Designation: D6666 – 04 (Reapproved 2014)

Standard Guide for
Evaluation of Aqueous Polymer Quenchants[1]

This standard is issued under the fixed designation D6666; 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 guide provides information, without specific limits, for selecting standard test methods for testing aqueous polymer quenchants for initial qualification, determining quality, and the effect of aging.

1.2 *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 requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D892 Test Method for Foaming Characteristics of Lubricating Oils
D1744 Test Method for Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent
D1747 Test Method for Refractive Index of Viscous Materials
D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
D2624 Test Methods for Electrical Conductivity of Aviation and Distillate Fuels
D3519 Test Method for Foam in Aqueous Media (Blender Test) (Withdrawn 2013)[3]
D3601 Test Method for Foam In Aqueous Media (Bottle Test) (Withdrawn 2013)[3]
D3867 Test Methods for Nitrite-Nitrate in Water
D4327 Test Method for Anions in Water by Suppressed Ion Chromatography
D5296 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography
D6482 Test Method for Determination of Cooling Characteristics of Aqueous Polymer Quenchants by Cooling Curve Analysis with Agitation (Tensi Method)
D6549 Test Method for Determination of Cooling Characteristics of Quenchants by Cooling Curve Analysis with Agitation (Drayton Unit)
E70 Test Method for pH of Aqueous Solutions With the Glass Electrode
E979 Practice for Evaluation of Antimicrobial Agents as Preservatives for Invert Emulsion and Other Water Containing Hydraulic Fluids
E2275 Practice for Evaluating Water-Miscible Metalworking Fluid Bioresistance and Antimicrobial Pesticide Performance

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *austenite, n*—solid solution of one or more elements in face-centered cubic iron (gamma iron) and unless otherwise designated, the solute is generally assumed to be carbon (**1**).[4]

3.1.2 *austenitizing, n*—forming austenite by heating a ferrous alloy into the transformation range (partial austenitizing) or above the transformation range (complete austenitizing). When used without qualification, the term implies complete austenitizing (**1**).

3.1.3 *aqueous polymer quenchant, n*—a solution containing water, and one or more water-soluble polymers including poly(alkylene glycol), poly(vinyl pyrrolidone), poly(sodium acrylate), and poly(ethyl oxazoline) (**2, 3**) and additives for corrosion and foam control, if needed.

3.1.4 *biodegradation, n*—the process by which a substrate is converted by biological, usually microbiological, agents into simple, environmentally acceptable derivatives. (**4**)

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[1] This guide 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, 2014. Published July 2014. Originally approved in 2001. Last previous edition approved in 2009 as D6666 – 04 (2009). DOI: 10.1520/D6666-04R14.

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

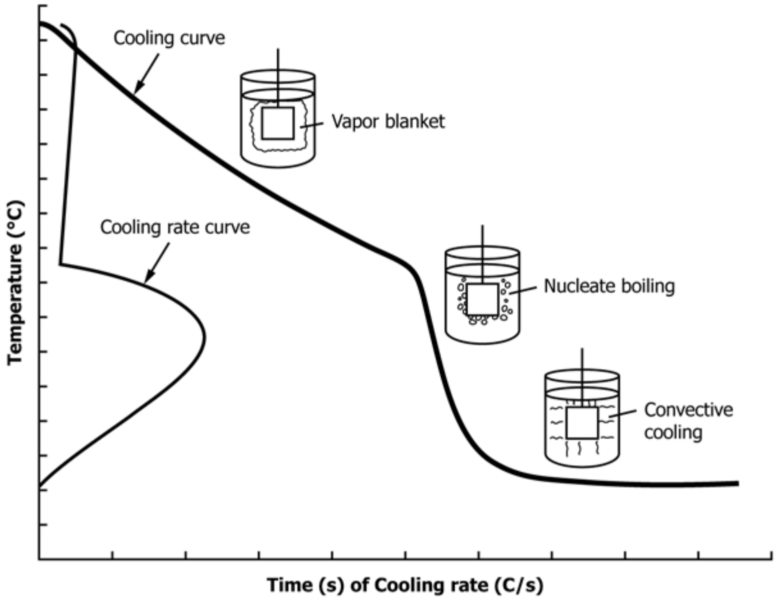


FIG. 1 Cooling Mechanisms of the Quenching Process

3.1.5 *biodeterioration, n*—loss of product quality and performance and could be regarded as the initial stages of biodegradation (see 3.1.4) , but in the wrong place at the wrong time, that is when the product is stored or in use. **(4)**

3.1.6 *convective cooling, n*—after continued cooling, and the interfacial temperature between the cooling metal and the aqueous polymer quenchant is less than the boiling point of the water in the quenchant solution at which point cooling occurs by a convective cooling process. For convective cooling, fluid motion is due to density differences and the action of gravity and includes both natural motion and forced circulation **(1, 5)**. This process is illustrated in Fig. 1.

3.1.7 *cooling curve, n*—a graphical representation of the cooling time (*t*)—temperature (*T*) response of the probe such as that shown in Fig. 1. **(5)**

3.1.8 *cooling curve analysis, n*—the process of quantifying the cooling characteristics of a quenchant medium based on the temperature versus time profile obtained by cooling a preheated metal probe assembly (see Fig. 2) under specified conditions which include: probe alloy and dimensions, probe and bath temperature, agitation rate, and aqueous polymer quenchant concentration.

3.1.9 *cooling rate curve, n*—obtained by calculating the first derivative (*dT/dt*) of the cooling time-temperature curve as illustrated in Fig. 1. **(5)**

3.1.10 *dragout, n*—solution carried out of a bath on the metal being quenched and associated handling equipment. **(1)**

3.1.11 *full-film boiling, n*—upon initial immersion of hot steel into a quenchant solution, a vapor blanket surrounds the metal surface resulting in full-film boiling as shown in Fig. 1. **(5)**

3.1.12 *nucleate boiling, n*—when the vapor blanket surrounding the hot metal collapses and a nucleate boiling process occurs as illustrated in Fig. 1. **(5)**

3.1.13 *quenchant medium, n*—any liquid or gas, or mixture, used to control the cooling of a metal to facilitate the formation of the desired microstructure and properties. **(1)**

3.1.14 *quench severity, n*—the ability of a quenchant medium to extract heat from hot metal. **(6)**

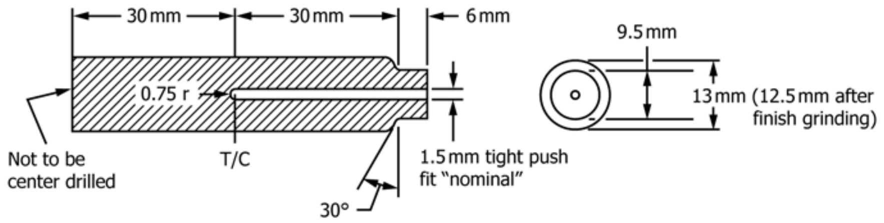
3.1.15 *transformation temperatures, n*—characteristic temperatures that are important in the formation of martensitic microstructure of steel including: A_{e1}—equilibrium austenitization phase change temperature; M_S—temperature at which transformation of austenite to martensite starts during cooling and M_f—temperature at which transformation of austenite to martensite is completed during cooling. **(1)**

4. Significance and Use

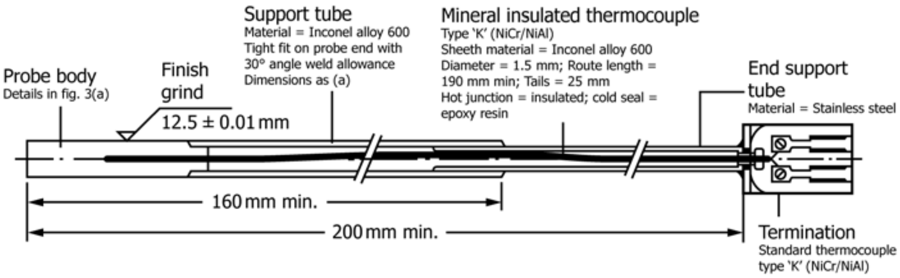
4.1 The significance and use of each test method will depend on the system in use and the purpose of the test method listed under Section 7. Use the most recent editions of the test methods.

