TESTOIL
MATERIAL IDENTIFICATION ANALYSIS

## Material Identification Analysis

| | | | | |
|---|---|---|---|---|
| Fluid Type: | Royal Purple Synfilm NGL 100 | Received: | 03/14/14 | |
| Machine MFG: | LeROI | Report: | 03/18/14 | |
| Machine MOD: | LG30 Series | Sample No: | 1366-1-3001 | |
| Machine Type: | Gas Compressor | Analyst: | HV | |
| Sample Source: | Filter and drain port from separator vessel | | | |

**Summary of Findings:**
The primary contaminants identified were water, fluid oxidation products, aromatic sulfonate, sulfate salt, and elemental sulfur. Water is often an ingress source and can lead to increased rates of fluid oxidation. Fluid oxidation products typically include molecules such as carboxylic acids and aromatic compounds which often form dark brown tacky deposits. These deposits are known to clog filters and cause mechanical issues such as improper operation of pumps, servo valves, heat exchangers, etc. It appears that the main issues occurring in the system are the following:

1. The fluid has experienced oxidation. This process was most likely accelerated by the presence of water and increased oxygen levels within the system.
2. Hydrogen sulfide gas may have mixed with water in the system forming a weak acid known as hydrosulfuric acid that can also increase the rate of fluid oxidation.
3. Elemental sulfur was still present in the system.
4. Sulfate salt most likely originated from sulfur in the system adhering to oxidation products, i.e. carboxylic acid to form the salt product.
5. The presence of aromatic sulfonate suggests the system has experienced external contamination from a cleaning/detergent product or other unknown source.

An inspection of machine components is recommended. In addition, due to the amount of debris and water found during the analysis a thorough flush of the system is recommended. It was reported that this system was not operating for a period of time during which oil analysis was not occurring. It would be appropriate to continue with routine oil analysis while the machine is down if there is a possibility of product contamination, faulty maintenance, or the heater exchangers remain on. Investigating other lubricant options with the machine manufacturer such as Royal Purple Acilube, engine oils, or crankcase fluids may help prevent these issues in the future as they are more capable of handling acid production.

**Background:**
Two samples were received for analysis: a lube sample from the oil piping and debris from the heat exchanger cleaning process. The machine operates 24 hours per day in a relatively clean environment except the oil is mixed with dirty landfill gas. The system has a capacity of approximately 110 gallons and temperatures inside the enclosure range from 60-100°F. It was reported the oil heat exchangers appear to be clogging which reduces flow and cooling. This causes the oil pump to run at high speeds and over the normal temperature range up to 200°F. Hydrogen sulfide gas is constantly interacting with lube in the system; however, the concentration of hydrogen sulfide is typically not more than 1000 ppm. At one point, the company became aware that elemental sulfur had been generated and recycled through the system. It was also reported that the machine was not operating for a period of time. During down time water is expected to accrue in the system. Upon startup oxygen levels were increased for one to two days. The goal of the analysis is to identify if the same materials identified in report 1225578 were included in this debris and oil sample.

**Analysis:**
The lube as received had a visible layer of dark grey/metallic debris on the bottom of the bottle. It was first screened for water and tested positive. A portion of the lube sample was washed with pentane in order to remove the lubricant from the debris. The remaining debris was then extracted into organic and inorganic portions using methylene chloride. During the separation of the organic material a sulfur crystal was extracted and crystalline material was adhered to the sides of the test tube. The debris appeared to be primarily inorganic.

*Figure 1. (From left to right) Picture of debris in lube as received, organics, inorganics, sulfur crystal.*

The debris as received consisted of a tan debris, lubricant, and water. The lubricant and water were first separated from the debris. The debris was further extracted into organic and inorganic fractions using methylene chloride. The debris appeared to be primarily inorganic.

