



# Standard Guide for Evaluation of Hydrocarbon-Based Quench Oil<sup>1</sup>

This standard is issued under the fixed designation D6710; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide covers information without specific limits, for selecting standard test methods for testing hydrocarbon-based quench oils for quality and 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 limitations prior to its use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D91 Test Method for Precipitation Number of Lubricating Oils
- D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- D94 Test Methods for Saponification Number of Petroleum Products
- D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
- D189 Test Method for Conradson Carbon Residue of Petroleum Products
- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D482 Test Method for Ash from Petroleum Products
- D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products
- D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
- D974 Test Method for Acid and Base Number by Color-Indicator Titration
- D1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter

D4530 Test Method for Determination of Carbon Residue (Micro Method)

D6200 Test Method for Determination of Cooling Characteristics of Quench Oils by Cooling Curve Analysis

D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

### 2.2 ISO Standards:<sup>3</sup>

ISO 9950 Industrial Quenching Oils—Determination of Cooling Characteristics—Nickel-Alloy Probe Test Method, 1995-95-01

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

#### 3.1.1 Quench Processing:

3.1.1.1 *austenitization, n*—heating a steel containing less than the eutectoid concentration of carbon (about 0.8 mass %) to a temperature just above the eutectoid temperature to decompose the pearlite microstructure to produce a face-centered cubic (fcc) austenite-ferrite mixture.

3.1.1.2 *dragout*—solution carried out of a bath on the metal being quenched and associated handling equipment.

3.1.1.3 *martempering, n*—cooling steel from the austenitization temperature to a temperature just above the start of merrtensite transformation ( $M_s$ ) for a time sufficient for the temperature to equalize between the surface and the center of the steel, at which point the steel is removed from the quench bath and air cooled as shown in Fig. 1. (1)<sup>4</sup>

3.1.1.4 *protective atmosphere, n*—any atmosphere that will inhibit oxidation of the metal surface during austenitization, or it may be used to protect the quenching oil, which may be an inert gas such as nitrogen or argon or a gas used for a heat treating furnace.

3.1.1.5 *quench media, n*—any medium, either liquid (water, oil, molten salt, or lead, aqueous solutions of water-soluble polymers or salt-brines) or gas or combinations of liquid and gas (air at atmospheric pressure, or pressurized nitrogen, helium, hydrogen) such as air-water spray, used to facilitate the

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<sup>2</sup> 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.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

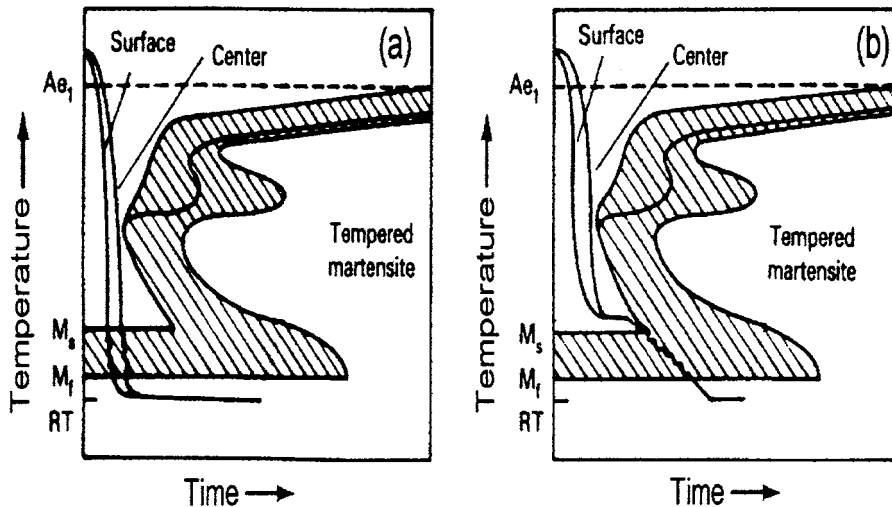


FIG. 1 (a) Conventional Quenching Cycle; (b) Martempering

cooling of metal in such a way as to achieve the desired physical properties or microstructure.

3.1.1.6 *quench severity, n*—the ability of a quenching oil to extract heat from a hot metal traditionally defined by the quenching speed (cooling rate) at 1300°F (705°C) which was related to a Grossmann H-Value or Quench Severity Factor (H-Factor). (2)

3.1.1.7 *quenching, n*—cooling process from a suitable elevated temperature used to facilitate the formation of the desired microstructure and properties of a metal as shown in Fig. 2.

