Standard Test Methods for Magnet-Wire Enamels[1]

This standard is issued under the fixed designation D3288/D3288M; 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 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	6 – 10
Determined Solids	16 – 22
Effective Solids	31 – 37
Flash Point	11 – 15
Infrared Analysis	45 – 50
Stack Loss	23 – 30
Viscosity	38 – 44

1.4 There is no known IEC equivalent document.

1.5 *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]

D29 Test Methods for Sampling and Testing Lac Resins (Withdrawn 2005)[3]
D56 Test Method for Flash Point by Tag Closed Cup Tester
D476 Classification for Dry Pigmentary Titanium Dioxide Products
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D1475 Test Method For Density of Liquid Coatings, Inks, and Related Products
D1711 Terminology Relating to Electrical Insulation
D5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation
E131 Terminology Relating to Molecular Spectroscopy
E168 Practices for General Techniques of Infrared Quantitative Analysis (Withdrawn 2015)[3]
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2975 Test Method for Calibration of Concentric Cylinder Rotational Viscometers

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these test methods, refer to Terminology D1711.

3.2 *Definitions of terms specific to this standard:*

3.2.1 For definitions of terms specific to this standard, see the individual test methods.

4. Significance and Use

4.1 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.

5. Sampling

5.1 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

6. Scope

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

[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 D09.12 on Electrical Tests.

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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.

*A Summary of Changes section appears at the end of this standard

7. Significance and Use

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

7.2 The preferred method is Procedure A, hydrometer method.

8. Procedure A—Hydrometer Method

8.1 Determine the specific gravity of the magnet-wire enamel in accordance with Test Method D1298 at 25.0 ± 0.1°C.

8.2 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 ± 0.1°C).

9. Procedure B—Weight per Gallon Method

9.1 Determine the weight per gallon of the magnet-wire enamel in accordance with Test Method D1475 at 25.0 ± 0.1°C.

10. Report

10.1 Report the following information:

10.1.1 Identification of the magnet-wire enamel, and

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

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

FLASH POINT

11. Scope

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

12. Terminology

12.1 *Definitions of Terms Specific to This Test Method:*

12.2 *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.

13. Significance and Use

13.1 The flash point reveals the upper temperature limit that is permissible for storage or use of a magnet-wire enamel without presenting a fire hazard.

14. Procedure

14.1 Determine the flash point in accordance with Test Method D56.

15. Report

15.1 Report the following information:

15.1.1 Identification of magnet-wire enamel, and

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

DETERMINED SOLIDS

16. Scope

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

17. Terminology

17.1 *Definitions of Terms Specific to This Test Method:*

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

18. Significance and Use

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

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

18.3 The stack loss (see 27.3) requires the Determined Solids value as an input.

19. Apparatus

19.1 *Forced-Convection Oven,* capable of maintaining 200 ± 3°C at the specified specimen location. Refer to Specification D5423 Type II for a representative oven.

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

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

20. Procedure

20.1 Preheat dishes to remove oil. Five minutes at 200 ± 3°C is adequate.

20.2 Test a minimum of two specimens.

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

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

20.5 Place the dish and its contents in a 200 ± 3°C forced-convection oven for 2 ± 0.1 h.

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

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

21. Report

21.1 Report the following information:

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

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

21.1.2 Number of tests and individual values,

21.1.3 Average determined solids of all tests made, and

21.1.4 Identification of the magnet-wire enamel.

22. Precision and Bias

22.1 *Precision:*

22.1.1 The results of all measurements on the sample typically agree within ± 0.5 %.

22.2 *Bias:*

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

STACK LOSS

23. Scope

23.1 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.

24. Terminology

24.1 *Definitions of Terms Specific to This Test Method:*

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

25. Significance and Use

25.1 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.

26. Apparatus and Reagent

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

26.2 *Laboratory Mixer or Drill Press.*

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

26.4 *Oven,* forced-convection, capable of maintaining 110 ± 2.5°C (refer to Specification D5423 Type II).

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

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

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

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

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

26.10 *Titanium Dioxide* (TiO_2), meeting the specifications outlined in Specification D476, Type III.

27. Procedure

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

27.2 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).

27.3 Measure the determined solids of the magnet-wire enamel in accordance with Sections 16 – 22, and the ash content of the solids in accordance with Test Methods D29.

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

27.5 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.

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

27.7 Apply this enamel in accordance with 27.1, 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.

27.8 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.

27.9 To remove moisture, place the weighing bottle containing the coating in a 110 ± 2.5°C forced-convention oven for 60 ± 2 min.

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

27.11 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.

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

27.13 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.

