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Standard Test Method for Determination of Cooling Characteristics of Aqueous Polymer Quenchants for Aluminum Alloys by Cooling Curve Analysis[1]

This standard is issued under the fixed designation D7646; 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~~ Scope*

1.1 This test method covers the description of the equipment and the procedure for evaluating quenching characteristics of aqueous polymer quenchants by cooling rate determination.

1.2 This test method is designed to evaluate aqueous polymer quenchants for aluminum alloys in a non-agitated system. There is no correlation between these test results and the results obtained in agitated systems.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
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 for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples

2.2 *ISO Standards:*[3]

ISO 3819 Laboratory Glassware—Beakers

2.3 *Japanese Industrial Standards:*[4]

JIS K 2242 Heat Treating Oil

[1] 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.

Current edition approved ~~May 1, 2019~~July 1, 2023. Published ~~July 2019~~August 2023. Originally approved in 2010. Last previous edition approved in ~~2014~~2019 as D7646 – 10 ~~(2014).~~(2019). ~~DOI:10.1520/D7646-10R19.~~DOI:10.1520/D7646-23.

[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, http://www.sae.org.

[4] Available from Japanese Standards Association (JSA), Mita MT Bldg., 3-13-12 Mita, Minato-ku, Tokyo 108-0073, Japan, http://www.jsa.or.jp.

***A Summary of Changes section appears at the end of this standard**

2.4 *Wolfson Engineering Group Specification:*[5]
Laboratory Tests for Assessing the Cooling Curve of Industrial Quenching Media

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aqueous polymer quenchant, n*—aqueous solution containing a water soluble polymer; typically including poly(alkylene glycol), poly(ethyl oxazoline), poly(sodium acrylate) and poly(vinyl pyrrolidone). 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.

3.2.2 *characteristic temperature, n*—transition temperature from vapor blanket phase (film boiling phase) to rapid cooling phase (nucleate boiling phase) on cooling curve.

3.2.3 *cooling curve, n*—cooling curve is a graphical representation of the cooling time (t)–temperature (T) response of the probe (see 7.3). An example is illustrated in Part B of Fig. 1.

3.2.4 *cooling curve analysis, n*—the process of quantifying the cooling characteristics of a heat treating oil based on the temperature versus time profile obtained by cooling a pre-heated metal probe assembly (see Fig. 2) under standard conditions.

3.2.5 *cooling rate curve, n*—The cooling rate curve is obtained by calculating the first derivative (*dT/dt*) of the cooling time–temperature curve. An example is illustrated in Part B of Fig. 1.

3.2.6 *quench severity, n*—the ability of a quenching medium to extract heat from a hot metal.

3.2.7 *quenchant, n*—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 Determine the silver rod probe assembly's cooling time versus temperature after placing the assembly in a furnace and heating to 500 °C and then quenching in an aqueous polymer quenchant solution. The temperature inside the probe assembly and the

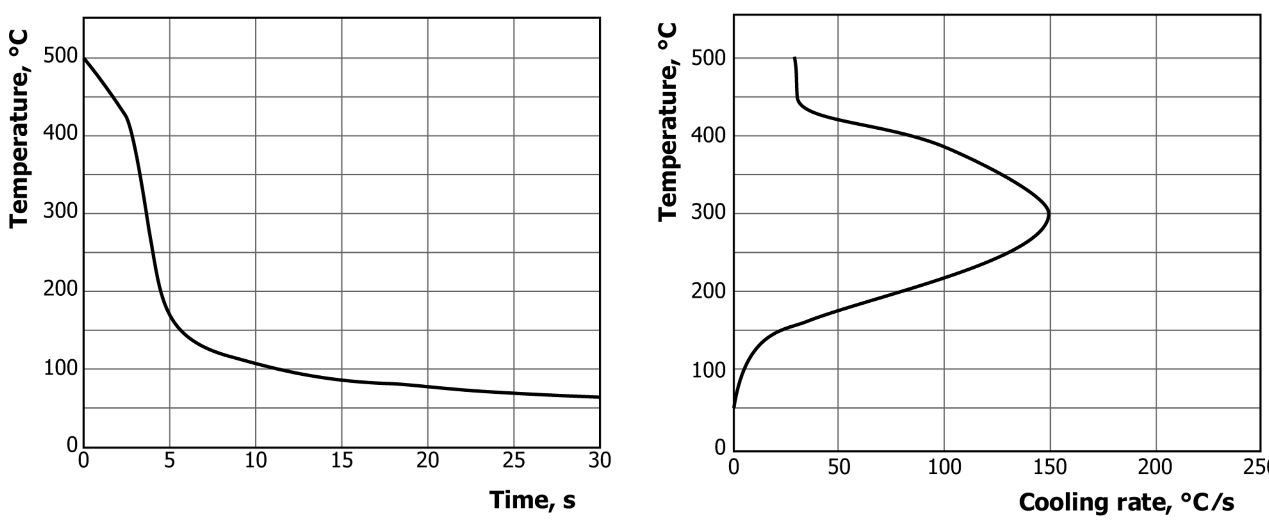

FIG. 1 Typical Temperature/Time and Temperature/Cooling Rate Plots for Test Probe Cooled in an Aqueous Polymer Quenchant

[5] Available from Wolfson Heat Treatment Centre, Aston University, Aston Triangle, Birmingham B4 7ET, England, http://www.sea.org.uk/whtc/.