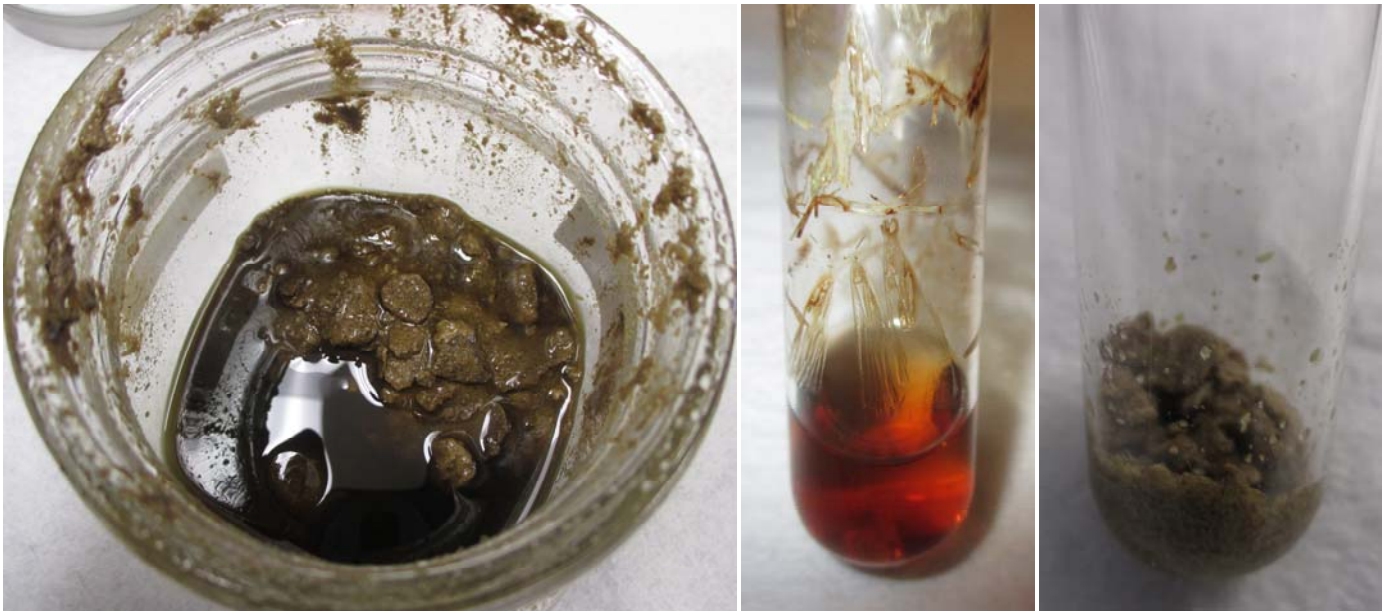


*Figure 2. (From left to right) Picture of debris as received, organics, inorganics.*

The organic portions extracted from the debris and lube samples were characterized using FTIR spectroscopy. They appear to have the same chemistry as the organic portions extracted from the previously received lube and filter samples. This chemistry included carboxylic acid, aromatic hydrocarbon, and aromatic sulfonate compounds. Carboxylic acid and aromatic hydrocarbon compounds are known degradation products of oxidation. Possible sources of the aromatic sulfonate were discussed with a colleague who does not believe the aromatic sulfonate originated from recycling materials or the hydrogen sulfide gas. Although the exact source is unknown at this time one source may be a commercial detergents or system cleaner. If a cleaner is the source it is possible that the cleaner was added to the system unknowingly by the operator (as can be the case with mislabeled or contaminated drums), or during the last system cleaning the detergent may not have been thoroughly flushed from the system. An MSDS of the system cleaner was provided in order to aid this analysis; however, more conclusions could not be made due to a lack of information regarding the basic chemical makeup of the cleaner used to flush the heat exchangers.

