alloys⁶

E 607 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique, Nitrogen Atmosphere⁷

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁸

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 **D 962**, 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 **E 34** and **E 607** or by atomic absorption.

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

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 *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.04.

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

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

⁶ *Annual Book of ASTM Standards*, Vol 03.05.

⁷ *Annual Book of ASTM Standards*, Vol 03.06.

⁸ *Annual Book of ASTM Standards*, Vol 14.02.

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 D 962.

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 D 235 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	D 962, Type 1	A	1.0
		B	1.0
		C	2.0
Aluminum paste	D 962, 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 D 962.

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 D 235, and the acetone shall conform to Specification D 329. 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 E 11, 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