



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(sodium 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**).

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.

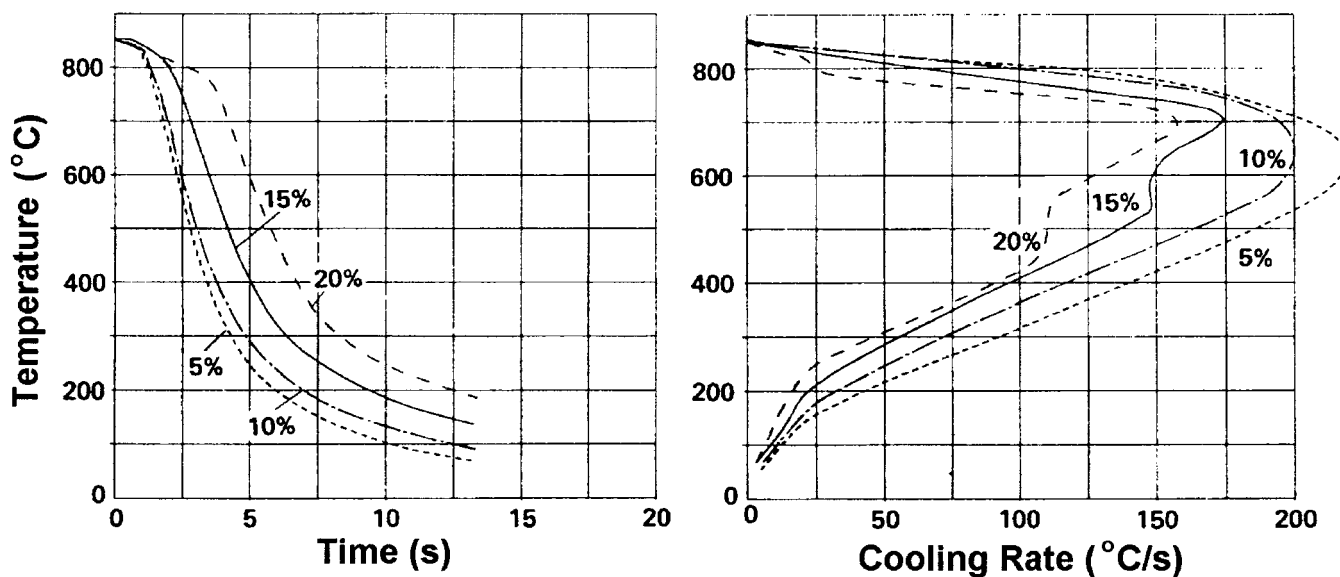


FIG. 1 Illustration of the Effect of Quenchant Concentration on Cooling Curve Performance for Poly(Alkylene Glycol) Quenchant at 30°C and 0.5 m/s

