

# **QUENCHANT FUNDAMENTALS: QUENCH OIL BATH MAINTENANCE**

By

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## **ABSTRACT**

It is essential that periodic analysis of quench oils be conducted to assure optimal performance. A quench oil testing strategy is discussed which includes various recommended and optional procedures. Following these recommendations will provide the heat treater with more uniform hardening, reduced distortion and cracking and significant improvements in operational safety.

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## **INTRODUCTION**

There are two primary functions of a quench oil. These include facilitation of the hardening of steel by controlling heat transfer during quenching and to enhance the wetting of steel during quenching to minimize the formation of undesirable thermal and transformational gradients which may lead to increased distortion and cracking.

The cooling processes encountered when cooling hot metal are illustrated in Figure 1 [1]. A vapor blanket surrounds the hot metal when it is first immersed into the oil. The stability of the vapor layer, and thus the ability of the oil to harden steel, is dependent on surface irregularities of the metal, the presence of oxides, surface wetting additives which accelerate the wetting process and destabilize the vapor blanket, and the molecular composition of the quench oil, including the presence of more volatile oil degradation by-products.

Upon further cooling, the vapor blanket collapses resulting in nucleate boiling which is the fastest heat transfer region. The point where this transition occurs and the rate of heat transfer in this region is dependent on the overall molecular composition of the oil.

When the temperature of the hot oil-steel interface is less than the boiling point of the oil, nucleate boiling will cease and convective cooling will begin. Heat transfer in this region is exponentially dependent on the viscosity of the oil which will vary with the degree of oil decomposition. Increasing oil

decomposition will result initially in a reduction of oil viscosity followed by increasing viscosity as the degradation process increases. Heat transfer rates decreases with lower viscosities and decreases with increasing viscosity.

Oil degradation is often accompanied by the formation of sludge and varnish. These by-products typically adsorb non-uniformly on the surface of the steel as it is being quenched. This will result in corresponding surface cooling rate variations and increased thermal gradients.

Another source of non-uniform heat transfer is quench oil contamination. For example, water may be introduced to the quench oil through a leak in the heat exchanger. Water, since it is not compatible with oil and possess different physical properties such as viscosity and boiling point will result in corresponding increases in thermal gradients.

To assure optimal quench process control, it is essential that quality variations throughout the lifetime of the oil be monitored and controlled. This is accomplished with quench bath maintenance procedure. This paper will outline a suggested maintenance procedures that will provide the necessary process quality controls.

## **DISCUSSION**

### **Physical Property Characterization**

There are numerous specific physical property characterization procedures that may be used. The objective here

is to provide a representative examples of testing procedures that may be used and to provide some insight into the meaning of the results obtained.

**Viscosity** - As discussed above, quenching performance of a quench oil is dependent on the viscosity of the oil. Due to degradation, oil viscosity changes with time. For process monitoring and the potential necessity of trouble-shooting, the heat treater should develop an historical record of viscosity variation in his tank such as the chart shown in Figure 2 [2].

**Water Content** - Water, from oil contamination or degradation, may cause soft spots, uneven hardness, and staining, perhaps worst of all, fires! If water-contaminated oil is heated a crackling sound may be heard. This is the basis of a qualitative field test for the presence of water in a quench oil. The most common laboratory tests for water contamination is either Karl Fisher analysis (ASTM D 1744) or by distillation. Automated detectors should be used with caution since the sensitivity levels of many of these instruments (>0.5%) are higher than allowed contamination limits of the quench oil.

**Flash Point** - The flash point is the temperature where the oil in equilibrium with its vapor produces a gas which is ignitable but does not continue to burn when exposed to a spark or flame source. There are two types of flash point values that may be determined: closed-cup or open-cup. In the closed-cup measurement, the liquid

and vapor are heated in a closed system. Traces of low-boiling contaminants may concentrate in the vapor phase resulting in a relatively low value. When conducting the open-cup flash point, the relatively low boiling by-products are lost during heating and have less impact on the final value. The most common open-cup flash point procedure is the "Cleveland Open-Cup" procedure described in ASTM D 92. The minimum flash point of an oil should be 90°C (160°F) above the oil temperature being used.