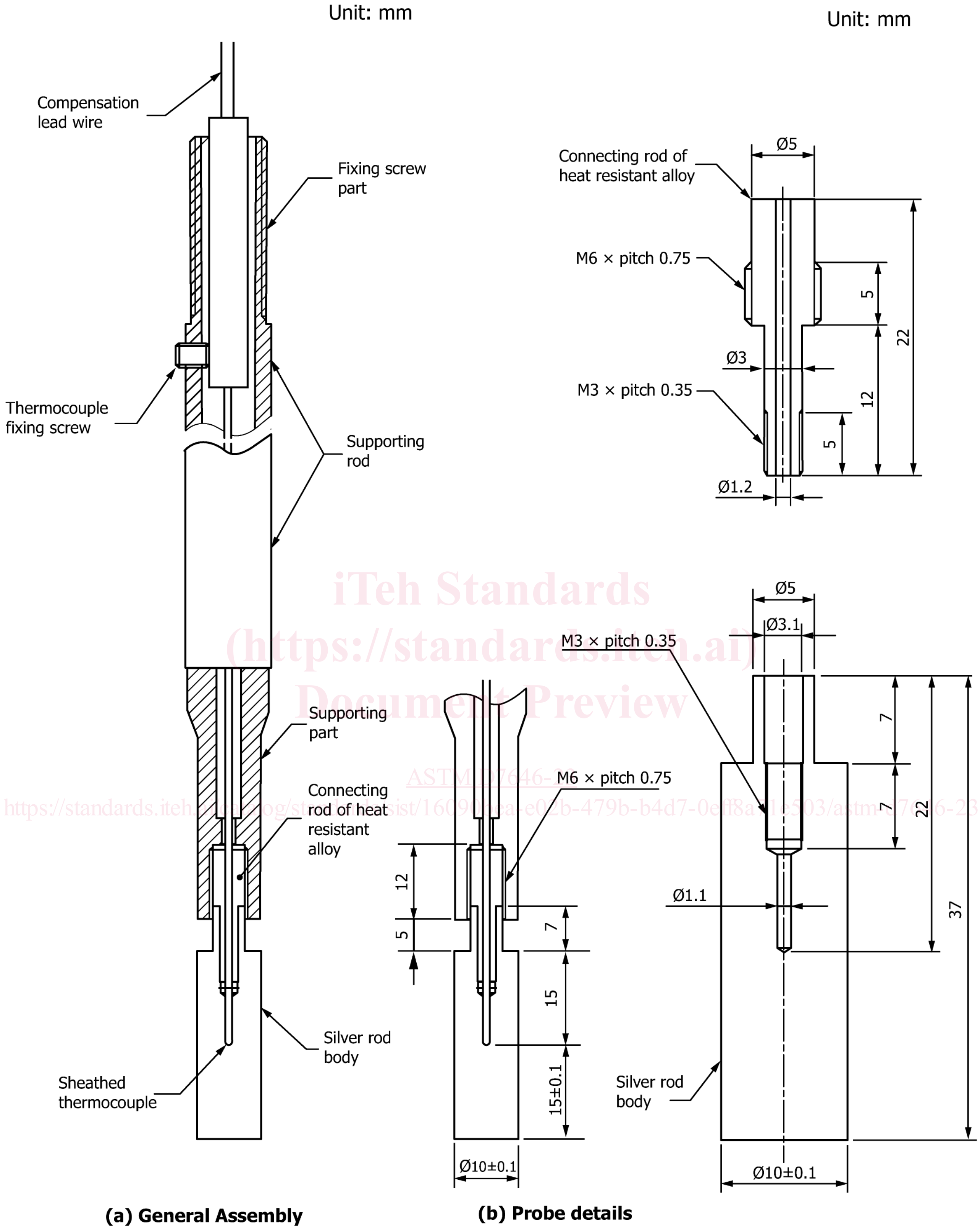

FIG. 2 Probe Details and General Probe Assembly

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.

5. Significance and Use

5.1 This test method provides a cooling time versus temperature pathway. 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.

