Designation: D 6549 – 00~~0~~1

An American National Standard

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 D 6549; 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 in SI units are to be regarded as the standard. The values 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:*

[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.

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E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[2]
E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[2]
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

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)**.[5] 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)**.

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.

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 to 857°C (1500 to 1575°F) may be used.

[2] *Annual Book of ASTM Standards*, Vol 14.03.
[3] Available from Society of Automotive Engineers, 400 Commonwealth ~~Drive,~~ Dr., Warrendale, PA 15096.
[4] Available from Wolfson Heat Treatment Centre, Aston University, Aston Triangle, Birmingham B4 7ET, England.
[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

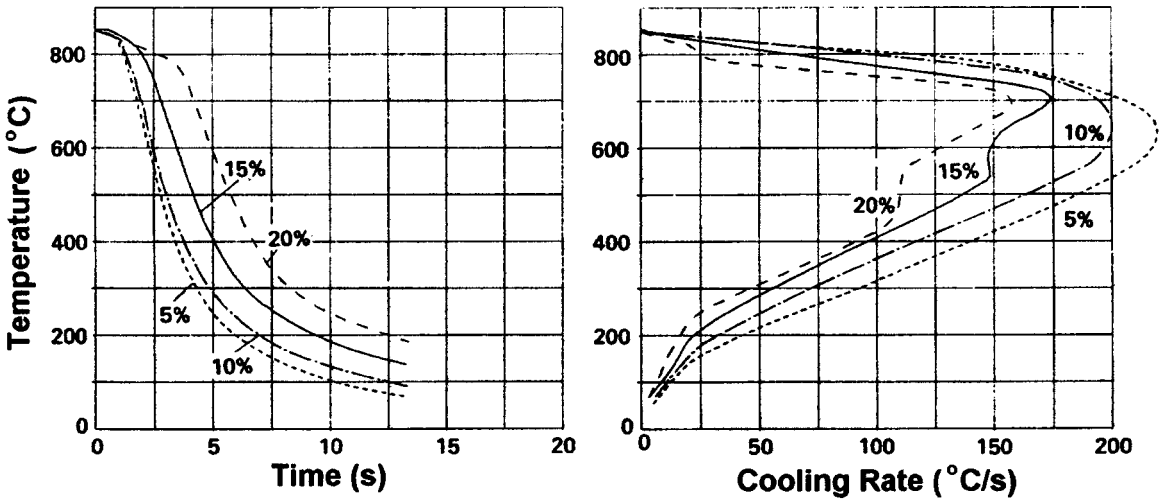


FIG. 1 Effect of Quenchant Concentration on Cooling Curve Performance for a Poly(Alkylene Glycol) Quenchant at 30°C and 0.5 m/s

