

Designation: D6482 – 06

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

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 (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 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:²

- 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:³
- AMS 5665 Nickel Alloy Corrosion and Heat Resistant Bars, Forgings and Rings
- 2.3 Japanese Industrial Standards:⁴

JIS K 2242 Heat Treating Oil

JIS K 6753 Di-2-ethylhexyl Phthalate

2.4 Wolfson Engineering Group:⁵

- Laboratory Tests for Assessing the Cooling Curve of Industrial Quenching Media
- 2.5 ASTM Adjuncts:
- ADJD6300 D2PP, Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products⁶

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(solium acrylate) and poly(vinyl pyrrolidone) (1,2).⁷ 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.

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

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¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.L0.06 on Nonlubricating Process Fluids.

Current edition approved Nov. 1, 2006. Published December 2006. Originally approved in 1999. Last previous edition approved in 2001 as D6482–01. DOI: 10.1520/D6482-06.

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 Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096.

⁴ Available from Japanese Standards Association, 1-24, Akasaka 4, Minato-ku, Tokyo 107 Japan.

⁵ Wolfson Engineering Group Specification, available from Wolfson Heat Treatment Centre, Aston University, Aston Triangle, Birmingham B4 7ET, England, 1980.

⁶ Available from ASTM International Headquarters. Order Adjunct No. ADJD6300.

 $^{^{7}}$ The boldface numbers in parentheses refer to the list of references at the end of this standard.

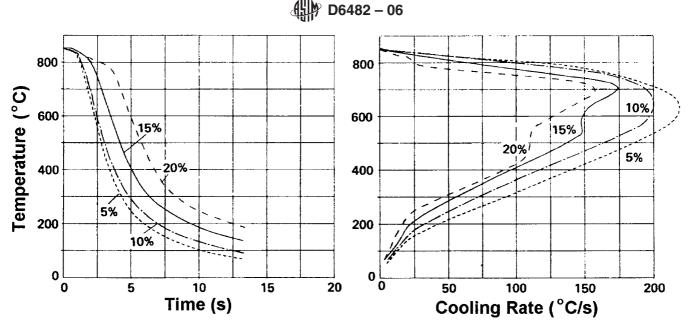


FIG. 1 Illustration of the Effect of Quenchant Concentration on Cooling Curve Performance for Poly(Alkylene Glycol) Quenchant at 30°C and 0.5 m/s

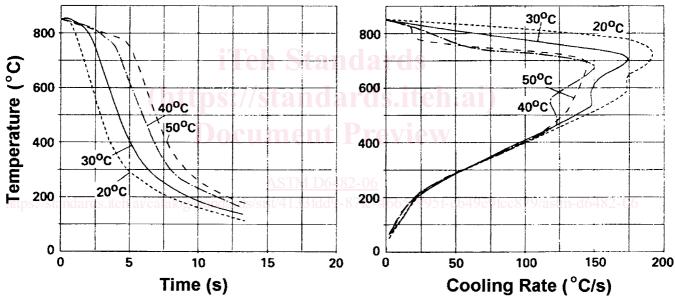


FIG. 2 Illustration of the Effect of Bath Temperature Variation on Cooling Curve Performance for 15 % Aqueous Solution of Poly (Alkylene Glycol) Quenchant at 0.5 m/s

3.1.6 *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, metalworking fluids, forging lubricants, and polymer degradation, may affect cooling curve results obtained by this test method for aqueous polymer quenchants. Contaminants, such as water,

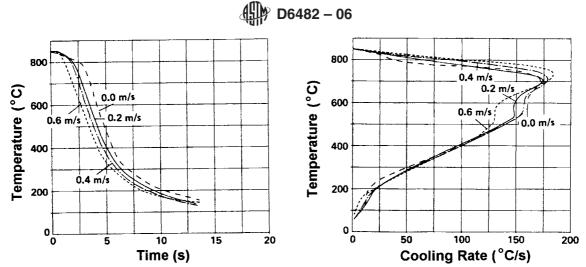


FIG. 3 Effect of Agitation Rate Variation on Cooling Curve Performance for a 15 % Aqueous Poly(Alkylene Glycol) Quenchant Solution at 30°C

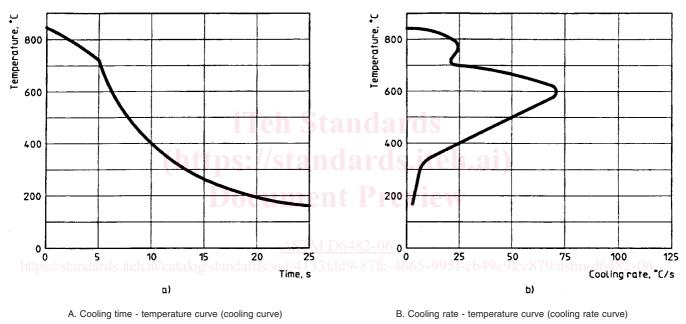


FIG. 4 Typical Temperature/Time and Temperature/Cooling Rate Plots for Test Probe Cooled in Quenching Oil

hydraulic fluids, sludge, additive loss, and oil degradation, may similarly affect the cooling curve behavior of oil 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 ± 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 $12.5 \pm 0.01 \text{ mm} (0.492 \pm 0.0004 \text{ in.})$ and a length of $60 \pm 0.25 \text{ mm} (2.362 \pm 0.01 \text{ in.})$ with a 1.45 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.

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

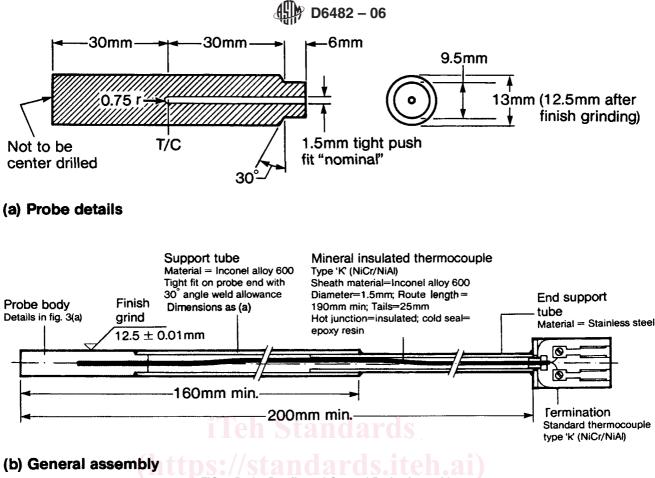


FIG. 5 Probe Details and General Probe Assembly

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 by 10^{-3} m³. 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.⁸

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 mm $\approx - \times 0.85$. The impeller is commercially available.

NOTE 3—This procedure is recommended for aqueous polymer quenchants. Quench oils are not compatible with the poly(methyl methacry-late) 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 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 dependent on the total fluid volume. Therefore, the cooling curve analysis shall be performed with the same volume of

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