Designation: D4016 – 14

Standard Test Method for Viscosity and Gel Time of Chemical Grouts by Rotational Viscometer (Laboratory Method)[1]

This standard is issued under the fixed designation D4016; 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 viscosity of catalyzed chemical grouts with a rotational viscometer (laboratory method), over the range from 1.0 mPa·s to 1000 mPa·s (1.0 to 1000 cP).

1.2 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.2.1 The procedures used to specify how data are collected/recorded or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose of obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.3 The values stated in SI units are the standard. Other units provided in parentheses are for the convenience of the user.

1.4 *This standard does not purport to address all of the safety problems, 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]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D6026 Practice for Using Significant Digits in Geotechnical Data

E1 Specification for ASTM Liquid-in-Glass Thermometers

E1142 Terminology Relating to Thermophysical Properties

3. Terminology

3.1 For common definitions of terms used in this standard, refer to Terminologies D653 and E1142.

4. Summary of Test Method

4.1 A rotating spindle is immersed in a container of catalyzed chemical grout. The test specimen viscosity is obtained from the drag on the spindle exerted by the test specimen, and the rotational speed of the spindle.

5. Significance and Use

5.1 This test is intended for materials that will penetrate soil voids and rock fissures. Viscosity alone is not necessarily an exact index of grout penetrability.

6. Apparatus

6.1 *Rotational Viscometer*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this method include:

6.1.1 A *drive motor* to apply a unidirectional rotational displacement to the specimen at a rate from 0.3 revolutions per minute (rev/min) to 60 rev/min constant to within 1 %.

6.1.2 A *force sensor* to measure the torque developed by the specimen.

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

6.1.4 A *geometry, spindle, tool or rotational element* to fix the specimen between the drive shaft and a stationary position with a range of 1 mPa•s to 1000 mPa•s.

NOTE 1—Each spindle typically covers a range of about 2 decades of viscosity. The spindle is selected so that the measured viscosity is between 20 and 80 % of the range of the spindle.

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

6.1.6 A *temperature sensor* to provide an indication of the specimen temperature over the range of 15°C (59°F) to 25°C (77°F) readable to within ± 0.1°C (0.2°F).

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.15 on Stabilization With Admixtures.

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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.

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

NOTE 2—Glass thermometers should not be used with silicates.

6.1.7 A *temperature bath* to provide a controlled isothermal temperature environment for the specimen applicable to the temperature range of this method.

6.1.8 A *temperature controller* capable of operating the temperature bath at an isothermal temperature over the range of 15°C (59°F) to 25°C (79°F) constant to within ± 0.5°C (± 1°F).

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

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

6.1.11 A *specimen container*, of approximately 600 mL capacity, to contain the specimen during testing.

NOTE 3—A 600-mL low form stainless steel Griffin Beaker has been found suitable for this purpose. The composition of the container must not react with the grout. Glass containers are not acceptable for silicates.

NOTE 4—Grout temperature must equal bath temperature at the start of the test. Temperature rise due to chemical reaction does not invalidate the test data.

7. Sample Preparation

7.1 The temperature of the grout components at time of testing shall be between 19.5°C and 20.5°C (68 ± 1°F). Grout temperature must equal bath temperature at the start of the test. Temperature rise due to chemical reaction does not invalidate the test data.

NOTE 5—Alternatively, three or more tests at various temperatures may be taken to define a viscosity versus temperature relationship. It is desirable to have such tests span the anticipated ground temperature.

7.2 Viscosity data should be taken on catalyzed grout solution that contains all of the components normally used for field application.

8. Procedure

8.1 Prepare the grout components separately and bring them to the required temperature.

8.2 Catalyze the grout in the 600 mL container.

8.3 Insert the spindle into the specimen up to the indicator mark on the shaft and level the viscometer.

8.4 Initiate the rotation of the spindle at the lowest speed available.

8.5 Increase the spindle rotational speed to that required to produce a reading nearest the midpoint of the torque scale. Readings between 20 and 80 % full scale are acceptable.

8.6 Record the viscosity (or torque and rotational speed) every 60 seconds.

NOTE 6—For materials whose gel time range is between 10 and 30 min, gel time should be set at about 20 min. Instrument readings of viscosity (versus time) should be taken at approximately every two minutes. For materials whose gel time range is less than 10 min, a gel time should be set between 50 and 100 % of gel time range, and readings taken at least every 2 min or, if possible, at 1-min intervals. For materials whose gel time range is over 30 min, a gel time should be selected at least 25 % above minimum, and readings taken at approximately equal time intervals to permit at least five readings prior to gelation.