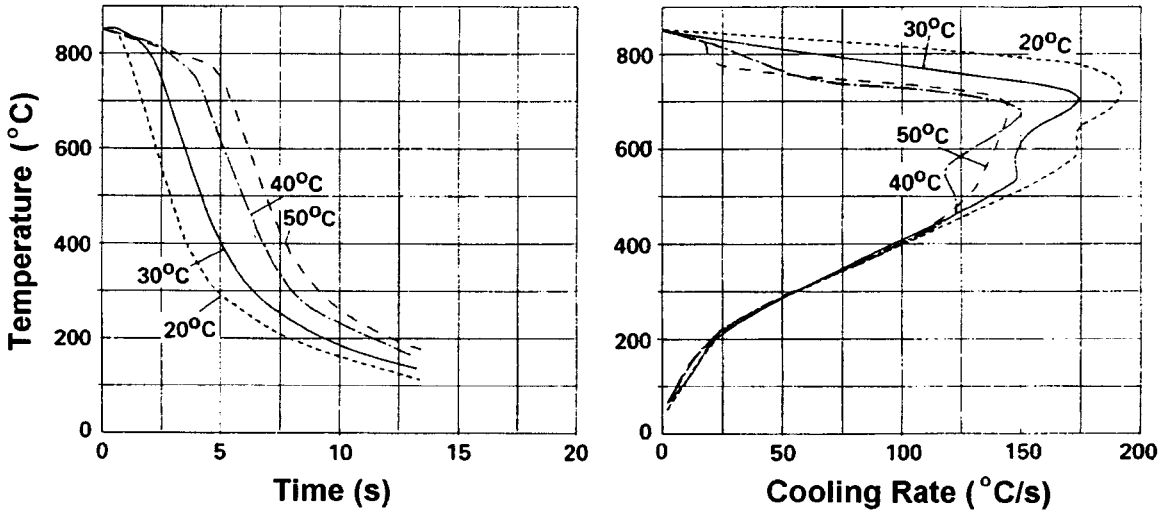


FIG. 2 Effect of Bath Temperature Variation on Cooling Curve Performance for 15 % Aqueous Solution of Poly(Alkylene Glycol) Quenchant at 0.5 m/s

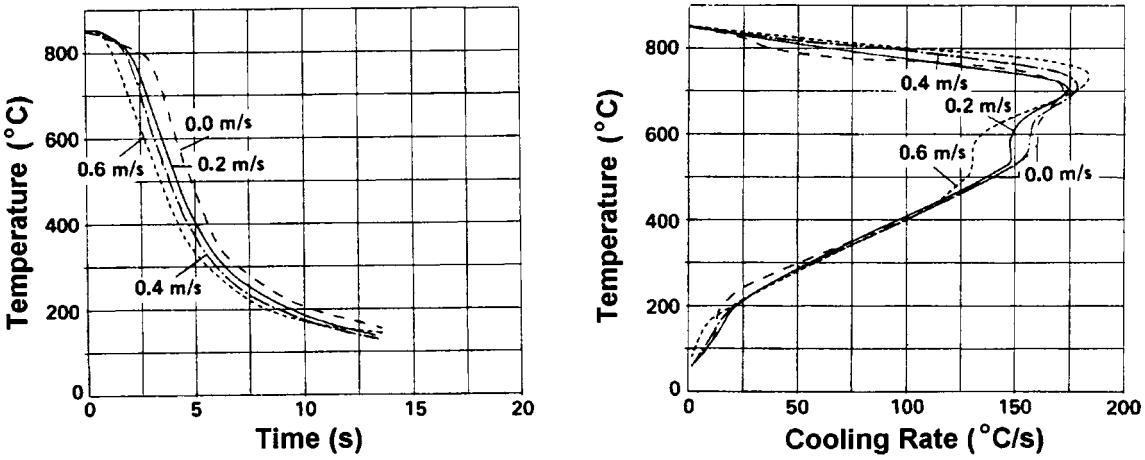


FIG. 3 Effect of Agitation Rate Variation on Cooling Curve Performance for a 15 % Aqueous Poly(Alkylene Glycol) Quenchant Solution at 30°C

