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Sampling Hot Heat Transfer Fluids: Simple Insights for Gaining a Representative Sample

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***Abstract* — Laboratory analysis provides insights into the oxidative status and decomposition by-products produced during the ageing process of a hot heat transfer fluid (HTF).**

Oxidative status is determined by assessing the total acid number of a hot HTF. Decomposition parameters measured in HTF analysis includes residual carbon content and volatile decomposition products (i.e., light-ends). The build-up of volatile gases in a hot HTF can be measured using open and closed flash temperatures. As a hot HTF thermally decomposes the composition of light-ends increases. This build-up leads to a decrease in the flash point temperature of a HTF and, if it drops below a critical level, presents a potential fire risk.

Hence, the accurate and repeated measurement of carbon, flash temperatures and total acid number is critical in the assessment of a thermal system's safety. The current paper indicates that laboratory test results are affected by the actual process of hot HTF sampling from a thermal system and this can lead to false measurements of HTF flash temperatures. The key finding herein was that exposure to air needs to be controlled to limit potentially false measurements of flash temperatures and for accurate conclusions to be reached regarding system safety.

***Index Terms* — Hot heat transfer fluid, thermal science and engineering, flash point temperature, combustion.**

I. INTRODUCTION

In normal production, the decomposition rate of a hot heat transfer fluid (HTF) is estimated to be about 0.03% per day or 10% per year [1]. In reality the rate of decomposition is not uniform and influenced by a number of factors including the type of HTF used, the thermal system design, the extent of oxidation and the operating temperature of the thermal system [1]. To maintain a safe working environment it is essential that the by-products of decomposition are monitored routinely and measured accurately.

Operating at high temperatures, sometimes in excess of 300^{o}C, means that over time it is normal for a HTF to decompose through a process of thermal cracking and/or oxidation. Thermal cracking is the process in which molecules breakdown in the absence of oxygen to form "light-ends" and "heavy-ends."

Light-ends are highly volatile decomposition products that are potentially combustible when a HTF is heated. The composition of these volatile decomposition products in a HTF can be assessed by measuring open (test method ASTM D92) and closed (ASTM D93) flash point temperatures. Heavy-ends are molecularly heavier compounds and congregate to form sticky carbon deposits of sludge. The formation of these carbon deposits can be assessed by measuring the percentage carbon residue (IP14) in a HTF.

Hot HTF's can also decompose through the process of oxidation. Oxidation is the result of chemical reactions in the presence of oxygen and influenced by temperatures. The by-product of oxidative reactions is the formation of carbon sludge and acids, which are potentially corrosive to a thermal system. By measuring total acid number (TAN; IP139) it is possible to determine the extent of HTF oxidation.

Standardized test methods mean that thermal decomposition products can be measured accurately on a repeated basis. However, the process of sampling is also extremely important and must not be overlooked.

II. WHY IS SAMPLING SO IMPORTANT?

It is important that a sample reflects the actual operational condition of the HTF. If the sample is not representative then it can potentially lead to the engineer drawing incorrect conclusions from the test results. The single most important parameter that is affected by incorrect sampling is the open and closed flash point temperature of a HTF.

What are open and closed flash point temperatures? There are two methods to assess flash point temperature – open and closed. Open flash tests simulates combustion vapors mixing with air and in this scenario, vapors are removed by air and the most volatile escape. Closed flash is the opposite where vapors do not mix with air which means the most volatile do not escape and are maintained in the hot HTF. The closed flash point temperature is lower than the open flash point temperature.

III. WHY MEASURE FLASH POINT TEMPERATURE?

Flash point temperature starts to fall when a HTF starts to decompose and is detected by measuring open and closed flash point temperatures. The gradual decline in flash point temperature means that a hot HTF system is not venting effectively and this indicates that highly volatile and combustible decomposition products are accumulating in the HTF. This ultimately leads to an increase in the potential fire risk of a hot HTF.

IV. HOW ARE HOT HEAT TRANSFER FLUIDS SAMPLED?

There are two principle ways to sample – take a live hot sample or a live cooled sample (please see Table 1). This can be done whilst the sampling container is open or closed to air.

