



Designation: ~~D6549 – 06 (Reapproved 2011)~~ **D6549 – 06 (Reapproved 2015)**

Standard Test Method for Determination of Cooling Characteristics of Quenchants by Cooling Curve Analysis with Agitation (Drayton Unit)¹

This standard is issued under the fixed designation D6549; 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 equipment and the procedure for evaluation of quenching characteristics of a quenching fluid by cooling rate determination.
- 1.2 This test method is designed to evaluate quenching fluids with agitation, using the Drayton Agitation Unit.
- 1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.
- 1.4 *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

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

2.2 *SAE Standards:*³

AMS 5665 Nickel Alloy Corrosion and Heat Resistant Bars, Forgings and Rings

2.3 *Other Standards:*⁴

Wolfson Engineering Group Specification Laboratory Tests for Assessing the Cooling Curve Characteristics of Industrial Quenching Media

2.4 *ASTM Adjuncts:*⁵

ADJD6300 D2PP, Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *aqueous polymer quenchant*—an aqueous polymer quenchant is an aqueous solution containing a water soluble polymer, typically including poly(alkylene glycol), poly(ethyl oxazoline), poly(sodium acrylate), and poly(vinyl pyrrolidone) (**1, 2, 3**).⁶ The quenchant solution also typically contains additives for corrosion and foam control, if needed. Quench severity of aqueous polymer quenchants is dependent on concentration and molecular weight of the specific polymer being evaluated, quenchant temperature, and agitation rate as shown in Fig. 1, Fig. 2, and Fig. 3, respectively.

3.1.2 *cooling curve*—the cooling curve is a graphical representation of the cooling time (t) versus temperature (T) response of the probe (see 7.3). An example is illustrated in Fig. 4.

3.1.3 *cooling curve analysis*—the process of quantifying the cooling characteristics of a quenchant based on the temperature versus time profile obtained by cooling a preheated metal probe assembly (see Fig. 4) under standard conditions (**1-7**).

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products—Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.L0.06 on Non-Lubricating Process Fluids.

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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 Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

⁴ Available from Wolfson Heat Treatment Centre, Federation House, Vyse St., Birmingham, B18 6LT, UK. http://www.sea.org.uk/whtc.

⁵ No longer available from ASTM International Headquarters.

⁶ The boldface numbers in parentheses refer to the list of references at the end of this standard.

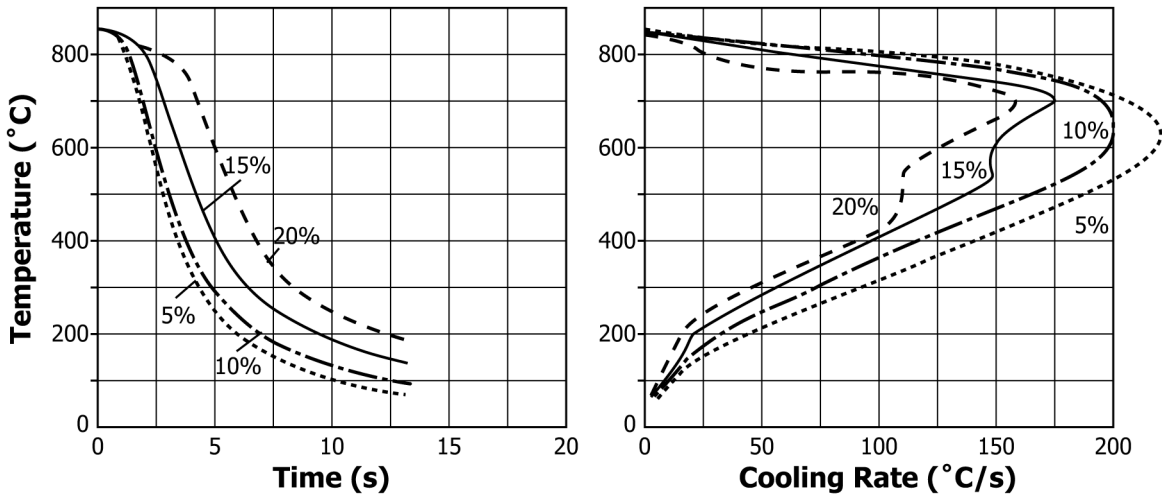


FIG. 1 Effect of Quenchant Concentration on Cooling Curve Performance for a Poly(Alkylene Glycol) Quenchant at 30°C and 0.5 m/s