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

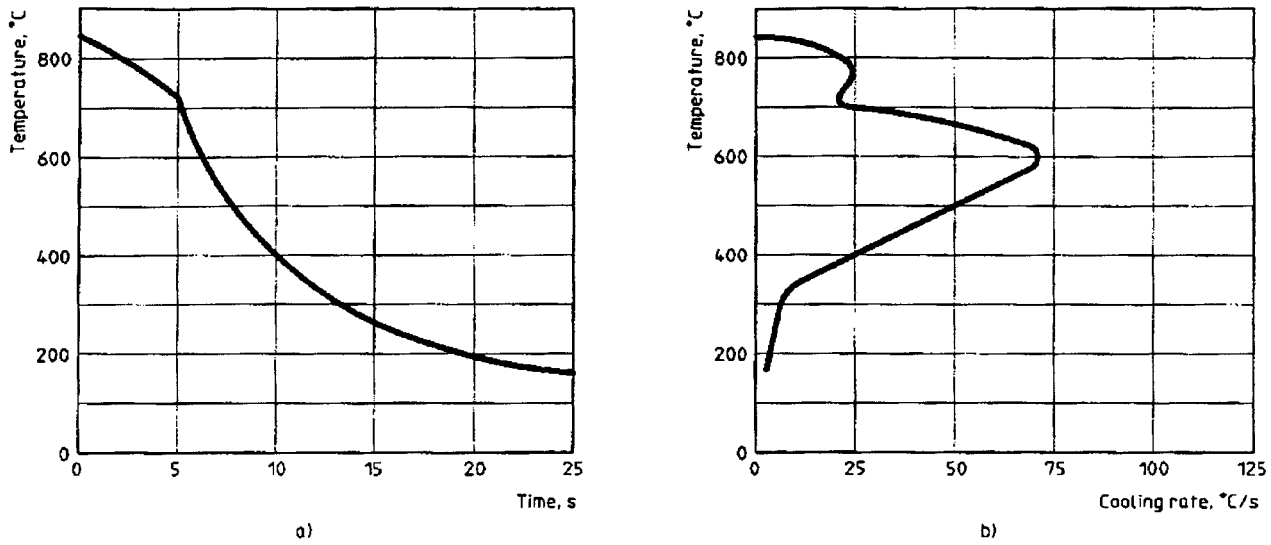
6.1 The presence of contaminants, such as oil, salt, metalworking fluids, forging lubricants, and polymer degradation, may affect cooling curve results obtained by this test method for aqueous polymer quenchants.

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 ±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 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 ± 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 in accordance with AMS 5665, which has a nominal



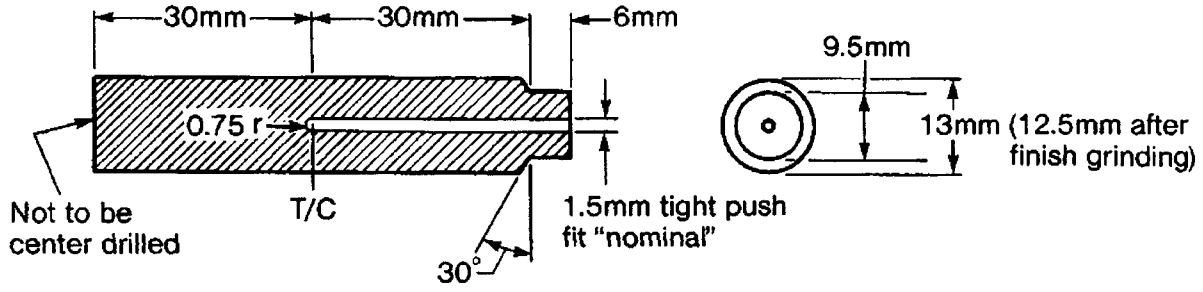
NOTE 1—(a.) Cooling Curve (b.) Cooling Rate Curve