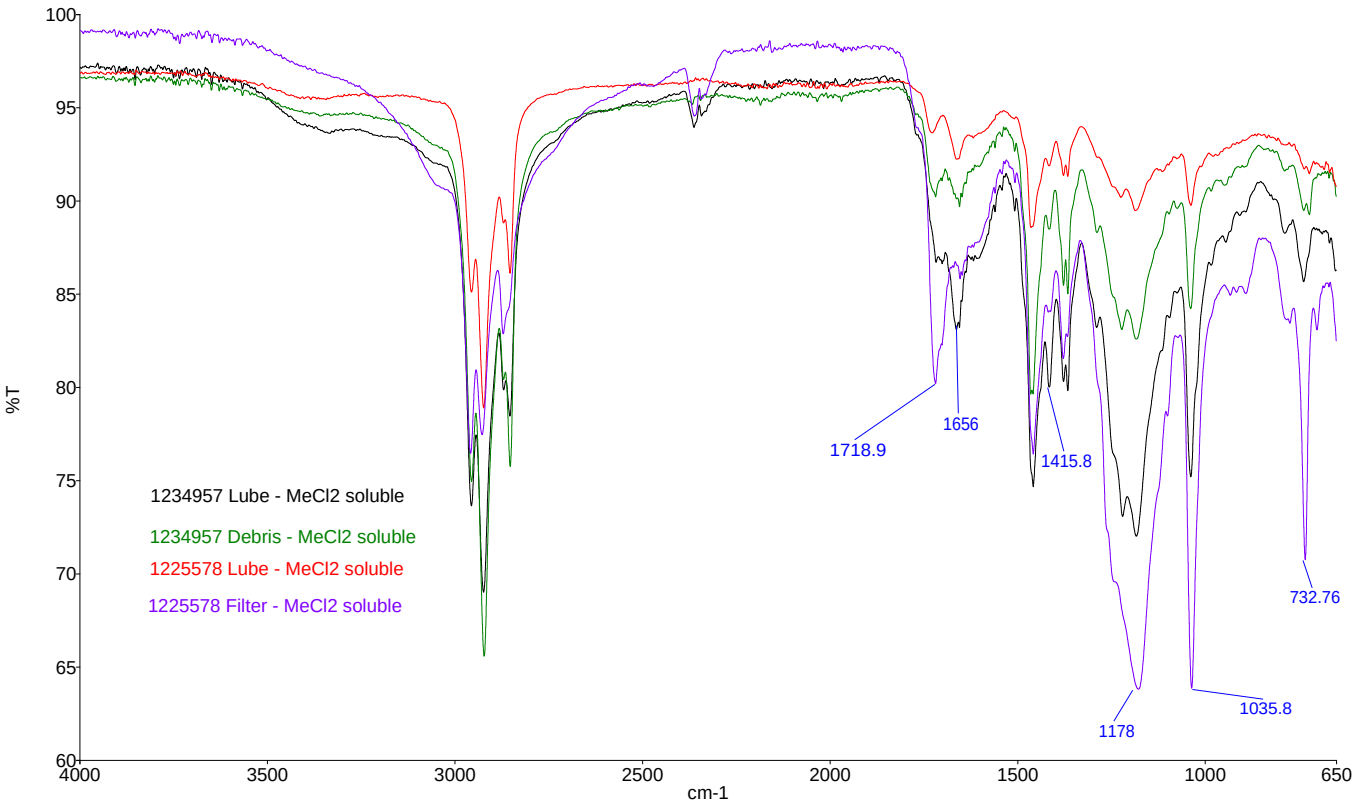



*Figure 3. FTIR spectra of organic portions from current lube, current debris, previous lube, and previous filter samples.*

The inorganics were characterized using XRF elemental spectroscopy. Primarily sulfur was detected in the inorganic fraction extracted from the debris. This is most likely detecting sulfur components from the sulfate salt and elemental sulfur. Primarily iron was detected in the inorganic fraction extracted from the lube. The iron may have originated from machine component wear as well as the SulfaTreat process. Compounds such as iron sulfides may have formed from a reaction between the iron and sulfur.

| | Concentration (%) | | | |
|---|---|---|---|---|
| Element | 1225578 Filter | 1225578 Lube | 1234957 Debris | 1234957 Lube |
| W | - | - | - | - |
| Fe | 58.50 | 53.90 | 7.30 | 71.20 |
| Co | - | - | - | - |
| Ni | - | - | - | - |
| Cu | - | - | - | - |
| Zn | - | - | - | - |
| Na | 2.60 | 2.00 | - | - |
| Mg | - | - | - | - |
| Al | 4.90 | - | - | 3.60 |
| Si | - | - | - | - |
| P | - | - | - | - |
| S | 21.20 | 36.90 | 86.70 | 11.20 |
| Cl | 2.30 | - | 4.00 | - |
| K | - | - | - | - |
| Ca | - | - | - | - |
| Mo | - | - | - | - |
| Ag | - | - | - | - |
| Cd | - | - | - | - |
| Sn | - | - | - | - |
| Pb | - | - | - | - |
| Ti | - | - | - | - |
| V | - | - | - | - |
| Cr | 2.70 | - | - | 4.90 |
| Mn | 4.00 | 2.00 | - | - |
| Ba | - | - | - | - |
| Sb | - | - | - | - |

*Table 1. XRF of inorganics from previous and current samples.*

The inorganic portions extracted from the debris and lube was then characterized using FTIR spectroscopy. Primarily, the same sulfate salt chemistry was detected. The elemental sulfur contamination most likely adhered to fluid oxidation products, i.e. carboxylic acid, to form the salt. Spectra collected from the 1225578 lube and 1234957 debris inorganic fractions did not provide much information. There are very small peaks in 1225578 sample typical of elemental sulfur and small peaks in the 1234957 sample indicating the presence of sulfate salt. However, a lack of significant peaks often indicates the material applied to the FTIR is not active in the infrared light region and totally inorganic.