5. Quenching Process

5.1 *Aqueous Polymer Quenchant Cooling Mechanisms*—Upon initial immersion of a heated metal into a solution of an aqueous polymer quenchant, an insulating polymer film, which controls the heat transfer rate from the hot metal into the cooler quenchant solution, forms around the hot metal which is separated by a vapor film (Fig. 3) **(7)** for the quenching process in a poly(alkylene glycol) quenchant. The overall heat transfer mediating properties of the film are dependent on both the film thickness (a function of polymer concentration) and interfacial film viscosity (a function of polymer type and bath temperature). The timing of film formation and subsequent film rupture and removal is dependent on the film strength of the polymer, agitation (both direction and mass flow), and turbulence of the polymer solution surrounding the cooling metal.



(a) Probe details



(b) General assembly

NOTE 1—From Wolfson Engineering Group Specification, available from Wolfson Heat Treatment Centre, Aston University, Aston Triangle, Birmingham B4 7ET, England, 1980.

FIG. 2 Schematic Illustration of the Probe Details and Probe Assembly

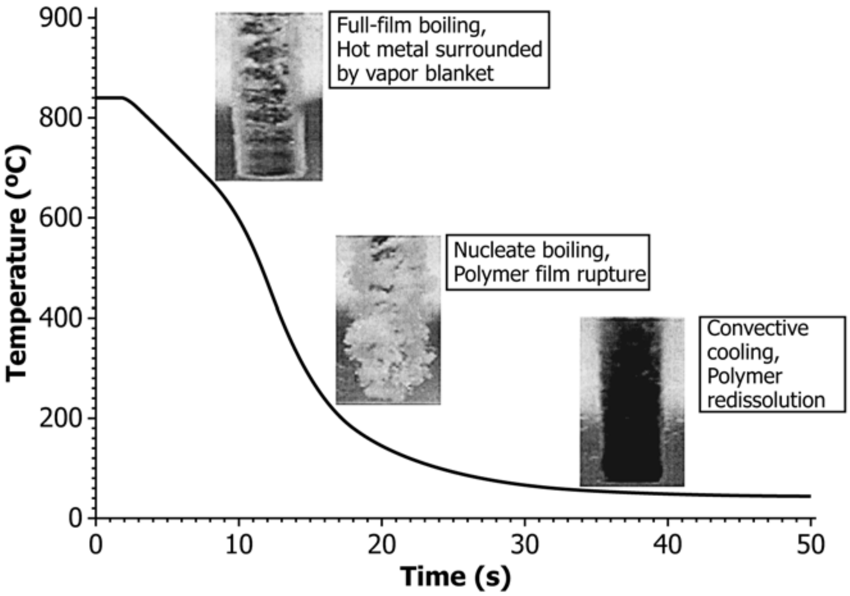
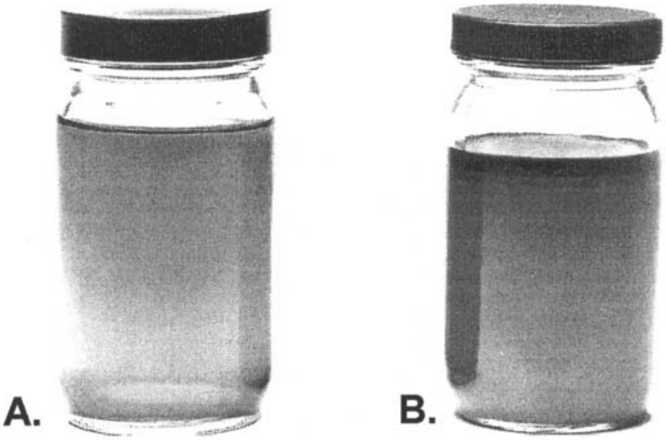


FIG. 3 Illustration of the Three Phases of Cooling

5.1.1 The cooling process that occurs upon initial immersion of the hot metal into the aqueous polymer quenchant is full-film boiling. This is frequently referred to as the vapor blanket stage. Cooling is slowest in this region. When the metal has cooled sufficiently, the polymer film encapsulating the hot metal ruptures and a nucleate boiling process results. The temperature at the transition from full-film boiling to nucleate boiling is called the Leidenfrost temperature. Cooling is fastest in this region. When the surface temperature of the cooling metal is less than the boiling temperature of water, convective cooling results. All three cooling mechanisms are superimposed on a cooling curve and illustrated in Fig. 3. (7)

6. Sampling

6.1 *Sampling*—Flow is never uniform in agitated quench tanks. There is always variation of flow rate and turbulence from top to bottom and across the tank. This means there may be significant variations of particulate contamination including



(A) New aqueous polymer quenchant solution.
(B) Used quenchant solution with oil contamination (see separated upper layer).

FIG. 4 Sample of Oil Contaminated Aqueous Polymer Quenchant

carbon from the heat treating process and metal scale. For uniform sampling, a number of sampling recommendations have been developed.

6.1.1 *Sampling Recommendations:*

6.1.1.1 *Minimum Sampling Time*—The circulation pumps shall be in operation for at least 1 h prior to taking a sample from the quench system.

6.1.1.2 *Sampling Position*—For each system, the well-mixed sample shall be taken from the same position each time that system is sampled. The position in the tank where the sample is taken shall be recorded.

6.1.1.3 *Sampling Values*—If a sample is taken from a sampling valve, then sufficient quenchant should be taken and discarded to ensure that the sampling valve and associated piping has been flushed before the sample is taken.

6.1.1.4 *Effect of Quenchant Addition as Make-Up due to Dragout*—It is important to determine the quantity and frequency of new quenchant additions, as large additions of new quenchant solution will have an effect on the test results, in particular, the cooling curve. If a sample was taken just after a large addition of new quenchant, this shall be taken into consideration when interpreting the cooling curve for this sample.

6.1.1.5 *Sampling Containers*—Samples shall be collected in new containers. Under no circumstances shall used beverage or food containers be used because of the potential for fluid contamination and leakage.