27.14 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.

27.15 Weigh the crucibles.

28. Calculation

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

$$\text{Let } F = (EA)/(AB + CD) \tag{2}$$

$$\text{Let } R = (100/F)(G - F)$$

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

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.

29. Report

29.1 Report the following information:

29.1.1 Identification of magnet-wire enamel,

29.1.2 Determined solids content of the magnet-wire enamel,

29.1.3 Percent ash content of the magnet-wire enamel solids,

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

29.1.5 Average percent stack loss to two decimal places.

30. Precision and Bias

30.1 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

31. Scope

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

32. Terminology

32.1 *Definitions of Terms Specific to This Test Method:*

32.2 *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.

32.3 *evaporative solids, of magnet wire enamels, n*—the percentage of liquid enamel that will be retained after removal of the solvents according to step 35.1 of this test procedure.

32.4 *simulated stack loss, n*—the percentage change in evaporative solids after additional lab oven heat exposures as described in step 35.2 of this procedure.

33. Significance and Use

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

34. Procedure

34.1 *Evaporative Solids:*

34.1.1 Test a minimum of two specimens.

34.1.2 Preheat the aluminum weighing pans to remove oil. Five min at 200 ± 3°C is adequate.

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

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

34.1.5 Add 1 ± 0.1 g 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*).

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

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

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

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

34.1.10 Record these result as *W1*.

34.2 *Simulated Stack Loss:*

34.2.1 Place the pans from 34.1.9 into the additional forced air convection laboratory ovens for the times and temperatures specified in Table 2.

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.

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

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

34.2.4 Record these results as *W2*.

35. Calculation

35.1 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 (34.1.10),
p = aluminum pan tare weight (34.1.4), and,
I = initial weight of the aluminum pan containing the liquid enamel (34.1.5).

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

$$\%\ Simulated\ Stack\ Loss\ (SSL) = \quad (4)$$

$$[(\ EVS - [(W2 - p)/(I - p) \times 100]\ )/EVS] \times 100$$

where:

W2 = weight of pan and dried contents after all of the heat exposures (34.2.4),
p = aluminum pan tare weight (34.1.4),
I = initial weight of the aluminum pan containing the liquid enamel (34.1.5), and
EVS = percent evaporative solids.

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	. . .	. . .

35.3 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 (34.2.4),
p = aluminum pan tare weight (34.1.4),
I = initial weight of the aluminum pan containing the liquid enamel (34.1.5), and
EVS = percent evaporative solids.

36. Report

36.1 Report the following information:

36.1.1 Identification of magnet-wire enamel,

36.1.2 Percent evaporative solids (*EVS*),

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

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

37. Precision and Bias

37.1 *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 E691. These results are provided for guidance.

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

VISCOSITY

38. Scope

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

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.

39. Significance and Use

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

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

40. Interferences

40.1 Temperature and container size are important factors in the accurate determination of rotational viscosity; deviation from the prescribed conditions will affect the accuracy of the results.

41. Apparatus

41.1 *Rotational Viscometer*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities include:

41.1.1 A *drive motor* to apply a unidirectional displacement of 2 to 60 r/min constant to ± 0.1 %.

41.1.2 A *force sensor* to measure the torque developed by the specimen readable to better than ±0.1 %.

41.1.3 A *coupling shaft* or other means to transmit the rotational displacement from the motor to the specimen.

NOTE 4—It is helpful to have a mark on the shaft to indicate appropriate test fluid level.

41.1.4 A *spindle, rotational element, geometry or tool* to fix the specimen between the drive shaft and a stationary position, with a right circular cylindrical shape (see Fig. 1) with a diameter of 18.8 mm and length of 65.1 mm.

41.1.5 A *temperature sensor or measuring device* to provide an indication of the specimen temperature over the range of 23 to 27°C readable to within ± 0.01°C

41.1.6 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required are torque, rotational speed, temperature and time.

NOTE 5—Manual observation and recording of data are acceptable.

41.1.7 A stand to support, level, and adjust the height of the drive motor, shaft and spindle.

41.1.8 A specimen container to contain the test specimen during testing.

NOTE 6—A 600 mL low form Griffin beaker and 1 qt wide-mouth pan cane have been found suitable

41.1.9 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

41.1.9.1 *Data analysis capability* to provide viscosity, stress or other useful parameters derived from the measured signals.

