

Reducing variability in heat transfer fluid (HTF) sample analysis results

Are the plots of your system’s fluid analysis test results lacking in statistical significance? With understanding of the factors contributing to variability in sample results, the desired, smooth trend lines are achievable. For statistical significance of trend lines, the p-values of a data set should be very small, preferably <0.05. Factors contributing to variability in heat transfer fluid analysis are examined in Table 1 for opportunities to improve data integrity. The table summarizes a list of common activities and their potential to raise or lower key fluid analysis test results.

Table 1. Common tests and their potential variability-inducing factors in fluid analysis

Activity	Test results						
	Viscosity	Acid number	Moisture	Insoluble solids	Low boilers %	High boilers %	Flash point
Collecting hot samples	✓	✓	✓		✓		✓
Environment (rain, dust, condensate drops)	✓		✓	✓			
High-temperature excursion	✓			✓	✓	✓	✓
Sample timing proximity:							
Following system venting	✓		✓		✓		✓
Following start-up			✓	✓			
Following fluid top-up	✓		✓		✓	✓	✓
Following reclamation	✓	✓	✓	✓	✓	✓	✓
Amid upset, troubleshooting	✓	✓	✓	✓	✓	✓	✓
Following vent condensate return	✓	✓	✓		✓	✓	✓
Change of sample location/port	✓		✓	✓	✓	✓	✓
Poor/no sample port flush	✓	✓	✓	✓	✓	✓	✓
Sampling an idle/"cold" system	✓	✓	✓				✓
Shared drain tank with another system	✓	✓	✓	✓	✓	✓	✓

✓ = The activity could raise or lower the indicated test result.

The activities in Table 1 are explained to provide guidance on reducing their potential to introduce variability in test results:

- 1. Collecting hot samples**—Depending on fluid temperature and duration prior to sealing the sample bottle, the fluid may experience evaporation of moisture and low boilers, increased flash point and viscosity, and oxidation that increases the acid number.
Optimal solution—For sample integrity and to eliminate thermal burn potential, allow the sample to cool to 60°C (140°F) after it is collected in the sample bottle.
- 2. Environment (rain, dust, condensate drops)**—Sampling in a dusty and humid environment can cause extraneous material to enter the sample bottle.
Optimal solution—Collect samples in a relatively clean and dry environment.

3. **High-temperature excursion**—This can cause thermal degradation of the fluid, possibly leading to changes in concentration of degradation products and their influence on viscosity, and it can cause an increase in insoluble solids (e.g., coke).
Optimal solution—*Conduct a sample analysis immediately following the event to assess impact to the HTF, and implement any needed fluid quality adjustments. After it is stabilized, sample at least one month later for a new baseline sample.*

4. Sample timing proximity

- a. **Sampling following system venting**—Successful venting will cause an immediate decrease in low boilers and moisture content as well as an increase in viscosity and flash point.
Optimal solution—*Collect routine sample(s) at least one month following system venting. See Technical Information Bulletin #4 for additional information on system venting.*
- b. **Sampling following start-up**—After extended shutdowns or when the expansion tank has been de-inventoried for inspection/repair, resuming flow can stir settled solids that can become entrained in the flowing liquid. Downtimes can also offer opportunities for moisture ingress if systems are not inerted.
Optimal solution—*Employ filtration during start-up and collect the first sample at least one month afterward.*
- c. **Sampling following fluid top-up**—Fresh fluid for top-up will be expected to have distinct and relatively favorable properties to have a positive impact on the in-service fluid properties. Small but noticeable dilution effects are expected and desired.
Optimal solution—*Vent immediately prior to adding top-up fluid for improved low-boiler removal and optimal quality improvement after fresh top-up addition.*
- d. **Sampling following reclamation**—The quality of reclaimed HTF can vary and is outside the scope of this text. When reclamation involves a complete de-inventorying of fluid and subsequent return to the system, all key properties are subject to change. If the system is not cleaned while empty, the remarks on activity 5 would apply. **Optimal solution**—*Perform analysis on the reclaimed fluid prior to adding to the system for awareness of its expected impact if added to existing in-service fluid. For significant fluid replacement (e.g., >30%), reset any statistical trends using the new system fill date.*
- e. **Sampling amid upset/troubleshooting**—Sampling is essential for troubleshooting and diagnosing system concerns. However, samples analyzed during events such as process contamination, exchanger leaks, and heater coil plugging represent deviations from normal system performance.
Optimal solution—*Analysis data of samples compromised by process upsets and equipment failures should be excluded or annotated as such for long-term statistical analysis.*
- f. **Sampling following vent condensate return**—Vent condensate is commonly collected in storage vessels, from which they may be properly disposed of. Some have chosen to return vent condensate into the heat transfer system. Since the vent condensate would typically be enriched in low boilers and moisture and possibly oxidized, its return into the HTF system would be expected to shift the composition proportionally, lowering flash point and viscosity and possibly increasing the fluid's acid number.
Optimal solution—*Vent condensate is the fluid enriched in degradation products that has been selectively removed from the HTF system and should not be returned into service.*

5. **Change of sample location/port**—Flow characteristics and distribution through a piping network can lead to opportunities for variation in composition and dispersion of insoluble solids. Depending on the cycling of process equipment demands for heating/cooling flow of the HTF, these variations can noticeably impact sample quality variations at different points in the system. Additionally, many expansion tank designs not only permit but are intended to concentrate low boilers and moisture.
Optimal solution—*Select an optimal sample port location on a flowing main header, preferably in a clean, dry location, and consistently sample from the same location.*
6. **Poor/no sample port flush**—A sample port that was last used perhaps a year or two earlier can contain residues that have been compromised with oxidation, condensation, and partial evaporation or seepage through the isolation valve. These concerns can raise challenges to sample analysis results and require an operator to unnecessarily collect a second fluid sample.
Optimal solution—*Assume that the sample port contains undesirable residues, and thoroughly flush into a bucket prior to filling the sample bottle with the fluid to be analyzed.*
7. **Sampling from an idle or “cold” system**—In cases where a system is down/idle and circulating flow is not provided, any excess water in a system may separate into a bottom or top layer, depending on solubility and relative density. This can introduce potentially significant error in moisture analysis results, and if the sample is high in moisture it can adversely affect integrity of other test results.
Optimal solution—*When collecting a sample from an idle or ‘cold’ system, ensure the system’s fluid is well mixed by thorough circulation before collecting the sample.*
8. **Shared drain tank with another HTF system**—Having a common drain tank with another system (e.g., primary and secondary HTF loops using the same fluid grade) creates an opportunity for intermixing of fluids in different systems. With varying degrees of stress, there is a potential for consequences of problems occurring in one system to occur in the second system as well.
Optimal solution—*Provide dedicated drain tanks for each HTF system. For significant returns of drain tank fluid into a system, it is recommended to sample the “before” and “after” system fluid to track the impact of the significant fluid addition/dilution. Additionally, if the drain tank was open to the atmosphere and had accumulated fluid for many weeks/months, it is advised to confirm quality of the fluid prior to its return into the system.*

By giving proper attention to factors that may contribute to variability in fluid sample analysis, resulting variability can be significantly reduced over time. The benefit is improved statistical relevance of the data and trending provided, supporting more informed fluid management decisions.



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