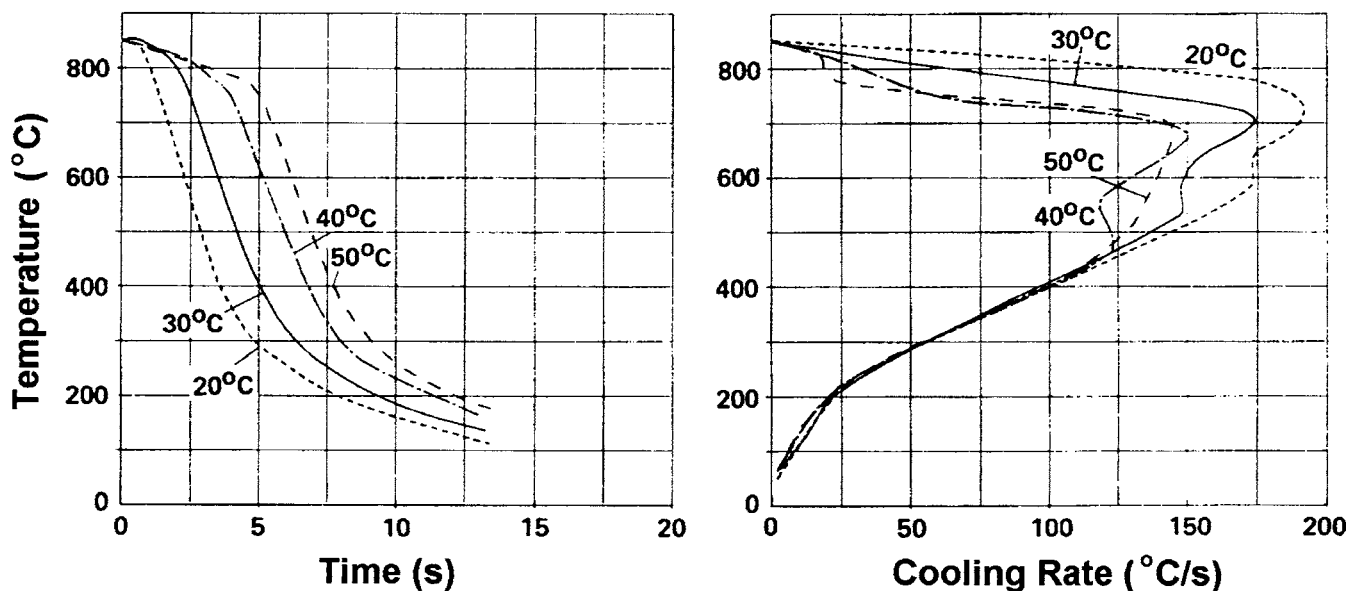


FIG. 2 Illustration of the Effect of Bath Temperature Variation on Cooling Curve Performance for 15% Aqueous Solution of Poly (Alkylene Glycol) Quenchant at 0.5 m/s