3.1.1.8 *transformation temperature, n*—characteristic temperatures that are important in the formation of martensitic microstructure as illustrated in Fig. 2;  $A_{e1}$ — 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.

### 3.1.2 Cooling Mechanisms:

3.1.2.1 *convective cooling, n*—after continued cooling, the interfacial temperature between the cooling metal surface and the quenching oil will be less than the boiling point of the oil, at which point cooling occurs by a convective cooling process as illustrated in Fig. 3.

3.1.2.2 *full-film boiling, n*—upon initial immersion of hot steel into a quench oil, a vapor blanket surrounds the metal surface as shown in Fig. 3. This is full-film boiling also commonly called vapor blanket cooling.

3.1.2.3 *Leidenfrost temperature, n*—the characteristic temperature where the transition from full-film boiling (vapor blanket cooling) to nucleate boiling occurs which is independent of the initial temperature of the metal being quenched as illustrated in Fig. 4. (3)

3.1.2.4 *nucleate boiling*—upon continued cooling, the vapor blanket that initially forms around the hot metal collapses and a nucleate boiling process, the fastest cooling portion of the quenching process, occurs as illustrated in Fig. 3.

3.1.2.5 *vapor blanket cooling, n*—See *full-film boiling* (3.1.2.2).

3.1.2.6 *wettability, n*—when a heated metal, such as the probe illustrated in Fig. 5, is immersed into a quenching medium, the cooling process shown in Fig. 6 occurs by initial vapor blanket formation followed by collapse, at which point the metal surface is wetted by the quenching medium. (4)

### 3.1.3 Quench Oil Classification:

3.1.3.1 *accelerated quenching oil, n*—also referred to as a fast or high-speed oil, these are oils that contain additions that facilitate collapse of the vapor blanket surrounding the hot metal immediately upon immersion into the quenching oil, as shown in Fig. 3.

3.1.3.2 *conventional quenching oil, n*—also called slow oils, these oils typically exhibit substantial film-boiling characteristics, commonly referred to as vapor blanket cooling due to relatively stable vapor blanket formation, illustrated mechanistically in Fig. 2.

3.1.3.3 *marquenching oils, n*—also referred to as marquenching oils or hot oils, these oils are typically used at temperatures between 95 to 230°C (203 to 446°F) and are usually formulated to optimize oxidative and thermal stability by the addition of antioxidants and because they are used at relatively high temperatures, a protective or non-oxidizing environment is often employed, which permits much higher use temperatures than open-air conditions.

3.1.3.4 *quenching oil, n*—although usually derived from a petroleum oil, they may also be derived from natural oils such as vegetable oils or synthetic oils such as poly(alpha olefin). They are used to mediate heat transfer from a heated metal, such as austenitized steel, to control the microstructure that is formed upon cooling and also control distortion and minimize cracking which may accompany the cooling process.

### 3.1.4 Cooling Curve Terminology:

3.1.4.1 *cooling curve, n*—a graphic representation of the temperature ( $T$ ) versus cooling time ( $t$ ) response of a probe. An example is illustrated in Fig. 3. (5)

3.1.4.2 *cooling curve analysis, n*—process of quantifying the cooling characteristics of a quenching oil based on the time-temperature profile obtained by cooling a preheated probe assembly (Fig. 5).

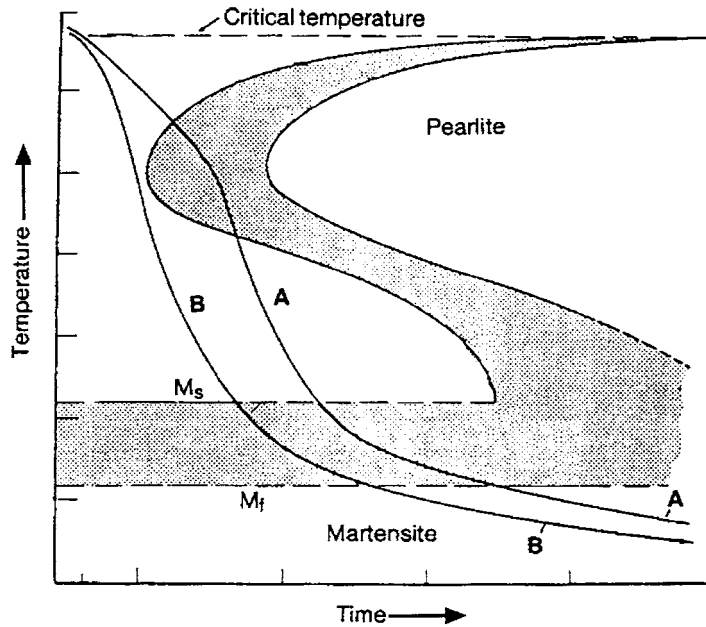


FIG. 2 Transformation Diagram for a Low-Alloy Steel with Cooling Curves for Various Quenching Media (A) High Speed Oil (B) Conventional Oil

