

Designation: D4217 - 07 (Reapproved 2013)

# Standard Test Method for Gel Time of Thermosetting Coating Powder<sup>1</sup>

This standard is issued under the fixed designation D4217; 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 ( $\epsilon$ ) 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 temperature<sup>2</sup>

#### 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.
- <sup>1</sup> 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 June 1, 2013. Published July 2013. Originally approved in 1982. Last previous edition approved in 2007 as D4217 07. DOI: 10.1520/D4217-07R13
- <sup>2</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

- 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,<sup>3</sup> having an electrically heated metal block with a polished surface capable of being maintained at temperatures between range 130 to 230°C (266 to 466°F) to within  $\pm 2$ °C ( $\pm 4$ °F). The temperature should be controlled by means of a thermoregulator.

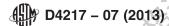
Note 1—There are expected differences in results between this method and ISO 8130–6 which requires a heating block with small depressions.

- 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 to 250°C (300 to 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.

<sup>&</sup>lt;sup>3</sup>The sole source of supply of the Model SS-200 hot plate known to the committee at this time is Thermo-Electric Co., 455 Route 30, Imperial, PA 15126. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.



- 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.

#### 7. Procedure

- 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 Verify the hot plate temperature 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 verification is only valid for the immediate location tested. Gel times should only be measured at the location that was verified.
  - 7.4 Carry out the gel time determination in duplicate.
- 7.5 Using the measuring spoon, transfer 1.25 ml (1/4 tsp) of the material under test onto the verified area of the hot plate.
- 7.6 As soon as the powder hits the plate, start the stop watch and begin stirring.
- 7.7 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.
- 7.8 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.

7.9 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.

#### 8. Report

- 8.1 Report the following information:
- 8.1.1 All details necessary to identify the product tested,
- 8.1.2 A reference to this standard,
- 8.1.3 The test temperature,
- 8.1.4 The amount of powder used if other than the default amount,
  - 8.1.5 The result of the test as indicated in section 7.9,
  - 8.1.6 Any deviation from the test method specified, and
  - 8.1.7 The date of the test.

## 9. Precision and Bias

- 9.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method D4217 for measuring gel time because adequate data has not been established. No activity is planned to develop such data.
- 9.2 *Bias*—This test method has no bias because the value for gel time is defined solely in terms of this test method.

#### 10. Keywords

10.1 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 (D4217 - 02) that may impact the use of this standard. (Approved June 1, 2007.)

(1) Section 7 and 8 combined for clarity.

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