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 over a heated length of not less than 120 mm and a probe positioned in the center of the heating chamber. The furnace shall be capable of maintaining the probe's temperature within 62.5 °C over the specimen length. The furnace, that is, the radiant tube heating media, shall be used with ambient atmosphere.

NOTE 1—Although the probe temperature is significantly lower 500 °C than the recommended furnace temperature capability 850 °C, this higher temperature capability is recommended since the same apparatus may be used for cooling curve analysis for steel alloys which is performed at 805 °C to 815 °C.

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 cooling rate with respect to temperature.

7.3 *Probe*—Shall be cylindrical, having a diameter of 10 mm ± 0.1 mm and a length of 30 mm ± 0.1 mm with a 1.0 mm sheathed Type K thermocouple in its geometric center. The probe shall be made of a silver of purity 99.99 % or more. The probe shall be attached to a support tube. See Fig. 2 for recommended manufacturing details. Preparation method for silver rod shall be as follows:

7.3.1 Screw the connecting rod of heat-resistant steel in the silver rod body.

7.3.2 Insert the sheath type thermocouple through the supporting rod and supporting part.

7.3.3 Screw the connecting rod of heat resistant steel in the supporting part as inserting the sheath type thermocouple in the central part of silver rod body.

7.3.4 Screw the supporting part in the supporting rod to connect.

7.3.5 Fix the thermocouple connecting part to the supporting rod by using a set screw while pushing the sheath type thermocouple in the direction of silver rod body. In such a case, take care so that the tip of thermocouple is completely pressed to the central part of silver rod body.

7.3.6 Heat the temperature of the silver rod body and supporting part at 700 °C to 800 °C, and coat the connecting part with the crystal of silver nitrate and joint them.

7.3.7 After cooling, finish the surface smoothly by using emery papers. Although coarser 320 grit paper may be used for initial cleaning, the final finish shall be provided using 500 grit emery paper.

7.4 *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 fluid.

7.5 *Sample Container*—300 mL beaker specified in ISO 3819.

7.6 *Temperature Measurement*—Any temperature detection device may be used that is capable of measuring quenching fluid temperature to within ±1 °C.

7.7 *Transfer Mechanism*—One of the following shall be used to transfer the heated probe from the furnace to the test fluid:

7.7.1 *Automated Transfer Mechanism*—The transfer from the furnace to the oil shall be completed within 3.0 s. Immerse the probe in the center, 0 mm to 5 mm, of the fluid container to a depth where there is 50 mm ± 2 mm of fluid above and below the probe when quenched. A mechanical stop shall be used for reproducibility of probe placement.

7.7.2 *Manual Transfer*—If manual transfer is used, the sample container shall be equipped with a fixture to ensure correct placement in the center of the fluid container and to the depth defined in 7.4. A timer shall be used to ensure a maximum transfer time of 3.0 s.

7.8 *Timer*—Graduated in seconds and minutes; may be part of a computer clock.

7.9 *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 fluid.

7.10 *Temperature Measurement*—Any temperature detection device may be used that is capable of measuring quenching fluid temperature to within ±1 °C.

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.

8.1.1 *Dioctylphthalate DOP (Di-2-ethylthexyl Phthalate)*—Used as primary reference quenching fluid for initial calibration and for periodic calibration of the probe. Properties of DOP used as reference fluid are as follows:

Density (20 °C): 0.986 g/m^3 ± 0.003 g/m^3
Refractive index (25 °C): 1.485 ± 0.003
Water content: Not greater than ~~0.1 mass%~~0.1 % by mass
Purity (GC method): Not lower than ~~97.0 mass%~~97.0 % by mass

(**Warning—**Potential acute and chronic health effects have been reported for D.O.P. and the user shall consult the Material Safety Data Sheet supplied with this material prior to use and appropriate safety precautions shall be implemented during use.)

8.1.2 A secondary reference fluid may be used provided that sufficient statistical cooling curve testing has been conducted so that results are traceable to the primary reference fluid such as that cited in JIS K 2242.

8.1.2.1 The ~~10 mass%~~ 10 % by mass of brine solution which is prepared by dissolving sodium chloride in distilled water has also been used as reference quenching fluid for initial calibration and for periodic calibration of the probe and the total system.

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

8.3 *Polishing Paper*—500 grit emery.

8.4 *Cloth*—Lint-free and absorbent.

9. Cleaning and Polishing

9.1 *Cleaning Used Probes*—Wipe probe with a lint-free cloth or absorbent paper after removal from the quenchant 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. (**Warning—**Do not use cleaning solvent near the furnace opening, especially with automated transfer mechanisms.). Water may be also be used as a cleaning solvent which may by followed by polishing (see 9.2).

9.2 *Polishing Used Probes Using Emery Paper*—Polish probe surface lightly at every trial using 500 grit emery paper until its metallic luster is recovered.