**Neutralization Number** - As an oil degrades, it forms acidic by-products. The amount of these by products may be determined by chemical analysis. The most common method is neutralization number. The neutralization number is determined by determining the net acidity of the same with a known amount of standard base such as potassium hydroxide (KOH). This is known as the "acid number" and is reported as milligrams of KOH per gram of sample (mg/g).

Oil oxidation may also be monitored and detected by infra-red spectroscopy. Figure 3 illustrates the spectral changes that occur after oil degradation.

**Sludge Formation** - One of the greatest problems encountered with quench oils is sludge formation. Although the various analyses above may indicate that a quench oil may be used, the presence of sludge may still be sufficient to cause non-uniform heat transfer, increased thermal gradients and increased cracking and distortion. Sludge may also plug filters and foul heat-exchanger surfaces. The loss of heat-exchanger efficiency may cause overheating, excessive

foaming and possible fires.

Sludge formation is caused by oxidation of the quench oil. The oxidation reactions lead to polymerized and cross-linked molecules which are insoluble in the oil. The relative amount of sludge present in a quench oil may be quantified by the "precipitation number". The precipitation number by ASTM D 91 is determined by the addition naphtha to the oil and determining the precipitate volume after centrifuging.

The relative propensity of sludge formation of a new and used oil may be compared providing an estimate of remaining lifetime. Experimental procedures that may be used include: Conradson carbon number, hot-panel coker test and the rotary bomb oxidation test.

**ICP Analysis** - When organometallic additives such as metal salts as quench rate accelerators, their potential loss by processes such as degradation and drag-out can be quantified by performing a direct analysis for the metal. One of the most common procedures is ICP (induction coupled plasma) spectroscopy. A typical ICP analysis report for a metal salt containing quench oil is shown in Figure 4.

### **Cooling Rate Characterization**

**GM Quenchometer** - One of the oldest tests to quantify the quench severity of an oil is the GM Quenchometer ("Nickel Ball") test which is conducted according to AMS D 3520 (see Figure 5). In this test, a 7/8 in. (22 mm) nickel ball is heated to 885°C (1625°F) and

then dropped into a wire basket suspended in a beaker containing 200 mL of the quench oil at 21-27°C (70-80°F). A timer is activated as the glowing nickel ball passes a photoelectric sensor. A horseshoe magnet is located outside the beaker as close as possible to the nickel ball, As the ball cools, it passes through the Curie point of nickel (354°C, 670°F), the temperature where it becomes magnetic. At this point, the ball is attracted to a magnet, activating a sensor that stops the timer. The time required for the nickel ball to cool from 885 - 354°C (1625-670°F) is recorded. Table 1 provides some illustrative GM Quenchemeter times for different quenchants.

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Table 1 - GM Quenchemeter Classification of Quench Oils

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<u>Quench Oil</u>	<u>time, s</u>
Fast Oil	8-10
Medium Oil	11-14
Slow Oil	15-20
Martempering Oil	18-25

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Although the GM Quenchemeter has been used to classify quench oils for approximately 40 years, its use is of only limited value. As illustrated in Figure 6, the GM Quenchemeter does not provide any information regarding the cooling pathway which must be known if the ability of a quench oil to harden steel is to be determined.

In view of this critical deficiency, GM Quenchemeter quench oil characterization is increasingly being replaced by the ISO 9950 cooling curve analysis procedure. Figure 7 illustrates the use of cooling curve analysis to identify cooling variations of a

quench oil oxidation over time. Figure 8 illustrates the effect of varying amounts of water on cooling of a non-accelerated quench oil. It should be noted however, that while cooling curve analysis provides an invaluable tool for monitoring and troubleshooting quench oil performance, physical property characterization is still required to identify the causes of the cooling behaviors observed.

