



Designation: D6482 – 06(Reapproved 2011)

# Standard Test Method for Determination of Cooling Characteristics of Aqueous Polymer Quenchants by Cooling Curve Analysis with Agitation (Tensi Method)<sup>1</sup>

This standard is issued under the fixed designation D6482; 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 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 Tensi agitation apparatus.

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:*<sup>2</sup>

**D6200** Test Method for Determination of Cooling Characteristics of Quench Oils by Cooling Curve Analysis

**E220** Test Method for Calibration of Thermocouples By Comparison Techniques

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

2.2 *SAE Standard:*<sup>3</sup>

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

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

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<sup>2</sup> 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.

<sup>3</sup> Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

2.3 *Japanese Industrial Standards:*<sup>4</sup>

**JIS K 2242** Heat Treating Oil

**JIS K 6753** Di-2-ethylhexyl Phthalate

2.4 *Wolfson Engineering Group:*<sup>5</sup>

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

2.5 *ASTM Adjuncts:*<sup>6</sup>

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

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *aqueous polymer quenchant*—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**).<sup>7</sup> 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 **Figs. 1-3**, respectively.

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

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. 5**) under standard conditions (**1**, **3**, **4**).

3.1.4 *cooling rate curve*—obtained by calculating the first derivative ( $dT/dt$ ) of the cooling time-temperature curve. An example is illustrated in **Fig. 4B**.

<sup>4</sup> Available from Japanese Standards Organization (JSA), 4-1-24 Akasaka Minato-Ku, Tokyo, 107-8440, Japan, http://www.jsa.or.jp.

<sup>5</sup> Wolfson Heat Treatment Centre, Federation House, Vyse St., Birmingham, B18 6LT, UK, http://www.sea.org.uk/whct.

<sup>6</sup> No longer available from ASTM International Headquarters.

