Designation: D 2471 – 99

An American National Standard

Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins[1]

This standard is issued under the fixed designation D 2471; 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 covers the determination of the time from the initial mixing of the reactants of a thermosetting plastic composition to the time when solidification commences, under conditions approximating the conditions of use. This test method also provides a means for measuring the maximum temperature reached by a reacting thermosetting plastic composition, as well as the time from initial mixing to the time when this peak exothermic temperature is reached. This test method is limited to reacting mixtures exhibiting gel times greater than 5 min.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with this test method.

1.3 *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.*

1.4 This test method applies to adhesives, caulks, sealants, encapsulating and potting compounds, and similar materials, as described in Table 1.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

E 1 Specification for ASTM Thermometers[3]

3. Termonology

3.1 *Definitions:*

3.1.1 *General*—Definitions of plastics terms used in this test method are in accordance with Terminology D 883.

4. Significance and Use

4.1 Since the gel time and the peak exothermic temperature of a reacting thermosetting plastic composition vary with the volume of material mixed at one time, it is essential that the volume be specified in any determination. By selection of an appropriate volume, gel time and peak exothermic data may be obtained in sufficiently precise and reproducible form or application evaluation, quality control, and material characterization of a thermosetting plastic composition. For most meaningful results, the cross sectional area of the material being examined, as well as other conditions of testing, should approximate as closely as possible the conditions of use of the material.

4.2 This test method is operator-dependent since it is simple to perform. It is of value for determining conditions required to produce an end product.

5. Apparatus

5.1 *Sample Containers*, to contain a volume of reacting thermosetting plastic in a cross sectional area representative of the conditions of application of the material. Suggested containers are the following:

5.1.1 *Aluminum Foil Dish*, approximately 7 cm (2.75 in.) in diameter and 1.4 cm (0.56 in.) deep.

5.1.2 *Paint Can*, open-top, ¼-pt, approximately 6.00 cm (2.375 in.) in diameter by 5.00 cm (2 in.) deep.

5.1.3 *Paint Can*, open-top, 1-pt, approximately 8.2 cm (3.25 in.) in diameter by 9.5 cm (3.75 in.) deep.

5.2 *Wooden Probe*—Applicator sticks approximately 0.24 cm (0.09 in.) in diameter by 15.2 cm (6.00 in.) long have been found satisfactory.

5.3 *Nonconducting Surface*, such as dry wood or corrugated casing.

5.4 *Temperature Measuring Devices*:

5.4.1 Any temperature recorder or indicator utilizing expendable thermocouples and accurate to approximately ±1 % of scale is adequate for all but the most precise characterizing tests.

NOTE 2—Previous versions of this test method have contained a paragraph detailing the possible use of a thermometer for temperature determination. Use of a thermometer is no longer recommended due to potential hazard of breakage as well as loss of the thermometer due to imbedment in the curing mass.

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.16 on Thermosetting Materials.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

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

TABLE 1 Sample Volumes Related to Application

Application	Test Volume	Working Volume
Thin-section applications, adhesives, tooling surface coats, dip coatings, laminating materials, impregnants, caulking and sealing compounds, small-volume encapsulating, and potting compounds	15 mL (0.5 fluid oz) in aluminum foil dish	60 mL (2 fluid oz)
Surface-casting systems, larger-volume encapsulating, and potting compounds	45 mL (1.5 fluid oz) in aluminum foil dish	90 mL (3 fluid oz)
Laminating materials, surface-casting systems	120 mL (4 fluid oz) in ¼-pt can	150 mL (5 fluid oz)
Mass-casting systems, potting compounds, quality control of any material normally mixed and used in this quantity	415 mL (14 fluid oz) (weighed directly into sample container) in 1-pt can	415 mL (14 fluid oz)

5.5 *Temperature-Controlled Bath*, capable of holding the temperature within ±0.1°C.

5.6 *Stop Watch*.