## **CONCLUSION**

It has been demonstrated that quench oil characterization is necessary to provide optimal quench process control. Quench oil characterization is readily performed by utilizing a series of physical property procedures including: viscosity, water content, neutralization number, precipitation number, flash point. In addition to physical property characterization procedures, cooling curve analysis should also be performed when needed.

## **REFERENCES**

1. For further information refer to: G.E. Totten, C.E. Bates and N.A. Clinton, ***Handbook of Quenchants and Quenching Technology***, 1993, Chapters 4 and 6, ASM International, Materials Park, OH.
2. R.T. von Bergen, *Heat Treat. Met.*, Vol. 2, 1991, p. 37-42.

Figure 1 - Illustration of the oil quenching process.

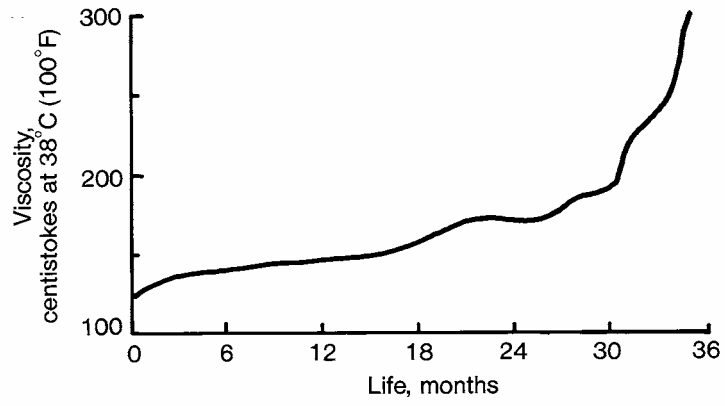
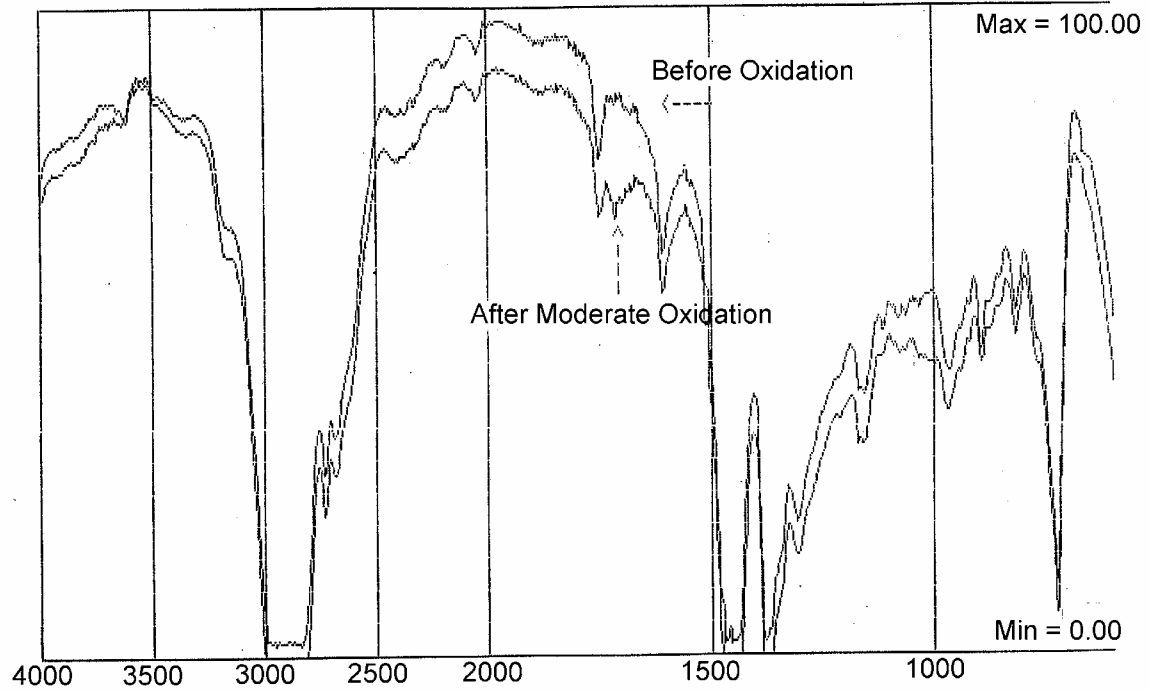
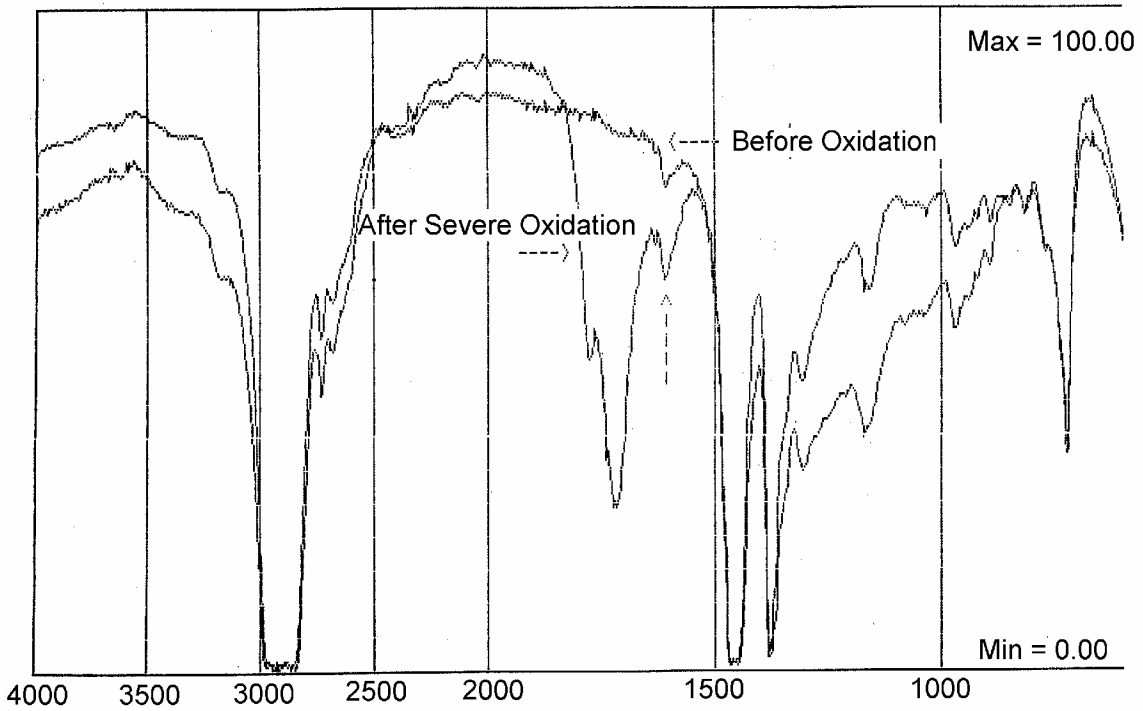


Figure 2 - Viscosity of a martempering oil as a function of time in use.



A.



B.

Figure 3 A. - IR Spectra of a new vs. moderately degraded quench oil and 3 B. - IR Spectra of a new vs. severely degraded quench oil.

Figure 4 - Typical Induction Coupled Plasma (ICP) analysis report  
for a metal containing quench oil.