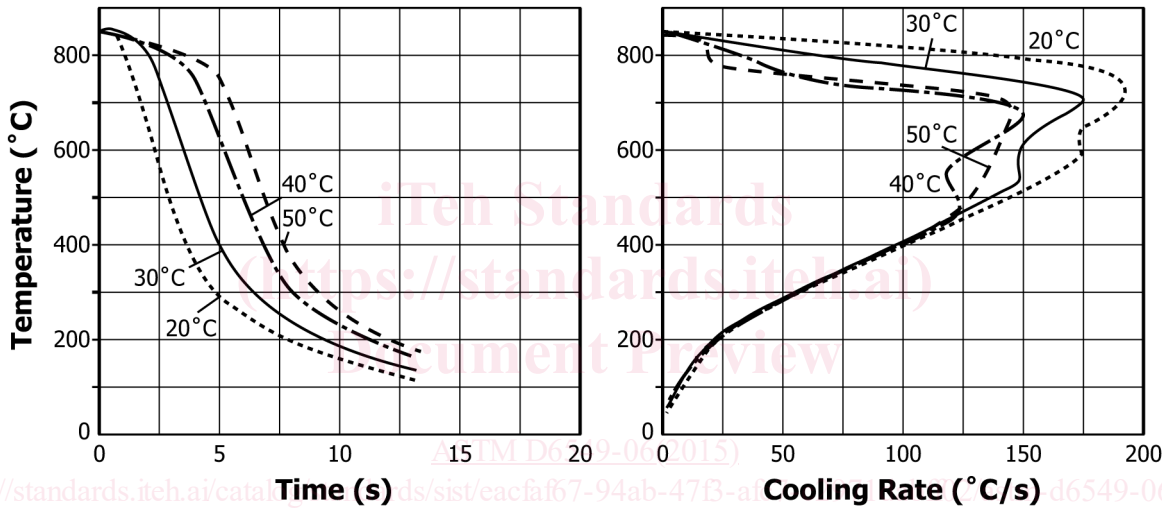


FIG. 2 Effect of Bath Temperature Variation on Cooling Curve Performance for 15 % Aqueous Solution of Poly(Alkylene Glycol) Quenchant at 0.5 m/s

