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An American National Standard

Standard Test Methods for Magnet-Wire Enamels[1]

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1. Scope

1.1 These test methods cover testing liquid enamel coatings used to produce film-insulated magnet wire.

1.2 The values stated in either lbs/gal or SI units are to be regarded separately as standard.

1.3 The test methods appear as follows:

	Sections
Density	5-9
Determined Solids	15-21
Effective Solids	30-36
Flash Point	10-14
Infrared Analysis	41-46
Stack Loss	22-29
Viscosity	37-40

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 29 Test Methods for Sampling and Testing Lac Resins[2]

D 56 Test Method for Flash Point by Tag Closed Tester[3,4]

D 476 Specification for Titanium Dioxide Pigments[2]

D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[3]

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products[5]

D 1638 Methods of Testing Urethane Foam Isocyanate Raw Materials[6]

D 5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation[7]

E 131 Terminology Relating to Molecular Spectroscopy[8]

E 168 Practices for General Techniques of Infrared Quantitative Analysis[8]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method [9]

3. Significance and Use

3.1 These tests are useful for specification and control purposes during the manufacture, purchase, and use of the magnet-wire enamels, and for determining uniformity of batches.

4. Sampling

4.1 Take a representative sample of liquid enamel and store for future testing. Store the sample at room temperature in a tightly sealed, nearly full container, unless otherwise specified. Use a container that is inert and impermeable to the wire enamel. These precautions avoid either the escape of solvent or reaction with the container and atmosphere. Glass and some metals are suitable materials. Copper, iron, and aluminum are unsatisfactory. After removing test specimens, use care to restore these storage conditions.

DENSITY

5. Scope

5.1 This test method covers the determination of the density of magnet-wire enamel in terms of specific gravity or weight per gallon.

6. Significance and Use

6.1 Density is useful for specification and control purposes during the manufacture and use of magnet-wire enamel.

6.2 The preferred method is Procedure A, hydrometer method.

[1] These test methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.10 on Magnet Wire Insulation.

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[2] *Annual Book of ASTM Standards,* Vol 06.03.

[3] *Annual Book of ASTM Standards,* Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 04.09.

[5] *Annual Book of ASTM Standards*, Vol 06.01.

[6] Discontinued; see 1990 *Annual Book of ASTM Standards*, Vol 06.03.

[7] *Annual Book of ASTM Standards*, Vol 10.02.

[8] *Annual Book of ASTM Standards*, Vol 14.01.

[9] *Annual Book of ASTM Standards*, Vol 14.02.

7. Procedure A—Hydrometer Method

7.1 Determine the specific gravity of the magnet-wire enamel in accordance with Test Method D 1298 at 25.0 ± 0.1°C.

7.2 If weight per gallon is required, multiply the specific gravity by the weight per gallon of distilled water at the same temperature (8.31 lb/gal at 25.0°C).

8. Procedure B—Weight per Gallon Method

8.1 Determine the weight per gallon of the magnet-wire enamel in accordance with Test Method D 1475 at 25.0 ± 0.1°C.

9. Report

9.1 Report the following information:

9.1.1 Identification of the magnet-wire enamel, and

9.1.2 When using Procedure A, report the specific gravity to the third decimal place, or

9.1.3 When using Procedure B, report the weight per gallon.

FLASH POINT

10. Scope

10.1 This test method covers the determination of the flash point of magnet-wire enamel.

11. Terminology

11.1 *Definitions of Terms Specific to This Standard:*

11.1.1 *flash point, of magnet-wire enamel*, *n*— the lowest temperature at which magnet-wire enamel gives off flammable vapor in sufficient quantity to ignite in air on application of a flame under specified conditions.

12. Significance and Use

12.1 The flash point reveals the upper temperature limit at which a magnet-wire enamel may be stored or used without presenting a fire hazard.

13. Procedure

13.1 Determine the flash point in accordance with Test Method D 56.

14. Report

14.1 Report the following information:

14.1.1 Identification of magnet-wire enamel, and

14.1.2 Flash point, degrees Celsius or Fahrenheit, preferably in degrees Fahrenheit.

DETERMINED SOLIDS

15. Scope

15.1 This test method covers the determination of a particular measured value for the solids content in a magnet-wire enamel.