<sup>7</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

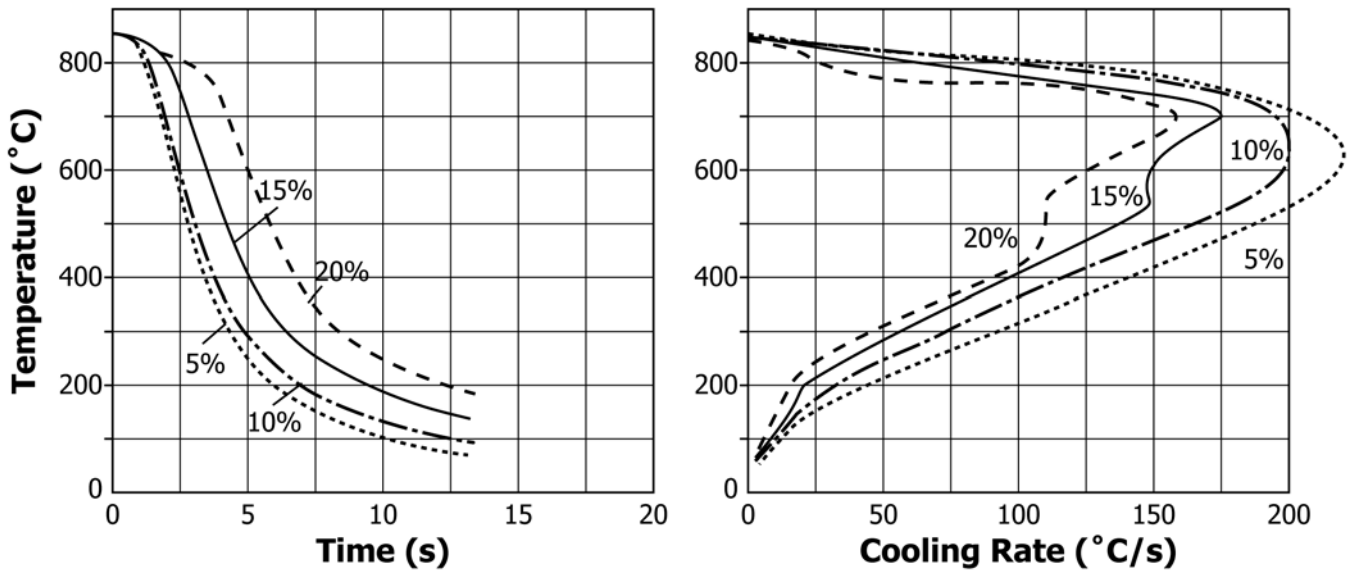


FIG. 1 Illustration of the Effect of Quenchant Concentration on Cooling Curve Performance for Poly(Alkylene Glycol) Quenchant at 30°C and 0.5 m/s

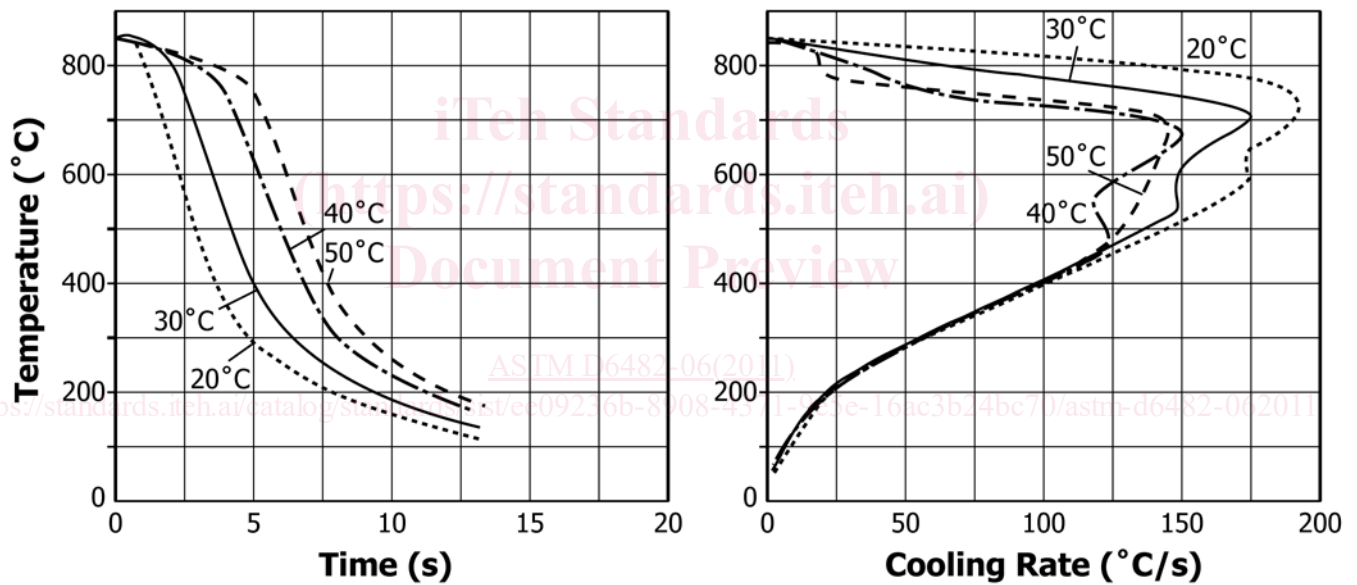


FIG. 2 Illustration of the Effect of Bath Temperature Variation on Cooling Curve Performance for 15 % Aqueous Solution of Poly (Alkylene Glycol) Quenchant at 0.5 m/s

3.1.5 *quench severity*—the ability of a quenching medium to extract heat from a hot metal (5).

3.1.6 *quenchant*—any medium, liquid or gas that may be used to mediate heat transfer during the cooling of hot metal.

#### 4. Summary of Test Method

4.1 The nickel alloy probe assembly’s cooling time versus temperature is determined after placing the assembly in a furnace and heating to 850°C (1562°F) and then quenching into 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 may be used to evaluate quench severity (see Note 1).