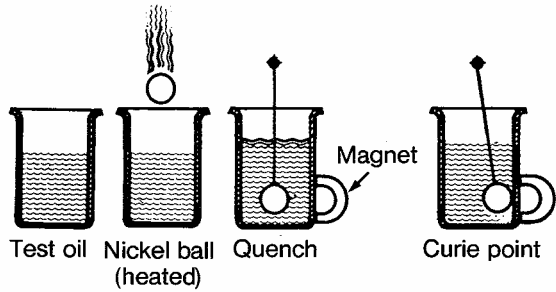
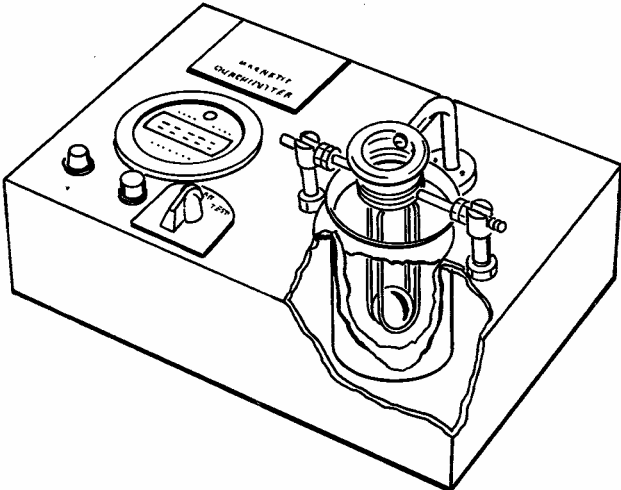


Figure 5 - Illustration of the GM Quenchometer test apparatus.

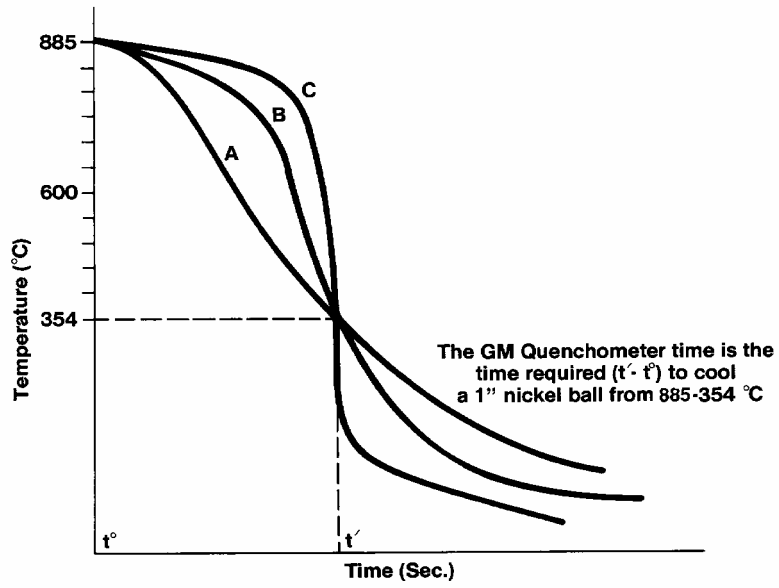


Figure 6 - Comparison of the GM Quenochometer test with cooling curve analysis.