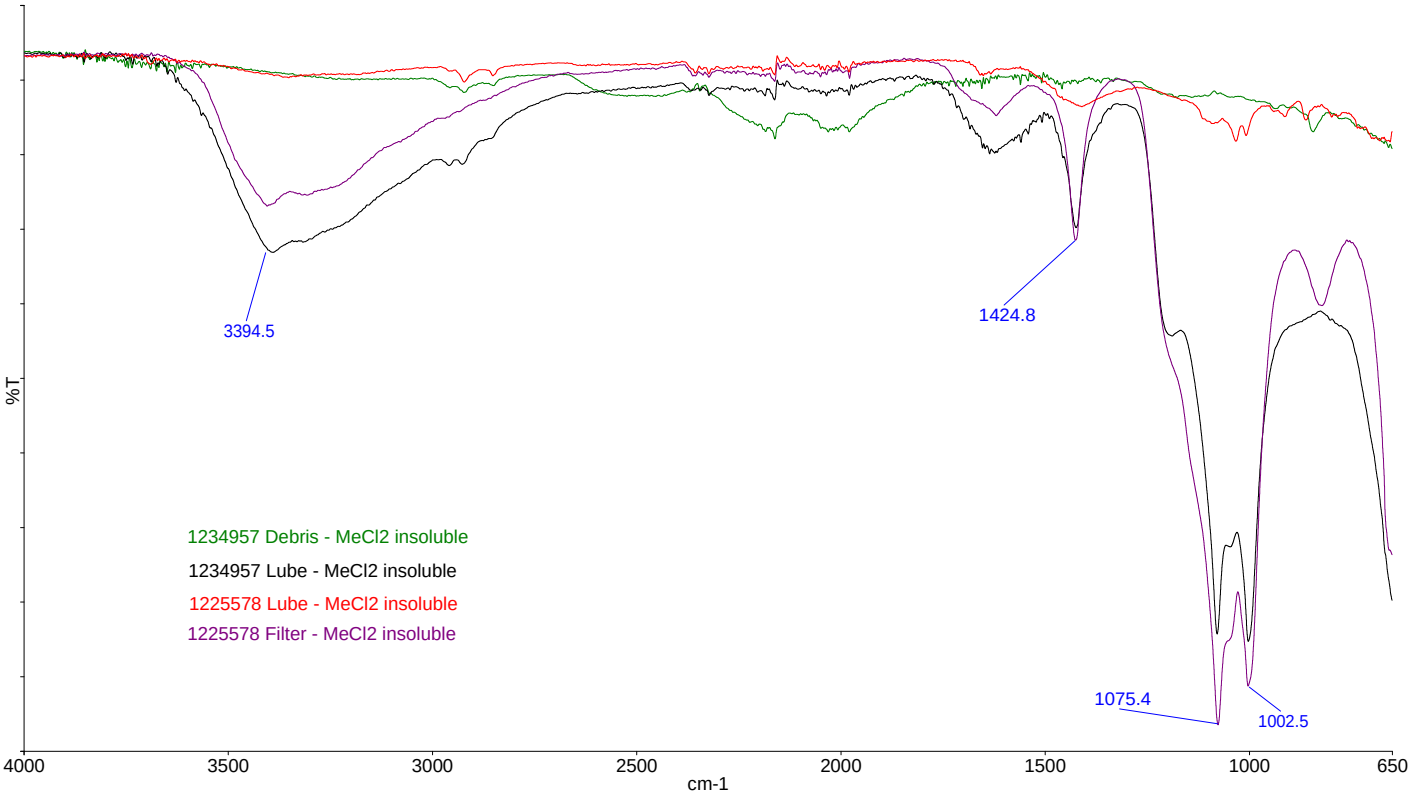



*Figure 4. FTIR spectra of inorganics from current and previous samples.*

The oil analysis history for this sample point exhibited interesting data that may point to when the oxidation issue began. TestOil has history on this compressor dating back to December 2012. The acid number and viscosity results do not yet appear significant enough in the compressor to cause machine issues; however, they have been steadily increasing since April 2013. This can be due to oxidation, formation of hydrosulfuric acid if water was present at the time, or both. Due to the harsh environment the lube in this compressor seems to experience it may be beneficial to discuss other lubricant options with the machine manufacturer. Other options may include the Royal Purple Acilube product, engine oil, or crankcase fluid. Another fluid may consist of an alkaline reserve that will provide better resistance to acid degradation. It is also recommended to keep in mind that when acid number and viscosity results are both steadily increasing it can be a sign that oxidation is taking place within the system.