NOTE 1—For production testing, the furnace temperature of 815 to 857°C (1500 to 1575°F) may be used.

#### 5. Significance and Use

5.1 This test method provides a cooling time versus temperature pathway that is directly proportional 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 comparison of quench severities of different quenchants, new or used.

#### 6. Interferences

6.1 The presence of contaminants, such as oil, salt, metal-working fluids, forging lubricants, and polymer degradation,

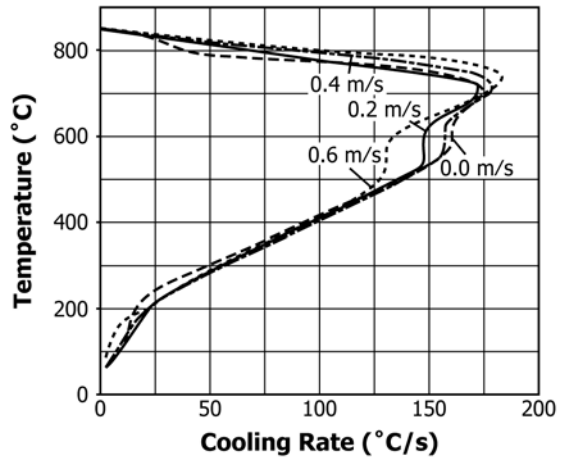
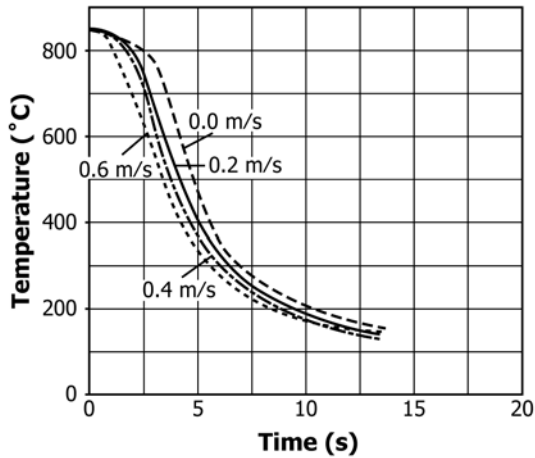
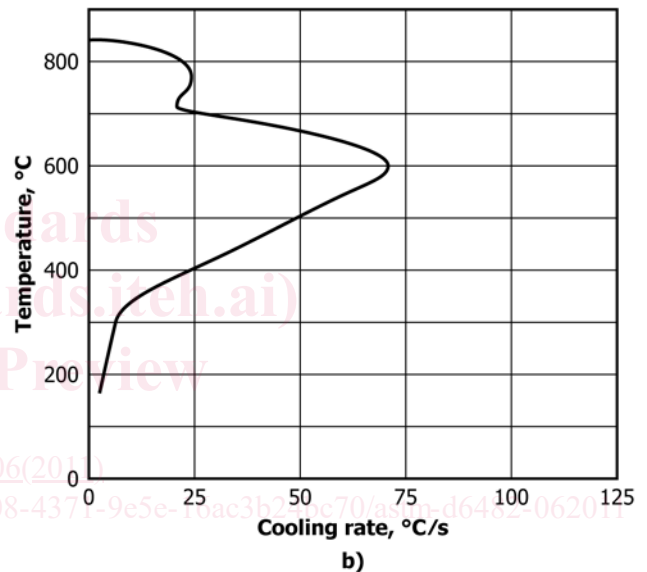
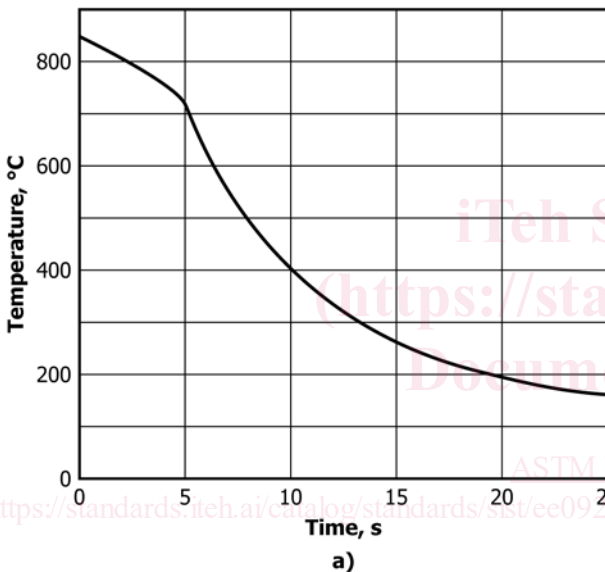