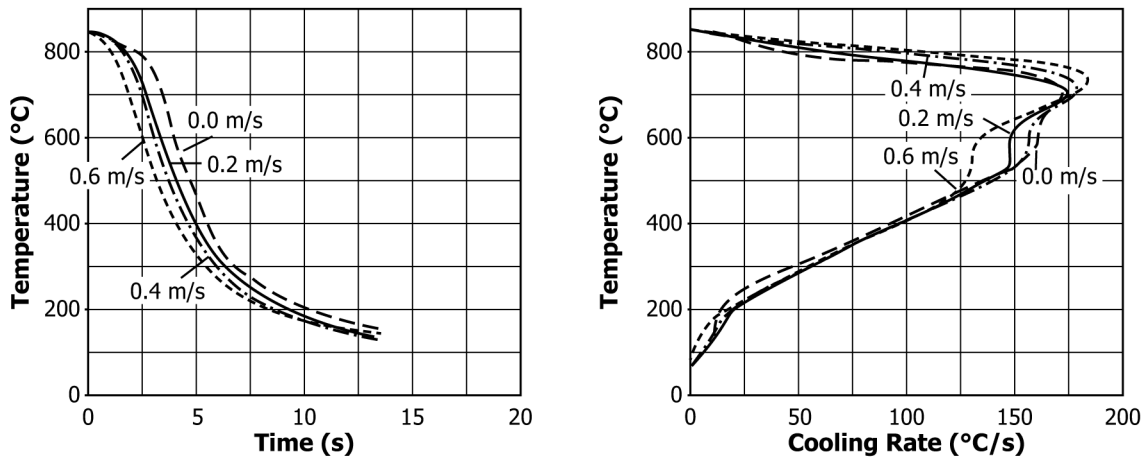
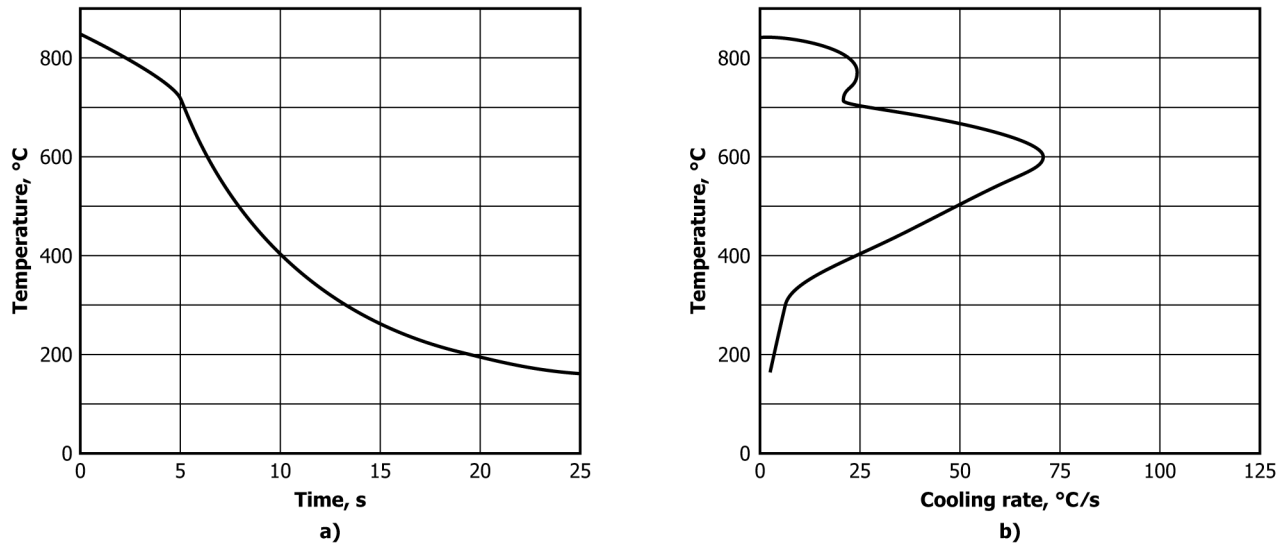


FIG. 3 Effect of Agitation Rate Variation on Cooling Curve Performance for a 15 % Aqueous Poly(Alkylene Glycol) Quenchant Solution at 30°C

3.1.4 *cooling rate curve*—the cooling rate curve is a graphical representation of first derivative of the cooling curve, the rate of temperature change (dT/dt) versus temperature. An example is illustrated in Fig. 4.



NOTE 1—a) Cooling Curve; b) Cooling Rate Curve

FIG. 4 Typical Temperature/Time and Temperature/Cooling Rate Plots for Test Probe Cooled in a Quenching Oil

3.1.5 *quenchant*—a quenching medium may be either a liquid or a gas. Gasses that are used as quenchants include air, nitrogen, argon, and hydrogen and, with the exception of air, which is used at atmospheric pressure, are used under pressure. Liquid quenchants include water, brine (most commonly dilute aqueous solutions of sodium chloride or sodium hydroxide), oil, molten salt, molten metal, and aqueous solutions of water soluble polymers. Water, brine, oil, and aqueous polymer quenchants are generally used with agitation.