41.1.9.2 A *level* to indicate the vertical plumb of the drive motor, shaft, and spindle.

41.1.9.3 A *guard* to protect the spindle from mechanical damage.

41.2 *Constant Temperature Bath* to maintain the temperature of the specimen and container at 25 ± 0.3°C

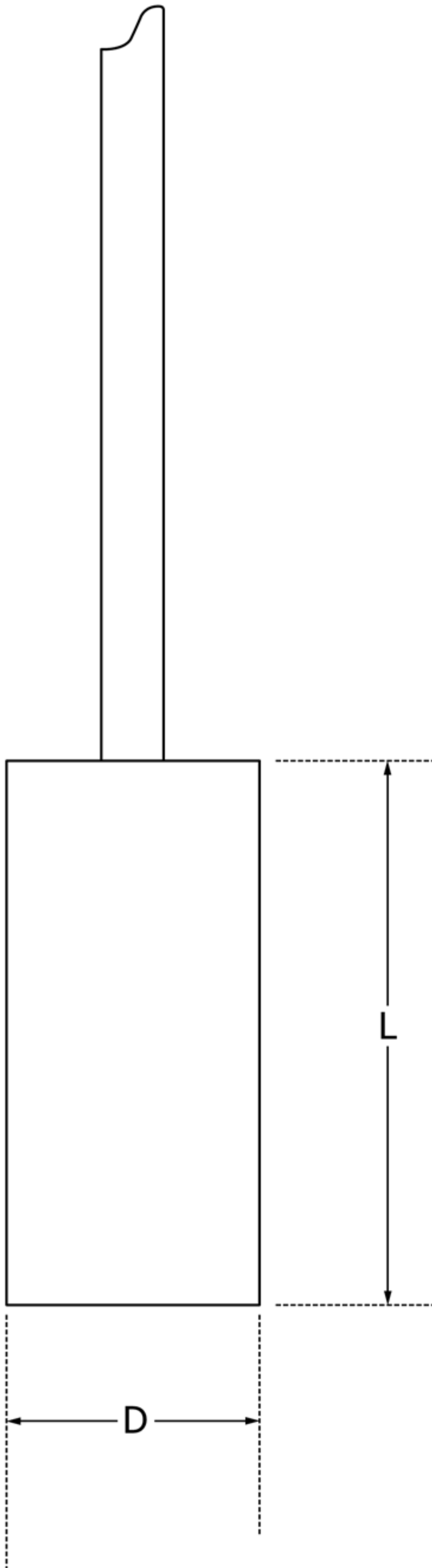

FIG. 1 Rotational Viscometer Spindle

42. Calibration

42.1 Periodical calibration of the viscometer is performed using Test Method E2975.

43. Procedure

43.1 Place sufficient sample into the container to cover the immersion mark on the viscometer spindle shaft.

43.2 Place the sample container in the constant temperature bath at 25 ± 0.3°C for a period of time sufficiently long for the test specimen to reach temperature equilibrium.

NOTE 7—Ensure that the heating medium fluid does not leak into the test specimen.

NOTE 8—Temperature equilibrium is achieved when the indicated temperature does not change for 10 min.

43.3 Immerse the viscometer spindle into the test specimen until the fluid level is the immersion mark on the shaft.

NOTE 9—Avoid air bubbles gathering under the spindle during immersion. If bubbles are observed, detach the spindle, keeping it in the test specimen, and stir until the bubbles are released then reattach the spindle.

43.4 Initiate the spindle rotation and adjust the rotational speed so that the torque read is near mid-scale.

43.5 Record the torque and rotational speed (or viscosity) after minimum of five rotations.

43.6 Determine the viscosity in mPa-s according to the procedure describe in the apparatus operations manual.

NOTE 10—1 cP = 1 mPa-s.

44. Report

44.1 Report the following information:

44.1.1 Identification of magnet-wire enamel,

44.1.2 Temperature of test,

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

44.1.4 A complete description of the rotational viscometer including supplier, apparatus model and spindle number,

44.1.5 Speed of rotation, and

44.1.6 Viscosity.

INFRARED ANALYSIS

45. Scope

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

46. Terminology

46.1 *Definitions:*

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

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

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

46.3.1 DISCUSSION—For additional definitions see Terminology E131.

47. Significance and Use

47.1 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 E168.

48. Procedure

48.1 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.

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

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

48.4 Conduct the test according to Practices E168.

49. Report

49.1 Report the following information:

49.1.1 Identification of the magnet-wire enamel, and

49.1.2 Variations in the infrared spectrum from the reference standard.

50. Precision and Bias

50.1 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.

51. Keywords

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

SUMMARY OF CHANGES

Committee D09 has identified the location of selected changes to this standard since the last issue (D3288 – 08) that may impact the use of this standard. (Approved Nov. 1, 2015.)

(1) Added experimental detail to viscosity measurement sections 40 – 43.