7. Recommended Test Procedures

7.1 *Performance-Related Physical and Chemical Properties:*

7.1.1 *Appearance*—Contamination of aqueous polymer quenchants by such fluids as hydraulic or quench oils may result in a non-uniform quench with thermal gradients sufficient to cause cracking or increased distortion, or possible staining, of the metal being quenched. The simplest test (and an excellent test) is to examine the appearance of an aqueous polymer quenchant in a clear glass container, such as a bottle. A sample of an oil-contaminated fluid is illustrated in Fig. 4. (**7**) However, if the oil readily separates from the aqueous polymer quenchant solution (Fig. 4), it may be removed by skimming. On the other hand, oil may form a milky-white emulsion which is not readily reclaimed by heat treaters.

7.1.1.1 Other problems that are easy to identify visually include carbon and sludge contamination which often results in cracking problems. Metal scale contamination is often identifiable by its magnetic properties by placing a magnet on the outside of the bottle next to the scale and determining if the scale exhibits any attraction for the magnet. Carbon, sludge, and scale may be removed from the quenchant by filtration or centrifugation. Alternatively, the quenchant mixture may be allowed to settle, the quenchant solution pumped off, and the separated solids then removed by shoveling. The amount of insoluble suspended solids or tramp oils may be quantified by a modification of Test Method D1796 where the aqueous quenchant is centrifuged without further dilution as described in the method. The amount of tramp oil in the quenchant is determined from the insoluble liquid layer at the top of the centrifuge tube and the volume of the insoluble sediment is taken from the bottom of the centrifuge tube.

7.1.2 *Refractive Index, (Test Method D1747)*—One of the most common methods of monitoring the concentration of aqueous polymer quenchants formulated using poly(alkylene glycol) coploymers is refractive index. As Fig. 5 (**7**) shows, there is a linear relationship between quenchant concentration and refractive index. The refractive index of the quenchant solution is determined using an Abbé refractometer (Test Method D1747) equipped with a constant temperature bath. Although the refractive index could potentially be used at any temperature within the control limits of the constant temperature bath, typically either 40°C or 100°F is selected.

7.1.2.1 Although refractive index is a relatively simple and a rapid method for determination of polymer quenchant concentration, it is not sensitive to low levels of polymer degradation and it is often significantly affected by solution contamination.

NOTE 1—Refractive index is typically unsuitable for aqueous polymer quenchants formulated with polymers with molecular weights greater than

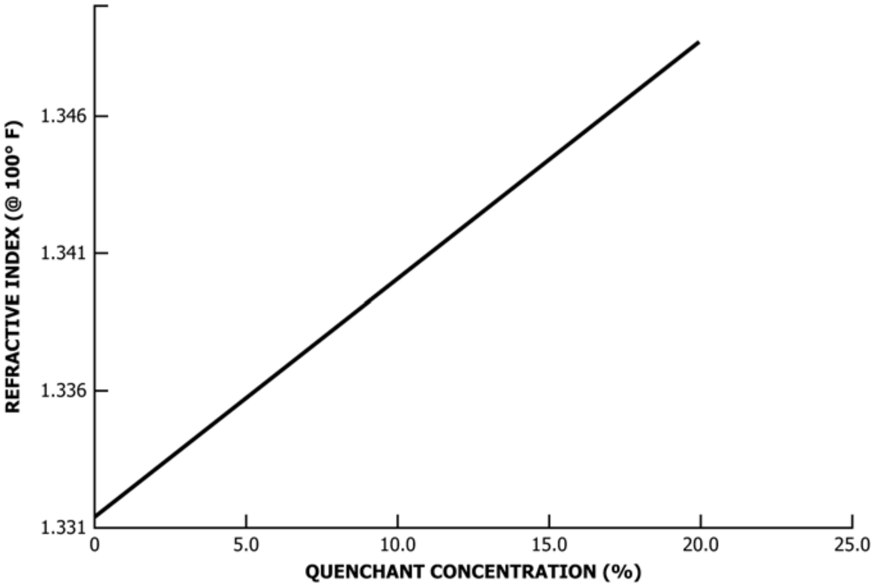


FIG. 5 Illustration of the Linear Relationship Between Refractive Index and Concentration

50 000 to 60 000 because the total concentration is relatively low. Small changes in polymer concentration may result even from normal use which impart significant process effects but the corresponding variation in refractive index may not be detectable.

NOTE 2—Although it is most desirable to use an Abbé refractometer because of its sensitivity, this is only practical in a laboratory environment. In the heat treating industry, for tankside monitoring and control, a temperature-compensated handheld refractometer (similar to the one illustrated in Fig. 6) is used. The hand-held refractometer is self-compensated for temperatures between 60 and 100ºF. Although there are various models available, the most common models provide arbitrary refractive index readings in Brix units over a 0 to 30º range. Typically, the smallest scale that can be read directly is in divisions of 0.2º as shown in Fig. 7. A concentration-refractive index curve obtained by a hand-held refractometer is shown in Fig. 8. **(7)** Hand-held refractometers are available whose scale readings correlate directly to the concentration of the polymer quenchant being used. This is particularly convenient for industrial tank-side use. However, since refractive index varies with contamination (such as dissolved salts) that may accumulate from evaporation of hard water, the actual quenchant concentration shall be verified periodically by other methods, and appropriate correction factors applied. In this case, the refractometer reading multiplied by the correction factor equals actual concentration.

7.1.3 *Viscosity, (Test Method D445)*—Aqueous polymer quenchant viscosity depends on the quenchant concentration and temperature as shown in Fig. 9. **(7)** Viscosity is readily determined using a Cannon-Fenske tube (see Fig. 10), stop-watch and constant temperature bath as described in Test Method D445.

7.1.4 *Comparison of Concentration by Refractive Index and Viscosity*—A useful procedure for monitoring variations in aqueous polymer quenchants, particularly poly(alkylene glycol) quenchants, is to compare the difference (delta) in the quenchant concentration value obtained by refractive index (C_R) and viscosity (C_V). **(8)**

$$\Delta = C_R - C_V \quad (1)$$

If the absolute value of the difference in delta is greater than 6-8, the source of this difference, contamination or degradation, should be determined.

7.1.5 *Water Content (Test Methods D95 and D1744)*—Aqueous polymer quenchants are composed of water, a water soluble polymer and an additive package to provide corrosion inhibition, foam control, and so forth. Therefore, determination of water content is necessary to establish the concentration of the quenchant in a way that is relatively insensitive to polymer degradation.

7.1.5.1 Water content may be determined by Karl Fisher analysis (Test Method D1744). The advantage of Karl Fisher analysis is that it is a direct measure of water content, whereas refractive index and viscosity are both indirect measurements that are substantially affected by either contamination (refractive index) or degradation (viscosity). In some cases, interferences may arise with Karl Fisher analysis and an alternative procedure is necessary such as distillation from toluene or some other water-insoluble solvent (Test Method D95), or evaporation.

7.1.6 *pH Determination, (Test Method E70)*—The performance of an aqueous polymer quench bath may be critically dependent on its pH. The pH of a quenchant solution may be determined by Test Method E70. There are many excellent commercially available sources of pH meters and glass electrodes. The choice of the instrument will be primarily affected by the desired precision of measurement. Electrodes used for pH measurement are designed for specific pH ranges and temperature; therefore, the solution pH and temperature shall be considered when the electrodes are selected for use.

7.1.6.1 For example, solution pH values of >10 for quenchants used in aluminum heat treating applications may be disastrous in view of potential caustic corrosion processes which may occur. The relatively simple determination of pH of an aqueous polymer quenchant may provide significant insight into potential polymer degradation, corrosion inhibitor depletion, and contamination.