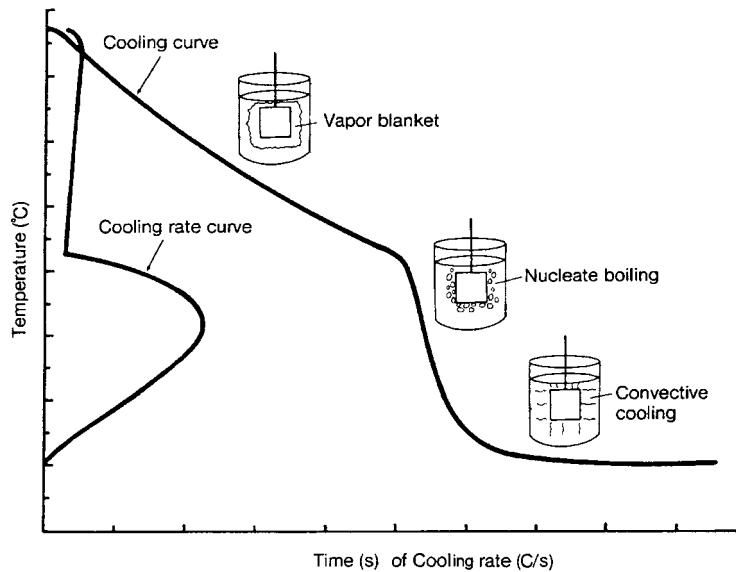


FIG. 3 Cooling Mechanisms for a Quenching Oil Superimposed on a Cooling Time-Temperature Curve and the Corresponding Cooling Rate Curve

3.1.4.3 *cooling rate curve, n*—the first derivative ( $dT/dt$ ) of the cooling time-temperature curve as illustrated in Fig. 3. (5)

#### 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 6. Use the most recent editions of the test methods.

#### 5. Sampling

5.1 *Sampling Uniformity*—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

that there may be significant variations of particulate contamination including sludge from oil oxidation and metal scale. For uniform sampling, a number of sampling recommendations have been developed.

##### 5.1.1 *Sampling Recommendations:*

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

5.1.1.2 *Sampling Position*—For each system, the sample shall be taken from the same position each time that system is sampled. The sample shall be taken at the point of maximum flow turbulence. The position in the tank where the sample is taken shall be recorded.

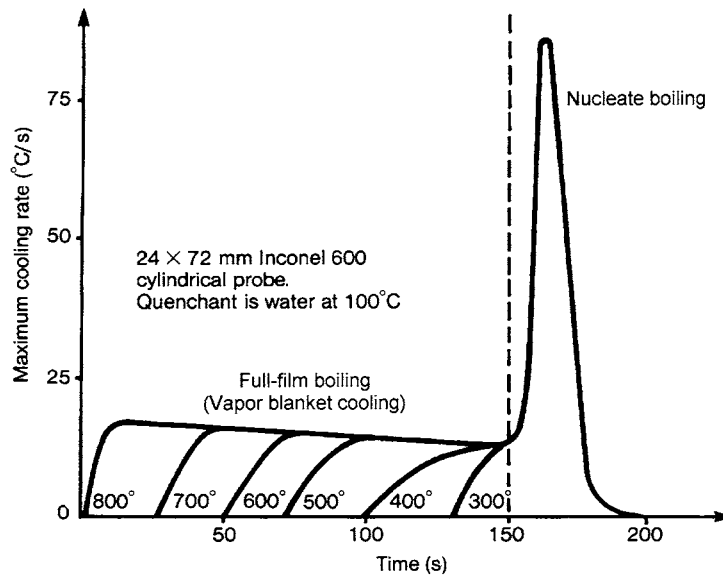
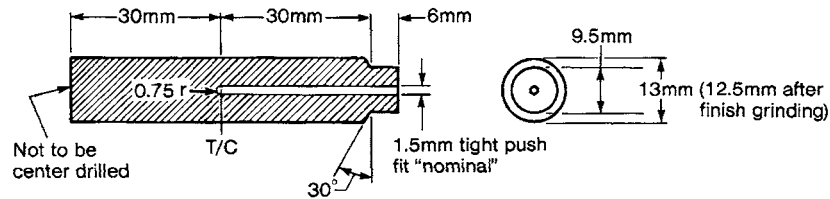
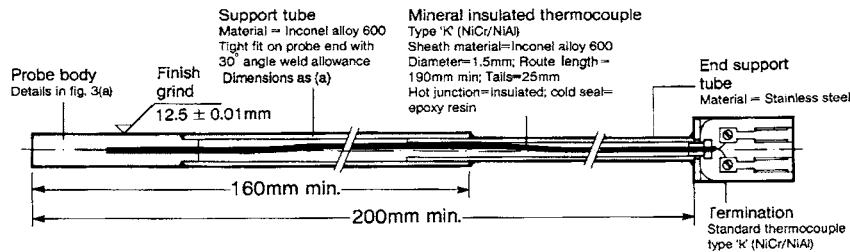