16. Terminology

16.1 *Definitions of Terms Specific to This Standard:*

16.1.1 *determined solids, of magnet-wire enamel*, *n*—the portion of a magnet-wire enamel which is not volatilized when exposed to specified conditions.

17. Significance and Use

17.1 Determined solids is one of the critical factors in a magnet-wire enamel that affects film build on a conductor.

17.2 The determined solids is also useful for control purposes during the manufacture and use of magnet-wire enamel and in determining uniformity of batches.

17.3 The Effective Solids Procedure (see 34.1 and 35.1) requires the Determined Solids value as an input.

18. Apparatus

18.1 *Forced-Convection Oven*, capable of maintaining 200 ± 2°C at the specified specimen location. Refer to Specification D 5423 for a representative oven.

18.2 *Weighing Dishes*, aluminum, approximately 2 in. (51 mm) in diameter, and ⅝ in. (16 mm) height.

18.3 *Analytical Balance*, capable of weighing to ±0.1 mg.

19. Procedure

19.1 Preheat dishes to remove oil. Five minutes at 200°C is adequate.

19.2 Test not less than two specimens.

19.3 Place a 2.0 g specimen (±0.1 mg) into a tared aluminum dish and weigh immediately.

19.4 The weighed specimen must thoroughly cover the entire bottom surface of the weighing dish. Accomplish this by warming the more viscous materials.

19.5 Place the dish and its contents in a 200 ± 2°C forced-convection oven for a predetermined period of time.

19.6 Remove the dish from the oven and cool to room temperature in a desiccator.

19.7 Weigh the dish and its contents (±0.1 mg).

20. Report

20.1 Report the following information:

20.1.1 Ratio of the weight of residue to that of the specimen, expressed as a percentage, as the determined solids content, *S*, calculated as follows:

$$S = (\text{Weight of residue/weight of specimen}) \times 100 \quad (1)$$

20.1.2 Number of tests and individual values,

20.1.3 Average determined solids of all tests made, and

20.1.4 Identification of the magnet-wire enamel.

21. Precision and Bias

21.1 *Precision*:

21.1.1 The results of all measurements on the sample should agree within ±0.5 %.

21.2 *Bias*:

21.2.1 Statements of bias are not applicable in view of the unavailability of a standard reference material for this property.

STACK LOSS

22. Scope

22.1 This test method covers the determination of the stack loss of magnet-wire enamel applied to AWG No. 18 (1.02-mm) electrical conductor using an inorganic material as a reference.

NOTE 1—With other sizes of electrical conductor, expect a variation in stack loss. This is particularly true with smaller diameter wire. Expect difficulty in removing the coating from fine wire.

23. Terminology

23.1 *Definitions of Terms Specific to This Standard:*

23.1.1 *stack loss, of magnet-wire enamel, n*— that portion of the magnet-wire enamel solids which are lost during the conductor-coating process.

24. Significance and Use

24.1 The stack loss of magnet-wire enamel will affect the increase in dimensions, the amount of enamel used, the weight increase, and the economics of applying the enamel to the conductor.

24.2 The Effective Solids procedure (see 33.1.1 and 34.1), requires the Stack Loss as an input but Stack Loss is in itself not an absolute value.

25. Apparatus and Reagent

25.1 *Laboratory Magnet-Wire-Coating Equipment*, that will duplicate production application conditions and a supply of bare conductor to be used for the test.

25.2 *Laboratory Mixer or Drill Press*.

25.3 *Muffle Furnace*, capable of maintaining 600°C.

25.4 *Oven*, forced-convection, capable of maintaining 110 ± 5°C (refer to Specification D 5423).

25.5 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

25.6 *Balance*, capable of weighing 2 kg (±1 g)

25.7 *Weighing Bottles*, tall-form cylindrical, glass.

25.8 *Crucibles*, high-form, high-temperature.

25.9 *Container*, at least 2 L in capacity.

25.10 *Titanium Dioxide* (TiO_2),[10] meeting the specifications outlined in Specification D 476, Type III.

