

Standard Test Method for Gel Time of Thermosetting Coating Powder¹

This standard is issued under the fixed designation D 4217; 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 This test method determines the length of time a thermosetting coating powder takes to gel on a polished metal surface at a specified temperature, such as 204°C (400°F). The determination of the gel time is a very simple method for the characterization and quality control of coating powders. However, the gel time determined by this method is not directly related to the time for the coating powder to cure in practical applications.

1.2 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 ISO Standards:

ISO 8130–6:1992 Coating powders—Part 6: Determination of gel time of thermosetting coating powders at a given temperature²

3. Terminology

3.1 Definitions:

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 *cure time of a coating powder*—the time required for a thermosetting coating powder to sufficiently chemically crosslink at a given temperature to provide the required coating properties.

3.1.3 *gel time of a coating powder*— the interval required at a given temperature for a coating powder to be transformed from a dry solid to a gel-like state.

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 in a continuous film by the application of heat or radiant energy.

3.1.5 *thermosetting*, *adj*—describing a material that, when heated per a minimum recommended cure condition, undergoes a chemical reaction and a permanent change to a more durable state capable of specific properties as designed for substrate protection or decoration, or both.

4. Significance and Use

4.1 This test method is useful for selecting coating powders that gel in the desired time at the specified temperature. The method is not useful for determination of cure time.

5. Apparatus

5.1 *Hot Plate*,³ having an electrically heated metal block with a polished surface capable of being maintained at temperatures between range 130-230°C (266-466°F) to within $\pm 2^{\circ}$ C ($\pm 4^{\circ}$ F). The temperature should be controlled by means of a thermoregulator.

NOTE 1—There are expected differences in results between this method and ISO 8130-6 which requires a heating block with small depressions.

5.2 Stopwatch or Timer, accurate to at least 1 s.

5.3 *Stirrer*, of very low heat capacity and of suitable size. Wooden stirrers with dimensions of 2 mm by 6 mm by 130 mm or $(\frac{1}{16}$ in. by $\frac{1}{4}$ in. by 5 in.) have been found suitable.

5.4 Surface Contact Thermocouple, suitable for use at $150-250^{\circ}C$ (300-480°F) and reading no greater than 1°C (2°F).

5.5 Measuring Spoon, of 1.25 cc (1/4 tsp) capacity.

5.6 *Scraper*, made of material softer than that of the heating block, for removing the test material from the heating block without scratching its surface.

6. Selection of Specimens

6.1 Obtain a representative sample of the coating powder.

6.2 Store the sample of coating powder in accordance with the manufacturer's recommendations, after sampling and prior to testing.

6.3 A specimen shall consist of approximately 1.25 ml ($^{1}/_{4}$ tsp) of coating powder.

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.51 on Powder Coatings.

Current edition approved July 10, 2002. Published September 2002. Originally published as D 4217 - 82. Last previous edition $D 4217 - 91 (1995)^{\epsilon_1}$.

² Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

³ Model SS-200 hot plate, available from Thermo-Electric Co., 455 Route 30, Imperial, PA 15126, or equivalent, has been found suitable.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

7. Calibration

7.1 Place the hot plate in a draft-free location or use a three-sided draft shield constructed to eliminate drafts.

7.2 Allow a minimum of 30 min for the hot plate to stabilize after reaching the temperature at which calibration is to be made.

7.3 Calibrate the hot plate with the surface contact thermocouple to $\pm 2^{\circ}$ C ($\pm 4^{\circ}$ F). The temperature of the hot plate will likely vary slightly over the hot plate surface. Thus the calibration is only valid for the immediate location tested. Gel times should only be measured at the location that was calibrated.

8. Procedure

8.1 Carry out the determination in duplicate.

8.2 Place the hot plate in a draft-free location or use a three-sided draft shield constructed to eliminate drafts.

8.3 Allow a minimum of 30 min for the hot plate to stabilize after reaching the temperature at which the measurement is to be made.

8.4 Using the measuring spoon, transfer $1.25 \text{ ml} (\frac{1}{4} \text{ tsp})$ of the material under test onto the calibrated area of the hot plate.

8.5 As soon as the powder hits the plate, start the stop watch and begin stirring.

8.6 Stir the molten material in small circular movements with the stirrer. When thickening starts, maintain the overall stirring action, and periodically lift the stirrer approximately 25 to 50 mm (1 to 2 in.) above the molten material to produce a filament of molten material. When filaments break and can no longer be drawn from the then gelled material, stop the timer and record the time to the nearest second. This is the gel time.

8.7 Clean the gelled material from the surface of the hot plate with the scraper. If the plate surface becomes pitted or scratched, polish it smooth or replace it.

8.8 Repeat the determination with a fresh sample. If the two results differ by less than 5 % of the lower value, calculate and report the arithmetic mean. If the difference between the two results exceeds 5 %, carry out a third determination and calculate and report the arithmetic mean of all three results to the nearest second. If the difference between the result of the third determination and those of the other two determinations is also greater than 5 %, state this and the individual results in the test report.

9. Report

9.1 Report the following information:

9.1.1 All details necessary to identify the product tested,

9.1.2 A reference to this standard,

9.1.3 The test temperature,

9.1.4 The amount of powder used if other than the default amount,

9.1.5 The result of the test as indicated in section 8.8,

9.1.6 Any deviation from the test method specified, and

9.1.7 The date of the test.

10. Precision and Bias

10.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method D 4217 for measuring gel time because adequate data has not been established. No activity is planned to develop such data.

10.2 *Bias*—This test method has no bias because the value for gel time is defined solely in terms of this test method.

11. Keywords

11.1 coating powder; gel time of coating powder; hot plate; powder coating; thermosetting

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue $(D 4217 - 91 (1995)^{\epsilon_1})$ that may impact the use of this standard. Minor changes to:

(1) Scope clarified and expanded in Section 1.

(2) Reference to ISO 8130-6 added in Section 2.

(3) Coating powder, cure time, powder coating, and thermosetting definitions added in Section 3.

(4) Apparatus details clarified and expanded in Section 5.

- (5) Sample details simplified in Section 6.
- (6) Calibration clarified and expanded in Section 7.
- (7) Procedure clarified and expanded in Section 8.

(8) Report clarified and expanded in Section 9.

(9) Precision statement revised.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).