FIG. 4 Leidenfrost Temperature and its Independence of the Initial Temperature of the Metal Being Quenched



(a) Probe details



(b) General assembly

NOTE 1—Measurements are nominal. (From Test Method D6200.)

FIG. 5 Probe Details and Probe Assembly

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

5.1.1.4 *Sampling From Tanks With No Agitation*—If samples are to be taken from bulk storage tank or a quench tank with no agitation, then samples shall be taken from the top and bottom of the bulk system or quench tank. If this is not possible and the sample can only be taken from the top, then the laboratory report shall state that the results represent a sample taken from the top of the bulk system or quench tank and may not be representative of the total system.

5.1.1.5 *Effect of Quenching Oil 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 quench oil 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 quench oil, this shall be taken into consideration when interpreting the cooling curve of this oil sample.

5.1.1.6 *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.

## 6. Recommended Test Procedures

6.1 *Performance-Related Physical and Chemical Properties*:

6.1.1 *Kinematic Viscosity, (Test Method D445)*—The performance of a quench oil is dependent on its viscosity, which

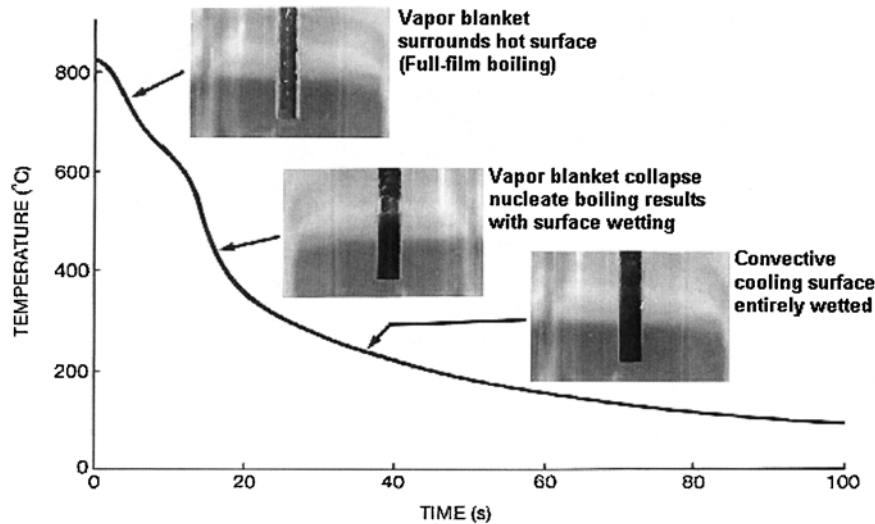


FIG. 6 Actual Cooling Process and Movement of the Wetting Front on a Metal Surface During a Quenching Process

varies with temperature and oil deterioration during continued use. Increased oil viscosity typically results in decreased heat transfer rates. (6) Oil viscosity varies with temperature which affects heat transfer rates throughout the process.

6.1.1.1 The flow velocity of a quench oil depends on both viscosity and temperature. Some quench oils are used at higher temperatures, such as martempering oils, also known as hot-oils. Although the viscosity of a martempering oil may not fluctuate substantially at elevated temperatures, the oil may become almost solid upon cooling. Thus, the viscosity-temperature relationship (viscosity index) of a quench oil may be critically important from the dual standpoint of quench severity and flow velocity.

6.1.1.2 Typically kinematic viscosity determination by Test Method D445 is used. Viscosity measurements are made at 40°C (104°F) for conventional or accelerated oils and also at 100°C (212°F) for martempering oils.

6.1.2 Flash Point and Fire Point (Test Method D92)—Use of a quench oil in an open system with no protective atmosphere shall be at least 60 to 65°C lower than its actual open cup flash point to minimize the potential for fire. General guidelines have been developed for use-temperatures of a quench oil relative to its flash point.