NOTE 7—For materials whose normal setting time is modified by prolonged agitation (this includes many silicate and acrylamide grouts) only one reading per sample should be taken, and multiple samples used to define the viscosity-time data.

NOTE 8—For all samples whose gel time permits more than one reading, at least one shall be taken at a different spindle speed, from that originally selected. If apparent viscosities differ by more than 20 % at different spindle speeds, the grout shall be reported as non-Newtonian.

NOTE 9—For non-Newtonian fluids it is recommended that, where possible, sufficient readings be taken so that a chart of spindle speed versus "effective" viscosity may be reported.

8.7 Prepare a display of viscosity versus time. Determine the time required to reach a viscosity of 100 mPa•s. Report this value as gel time (see Fig. 1).

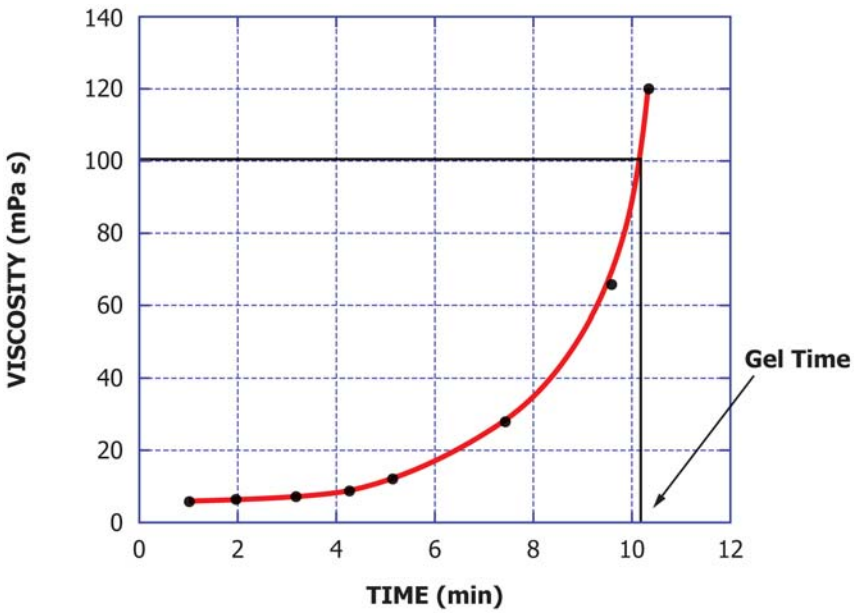

FIG. 1 Determination of Gel Point

NOTE 10—Other viscosity endpoint may be taken as the gel point but shall be reported.

9. Report

9.1 The methodology used to specify how data are recorded, as given below, is covered in 1.2.

9.2 Record as a minimum the following general information:

9.2.1 Sample identifying information, such as manufacturer, product number, lot number, etc.

9.2.2 Any special sample selection and/or preparation process.

9.3 Record as a minimum the following test specimen data:

9.3.1 The volume of the specimen, to three significant digits.

9.4 Record as a minimum the following test boundary conditions:

9.4.1 Key apparatus parameters such as spindle identification, calibration constants, and range settings.

9.5 Record as a minimum the following experimental data:

9.5.1 Torque, rotational speed, determined viscosity, all reported to three significant digits.

10. Precision

10.1 No interlaboratory study of this method has been performed to determine the within laboratory repeatability, the between laboratory reproducibility, or the bias. Participants for such an interlaboratory test are needed. Anyone wishing to participate in such an interlaboratory test may contact the Committee D18 staff manager.

11. Keywords

11.1 chemical grout; coefficient of viscosity; gel point; grouting; Newtonian fluids; non-Newtonian fluids; rotational viscometer; viscosity

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (2008) that may impact the use of this standard. (Approved June 15, 2014)

(1) Changed title.
(2) Added 1.2 and 1.3 and renumbered subsequent subsections.
(3) Added Terminology to 2.1.
(4) Added reference to Terminology E1142 to 3.1 and deleted 3.2.1 and 3.2.2.
(5) Changed 4.1.
(6) Deleted old Note 1 in 5.1 and renumbered subsequent notes.
(7) Changed 6.1 and added 6.1.1 to 6.1.11.
(8) Changed 6.2 to Note 3.
(9) Deleted 6.3 and 6.4.
(10) Changed SI units to be standard and customary units to be informational.
(11) Replaced 8.2 to 8.6.
(12) Added 8.7, Note 10, and Fig. 1.
(13) Replaced Section 9.
(14) Replaced Section 10.
(15) Added terms to Keywords (Section 11).