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

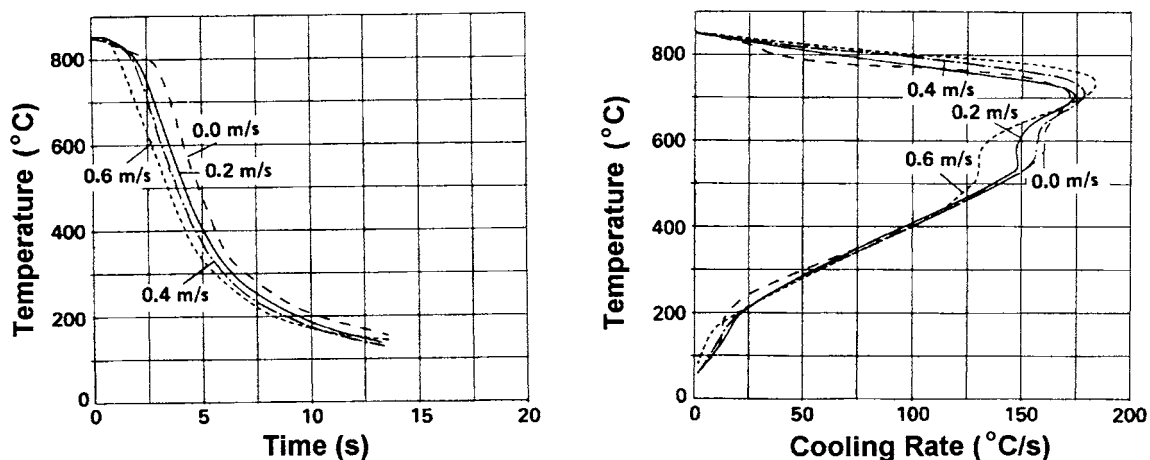
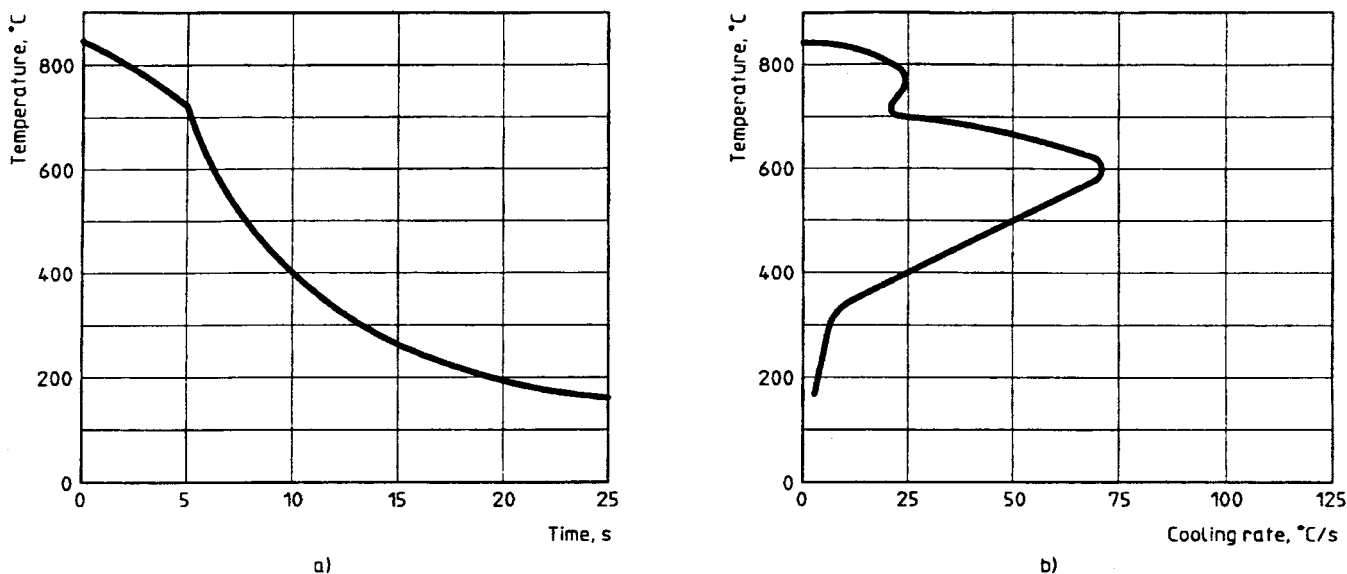


FIG. 3 Effect of Agitation Rate Variation on Cooling Curve Performance for a 15 % Aqueous Poly(Alkylene Glycol) Quenchant Solution at 30°C



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

hydraulic fluids, sludge, additive loss, and oil degradation, may similarly affect the cooling curve behavior of oil quenchants.

7. Apparatus

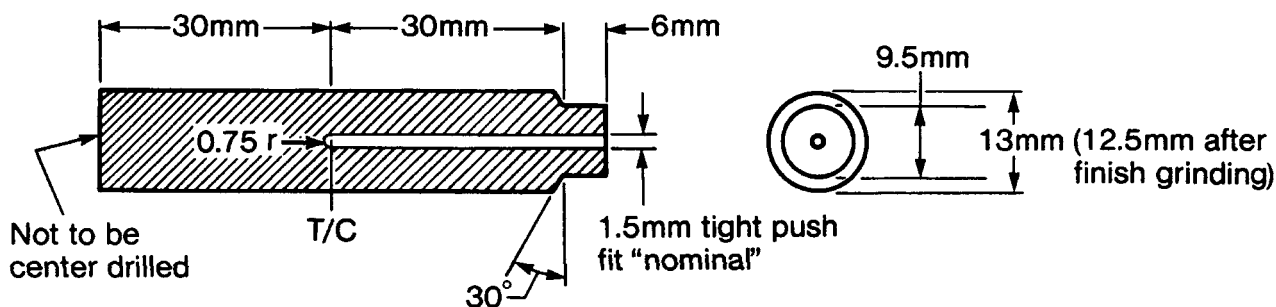
7.1 Furnace—Use a horizontal or vertical electrical resistance tube-type furnace capable of maintaining a constant minimum temperature of 850°C (1562°F) over a heated length of not less than 120 mm (4.72 in.) and a probe positioned in the center of the heating chamber. The furnace shall be capable of maintaining the probe's temperature within $\pm 2.5^\circ\text{C}$ (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 oil sample tested, producing a record of