NOTE 1—There are various manufacturer-dependent guidelines for relating the suitability for use of a used quenching oil with respect to its flash point and they shall be followed. In the absence of such guidelines, it is recommended that the use temperature of a quenching oil in an open system with no protective atmosphere shall be more than 60 to 65°C (140 to 149°F) below its actual open-cup flash point. In closed systems where a protective atmosphere is used, the use temperature of the used quenching oil shall be at least 35°C (95°F) lower than its actual open-cup flash point.

6.1.3 Density (Test Methods D1298 and D4052)—The density of materials of similar volatility is dependent on the chemical composition, and in the case of quenching oils, the type of basestock used in formulation. The oxidative stability of quenching oils is also dependent on similar chemical composition trends, and thus density (or relative density) is an

indirect indicator of oxidative stability. Density (or relative density) is measured at, or converted to, a standard reference temperature, normally either 15°C or 60/60°F, and these should be quoted alongside the result.

6.1.3.1 Test Method D1298 uses a hydrometer plus thermometer for measurement while Test Method D4052 uses a digital density meter based on an oscillating U-tube.

NOTE 2—Density or relative density are of limited value in the assessment of quality of a quenching oil.

6.2 Aged Fluid Properties—In addition to significant changes in fluid viscosity, oil degradation by thermal and oxidative processes may result in the formation of undesirable levels of volatile by-products, sludge formation, metal-staining products and particulates, all of which may result in loss of control of the quenching process.

6.2.1 Acid Number (Test Methods D664 and D974)—Quench oil oxidation results in the formation of carboxylic acids and esters. These by-products are similar to compounds that may be used as rate accelerating additives. These acids and esters significantly affect the viscosity and viscosity-temperature relationship of the oil, which in turn affect quench severity. Carboxylic acids may also act as wetting agents and increase the quench rate by increasing the wettability of the quench oil on the metal surface. (7)

6.2.1.1 Oxidation of the oil may be monitored by tracking changes in the acid number. Because the fresh oil may be either alkaline or acidic, depending on the additives present, the absolute value of the acid number itself is not indicative of quality. However, changes in the acid number from the initial condition may be used to indicate the degree of oxidation. Increasing acid numbers generally indicate increasing amounts of aforementioned by-products. The acid number is determined by titrating the acidity of a sample of known size with a known amount of standard base (Test Methods D664 or D974. The test is performed by dissolving the oil in a mixture of toluene and isopropanol,) to which has been added a small amount of

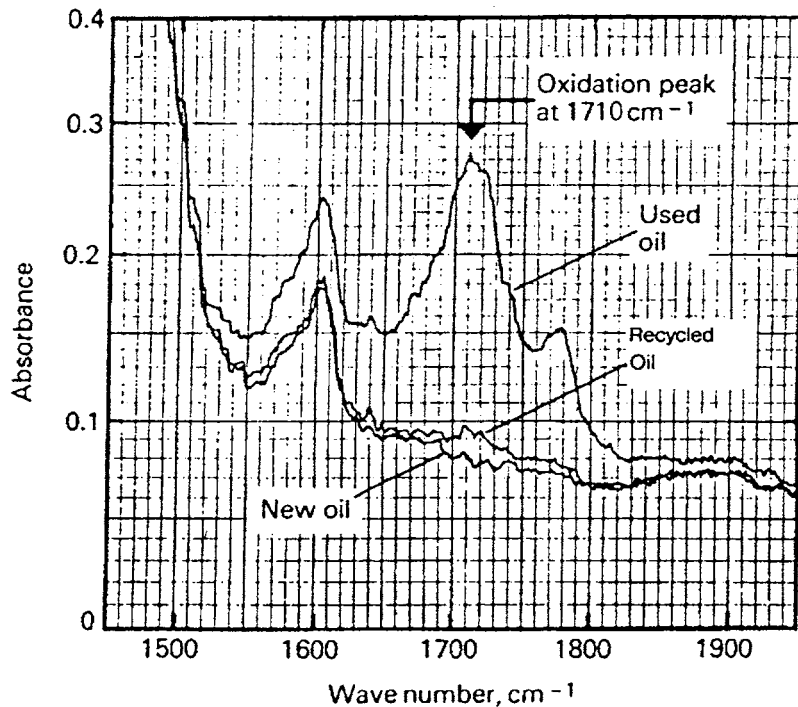


FIG. 7 Infrared Spectral Identification of Oxidation of a Used Quenching Oil

water, then titrating it with a standard solution of potassium hydroxide (KOH). The endpoint may be determined colorimetrically with a pH-sensitive indicator. The acid number (AN) is reported in units of milligrams of KOH per gram of sample (mg/g).