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

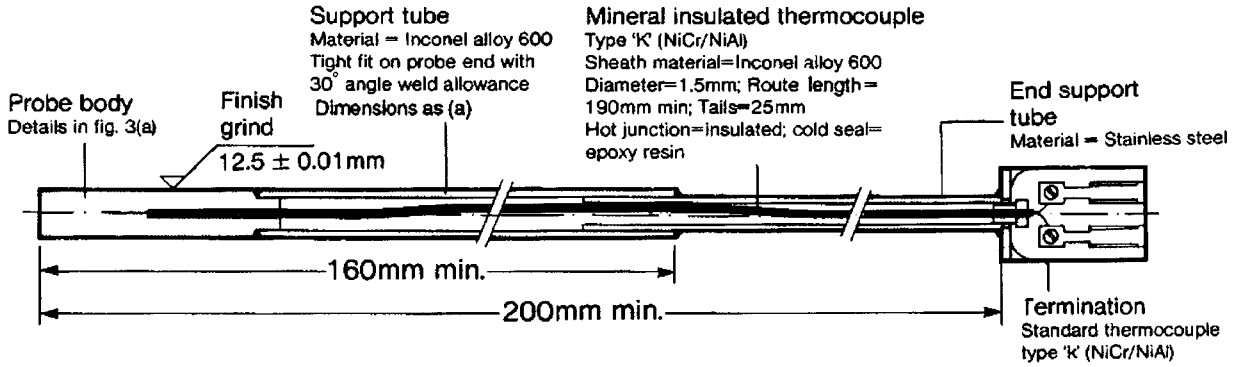
composition of 76.0 % Ni, 15.5 % Cr, 8.0 % Fe, 0.08 % C, and 0.25 % maximum Cu. The probe shall be attached to a support tube with a minimum length of 200 mm (7.874 in.). The thermocouple sheathing and the support tube shall be the same material as the probe (see Note 2). See Fig. 5 for other manufacturing 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*:



(a) Probe details



(b) General assembly

NOTE 1—Dimensions above are nominal.

FIG. 5 Probe Details and General Probe Assembly

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).

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.

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*, used for initial calibration and for periodic calibration verification. Data collected from quench tests with the reference fluid shall be evaluated for compliance to the specified values for the six primary characteristics. These characteristics, as defined in Wolfson Engineering Group Specification, are as follows:

Time to cool to 600°C (1112°F)	12-14 s
Time to cool to 400°C (752°F)	19-21 s
Time to cool to 200°C (392°F)	50-55 s
Maximum cooling rate	47-53°C/s (85-95°F/s)
Temperature of the maximum cooling rate	490-530°C (914-986°F)
Cooling rate at 300°C (572°F)	6-8°C/s (10.8-14.4°F/s)

8.1.1 If results do not comply with the specified ranges, the probe shall be replaced or reconditioned (see 9.3) or system adjustments made. Compliance to the specified limits of the primary reference fluid is critical for establishing the validity of subsequent test results. It has been shown that the test method has an excellent level of repeatability and reproducibility when the probe and system are shown to be in calibration **(9, 10)**.