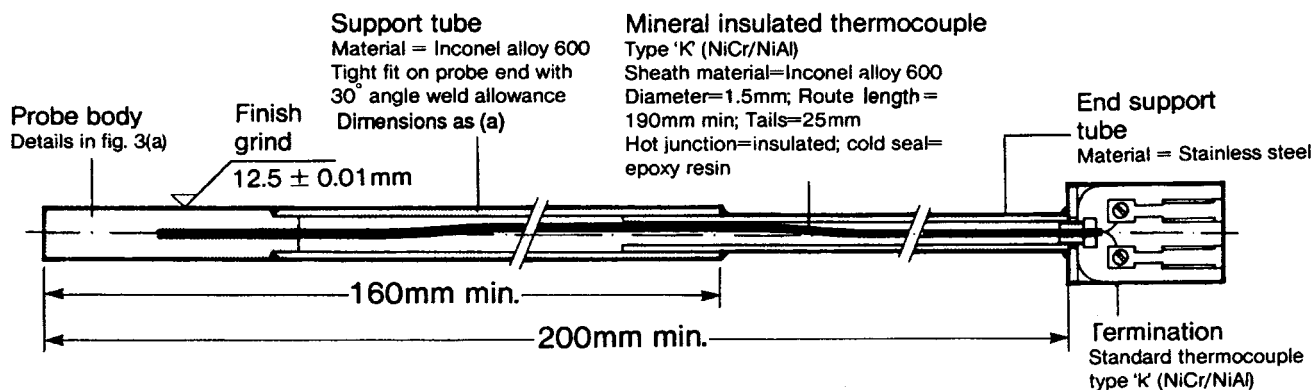
variation in the test probe assembly of temperature with respect to time and of cooling rate with respect to temperature.

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.

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



(a) Probe details



(b) General assembly

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 $1.5 \times 10^{-3} \text{ m}^3$. 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.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 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 assembly shall be rinsed with water at least three times to ensure that no quenchant residue or detergent solution remains.

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.