NOTE 3—The quenching oil supplier will recommend a maximum limit for used oil AN value for the quenching oil being used. In the absence of such a value, it is recommended that the AN not exceed 2.00 mg KOH/g for a used quenching oil.

6.2.2 *Infrared Spectroscopy*—An alternative method that is being used increasingly to identify and quantify oil oxidation, even in the presence of additives, is infrared (IR) spectroscopy. (8) Fig. 7 provides an illustration of the use of IR spectral analysis to identify oil oxidation. (9) Mang and Jünemann monitored the IR stretching vibrations of C=O at 1710 cm<sup>-1</sup>, for carboxylic acids contained in oxidized oil. IR analysis has been used to detect and quantify other carbonyl-containing compounds: (10)

- Metal carboxylate salts—1600 and 1400 cm<sup>-1</sup>
- Carboxylic acids—1710 cm<sup>-1</sup>
- Metal sulfates—1100 and 1600 cm<sup>-1</sup>
- Esters—1270 and 1735 cm<sup>-1</sup>

NOTE 4—These values for infrared vibrational frequencies for oxidized oil should be considered as illustrative examples since these frequencies may vary somewhat, depending on the chemical structure of the component being oxidized. There are a number of authoritative references that may be consulted to confirm these frequencies for oxidized lubricating oils, including (11,12).

6.2.3 *Saponification Number (Test Method D94)*—Oil degradation may produce both acids and ester by-products. The acid number quantifies the amount of acidic degradation by-products in the oil, whereas the saponification number is a measure of the presence of esters or fatty esters in the oil. The

saponification number of an oil is determined (Test Method D94) by heating a sample of the oil with a known amount of basic reagent and measuring the amount of reagent consumed. Because some quench oils are formulated with components that also have saponification numbers, it is necessary to monitor trends over time than to rely on an absolute value. (13) An increase in the acid number and the saponification number indicates an increased propensity to sludge formation. It has been suggested, that if the results of other tests are satisfactory, that saponification numbers below 3 mg KOH/g oil may be acceptable. (14)

6.3 *Contamination:*

6.3.1 *Water Content (Test Method D6304)*—The presence of water in a quench oil, which may be present due to condensation or a leaking heat exchanger, presents a potentially serious problem. Water concentrations as low as 0.1 % may cause the bath to foam during the quenching process, greatly increasing the risk of fire. Overflowing oil from the foaming bath may result in a more serious fire than if the flames were contained by the bath, as the oil may contact nearby furnaces or other ignition sources. If a sufficient amount of water accumulates in a hot bath, an explosion caused by steam generation may result. (15)

NOTE 5—The problem of water contamination in the quench bath, with respect to foaming, is illustrated in Fig. 8 where it is shown that 1 mL of water becomes 1700 mL of vapor when vaporized at atmospheric pressure (near instantaneously).

NOTE 6—The amount of foaming that does occur is often dependent on the degree of agitation. Some baths may be agitated to the point where the quenching oil is nearly splashing on the floor. In such baths, the water vapor is released even faster causing a greater potential foaming problem.

6.3.1.1 The presence of water in a quench oil may also produce variable cooling properties depending on the nature

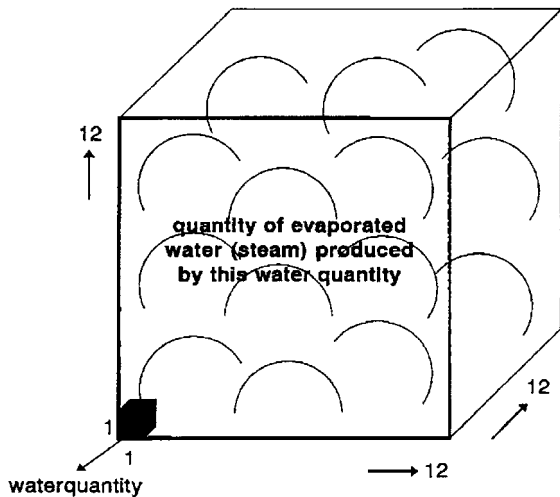


FIG. 8 Volumetric Expansion of 1 mL of Liquid Water to 1700 mL of Water Vapor

and amounts of cooling rate-accelerating additives present in the oil. The magnitude and direction of these effects depend on the particular quench oil and the amount of water present in the oil. Water contamination may also result in staining of the part being quenched, uneven hardness, and soft spots. A quantitative test for water contamination below 1000 mg/kg (0.1 mass %) is Test Method D6304, which uses the reaction of water with Karl Fisher chemical reagent as its basis, and a coulometric end point as the measurement. Higher water contents can be quantified by distillation Test Method D95.