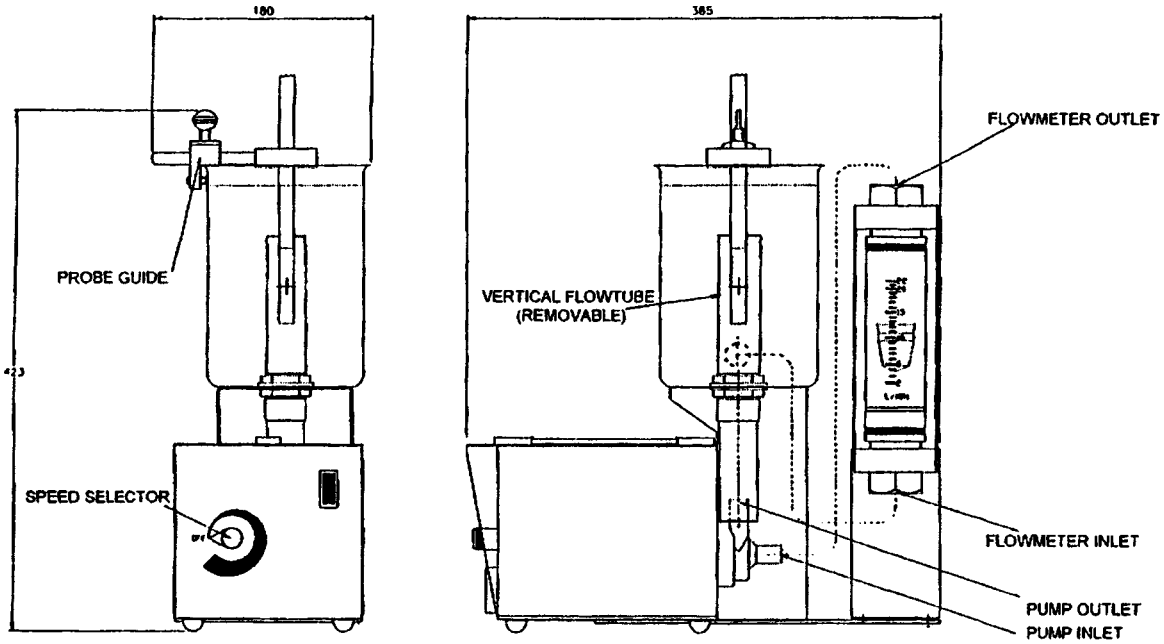


FIG. 6 Drayton Agitation Unit

FIG. 7 Commercially Available Drayton Agitation Unit

8.1.2 A secondary reference fluid may be used, provided that sufficient statistical cooling curve testing has been conducted so that the results are (*1*) traceable to the primary reference fluid and (*2*) compared on the basis of the six primary cooling characteristics.

8.1.3 Reference fluids shall be stored in a sealed container when not in use and shall be replaced after 200 quenches or two years, whichever is sooner.

8.2 *Polishing Paper*, 600 grit emery.

8.3 *Cotton Cloth or Paper*, lintless and absorbent.

9. Cleaning and Conditioning

9.1 *Cleaning Used Probes*—Wipe the probe with a clean, wet, lintless cotton cloth or absorbent paper after removal from the quenchant and prior to returning to the furnace. Unmounted probes may be cleaned in the same manner or, alternatively, washed under a stream of water, and then wiped dry. (**~~WARNING~~Warning**—The probe shall always be considered hot as a temperature below visual hot temperatures can still cause injury to the skin or ignition of the cloth or paper used in cleaning.)

9.2 *Conditioning New Probes*—Condition the probe prior to its initial use by carrying out a minimum of six trial quenches, or a grater number if required to achieve consistency, using a clean, neutral, general purpose hydrocarbon oil. Clean the probe assembly between quenches, as specified in 9.1. Quench the probe in the reference quenching fluid and check in accordance with 12.3. If the probe does not meet the requirements of 12.3, recondition in accordance with 9.3 and then recalibrate again in accordance with 12.3. Do not use probes that do not meet these requirements.

9.3 *Probe Reconditioning*—The probe shall be reconditioned when the probe calibration, as described in 12.3, does not meet the calibration limits of the six cooling characteristics specified for the reference fluid. Recondition the probe by polishing with emery paper. Although coarser 320-grit paper may be used for initial polishing, the final finish shall be provided by use of 600-grit emery paper. Following this procedure, the probe shall be quenched until satisfactory cooling curve results are obtained from the reference fluid.

10. Sampling

10.1 Take care that the gross media, from which the sample is taken to fill the agitation unit, is well mixed to ensure that the sample is representative of the media being tested. Any containers used to secure the quenchant sample must be clean and dry.

11. Preparation of Apparatus

11.1 Preheat furnace to 850 ± 2°C (1562 ± 4°F), or alternatively, to 815 to 857°C (1500 to 1575°F) for production testing.

11.2 Connect a dry, conditioned, calibrated probe in accordance with the equipment manufacturer's instructions, and insert in furnace.

11.3 Heat or cool the aqueous polymer quenchant to the desired temperature if production testing is being performed. Continuously agitate the quenchant sample at the desired flow rate while the sample is being heated. If the primary reference quenching fluid is being tested, heat it to 40 ± 2°C (104 ± 3.6°F), but do not agitate the fluid during the calibration test.

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 850 ± 2°C (1562 ± 4°F) and allow to equalize. Compare the outputs of both the furnace and probe thermocouples by any calibrated temperature-measuring device capable of the required accuracy as described in Test Method E 220 and Tables E 230.

