Designation: D7646 – 10 (Reapproved 2014)

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

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 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 *ISO Standards:*[3]

ISO 3819 Laboratory Glassware—Beakers

2.3 *Japanese Industrial Standards:*[4]

JIS K 2242 Heat Treating Oil

2.4 *Wolfson Engineering Group Specification:*[5]

Laboratory Tests for Assessing the Cooling Curve of Industrial Quenching Media

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *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.

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

3.1.3 *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.

3.1.4 *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 preheated metal probe assembly (see Fig. 2) under standard conditions.

3.1.5 *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.

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

[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 Dec. 1, 2014. Published February 2015. Originally approved in 2010. Last previous edition approved in 2010 as D7646 – 10. DOI:10.1520/D7646-10R14.

[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, International, 400 Commonwealth Dr., Warrendale, PA 15096-0001.

[4] Available from Japanese Standards Association, 4-1-24, Akasaka Minato-ku, Tokyo 107–8440, Japan.

[5] Available from Wolfson Heat Treatment Centre, Aston University, Aston Triangle, Birmingham B4 7ET, England.

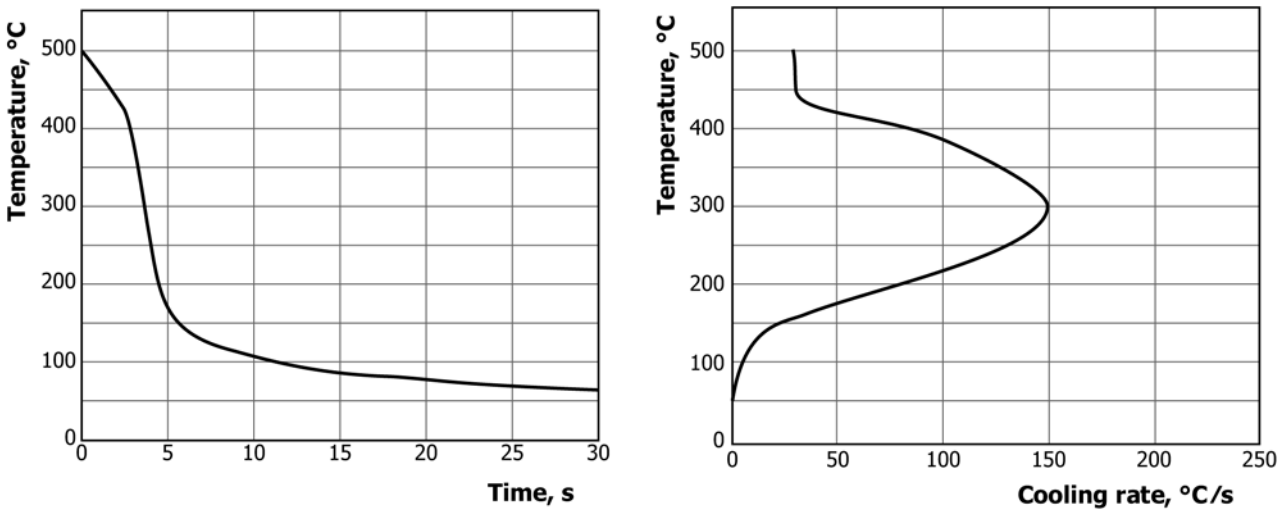

FIG. 1 Typical Temperature/Time and Temperature/Cooling Rate Plots for Test Probe Cooled in an Aqueous Polymer Quenchant

3.1.7 *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 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, 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 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 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 ± 0.1 mm and a length of 30 ± 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 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.