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

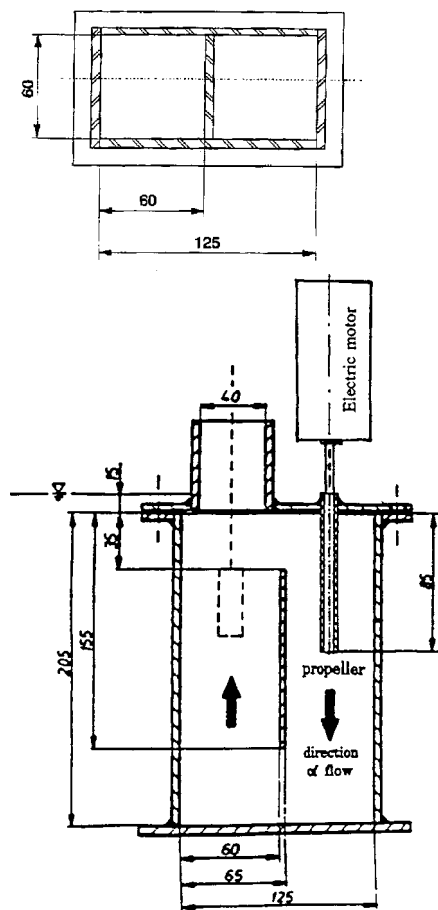


FIG. 6 Schematic Drawing of Tensi Agitation Assembly

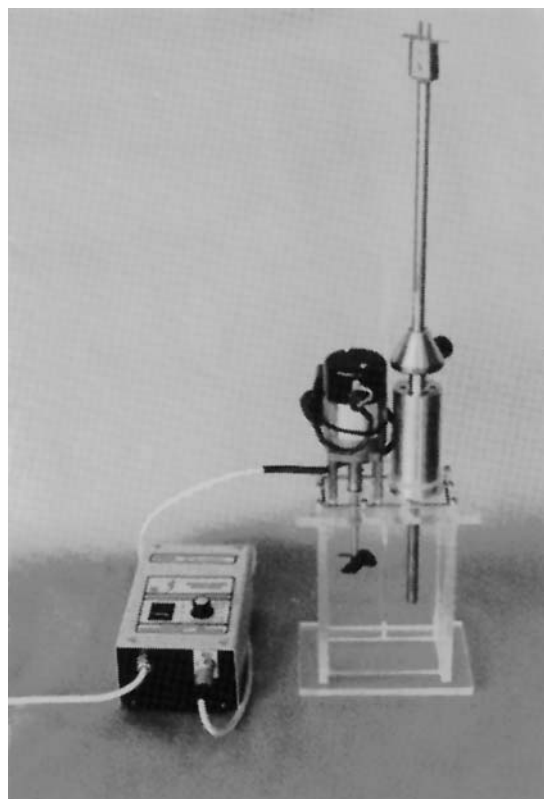


FIG. 7 Commercially Available Tensi Agitation Assembly

fluid. The fluid shall be level with the lower distance ring in the support tube, as shown in Fig. 10.

7.5 *Temperature Measurement*—Any temperature detection device may be used that is capable of measuring quenching fluid temperature to within $\pm 1^\circ\text{C}$ (1.8°F).

7.6 *Transfer Mechanism*—The heated probe is transferred manually to the Tensi agitation assembly, which shall be equipped with a fixture to ensure correct placement in the center of the quenching chamber, as illustrated in Figs. 6 and 7. A timer shall be used to ensure a maximum transfer time of 3.0 s.

7.7 *Timer*, graduated in seconds and minutes, and may be part of a computer clock.

8. Reagents and Materials

8.1 *Reference Quenching Fluid*—Use a reference quenching fluid for initial and regular probe calibration to determine if the probe will give results consistent to those obtained during initial break-in (see Test Method D6200). If the maximum cooling rate is greater than $\pm 3\%$, the probe shall be reconditioned (see 9.3). Cooling curve results shall be traceable to a primary standard fluid, such as that cited in Wolfson Engineering Group Specification or JIS K 2242 and JIS K 6753. The reference fluids shall be stored in a sealed container when not

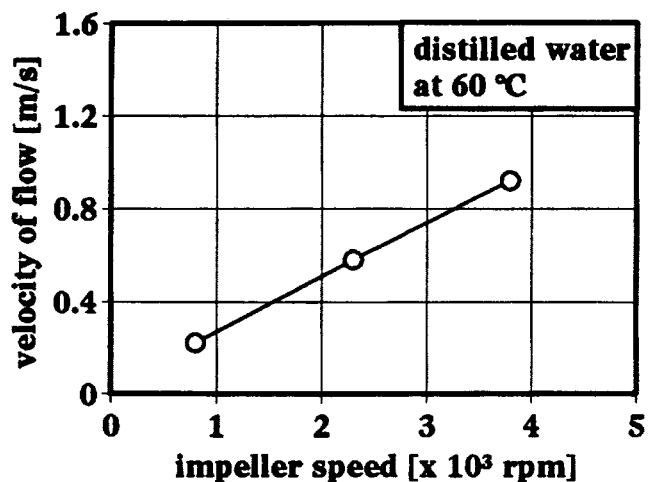
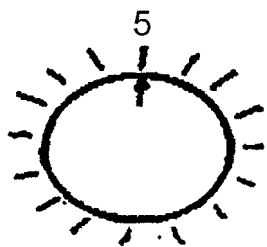


FIG. 8 Correlation of Flow Velocity Through Quenching Chamber and Impeller Rotational Speed for Water

in use and shall be replaced after 200 quenches or two years, whichever is sooner. Distilled or deionized water at 50°C may also be used.