6. Conditioning

6.1 Place all components in suitable containers in the temperature-controlled bath at the test temperature for at least 4 h prior to being tested, or for however much longer time is needed for all parts of the sample to reach the test temperature within 0.5°C (1.0°F). Condition all containers and probes to be used in the test at the test temperature at the same time.

6.2 If experience has shown that less than 4 h conditioning is required for all parts of the sample to reach the test temperature, report the time of conditioning.

7. Procedure

7.1 When all components have reached the test temperature (Note 2), agitate each component slowly and separately with a stirring rod or mixing paddle for at least 3 min, avoiding the entrapment of air.

NOTE 3—The test temperature shall be 23.0 ± 1.0°C (73.4 ± 1.8°F), unless otherwise specified.

7.2 Combine the components in the recommended ratio to provide a convenient working quantity.

NOTE 4—For accurate measurements of quantities of components, the individual components should be weighed, using the specific gravities of the respective components to determine the weights needed to make up the required volume.

NOTE 5—Table 1 lists suggested test volumes and working volumes for materials to be used in various applications.

7.3 Start the stop watch, and mix the components thoroughly for 3 min, avoiding air entrapment by slow agitation with a stirring rod or mixing paddle. To avoid transfer of heat, do not hold the container by hand during the mixing operation. Record the start of mixing as the "starting time."

7.4 Transfer the appropriate test volume (see Note 4) of the mixed components immediately to an appropriate sample container which has been previously conditioned at the test temperature.

7.5 Place the sample container on a nonconducting surface in still air at the test temperature.

NOTE 6—If it is desirable, because of the nature of the application, to use a conducting surface of high heat capacity instead of a nonconducting surface, include a description of the surface in the report. If, because of the nature of an application, it becomes desirable to measure gel time and peak exothermic temperature in a temperature-controlled bath, the conditions used *must* be noted in the report. **Warning**—Gel times and peak exothermic temperatures observed in a temperature-controlled bath are functions of the total system, including the quantity and nature of the coolant, as well as the nature of all components inserted into the bath. These properties are *not* functions of the resincuring agent system alone.

7.6 Insert a thermocouple, or other temperature-measuring device, into the geometric center of the reacting mass, and record the observed temperature changes to the end of the test.

7.7 *Every 15 s*, probe the center surface of the reacting mass, with the applicator stick perpendicular to the material surface.

NOTE 7—Use of a mechanical gel time meter is feasible in the larger sample sizes. However, the results obtained with the various mechanical gel time meters have not been consistent with results obtained with hand probing. If a mechanical gel time meter is used, include this information in the report.

7.8 When the reacting material no longer adheres to the end of a clean probe, record the "gel time" as the elapsed time from the start of mixing.

7.9 Continue recording the time and temperature until the temperature starts to drop. Record the highest temperature reached as the "peak exothermic temperature." Record the "peak exothermic time" as the elapsed time from the start of mixing.

8. Report

8.1 Report the following information:

8.1.1 Identification of the thermosetting plastic composition being tested,

8.1.2 Time of conditioning, if less than 4 h,

8.1.3 Method of mixing (by hand or mechanical mixer),

8.1.4 Volume of sample tested,

8.1.5 Thickness of sample tested,

8.1.6 Test temperature, to the nearest 1°C (or 2°F).

8.1.7 Gel time, to the nearest 0.5 min,

8.1.8 Peak exothermic temperature, to the nearest 1°C (or 2°F), and

8.1.9 Peak exothermic time, to the nearest 1 min.

9. Precision and Bias

9.1 The operator-dependent nature of this test method does not lend itself to a meaningful development of a precision statement. In addition, the small number of laboratories using

this test method also precludes attempting to develop such a statement.

9.2 There are no standard reference materials upon which to base an estimate of bias for this test method.

10. Keywords

10.1 caulks; gel time; peak exothermic temperature; resin, thermosetting; sealants

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this test method. This section also may include descriptions of the changes or reasons for the changes, or both.

D 2471–99:

(*1*) Editorially changed scope for clarification.

(*2*) Added summary of changes section.

(*3*) Changed SI units statement.