FIG. 3 Effect of Agitation Rate Variation on Cooling Curve Performance for a 15 % Aqueous Poly(Alkylene Glycol) Quenchant Solution at 30°C



A. Cooling time - temperature curve (cooling curve)

B. Cooling rate - temperature curve (cooling rate curve)

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

may affect cooling curve results obtained by this test method for aqueous polymer quenchant. Contaminants, such as water, hydraulic fluids, sludge, additive loss, and oil degradation, may similarly affect the cooling curve behavior of oil quenchant.

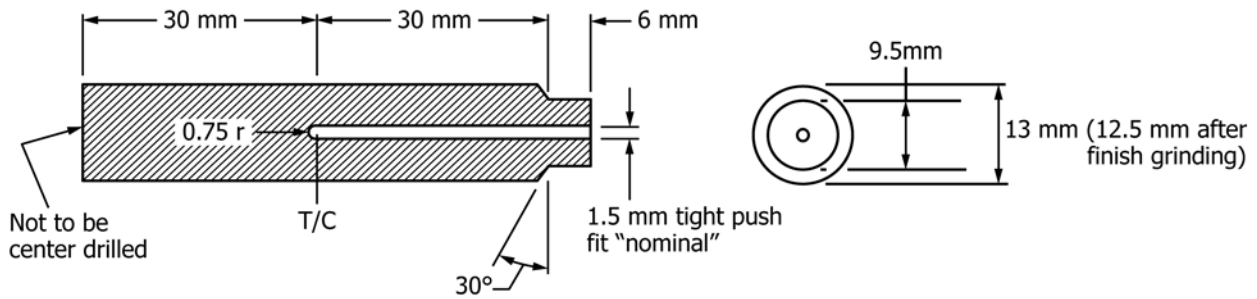
7. Apparatus

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 ±2.5°C (4.5°F) over the specimen length. The furnace, that is, the radiant tube heating media, shall be used with ambient atmosphere.

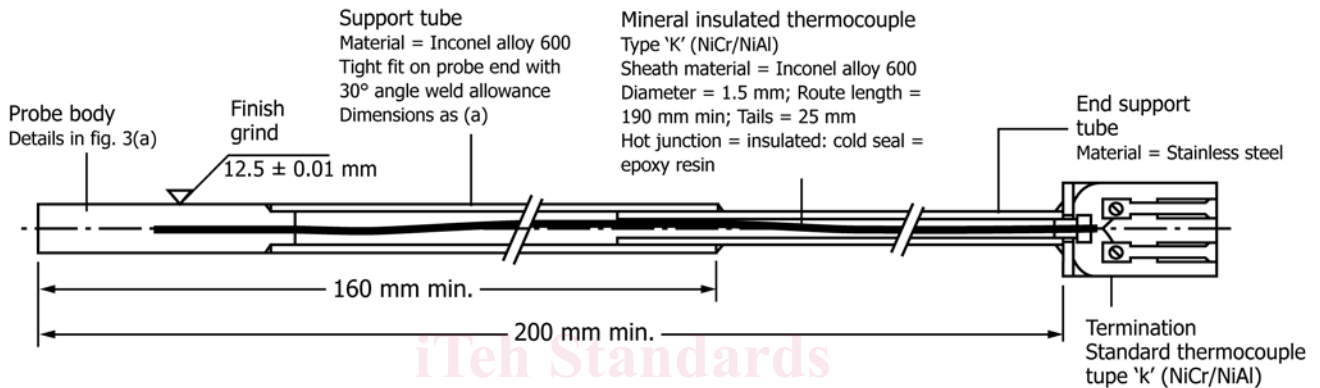
7.2 Measurement System—The temperature-time measurement system shall be a computer based data acquisition system