NOTE 5—If a reference fluid other than distilled water is used, the agitation device described here shall not be used. Instead, a suitable compatible container consistent with the specified requirement being followed shall be used.

8.2 *Cleaning Solvent*—A hydrocarbon solvent that will evaporate at room temperature, leaving no residue. (**Warning**—Flammable. Harmful if inhaled.)



1000 rpm

FIG. 9 Potentiometer Setting

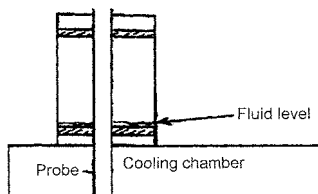


FIG. 10 Fluid Level

8.3 *Polishing Paper*, 600 grit emery.

8.4 *Cloth*, lintless and absorbent.

9. Cleaning and Conditioning

9.1 *Cleaning Used Probes*—Wipe probe with a lintless cloth or absorbent paper after removal from the oil and prior to returning to the furnace. (**Warning**—The probe shall always be considered hot, as temperature below visual hot temperatures can still cause injury to the skin.) A cleaning solvent may be used, but care should be taken that the probe is below 50°C (122°F). (**Warning**—Do not use cleaning solvent near the furnace opening, especially with automated transfer mechanisms.)

9.2 *Conditioning New Probes*—Condition the probe prior to its initial use with any quenchant by carrying out a minimum of six trial quenches, or a greater number if required to achieve consistency, using a general purpose hydrocarbon oil. Consistency shall mean the last two tests shall have maximum cooling rates within $\pm 2\%$ in temperature and cooling rate. Clean the probe assembly between quenches as specified in 9.1. Quench the probe in the reference quenching fluid and check according to 12.3. If the probe does not meet the requirements of 12.3, recondition according to 9.3 and then recalibrate again according to 12.3. Do not use probes that do not meet these requirements.

9.3 Probe Reconditioning:

9.3.1 The probe shall be reconditioned when the probe calibration according to 12.3 does not meet the calibration limits ($\pm 3\%$ at the maximum cooling rate) of the reference fluid. Recondition the probe by cleaning with emery paper. Although coarser 320-grit paper may be used for initial cleaning, the final finish shall be provided using 600-grit emery paper. Following this surface cleaning procedure, the probe shall be quenched until repeatable cooling curve results of a reference oil are obtained.

9.3.2 An alternative is to recondition the probe after every run. Before testing a set of aqueous polymer quenchant solutions, the probe is quenched into the reference fluid after

surface conditioning. If the maximum cooling rate of the reference fluid is within $\pm 3\%$ of the calibration limit, the probe may be used for further testing. When testing, the probe is cleaned prior to each run. After testing the set of fluids is completed, the probe is quenched into the reference fluid to ensure that it is still within calibration.

10. Sampling

10.1 Sampling shall be in accordance with 7.5. Take care to ensure the sample is representative of the oil being tested. Use a clean and dry sample container.

11. Preparation of Apparatus

11.1 Preheat furnace to 815 to 857°C, (1500 to 1575°F).

11.2 Connect a dry, conditioned, calibrated probe to the transfer mechanism in accordance with equipment manufacturer's instructions.

11.3 *Removal of Air*—After filling the apparatus with the quenchant to be tested, tilt to both sides to release any air that may be trapped in the shaft packing box. If air is mixed with the fluid, test results may be influenced.

11.4 The aqueous polymer quenchant shall be heated or cooled to the desired temperature if production testing is being performed, or to $40 \pm 2^\circ\text{C}$ ($104 \pm 3.6^\circ\text{F}$) if the reference quenching fluid is being tested. Continuously agitate the test sample when heating.