3.1.6 *quench severity*—the ability of a quenching medium to extract heat from a hot metal (8).

4. Summary of Test Method

4.1 This test method determines the cooling time versus temperature of a standard nickel alloy probe assembly after it has been heated in a furnace to 850°C (1562°F) and then quenched in an aqueous polymer quenchant solution. The temperature inside the probe assembly and the cooling times are recorded at selected time intervals to establish a cooling temperature versus time curve. The resulting cooling curve (profile) may be used to evaluate quench severity (see Note 1).

NOTE 1—Where appropriate for production testing, a furnace temperature from 815°C (1500°F) to 857°C (1575°F) may be used.

5. Significance and Use

5.1 This test method provides a cooling time versus temperature curve (profile) that can be related to physical properties, such as the hardness obtainable upon quenching of a metal. The results obtained by this test method may be used as a guide in quenchant selection or as a comparison of quench severities of different quenchants, new or used.

6. Interferences

6.1 The presence of contaminants, such as oil, salt, metalworking fluids, forging lubricants, and polymer degradation, may affect cooling curve results obtained by this test method for aqueous polymer quenchants.

7. Apparatus

7.1 *Furnace*—Use a horizontal or vertical electrical resistance tube-type furnace capable of maintaining a constant minimum temperature of 850°C (1562°F) over a heated length of not less than 120 mm (4.72 in.) and a probe positioned in the center of the heating chamber. The furnace shall be capable of maintaining the probe's temperature within $\pm 2.5^{\circ}\text{C}$ (4.5°F) over the specimen length. The furnace, that is, the radiant tube heating media, shall be used with ambient atmosphere.

7.2 *Measurement System*—The temperature-time measurement system shall be a computer based data acquisition system capable of providing a permanent record of the cooling characteristics of each sample tested, producing a record of variation in the test probe assembly of temperature with respect to time and cooling rate with respect to temperature.

7.3 *Probe*—The probe shall be cylindrical, having a diameter of $12.5 \pm 0.01\text{ mm}$ ($0.492 \pm 0.0004\text{ in.}$) and a length of $60 \pm 0.25\text{ mm}$ ($2.362 \pm 0.01\text{ in.}$) with a $1.45\text{ to }1.65\text{ mm}$ ($0.057\text{ to }0.065\text{ in.}$) sheathed Type K thermocouple in its geometric

center. The probe shall be made of a nickel Alloy 600 (UNS N06600), purchased in accordance with AMS 5665, which has a nominal composition of 76.0 % Ni, 15.5 % Cr, 8.0 % Fe, 0.08 % C, and 0.25 % maximum Cu. The probe shall be attached to a support tube with a minimum length of 200 mm (7.874 in.). The thermocouple sheathing and the support tube shall be the same material as the probe (see Note 2). See Fig. 5 for other manufacturing requirements.

NOTE 2—Care shall be taken that the probe specimen is not damaged as surface irregularities will influence results of the test.

7.4 Drayton Agitation Unit:

7.4.1 Construction—The sample container, a 2000-mL stainless steel beaker that is the same as the standard container used in nonagitated cooling curve test, is modified to provide upward or axial flow of the quenchant past the probe. This flow occurs through a vertical flow tube located in the geometric center of the container. As shown in Fig. 6, the unit includes a variable speed dc drive centrifugal pump and large diameter flowmeter for direct measurement of flow velocity. It is noted that the flow tube is removable, which will provide a more turbulent flow pattern.

7.4.2 Cleaning—The agitation assembly shall be cleaned prior to use with a detergent solution. After cleaning, the assembly shall be rinsed with water at least three times to ensure that no quenchant residue or detergent solution remains.

7.4.3 Flow Velocity—The variable speed pump and flow meter allow reproducible setting of quenchant flow through the tube. The flowmeter is calibrated for water at 25°C. Flow velocity for other fluids will vary with fluid viscosity and temperature.