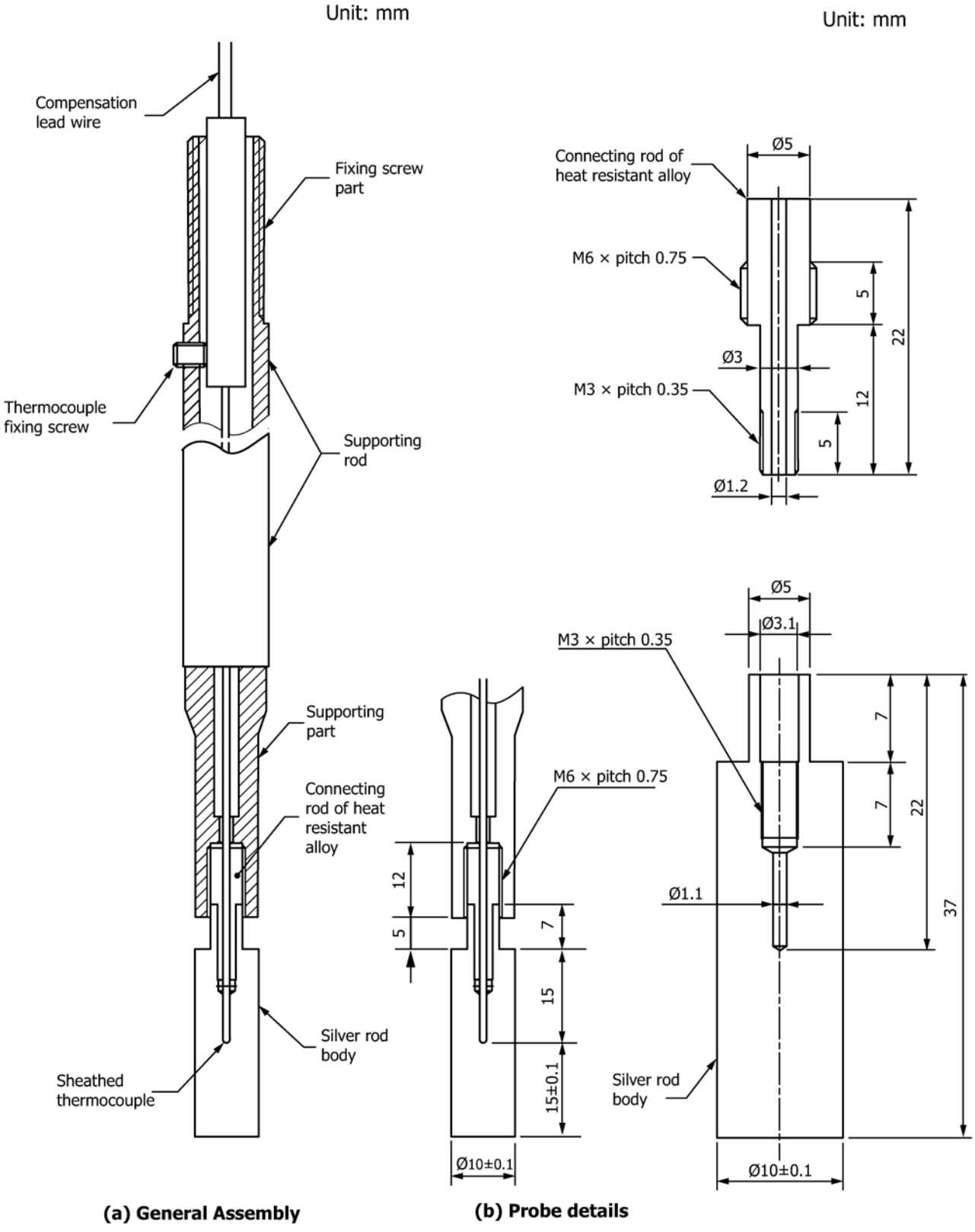

FIG. 2 Probe Details and General Probe Assembly

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 to 5 mm, of the fluid container to a depth where there is 50 ± 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 ± 0.003 g/m^3
Refractive index (25°C): 1.485 ± 0.003
Water content: Not greater than 0.1 mass%
Purity (GC method): Not lower than 97.0 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% 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.

10. Sampling

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

11. Preparation of Apparatus

11.1 Preheat furnace to 520 to 550°C.

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

11.3 The aqueous polymer quenchant shall be heated or cooled to the desired temperature if production testing is being performed, or to 80°C if the reference fluid dioctylphthalate (DOP) is being tested.

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 15 mm from the end of the probe. Heat the probe and calibrated thermocouple to the selected furnace temperature of 510 ± 5°C, 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.

12.1.2.1 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. The dioctylphthalate (DOP) and the 10 mass% of brine solution, which is prepared by dissolving sodium chloride in distilled water, shall be used.

(1) Probe Calibration by DOP—Employ DOP at 80°C, and record the cooling curve from 810 ± 5°C. In such cases, the characteristic temperature shall be 495 ± 10°C and the cooling time in seconds from 800°C to 400°C shall be 5.0 ± 0.3 s.

(2) Probe Calibration by Brine Solution—Employ 10% brine solution at 30°C and record the cooling curve form 810 ± 5°C. In such a case, the cooling time in seconds from 600°C to 300°C shall be within 0.3 s.

When these calibration references are not satisfied, disassemble the silver probe and assemble it again, polish the surface to make it flat, and calibrate it again.

12.2 *Equipment Calibration*—Calibrate desired recording mechanism, as described in Annex A1 in Test Method D6200.

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 12.1.

13. Procedure

13.1 Place the probe in the preheated furnace. Bring the probe temperature to the required temperature of 510 ± 5°C, and soak at this temperature for at least 2 min.

13.2 Transfer rapidly the probe to the center of the quenchant sample, activating the data collection equipment at the same time. At this time, the silver probe shall be immersed to the depth where its lower end is 15 mm above the bottom of the container (**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 the desired lower temperature, remove it from the fluid 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 quenchant 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 quenchant compared to another quenchant, 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 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 curves.

14.2 *Cooling Time*—Based on the cooling curve, measure the cooling time in seconds from 350°C to 150°C to the nearest 0.1 s, and take them as the cooling performance.

14.3 *Characteristic Temperature*—Based on the cooling curve, measure the characteristic temperature (the transition temperature from vapor blanket phase to rapid cooling phase), if vapor blanket phase exists on cooling curve. The tangential line crossing method is usual way to decide the value of characteristic temperature.

15. Report

15.1 The report shall include the 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 through 15.1.3. Additional values shall be reported as required by the purchaser.

15.1.1 From the time/temperature graph, report time from 350°C to 150°C to the nearest 0.1 s.

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

15.1.2.1 Maximum cooling rate, °C/s.

15.1.2.2 Temperature at the maximum cooling rate, °C, and

15.1.2.3 Cooling rate at 300°C.

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 The precision of this test method, as determined by statistical examination of interlaboratory test from 810 ± 5°C based on non-agitated DOP (dioctylphthalate) that have been heated to 80°C.

16.1.1 *Repeatability*—When two tests are carried out, in the same laboratory, by the same person, using the same apparatus, with the same sample, and on the different day or time, the discrepancy between two test results shall not exceed 5°C, in characteristic temperature and 0.3 s in cooling duration from 800°C to 400°C.

16.1.2 *Reproducibility*—When two tests are respectively carried out, in different laboratories, by different persons using different apparatus, and with the same sample, the discrepancy between two test results shall not exceed 10°C in characteristic temperature and 0.5 s in cooling duration from 800°C to 400°C.

16.1.3 *Bias*—The evaluation of cooling characteristics of quench oils 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; quenchant