7.1.6.2 Polymer degradation is typically accompanied by the formation of acidic by-products which will decrease pH.

(A) Application of the aqueous polymer quenchant to the refractometer.
(B) Visual reading of the refractometer scale to determine refractance value.

FIG. 6 Typical Hand-Held Refractometer

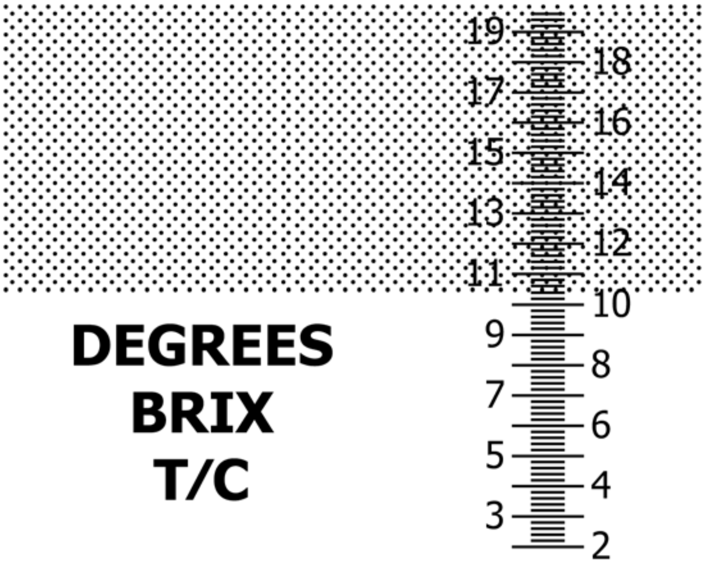


FIG. 7 Illustration of the Degrees Brix Refractive Index Scale Used for the Hand-Held Refractometer

Some polymer quenchants, particularly when containing sodium nitrite as a corrosion inhibitor, cannot be used below pH 7.0 without increasing the polymer degradation rate.

7.1.6.3 Some aqueous polymer quenchants contain amine or amine/fatty acid mixtures as corrosion inhibitors. If a substantial decrease in the concentration of these inhibitors occurs, a decrease in pH will result. Thus, pH determination may be a useful indicator of corrosion protection of some quenchants.

7.1.6.4 In some cases, the quench bath may be contaminated by ammonia which is used in some heat treatment processes. Clearly, pH is an excellent indicator of potential ammonia contamination. Quench baths may be contaminated by various

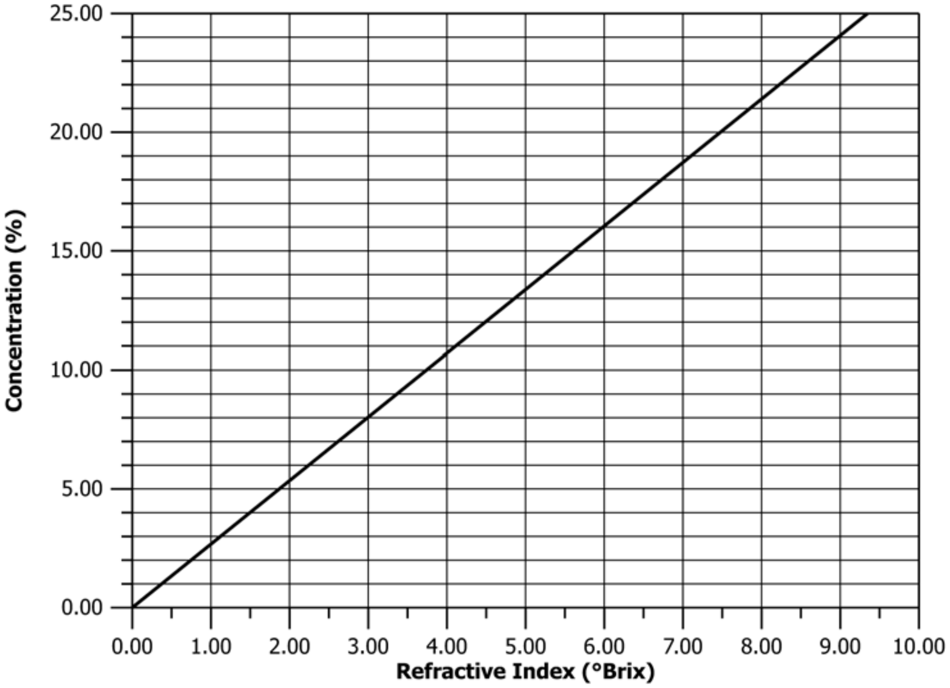


FIG. 8 Typical Refractive Index (Degrees Brix) Versus Quenchant Concentration Relationship

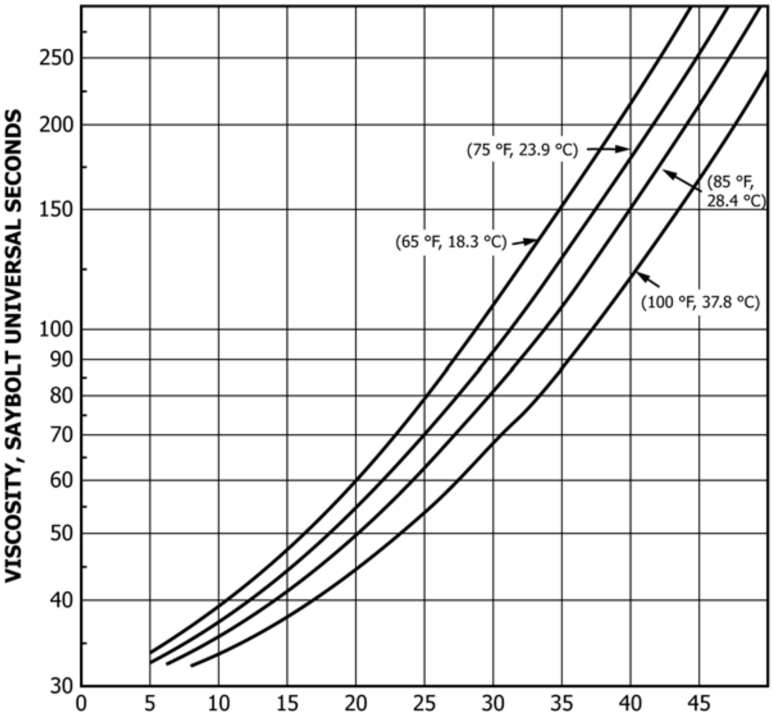


FIG. 9 Quenchant Viscosity as a Function of Concentration and Temperature

water-soluble hydraulic and metalworking fluids which may significantly affect the resulting pH of the aqueous polymer quenchant.