12. Calibration and Standardization

12.1 Probe:

12.1.1 Check the accuracy of the probe thermocouple by attaching a previously calibrated thermocouple to the outer surface of the probe. Locate the tip of the calibrated thermocouple 30 mm (1.181 in.) from the end of the probe. Heat the probe and calibrated thermocouple to the selected furnace temperature of 845 to 855°C (1553 to 1571°F), and allow to equalize. Compare the outputs of both the furnace and probe thermocouples by any calibrated temperature measuring device capable of required accuracy, as described in Test Method E220 and Specification E230.

12.1.2 *Frequency of Probe Calibration*—Calibrate the probe against a reference quenching fluid before each set of test runs.

12.2 *Equipment Calibration*—Calibrate desired recording mechanism, as described in Annex A1.

12.3 *Total System Calibration*—Calibrate the system with a reference quenching fluid (see 8.1) following the procedure described in Section 13. Calibrate the system prior to using a new probe for testing and before and after each new set of test runs. The limits of the results obtained on the reference fluid shall be established for each reference fluid prior to use, as described in 8.1. The limits shall include, as a minimum, the following values: maximum cooling rate ($^\circ\text{C/s}$, $^\circ\text{F/s}$); the temperature at the maximum cooling rate ($^\circ\text{C/s}$, $^\circ\text{F/s}$); cooling rate ($^\circ\text{C/s}$, $^\circ\text{F/s}$) at 300°C (572°F); and the time in seconds from immersion to three different temperatures, such as 600°C (1112°F), 400°C (752°F), and 200°C (392°F). If the limits are greater than $\pm 3\%$ of those previously established for the reference fluid, the probe shall be reconditioned (see 9.3) and calibrated, as described in Section 13.

13. Procedure

13.1 Place the probe in the preheated furnace. Bring the probe temperature to the required temperature of 845 to 855°C (1500 to 1575°F), and soak at this temperature for at least 2 min.

13.2 Transfer the probe to the center of the quenchant sample, activating the data collection equipment at the same time. (**Warning**—Electric resistance type furnaces may have to be turned off prior to the transfer from the furnaces to the sample when interference with the data collection device is noted.)

13.3 Hold the probe assembly without movement, with the mechanical transfer device or a holding fixture.

13.4 When the temperature of the probe has reached 200°C (392°F) or the desired lower temperature, remove it from the oil and clean, as described in 9.1.

13.5 Run test in duplicate for reproducibility verification, using the same probe and the same sample of the oil returned to the same temperature prior to the start of the test. The final data that is reported may be averaged to produce the final cooling curve data, or the results from both runs may be reported individually. Duplicate testing is not required when the cooling curves for aqueous polymer quenchant being tested are essentially the same as that curve to which the test cooling curve is being compared.

14. Interpretation of Results

14.1 *Cooling Curves*—Cooling curves and cooling rate curves are obtained for comparison reasons, that is, the oil compared to another oil, a control sample, or previously recorded curves. The test may show the effect of oxidation, the presence of additives and their concentrations, or contamination on the cooling characteristics of a quenching oil. Changes in aqueous polymer quenchant's chemical or physical properties cause changes in its heat extraction capabilities, either speeding up or slowing down part or all of the curve (see Fig. 5).

15. Report

15.1 The report shall include the quenching conditions, including the quenchant name and identification number, concentration, quenchant temperature, impeller speed, and cooling time-temperature and cooling rate-temperature curves for the submitted sample. Recommended data to be reported for each test run are provided in 15.1.1-15.1.3. Additional values shall be reported as required by the purchaser.

15.1.1 From the time/temperature graph, report the time to the nearest 0.1 s at 600°C (1112°F), 400°C (752°F), and 200°C (392°F).