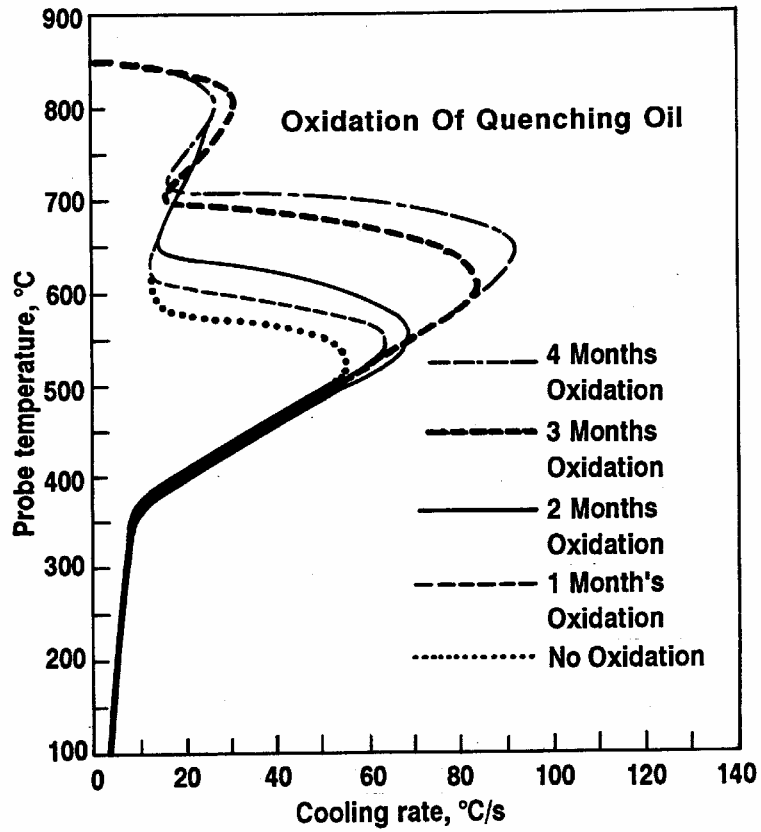


Figure 7 - Effect of quench oil oxidation on cooling rates.

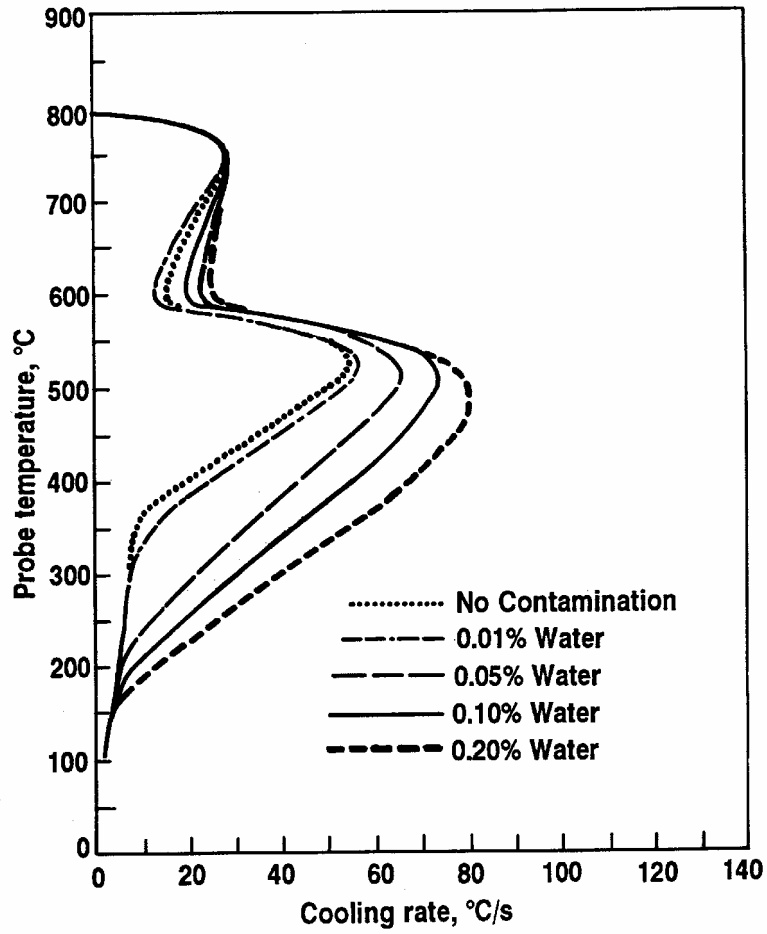


Figure 8 - Effect of water contamination on a normal speed quench oil.