7.1.7 *Conductance, Test Method D2624*—One of the most common and most deleterious contaminants of an aqueous polymer quenchant is hard metal ions (Ca $^{+2}$, Mg^{+2}, Mn^{+2}, and

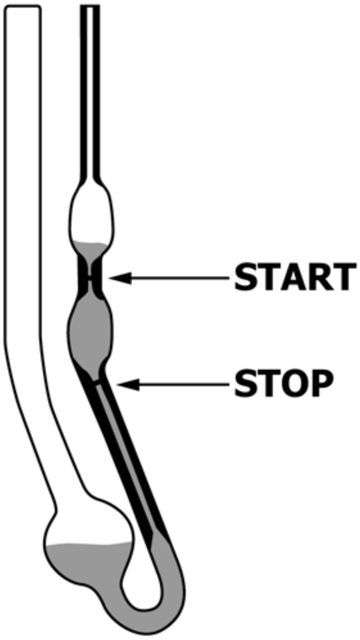


FIG. 10 Cannon-Fenske Tube

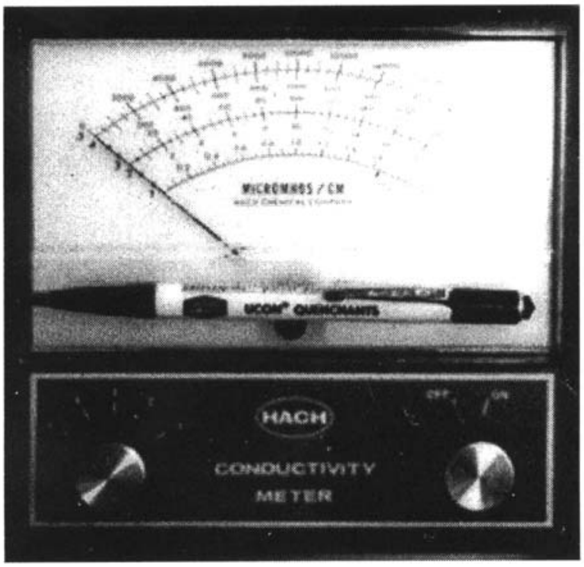


FIG. 11 Commercially Available Portable Conductivity Meter

Fe^{+3}). The presence of hard metal ions will lead to corresponding increases in cooling rates. To extend the lifetime of the quenchant, it is often recommended that either distilled or deionized water be used for initial polymer quenchant dilution; make-up water is added periodically due to normal evaporation processes. Typically, it is recommended that the conductivity of the water used for this purpose not exceed 15 µmho/cm. (**7**)

7.1.7.1 Another source of ionic contamination that will result in cooling rate increases is salts from molten salt furnaces (baths). These salts may contaminate an aqueous polymer solution by adhering to the part upon removal from the salt pot. When subsequently immersed into the aqueous polymer quenchant solution, the residual salt from on the part dissolves adding to the total ionic contamination in the quenchant solution.

7.1.7.2 Increased ionic contamination may also result if excessive corrosion inhibitor is added to the quench system.

7.1.7.3 Since metal ions may result in increased cooling rates which may potentially cause cracking of the metal due to increased thermal and transformational stresses, it is important to monitor the variation in the ionic content of an aqueous polymer quenchant. This is easily done because increasing ionic content results in increasing electrical conductance.

7.1.7.4 Any equipment capable of giving a conductivity reading almost instantaneously with the application of voltage across the two electrodes comprising the conductivity cell (described in Test Method D2624) is acceptable. A typical portable conductivity meter is illustrated in Fig. 11. The procedure followed is the same as that described in Test Method D2624, except that the aqueous polymer quenchant being analyzed is used.

7.1.8 *Separation Temperature (Cloud Point)*—Some aqueous polymer quenchants exhibit a characteristic temperature above which the water soluble polymer becomes mostly insoluble in the aqueous medium. (**9**) This reversible process, which is shown in Fig. 12, (**7**) is sometimes called the separation temperature or cloud point.

7.1.8.1 The separation temperature is determined by heating a solution of the aqueous polymer quenchant and noting the temperature where the fluid becomes sufficiently cloudy so that the thermometer is no longer visible. Although some salts may affect the separation temperature, oxidative degradation of the polymer is the most common problem. Degradation, which causes the separation temperature to rise 2 to 4°C (4 to 7°F) over the lifetime of the quenchant bath, is not unusual. A larger increase or sudden change in separation temperature is cause for concern.

7.1.9 *Corrosion Inhibitor*—Because polymer quenchants are water based, they must be formulated with a corrosion inhibitor(s). Corrosion inhibitors protect the tank, fixtures, and parts being quenched by either surface passivation or protective film formation. Depletion of the inhibitor during use is to be expected, and periodic replenishment of the corrosion inhibitor is required to maintain adequate protection of the quenching bath, parts being heat treated, and fixtures.

7.1.9.1 Many polymer quenchants use sodium nitrite as the corrosion inhibitor. The concentration of sodium nitrite may be determined according to Test Method D3867, a cadmium reduction method which may be performed manually or by an automated procedure. Alternatively, nitrite anion may be quantitatively determined by ion chromatography according to Test Method D4327.

NOTE 3—The concentration of nitrite in a quenchant may also be determined by a relatively simple color test using a commercially available test kit. A tablet, furnished with the test kit, is dissolved in a specific volume of the solution, and the resulting color of the quenchant solution is compared to a standard color chart of known concentrations of sodium nitrite as shown in Fig. 13.

7.1.9.2 Although sodium nitrite is a commonly used corrosion inhibitor in the formulation of aqueous polymer quenchants, there is an increasing trend in the marketplace for

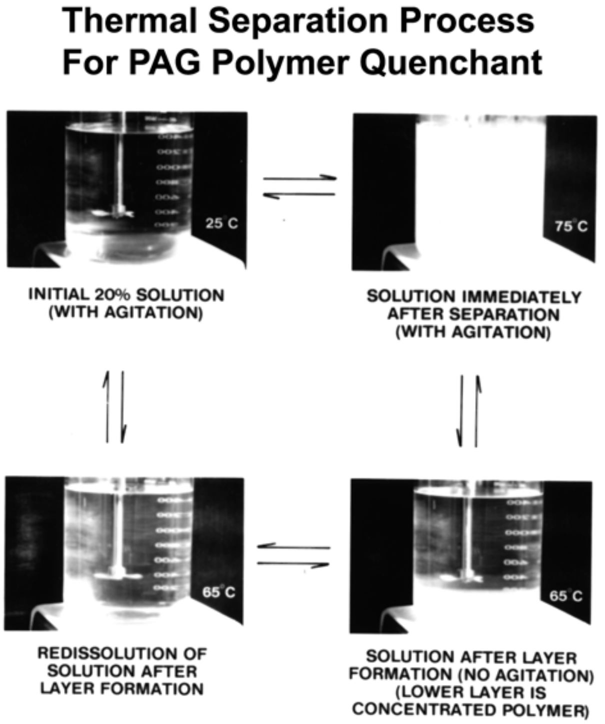


FIG. 12 Illustration of a Characteristic Reversible Separation Temperature Above Which the Aqueous Solution of a Poly(alkylene glycol) Quenchant Becomes Heterogeneous