26. Procedure

26.1 Determine the optimum conditions for applying the magnet-wire enamel using laboratory coating equipment.

26.2 Condition the crucible in a muffle furnace maintained at 600 ± 20°C to a constant weight (Note 2), and immediately place it in a desiccator for storage.

NOTE 2—In practice, crucibles will come to constant weight at 600°C, if held in the muffle furnace for 14 to 16 h (overnight).

26.3 Measure the determined solids of the magnet-wire enamel in accordance with Sections 15-21, and the ash content of the solids in accordance with Test Methods D 29.

26.4 Weigh into the container 1000 ± 1 g of the magnet-wire enamel.

26.5 Weigh into the container an amount of TiO_2 equal to the weight (±1.0 g) of the solids in the 1000-g specimen of the magnet-wire enamel.

26.6 Mix the contents in the container until the TiO_2 is completely dispersed in the wire enamel.

26.7 Apply this enamel in accordance with 26.1, using the same conditions and obtaining the same increase in build. Within 2 h of applying the enamel to the conductor, completely stir the enamel to ensure dispersion.

26.8 Remove this coating from the wire by snapping and twisting the wire or by other suitable means. Place the removed coating in a weighing bottle. For the coatings that are difficult to remove, try chilling the wire before snapping. In all cases, take care to prevent including any of the metal conductor.

26.9 To remove moisture, place the weighing bottle containing the coating in a 110°C forced-convention oven for 60 ± 2 min.

26.10 Remove the weighing bottle and contents from the oven and allow it to cool to room temperature in a desiccator.

26.11 Weigh two conditioned crucibles and weigh into each 0.5 to 0.6 g of the dried coating from the weighing bottle. Make all weighings to the nearest 0.1 mg.

26.12 Weigh two conditioned crucibles and weigh into each 0.5 to 0.6 g of TiO_2.

26.13 Place all four crucibles in the cold muffle furnace. Start the furnace, allowing the temperature to come to 600°C in 1 to 2 h.

26.14 Leave the crucibles in the muffle furnace at 600± 20°C until they reach a constant weight (Note 2). Remove the crucibles and allow them to cool in a desiccator to room temperature.

26.15 Weigh the crucibles.

27. Calculation

27.1 Calculate the percent stack loss, *L*, of the magnet-wire enamel as follows:

$$\text{Let } F = E[A/AB + CD] \tag{2}$$

$$\text{Let } R = 100[G - F/F]$$

$$\text{Then } L = 100 - R$$

where:

A = TiO_2 mixed with the wire enamel, g,
B = percent of TiO_2 ash, expressed as a decimal,
C = solids in the wire-enamel specimen, g,
D = percent ash of the wire-enamel solids, expressed as a decimal,
E = weight of ash in the coating specimen, g
F = corrected ash weight, g,
G = original weight of coating specimen before ashing, g, and
R = retention of coating.

28. Report

28.1 Report the following information:

28.1.1 Identification of magnet-wire enamel,

28.1.2 Determined solids content of the magnet-wire enamel,

28.1.3 Percent ash content of the magnet-wire enamel solids.

28.1.4 Average percent retention of coating to two decimal places, and

28.1.5 Average percent stack loss to two decimal places.

[10] Titanox RA-50 or Titanox 2032, available from the Titanium Pigment Corp., 111 Broadway, New York, NY 10006, has been found satisfactory for this method.

29. Precision and Bias

29.1 This test method has been in use for many years, but no statement of precision has been made and no activity is planned to develop such a statement.

EFFECTIVE SOLIDS

30. Scope

30.1 This test method covers the determination of the percentage of liquid enamel that will be retained on the metal conductor in the finished product.

31. Terminology

31.1 *Definitions of Terms Specific to This Standard:*

31.1.1 *effective solids, of magnet-wire enamel*, *n*—the percentage of the liquid enamel retained after the removal of the solvents and the additional oven bakes that simulate the stack loss that occurs during the enameling manufacturing process for magnet wire.

31.1.2 *evaporative solids, of magnet wire enamels*, *n*—the percentage of liquid enamel that will be retained after removal of the solvents according to step 34.1of this test procedure.