15.1.2 From the temperature/cooling rate graph, report the following:

15.1.2.1 Maximum cooling rate, °C/s (°F/s),

15.1.2.2 Temperature at the maximum cooling rate, °C (°F), and

15.1.2.3 Cooling rate at 300°C (572°F).

15.1.3 Report the following information:

15.1.3.1 Date,

15.1.3.2 Identification of sample,

15.1.3.3 Reference to the test method,

15.1.3.4 Cooling curves and cooling rate curves, including calibration curves for the reference fluid

15.1.3.5 Statement of results, and

15.1.3.6 Any modifications to test methods.

16. Precision and Bias

16.1 Four cooperators tested aqueous polymer quenchants of PAG and PVP type. The quenchants were heated to temperatures of 30 and 50°C. Agitation rates were based on propeller rotation speeds of 800 and 1500 rpm. Test results for the six primary cooling curve characteristics ranged from:

Maximum Cooling Rate	107 to 169°C/s
Temperature of the Maximum Cooling Rate	551 to 668°C
Cooling Rate at 300°C	37 to 59°C/s
Time to Cool to 600°C	2.5 to 6.2 s
Time to Cool to 400°C	4.6 to 8.5 s
Time to Cool to 200°C	10.0 to 17.7 s

16.1.1 Cooling characteristic ranges are based on the overall average test response for each fluid. Individual results from a given laboratory exceed the limits listed above.

16.1.2 The four cooperators verified the calibration of their units in accordance with 12.3. The statistical analysis of data from this interlaboratory report can be obtained from ASTM International.⁹

16.2 The precision of this test method was determined by statistical examination of interlaboratory test results using ADJD6300.⁶ Additional variation may be encountered when testing different types and concentrations of quenching fluids or testing at conditions outside that described in 16.1. It is noted that due to the limited number of participants, coupled with the scatter in test results, only two characteristics were shown to have statistically significant repeatability and reproducibility. Even these parameters exhibit higher than desired levels of variation, which suggests that the test should be used only for analysis when running side by side comparisons.

16.2.1 *Repeatability*—The difference in successive results obtained by the same operator with the same apparatus under constant operating conditions in the same sample would, in the long run, in the normal and correct operation of the test method exceed the following deviation in one case in twenty:

Time to Cool to 600°C	1.3492 s
Time to Cool to 400°C	1.4855 s

16.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test specimens would in the long run exceed the following in only one case in twenty:

Time to Cool to 600°C	3.7095 s
Time to Cool to 400°C	4.5392 s

16.3 *Bias*—The evaluation of cooling characteristics of quenching fluids by this test method has no bias because the cooling characteristics can be defined only in terms of this test method.

17. Keywords

17.1 cooling curve; cooling rate; cooling time; quench oil

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1606.

ANNEX

(Mandatory Information)

A1. EQUIPMENT CALIBRATION

A1.1 Computer

A1.1.1 Using a potentiometer, supply an electromotive force to the sheathed type *K* ungrounded thermocouple leadwire or connector to the following equivalents: 200°C (392°F), 500°C (932°F), and 850°C (1562°F).

A1.1.2 The resultant readout on the system should be $\pm 2.25\%$ of the electromotive force's equivalent temperature (see Specification E230).

A1.1.3 The time axis of the data collection system shall be checked by a stopwatch at the commencement of each series of tests, but shall not exceed 50 tests. The error shall not exceed 0.5 %.

A1.2 Data Acquisition and Plotting

A1.2.1 The probe thermocouple output is sampled, digitized, and stored in the memory of the computer. The type *K*

thermocouple leadwire shall have an electronic zero reference junction in the hookup. The frequency of sample data point collection should not be less than five times per second (sample period of 125 ms or less) and the data collection time should be 60 s or greater.

A1.2.2 The temperature-time plot may be produced either on-line during the test or off-line after the test.

A1.2.3 The cooling rate is calculated by numerical differentiation of the probe thermocouple output temperature. The cooling rate shall be calculated by a software program in the microprocessor or from a disc. The overall accuracy of the measurement system shall not exceed the limits described in 12.3.

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