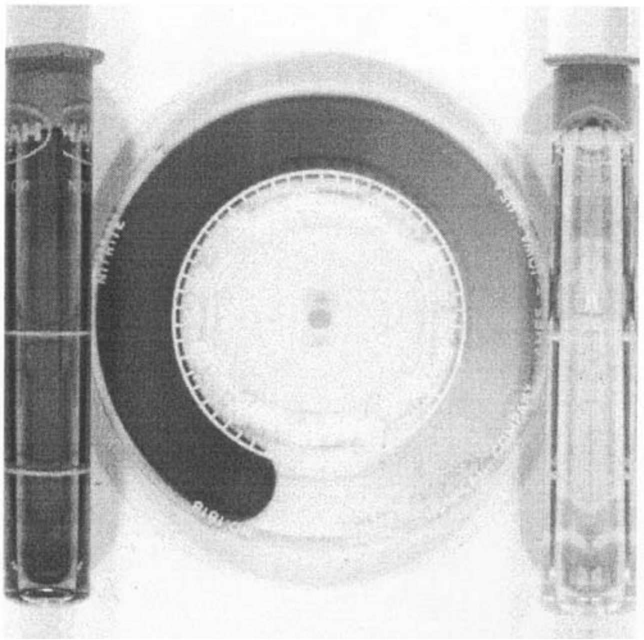


FIG. 13 Illustration of a Portable Color Test To Determine Sodium Nitrite Concentration

so-called non-nitrite quenchants. Many of the non-nitrite inhibitor systems are based on various amines or amine-fatty acid combinations. Due to the specificity of the required analytical procedures for determination of each non-nitrite inhibitor package that may be used, it is most common to submit samples periodically to the quenchant manufacturer for analysis.

7.1.10 *Foam Testing, Test Methods D892, D3519, and D3601*—A commonly encountered problem in production quench tanks is excessive foaming. Excessive foaming is detrimental because it may potentially lead to cracking or increased distortion, or both. Relative foaming propensity for one fresh quenchant, compared to another or a used quenchant compared to a fresh quenchant or two used quenchant samples, may be readily determined by various tests including Test Methods D892 (using a gas diffusion tube), D3519 (blender test), or D3601 (bottle test).

7.1.11 *Polymer Molecular Weight Analysis, Test Method D5296*—As a test for polymer degradation, gel permeation chromatography (GPC), also known as size exclusion chromatography (SEC), provides the most unambiguous results. This chromatography technique, described in Test Method D5296, separates polymers on the basis of their molecular size.

NOTE 4—Test Method D5296 may be modified for use with various nonionic polymers such as those based on poly(alkylene glycol). However, many water-soluble polymers are not readily soluble in solvents other than water. In this case, an aqueous GPC procedure, a procedure where water is used as the solvent for GPC analysis, will need to be developed.

7.1.11.1 Illustrative chromatograms for a fresh undegraded polymer from a polymer quenchant and a badly degraded polymer are superimposed in Fig. 14. **(7)**

NOTE 5—Another method of polymer degradation analysis is to compare, by superimposition, the SEC chromatogram for the fresh and used polymer for the specific the quenchant of interest (Fig. 14). **(7)** The total area under the peak for the degraded polymer is normalized to that of the area under the fresh polymer peak. The resulting *area shift* of the broader-peaked degraded polymer curve is a quantitative measure of degradation. Although the area shifts may vary widely, typically values greater than 10 % are cause for concern.

7.1.12 *Microbiological Stability:*

7.1.12.1 *Fluid Biodeterioration Processes*—Fluid biodeterioration is the result of microbial growth utilizing the additives as a substrate, such as the water-soluble polymer used to formulate an aqueous polymer quenchant, in their metabolic processes in the presence of bacteria or fungi to yield biomass. **(10)** If this degradation process is not inhibited, enormous quantities of biomass may be present in the system in the form of sludge or microbial scums, which are composed of dead cells, gelatinous slimes, and fungal threads. It has been reported that a bacterial cell may double in size and divide into two new cells every 15 min until a limiting condition is encountered. **(10)**

(*1*) In addition to solid by-products, obnoxious gases may be formed from the biodegradation of certain additives acting as microbial nutrients. For example, nitrites and nitrates which are converted to ammonia and sulfur or sulfate which is converted to hydrogen sulfide (H_2S), which exhibits a characteristic *rotten egg* odor. **(11)**

(*2*) Biodeterioration processes that occur in the presence of air (oxygen) are enhanced by system agitation and are designated as *aerobic* processes. However, biodeterioration processes may also occur without air (oxygen) being present.

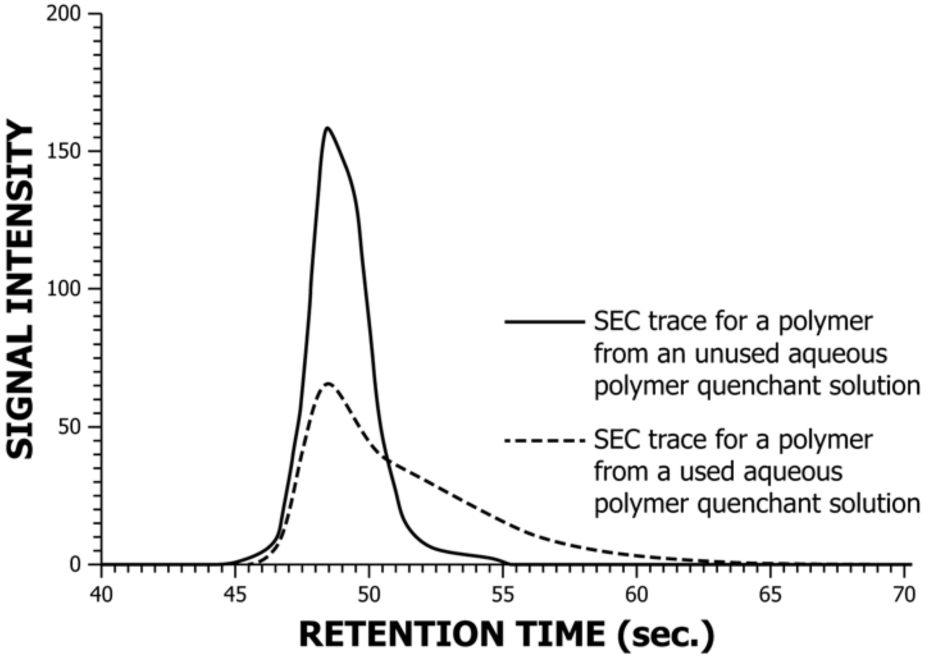


FIG. 14 Size-exclusion Chromatography Data for a Fresh and Severely Degraded Poly(alkylene glycol) Quenchant

These are called *anaerobic* processes which are inhibited by aeration provided by system agitation. Most aqueous polymer quenchants undergo biodeterioration by an anaerobic process.

7.1.12.2 *Biodeterioration Monitoring Procedures (Test Method E979 and Practice E2275)* —There are four strategies for monitoring microbial contamination: (*1*) gross, (*2*) physical, (*3*) chemical and (*4*) microbiological. Gross detection procedures include visual observation of slimes or detection of foul odors. Physical detection procedures include the observation of haze and visible, nonmetallic particulate matter in the fluid. Chemical tests that are often used include pH. Sudden decreases in pH indicate a strong potential for microbial contamination. **(12)**

(*1*) The fourth procedure is to conduct a microbial test. One test is to directly observe the microbial species on a glass slide under a microscope. **(12)** Currently, there are three standard bench test procedures that may be used for monitoring resistance to microbial growth (Test Method E979 and Practice E2275). Alternatively, a commercial dip-slide test that is coated with a microbial growth media is often used in the heat treat shop. This is called a *viable titer method*, in which the population densities of the microbial species are estimated after incubation for 24 to 72 h as illustrated in Fig. 15. Viable titer procedures may not detect microbial species that do not form colonies and, therefore, may not correlate with biodeterioration processes.