31.1.3 *simulated stack loss*, *n*—the percentage change in evaporative solids after additional lab oven heat exposures as described in step 34.2 of this procedure.

32. Significance and Use

32.1 In determining the cost of a magnet-wire enamel, only that portion of the enamel that is retained on the conductor is of value.

33. Procedure

33.1 *Evaporative Solids*:

33.1.1 Test not less than two specimens.

33.1.2 Preheat the aluminum weighing pans to remove oil. Five min at 200 ± 2°C is adequate.

33.1.3 Remove the pans from the oven and cool to room temperature in a desiccator.

33.1.4 Measure the tare weight (*p*) of the dried aluminum pan to an accuracy of ± 0.1 mg.

33.1.5 Add 1 ± 0.1 grams of liquid magnet wire enamel into each tared aluminum weighing pan and measure to an accuracy of ± 0.1 mg (total of pan and contents = *I*).

33.1.6 Distribute the material evenly over the bottom of the pan.

33.1.7 Place the pan and its contents into a forced air convection laboratory oven for the time and temperature specified in Table 1.

33.1.8 Remove the pan from the oven and cool to room temperature in a desiccator.

33.1.9 Weigh the pan and its dried contents (± 0.1 mg).

33.1.10 Record these result as *W1*.

33.2 *Simulated Stack Loss*:

33.2.1 Place the pans from 33.1.9 into the additional forced air convection laboratory ovens for the times and temperatures specified in Table 2.

TABLE 1 Conditions for Determining Evaporative Solids

Magnet Wire Enamel Type	Oven Temperature (± 2°C)	Bake Time (-0, +2) Min
Polyester	200°C	30 min
Amideimide	200°C	60 min
Esterimide	200°C	30 min
Polyimide	200°C	60 min
Polyvinyl formal	200°C	30 min
Polyurethane	200°C	30 min
Polyimide	200°C	30 min

TABLE 2 Conditions to Simulate Stack Loss

Magnet Wire Enamel Type	Oven 1 Temperature (± 2°C)	Time in Oven 1: (-0, +2) Min	Oven 2 Temperature (± 2°C)	Time in Oven 2: (-0, +2) Min
Polyester	250°C	20 min	300°C	15 min
Amideimide	250°C	20 min	300°C	15 min
Esterimide	250°C	20 min	300°C	15 min
Polyimide	250°C	20 min	300°C	15 min
Polyvinyl formal	250°C	20 min	. . .	. . .
Polyurethane	250°C	20 min	. . .	. . .
Polyimide	250°C	15 min	. . .	. . .

NOTE 3—When running simulated stack loss, pans do not have to go directly from one oven to the other. Do not leave the pans in the oven if you are changing temperatures.

33.2.2 Remove the pans from the oven and cool to room temperature in a desiccator.

33.2.3 Weigh the pan and their dried contents (± 0.1 mg).

33.2.4 Record these results as *W2*.

34. Calculation

34.1 Calculate the evaporative solids percentage (± 0.1 %) of the magnet wire enamel as follows:

$$\%\ Evaporative\ Solids\ (EVS) = [(W1 - p) / (I - p)] \times 100 \quad (3)$$

where:
W1 = weight of pan and dried contents after evaporative solids heat exposure (33.1.10),
p = aluminum pan tare weight (33.1.4), and,
I = initial weight of the aluminum pan containing the liquid enamel (33.1.5).

34.2 Calculate the simulated stack loss percentage (± 0.1%) of the magnet wire enamel as follows:

$$\%\ Simulated\ Stack\ Loss\ (SSL) = (\ EVS - [(W2 - p) / (I - p) \times 100]\ ) / EVS \times 100 \quad (4)$$

where:
W2 = weight of pan and dried contents after all of the heat exposures (33.2.4),
p = aluminum pan tare weight (33.1.4),
I = initial weight of the aluminum pan containing the liquid enamel (33.1.5), and
EVS = percent evaporative solids.

34.3 Calculate effective solids percentage (± 0.1%) of the magnet wire enamel as follows:

$$\%\ Effective\ Solids\ (EFS) = [(W2 - p) / (I - p)] \times 100 \quad (5)$$

where:

W2 = weight of pan and dried contents after all of the heat exposures (33.2.4),
p = aluminum pan tare weight (33.1.4),
I = initial weight of the aluminum pan containing the liquid enamel (33.1.5), and
EVS = percent evaporative solids.