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 test procedure described in Section 13. It is noted that this calibration shall be performed in the static or unagitated condition. Calibrate the system prior to using a new probe for testing and before and after each new set of test runs. The probe and system shall be considered to be in calibration if the results conform to the limits prescribed for the six primary cooling characteristic values, as described in 8.1. If the results deviate beyond the limits prescribed for the reference fluid, the system shall not be considered as being in calibration. The probe may need to be reconditioned (see 9.3). Alternately, when results deviate from the prescribed limits, it is also appropriate to examine the test setup and procedure for compliance to this test method and the manufacturer's recommended practice.

13. Procedure

13.1 Bring the probe temperature to the required temperature of 850 ± 2°C (1562 ± 4°F), or to the alternate appropriate temperature for production testing, and hold 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 test is complete (generally within 60 s) and the probe has reached a desired safe lower temperature, 200°C (392°F) maximum, remove it from the quenchant and clean, as described in 9.1.

13.5 Run the test in duplicate for reproducibility verification, using the same probe and the same quenchant sample after it has cooled to the same temperature prior to the start of the test. 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 measured to compare a quenchant to a control sample or to compare one quenchant to another quenchant or to compare to 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 the quenchant. 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 Figs. 4 and 6).

15. Report

15.1 The report shall include the quenching conditions, including the quenchant name and identification number, concentration, quenchant temperature and agitation rate, 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).

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.

15.1.3.6 Any modifications to test methods.

16. Precision and Bias

16.1 The precision and bias of the procedure in Test Method D 6549 for determining cooling characteristics of a quench oils are being determined.

17. Keywords

17.1 cooling curve; cooling curve analysis; cooling rate; cooling time; quenchant

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 ±2.25 % of the electromotive force's equivalent temperature (see Tables E 230).

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 not to 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 electronic circuitry shall include Type K thermocouple linearization and ice point correction. 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 disk. The overall accuracy of the measurement system shall not exceed the limits of 12.3.

REFERENCES

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(**2**) Tensi, H.M., Stich, A., and Totten, G.E., "Quenching and Quenching Technology," Chapter 4 in *Heat Treatment of Steel Handbook*, G.E. Totten and M.A.H. Howes, Eds., Marcel Dekker, NY. 1997, pp. 157-249.

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(**4**) Tamura, I., Shimizu, N., and Okada, T., "Engineering Method to Judge the Quench Hardening of Steel from Cooling Curves of Quenchants," 7th ASM Heat Treating Conference/Workshop, Chicago, Il, May 10-12, 1983.

(**5**) Moore, D.L., and Crawley, S., "Applications of Standard Quenchant Cooling Curve Analysis," Second ASM Heat Treating and Surface Engineering Conference, Dortmund, Germany, June 2-4, 1993.

(**6**) Totten, G.E., Bates, C.E., and Clinton, N.A., "Cooling Curve Analysis", Chapter 3 in *Handbook of Quenchants and Quenching Technology*, ASM International, Materials Park, OH, 1993, pp. 69-128.

(**7**) Tensi, H.M., "Determination of Quenching Power of Various Fluids," Chapter 8 in *Theory and Technology of Quenching - A Handbook*, B. Liscic, H.M. Tensi, and W. Luty, Eds., SpringerVerlag, New York, NY, 1992, pp. 208-247.

(**8**) Boyer, H.E., and Cary, P.R., *Quenching and Distortion Control*, ASM International, Materials Park, OH, 1988, p. 162.

(**9**) Guisbert, D.A., "Precision and Accuracy of the Continuous Cooling Curve Test Method," *Proceedings of the 16th ASM Heat Treating Society Conference and Exposition*, March 19-21, 1996, Cincinnati, OH, pp. 435-441.

(**10**) Guisbert, D.A., and Moore, D.L., "Influence of Test Conditions on Cooling Curve Test Results," *Proceedings of the 15th International Automotive Heat Treating Conference*, ASM International, Puerto Vallarta, Mexico, 1998, pp. 267-280.