7.1.13 *Quench Severity Measurement :*

7.1.13.1 *Cooling Curve Analysis (Test Methods D6482 and D6549)*—Cooling curve analysis provides a cooling time versus temperature pathway which is directly proportional to physical properties such as hardness obtainable upon quenching of metal. Aqueous polymer quenchants are typically used with agitation and it is recommended that cooling curve analysis of this class of quenchants be performed according to Test Method D6482 (Tensi Method, see agitation device shown in Fig. 16) or Test Method D6549 (Drayton Method, see agitation device shown in Fig. 17). The results obtained by these tests may be used as a guide in quenchant selection or comparison of different quenchants or dilutions of the same quenchant, whether new or used.

(*1*) Cooling curve analysis of an aqueous polymer quenchant by Test Methods D6482 or D6549 is conducted by placing the probe assembly (Fig. 2) into a furnace and heating to 850°C (1562°F) . The heated probe is then immersed into the agitated quenchant solution at a known agitation rate and desired temperature. The temperature inside the probe assembly and cooling times are recorded at selected time intervals to establish a cooling temperature versus time curve.

(*2*) A series of cooling rate curve comparisons illustrating the effect of aqueous polymer quenchant concentration and bath temperature are illustrated in Figs. 18 and 19 respectively. The effect of agitation is illustrated in Fig. 20. The cooling curve performance of a severely degraded sample of the same poly(alkylene glycol) polymer quenchant relative to a fresh quenchant is shown in Fig. 21.

8. Keywords

8.1 cooling curve; cooling rate; cooling time; polymer quenchants

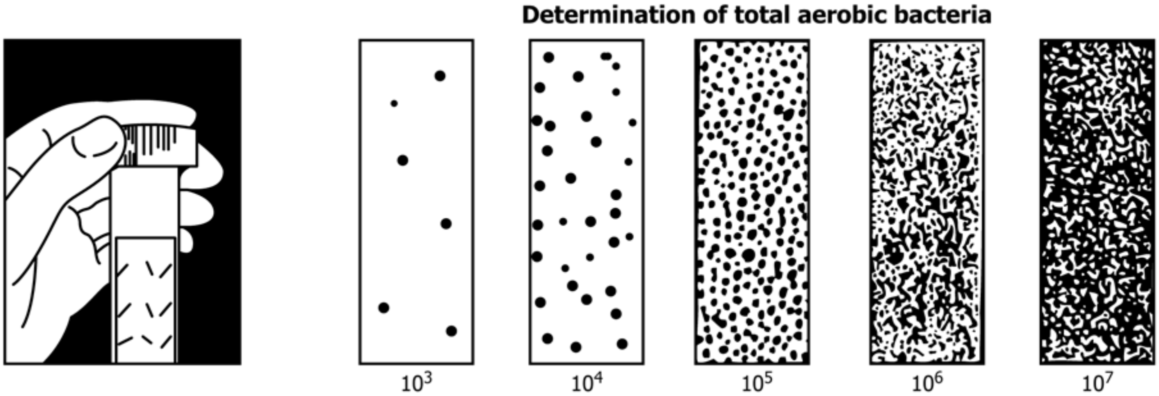


FIG. 15 Dip-Slide Test for Bacteria and Fungi Detection

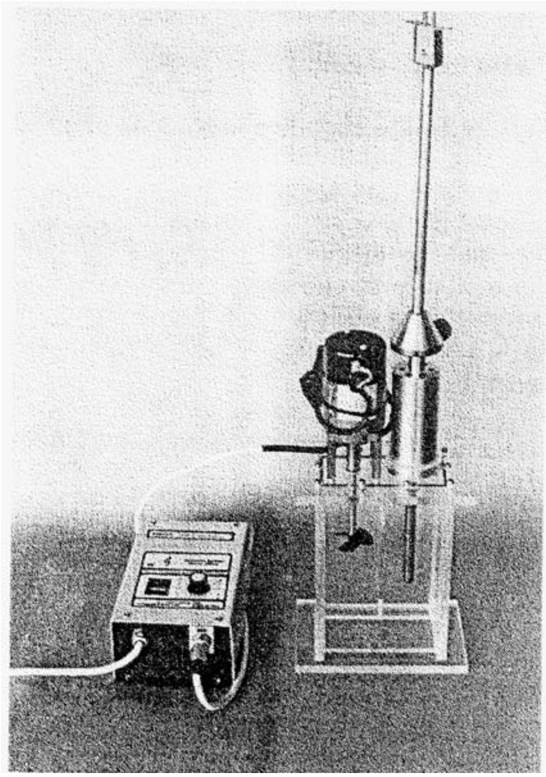


FIG. 16 Illustration of Commercially Available Quenchant Agitation Systems (Tensi Agitation System)

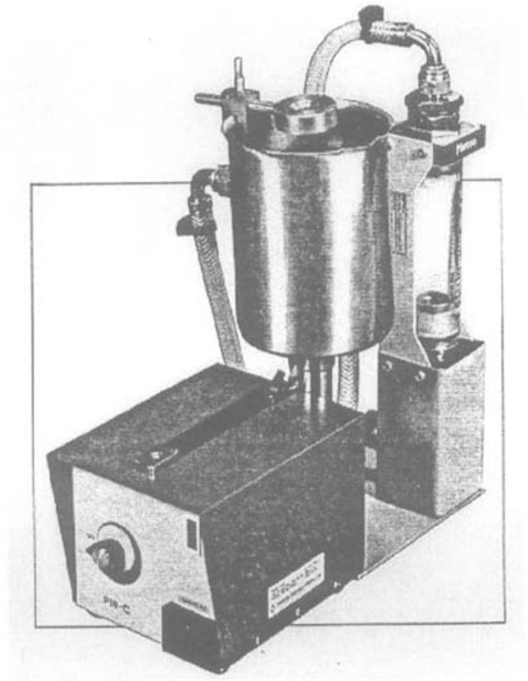


FIG. 17 Illustration of Commercially Available Quenchant Agitation System (Drayton Agitation System)

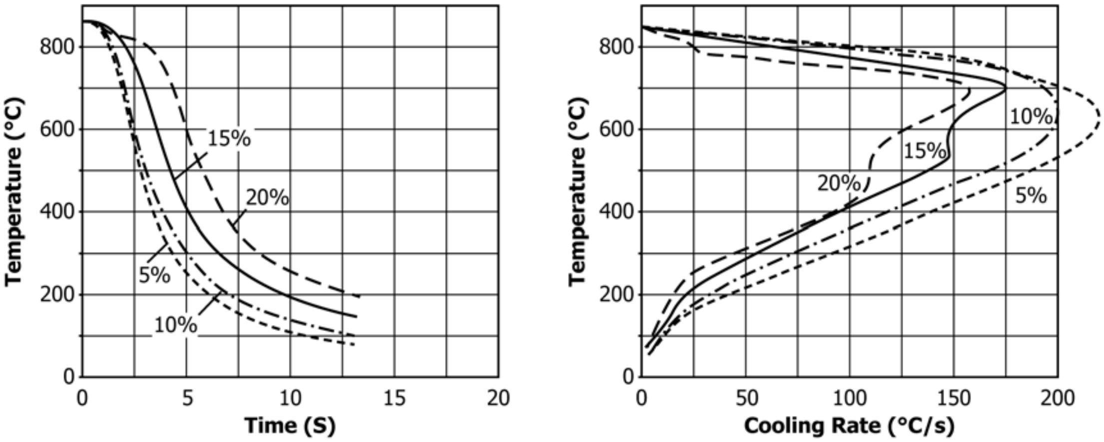


FIG. 18 Illustration of the Effect of Quenchant Concentration on Cooling Curve Performance for a Poly(alkylene glycol) Quenchant at 30°C and 0.5 m/s