NOTE 7—A common qualitative field test for water-induced foaming is the so-called “*crackle test*” which is conducted by heating a sample of the quenching oil and listening for an audible crackling sound. (14) If the oil is contaminated with sufficient water to potentially cause foaming of hydrocarbon-based quench oils, a crackling sound will be heard before the quenching oil has reached its smoke-point. Applicability of the “*crackle test*” for other quench oil properties has not been established.

6.3.2 *Carbon Residue (Test Methods D189, D524, and D4530)*—One of the greatest problems encountered when using a quenching oil is the formation and accumulation of sludge. Although the various analyses procedures including viscosity, neutralization number, and saponification number may indicate that a quench oil is adequate for continued use, the amount of sludge buildup in the tank may demand that the system be drained and cleaned. Cleaning and sludge disposal are growing problems for the heat treating industry. Therefore, determination of the sludge-forming potential of a quench oil prior to use is important.

6.3.2.1 One contributor to the sludge forming potential of quenching oils is the carbon residue which consists of controlled pyrolyzed material after combustion in insufficient oxygen for complete conversion. Test Methods D189, D524 and D4530 use different procedures for achieving this controlled pyrolysis, and give slightly different results, which can be interrelated. Test Method D189 uses a crucible, gas burner, and specially designed cover and hood, whereas Test Method D524 uses a glass ampoule heated in a metal block. Test Method D4530 uses a small vial in a carousel heated under a fixed flow of nitrogen.

NOTE 8—All carbon residue results are affected by the presence of inorganic additives which may be present in finished quenching oil formulations.

NOTE 9—In some heat treating operations, steel is austenitized in air which causes the increased formation of metal oxide scale which will act as a contaminant in the oil. If this occurs, the Conradson carbon residue number may be abnormally high and misleading.

6.3.3 *Precipitation Number (Test Method D91)*—Sludge formation in a quenching oil is caused by oxidation of various components in the oil, leading to polymerization and cross-linking reactions. These cross-linked and polymerized by-products are sufficiently high in molecular weight to cause them to be insoluble in the oil. Besides oil oxidation, other sources that contribute to sludge are dirt, carbon residue formation, and soot from the heat treating furnace. It is important to maintain the particle sizes in the quench oil to  $< 1 \mu$  to optimize quenching performance. (16)

6.3.3.1 Sludge can plug filters and foul heat-exchanger surfaces. The loss of heat-exchanger efficiency may result in overheating of the quenchant and possibly a fire. (17) Increasing sludge formation often indicates oxidation of the oil. In addition, sludge may adsorb on a part, causing nonuniform heat transfer during the quenching process.

6.3.4 *Particulate Contamination*—It is important to maintain particulate contamination to  $< 1 \mu$  by adequate filtration procedures to optimize quenching performance.

NOTE 10—Precipitation numbers of  $> 0.5 \%$  are typically associated with an acid number of  $> 0.5 \text{ mg KOH/g}$  by Test Method D974.

6.3.4.1 Sludge formation may be accompanied by increased volatile oxidation by-product formation which may cause a simultaneous increase in fire potential. The viscosity of a quench bath also changes with the formation of sludge, affecting both heat transfer and quench severity.

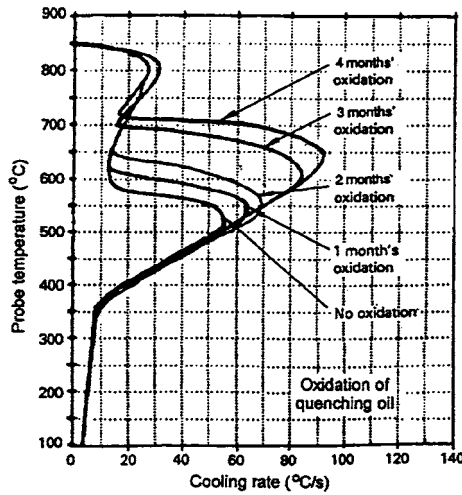
6.3.4.2 One method to measure sludge forming potential of a quench oil is to determine the precipitation number (Test Method D91). The precipitation number of the oil is measured by adding naphtha solvent to the oil sample and determining the volume of the precipitate (sludge) after centrifuging. Precipitation numbers as low as 0.2 % may produce staining of normally bright surfaces. However, staining is more commonly observed with a precipitation number of  $> 0.5 \%$ .