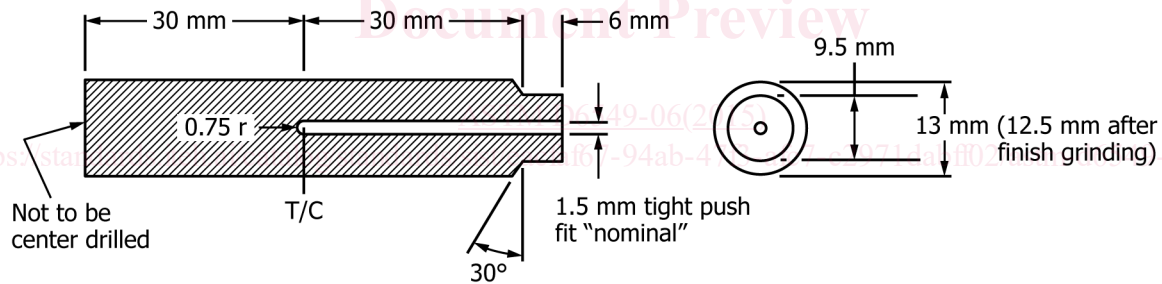
7.4.4 Fluid Volume—The resulting cooling curve is influenced by the temperature rise during the quench, which is dependent on the total fluid volume. Therefore, the cooling curve test shall be performed with a fixed volume of fluid.

7.5 Temperature Measurement—Any temperature detection device may be used that is capable of measuring quenching fluid temperature to within ±1°C (1.8°F); ±1 °C (1.8 °F).

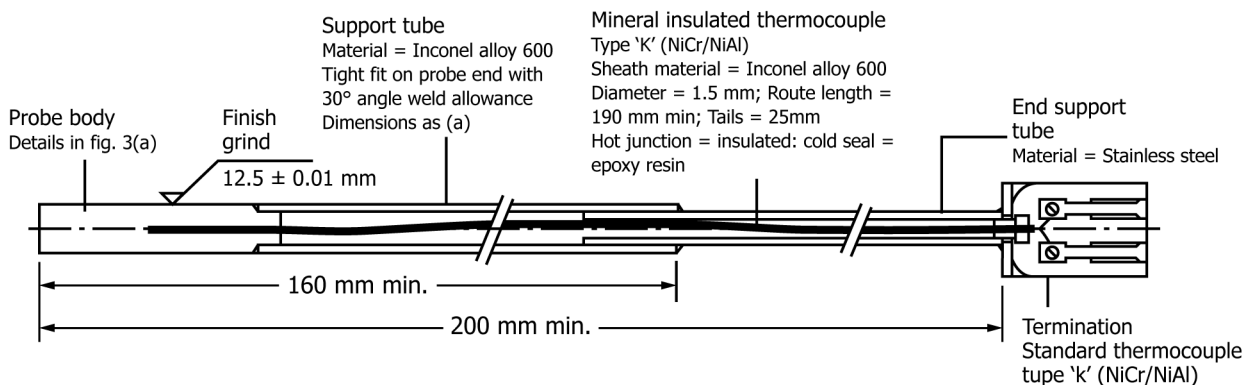
7.6 Transfer Mechanism—One of the following shall be used to transfer the heated probe from the furnace to the test fluid.

7.6.1 Mechanical Transfer—The agitation unit is positioned with the center of the test chamber coincident with the probe centerline. The transfer mechanism is set to deliver the probe to the vertical center of the sample.

7.6.2 Manual Transfer—The probe is transferred to the agitation unit through a probe guide, which is set (1) to the test chamber centerline and (2) with a preset stop that causes the probe to rest in the vertical center of the sample. The unit is illustrated further in the sketch and photograph of Fig. 6 and Fig. 7, respectively. A timer shall be used to ensure a maximum transfer time of 3.0 s.



(a) Probe details



(b) General assembly

NOTE 1—Dimensions above are nominal.

FIG. 5 Probe Details and General Probe Assembly