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Table 1. A Simple Matrix Depicting the Basic Options for Sampling a Heat Transfer Fluid

	Cold* HTF sample	Hot** HTF sample
Open (to air)		
Closed (to air)		

*Note: *, taken at ambient temperature; **, taken at operational temperature.*

Global Heat Transfer typically takes hot samples that are closed to atmospheric air (see Figure 1). This is done using a sampling device that isolates a sample of the hot HTF.

Fig 1. A Photograph of the Isolation Device Used by Global to Sample Hot Heat Transfer Fluids.

V. WHY TAKE A CLOSED HOT SAMPLE?

In the past Global worked with a client to demonstrate the importance of closed sampling of hot HTF's. The client in question was using a mineral HTF and two live samples were taken - into a closed (i.e., closed, hot sample) or open isolation device (i.e., open, hot sample) – and closed flash point temperature was then tested according to ASTM D93. The results are compared in the below Figure and show that the open sample provided a much higher sample result than measured using the closed sampling approach (170 versus 112°C, respectively).

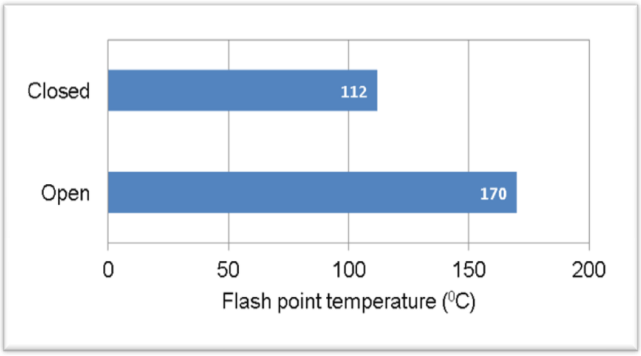

Fig 2. Flash Point Temperatures Recorded Following Open and Closed Sampling from the Same Heat Transfer System.

VI. DOES THE HIGHER VALUE RECORDED WITH THE OPEN HOT SAMPLE HAVE ANY SIGNIFICANCE?

The single explanation for this is that the open sampling technique means that vapors from the hot HTF are allowed to escape and mix with air. This means that an artificially high flash point temperature was recorded when in fact the flash point temperature was 112°C. Global Heat Transfer would normally give a cautionary rating to a closed flash point temperature between ≤130 and >110°C. This means that the client should start planning an intervention to mitigate the decline in closed flash point temperature. In contrast, a closed flash point temperature of 170°C would be given a satisfactory rating and would require no action would be recommended to the client. This is clearly incorrect as it was masking the potential fire hazard represented by accumulating combustible gases in the hot HTF.

VII. CONCLUSION – SUMMARIZING THE IMPORTANCE OF GAINING A REPRESENTATIVE HOT HEAT TRANSFER SAMPLE

Correct and representative sampling of a live hot HTF is extremely important. If samples are not managed correctly, they can give erroneous results that could have catastrophic consequences. When taking a sample the objective is that the test results reflects the state of the hot HTF in situ and either gives a true positive or negative result in the context of a flash point temperature (see Table 2). Above we have explored the impact of open and closed sampling and what we see is that with open hot samples there is the potential to gain false negative results (see Table 2). This result was not due to the test method itself but rather the impact of the sampling technique. Hence, sampling can affect the predictive nature of test results and this has implications for the safe management of hot HTF systems.

Table 2. The Predictive Nature of Testing is Dependent on Gaining a Representative hot Heat Transfer Fluid Sample.

		Thermal hot HTF System	
		Has a flash point temperature issue	Has no flash point temperature issue
Test Result for hot HTF	Predict flash point temperature issue	True positive *e.g., Case with the closed hot sample*	False positive
	Predict no flash point temperature issue	False negative *e.g., Case with the open hot sample*	True negative

Note: Testing should ideally fall in the upper-left or lower-right quadrant. The predictive nature of testing, as shown in Figure 2, can lead to the recording of false negative results and this can

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have potentially disastrous consequences in terms of managing thermal system safety.

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