6.3.5 *Ash Content (Test Method D482)*—Although mineral oil basestock possess very low ash values, many formulated quench oils contain metallic components which contribute to ash. If the ash content in a bath filled with a formulated quenching oil is decreasing, it is likely that an ash-containing additive is being removed by dragout or some other process. If the ash content is increasing, the additive is either accumulating in the bath or metallic contamination is increasing, perhaps in the form of scale accumulation.

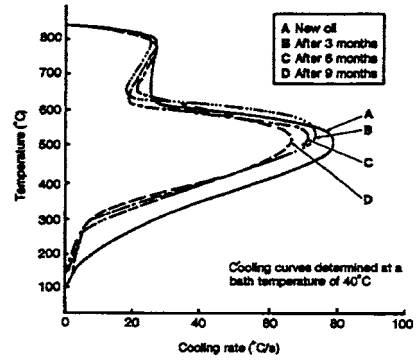
6.3.5.1 Ash contents are determined by Test Method D482 which involves heating a quenching oil in a muffle furnace at  $775^\circ\text{C}$  ( $1427^\circ\text{F}$ ) under conditions that burn off organic compounds but leave metallic species such as metal oxides or hydroxides.

#### 6.4 *Quenching Properties:*

6.4.1 *Cooling Curve Analysis (Test Method D6200 and ISO 9950)*—The most common method in use throughout the world



A.



B.

FIG. 9 Illustration of the Effect of Oil Oxidation on the Cooling Rate Curve (A) Conventional Quenching Oil (B) Accelerated Quenching Oil

to evaluate the cooling properties of a quenching oil is cooling curve analysis. 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. The results obtained by this test may be used as a guide in heat treating oil selection or comparison of quench severities of different heat treating oils, new or used.

6.4.1.1 Cooling curve analysis of a quenching oil, according to Test Method D6200 and ISO 9950, is conducted by placing the probe assembly illustrated in Fig. 5. into a furnace and heating to 850°C (1562°F). The heated probe is then immersed into at least 700 mL of the quenching oil, typically at 40°C, or other preferred temperature. The temperature inside the probe assembly and cooling times are recorded at selected time

intervals to establish a cooling temperature versus time curve. From the temperature-time curve, the cooling rate is derived.

6.4.1.2 A series of cooling rate curve comparisons illustrating the effect of oil oxidation on a conventional quench oil and an accelerated quenching oil are illustrated in Fig. 9. The effect of water contamination on a conventional quenching oil and an accelerated quenching oil are illustrated in Fig. 10. The maximum cooling rate will shift in proportion to the water content of the oil.

## 7. Keywords

7.1 cooling curve; cooling rate; cooling time; oxidation; quenching oils; water contamination



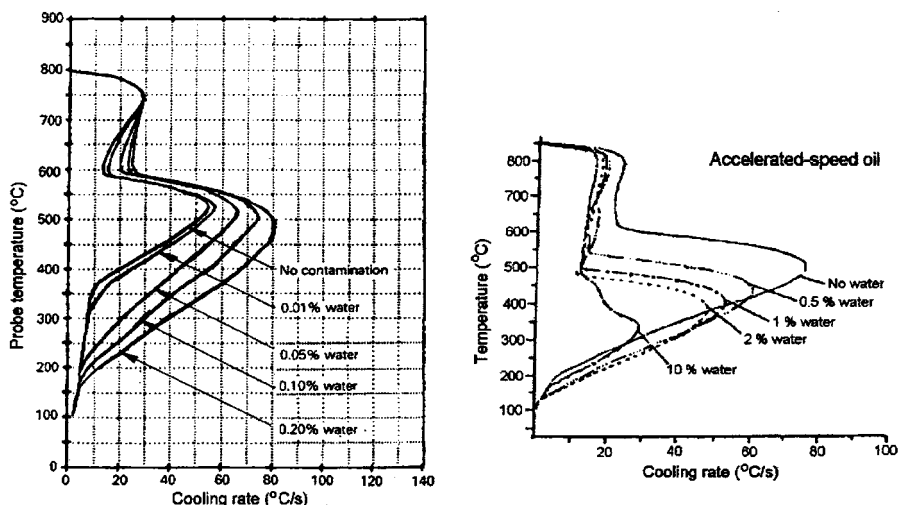


FIG. 10 Illustration of the Effect of Water Content on the Cooling Rate Curve (A) Conventional Quenching Oil (B) Accelerated Quenching Oil

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