| Lab Number | Date | Acid Number | Viscosity |
|---|---|---|---|
| 1225577 | 2/18/2014 | 0.25 | 101.3 |
| 1052924 | 9/20/2013 | 0.20 | 94.5 |
| 994535 | 5/20/2013 | 0.11 | 94.2 |
| 978124 | 4/16/2013 | 0.10 | 94.5 |
| 963575 | 3/14/2013 | 0.19 | 90.8 |
| 954159 | 2/21/2013 | 0.17 | 88.5 |
| 938812 | 1/22/2013 | 0.26 | 95.0 |
| 921762 | 12/5/2012 | 0.22 | 88.1 |

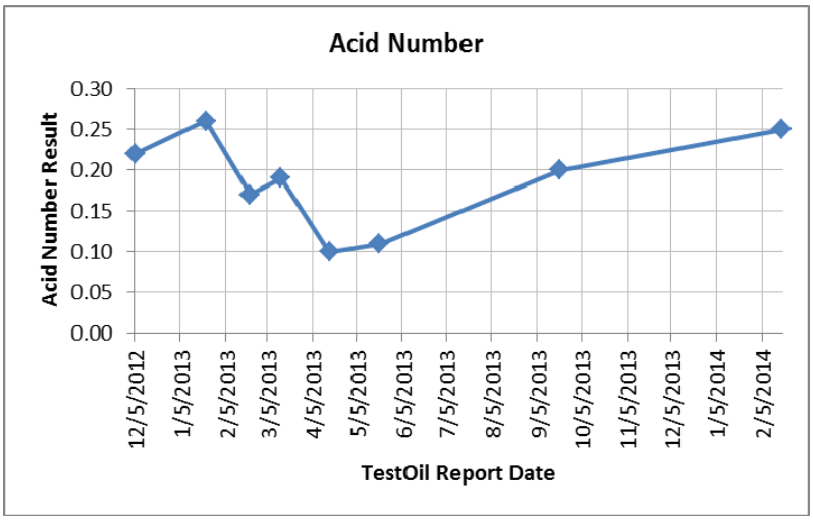
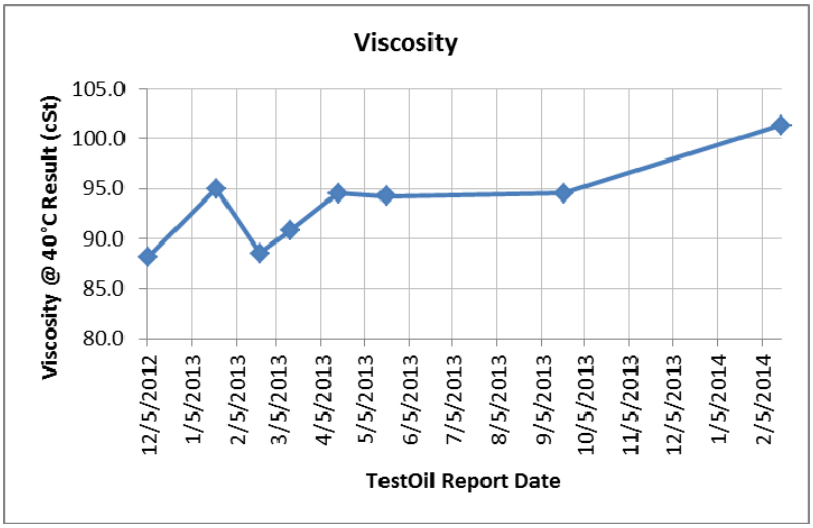



*Table 2. Historical acid number and viscosity data.*




Please refer to the Summary of Findings section located at the beginning of the report for a conclusion of the results. Analysis completed by Heather Vercillo. For questions please e-mail hvercillo@testoil.com.