35. Report

35.1 Report the following information:

35.1.1 Identification of magnet-wire enamel,

35.1.2 Percent evaporative solids (*EVS*),

35.1.3 Percent simulated stack loss (*SSL*), and

35.1.4 Percent effective solids (*EFS*) to two decimal places.

36. Precision and Bias

36.1 *Precision*:

Table 3 lists the results based on a round robin test four laboratories and eight materials. Each test result was the average of three specimens. Each laboratory obtained one test result for each material. due to the limited number of laboratories involved in testing, the results were not obtained in accordance with Practice E 691. These results are provided for guidance.

36.2 *Bias*:

No information can be presented on bias for determining effective solids as no material having an accepted reference value is available.

VISCOSITY

37. Scope

37.1 This test method covers the determination of the viscosity of magnet-wire enamels at low-shear rates using a Brookfield viscometer.

38. Significance and Use

38.1 Viscosity is important in determining the type of application best suited for a magnet-wire enamel.

38.2 Viscosity is also useful for control purposes during the manufacture and use of magnet-wire enamel, and in determining the uniformity of batches.

39. Procedure

39.1 Determine the viscosity in accordance with Methods D 1638, except measure the specimen at 25 ± 0.1°C.

NOTE 4—If the Brookfield viscometer is used without the guard, it must be restandardized in a single-size container.

TABLE 3 Percent Effective Solids[A]

Material	Average	Standard Deviation
Polyester	35.53	0.62
THEIC Polyester	36.66	0.32
Polyurethane	23.73	1.62
Formvar	18.51	0.15
Polyimide	15.01	0.10
Amideimide	23.89	0.30
Esterimide	36.87	0.23
Nylon	19.18	0.33

[A] Round robin results are in percentages.

40. Report

40.1 Report the following information:

40.1.1 Identification of magnet-wire enamel,

40.1.2 Temperature of test,

40.1.3 Determined solids and solvent used, if sample was diluted,

40.1.4 Model of Brookfield viscometer,

40.1.5 Speed of rotation,

40.1.6 Spindle number, and

40.1.7 Viscosity, in centipoises.

INFRARED ANALYSIS

41. Scope

41.1 This test method covers the testing of magnet-wire enamel by the use of infrared spectroscopy.

42. Terminology

42.1 *Definitions:*

42.1.1 *absorption spectrum, n*—a plot, or other representation, of absorbance, or any function of absorbance against wavelength, or any function of wavelength.

42.2 *infrared, n*—the region of the electromagnetic spectrum from approximately 0.78 to 300 µm.

42.3 *infrared spectrometer, n*—an instrument which measures infrared spectrum.

42.3.1 DISCUSSION—For additional definitions see Terminology E 131.

43. Significance and Use

43.1 Infrared spectroscopy is useful for specification and control purposes during the manufacture and use of magnet-wire enamel. The infrared spectrum also provides information concerning the generic composition of the magnet-wire enamel. For further information see Practices E 168.

44. Procedure

44.1 Prepare magnet-wire polymers as cast films from their enamel solutions. Place a drop of enamel on the surface of an IR transparent material, generally an alkali halide crystal. Coat the drop as a thickness gradient across the crystal. This gives the option of selecting a spot on the crystal which gives the proper absorption maximum for the bands of interest.

44.2 Bake the crystal in a forced-convection oven for 30 min at 200°C.

44.3 After the crystal has cooled to ambient conditions, run a survey scan and select the proper thickness for the final scan.

45. Report

45.1 Report the following information:

45.1.1 Identification of the magnet-wire enamel, and

45.1.2 Variations in the infrared spectrum from the reference standard.

46. Precision and Bias

46.1 This test method has been in use for many years, but no statement of precision and bias has been made and no activity is planned to develop such a statement.

47. Keywords

47.1 density; determined solids; effective solids; enamels-magnet wire; evaporative solids; flash point; infrared spectroscopy; magnet wire enamels; simulated stack loss; stack loss; viscosity