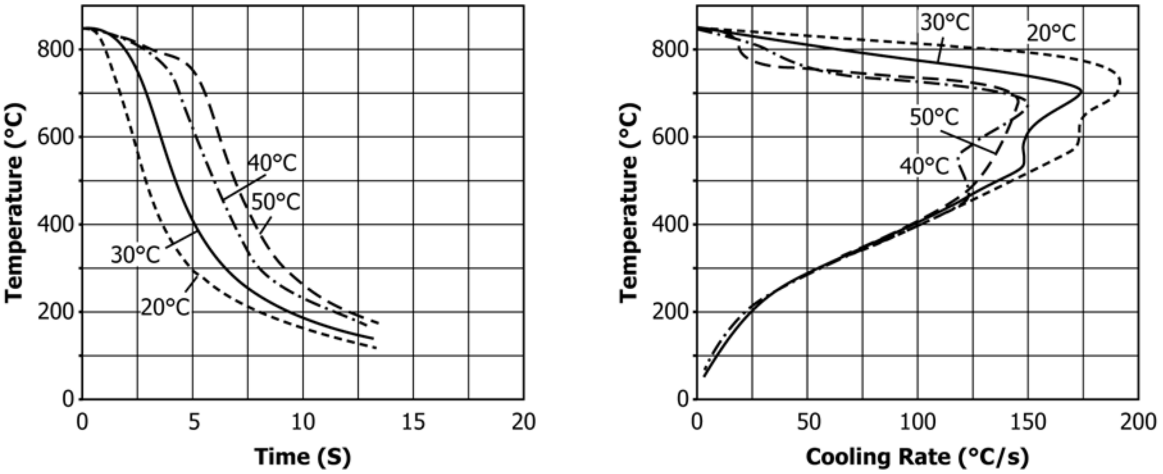


FIG. 19 Illustration of the Effect of Bath Temperature Variation on Cooling Curve Performance for 15 % Aqueous Solution of Poly(alkylene glycol) Quenchant at 0.5 m/s

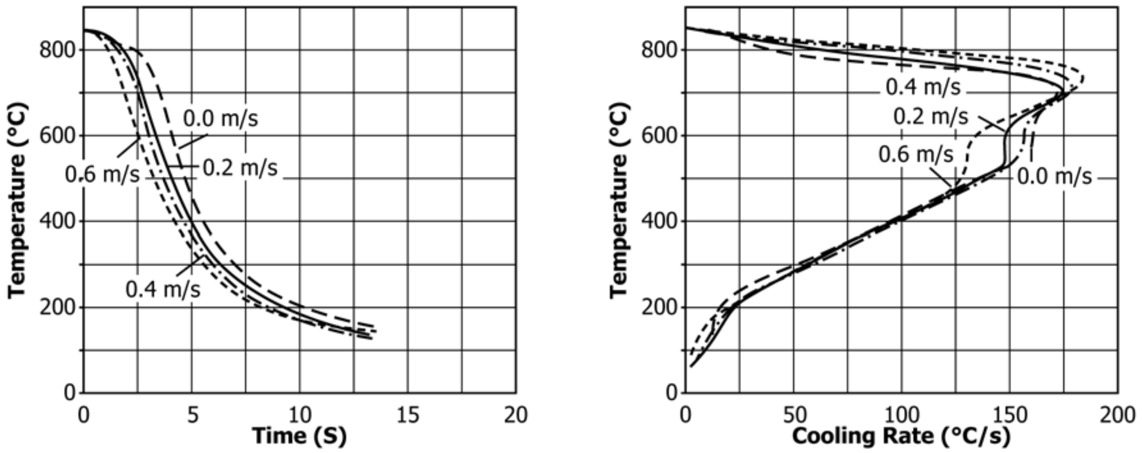


FIG. 20 Illustration of the Effect of Agitation Rate Variation on Cooling Curve Performance for 15 % Aqueous Solution of a Poly(alkylene glycol) Quenchant at 30°C

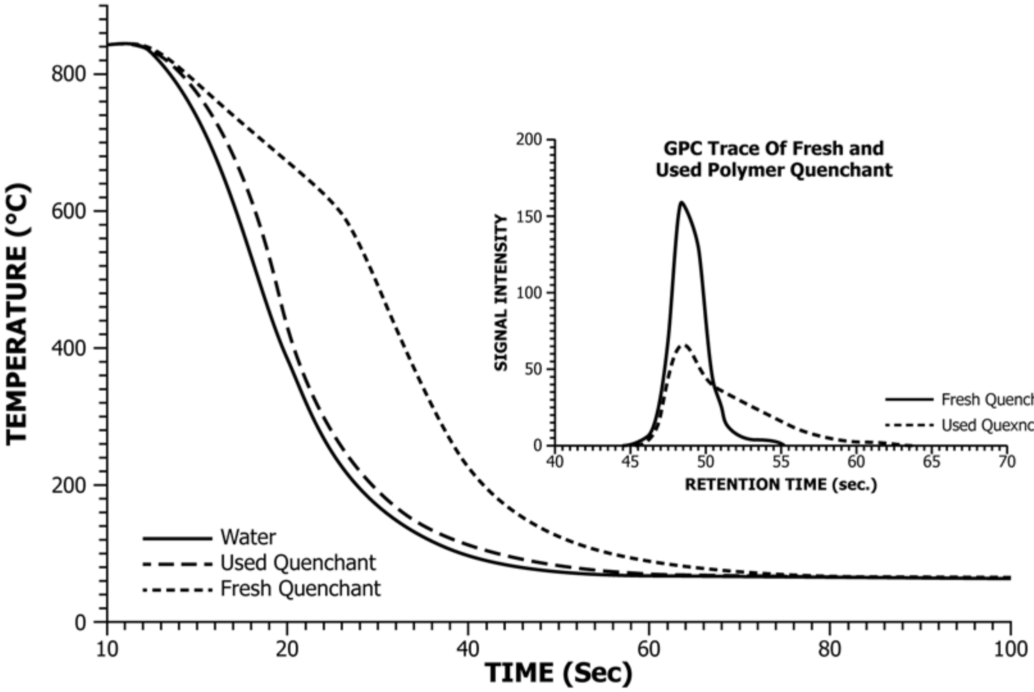


FIG. 21 Illustration of Cooling Curve Performance of a Severely Degraded Aqueous Polymer Quenchant Compared to Water and a Fresh Solution of the Same Polymer Quenchant at the Same Concentration, Bath Temperature and Agitation

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