capable of providing a permanent record of the cooling characteristics of each oil sample tested, producing a record of variation in the test probe assembly of temperature with respect to time and of cooling rate with respect to temperature.

7.3 Probe, shall be cylindrical, having a diameter of 12.5 ± 0.01 mm (0.492 ± 0.0004 in.) and a length of 60 ± 0.25 mm (2.362 ± 0.01 in.) with a 1.45 to 1.65 mm (0.057 to 0.065 in.) sheathed type K thermocouple in its geometric center. The probe shall be made of a nickel alloy 600 (UNS N06600) purchased to SAE specification (see AMS 5665), that 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. 4 for recommended manufacturing details.



(a) Probe details



(b) General assembly

FIG. 5 Probe Details and General Probe Assembly

NOTE 2—Exercise care that the probe specimen is not damaged because surface irregularities will influence the results of the test.

7.4 Tensi Agitation Assembly:

7.4.1 Construction:

7.4.1.1 The 125 by 60 by 60-mm Tensi agitation assembly is illustrated in Fig. 6. The volume of the assembly is approximately  $1.5 \times 10^{-3} \text{ m}^3$ . This assembly may be constructed from glass or any transparent and temperature-resistant synthetic material that is chemically compatible with the quenching fluids to be evaluated. Alternatively, the agitation assembly, illustrated in Fig. 7, may be purchased assembled.<sup>8</sup>

7.4.1.2 Quenchant agitation is provided by an impeller mixer. The three-blade impeller is 50 mm in diameter with a pitch setting  $42 \text{ mm} \approx \_ \times 0.85$ . The impeller is commercially available.

NOTE 3—This procedure is recommended for aqueous polymer quenchant. Quench oils are not compatible with the poly(methyl methacrylate) materials used for construction of this apparatus.

7.4.2 Cleaning—The agitation assembly shall be cleaned prior to use with a detergent solution. After cleaning, the

<sup>8</sup> The sole source of supply of the Tensi agitation apparatus, fully assembled, known to the committee at this time is IVF, The Swedish Institute of Production Engineering Research, Argongatan 30, S-431 53 Mölndal, Sweden. 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,<sup>1</sup> which you may attend.

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—Correlation of flow velocity through the quenching chamber and impeller rotational speed for water is illustrated in Fig. 8. Flow velocity for other fluids will vary with fluid viscosity.

7.4.3.1 Impeller Speed—Fluids shall be controlled by the rotational speed of the impeller. Standard impeller speed of 1000 r/min is recommended and is obtained from a plot of revolutions per minute versus potentiometer setting as illustrated in Fig. 9 and described as impeller speed calibration: Impeller speed shall be determined using an optical tachometer. Optional tachometers operate by emitting and receiving light to and from a reflector fastened on to the impeller shaft. A typical calibration plot is illustrated in Fig. 8.

NOTE 4—The impeller velocity will depend to some extent on the viscosity of the quenchant solution. However, the variation was found to be minimal over a wide range of viscosities from water to a polymer quenchant at 30 % by volume.

7.4.3.2 Flow Direction—The correct fluid flow direction is illustrated in Fig. 6. However, if the wiring of the electrical motor is reversed, it is possible that the flow direction will also be reversed. If this occurs, which is easily detected visually, the polarity of the electrical motor is reversed by reversing the two wire leads to the motor.

7.4.4 Fluid Volume—The resulting cooling curve will be dependent on the temperature rise during the quench, which is