

# Bureau Veritas

## Oil Analysis



## Grease Analysis - Whitepaper

Monitoring Machine Condition  
with Grease Analysis



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VERITAS**

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## Introduction

Lubricating greases are usually a solid or semi-solid mixture of a fluid lubricant, a thickener and additives. The lubricating fluid that actually performs the role of lubrication can have a mineral, synthetic or even vegetable origin. The thickener determines the consistency of the grease. Thickeners are usually organic or inorganic soaps or thickeners with most commercial greases being composed of a mineral oil and a thickening soap. Finally, additives protect the grease-lubricated surfaces.

The function of grease is to remain in contact with the surfaces and to lubricate them without leakage due to centrifugal forces or pressures. At the same time, the grease must be able to flow throughout all components it is intended to lubricate. However, it is important to be aware of the disadvantages of using greases as lubricants over lubricating oils:

- Greases have lower cooling and heat transfer capacities than oils.
- The high viscosity and poor dissipation characteristics of greases impose component speed limitations and generate excessive heat.
- Greases have less storage stability – the base oil tends to separate from the thickener.
- The pose compatibility problems. When mixing greases, always consider the compatibility of each base oil, additive and thickener.
- Thickeners are susceptible to oxidation which can significantly affect their stability.
- Unlike oils, greases permanently suspend contamination exposing component surfaces to these particles and their abrasive effects.
- Volume control is often difficult – especially during re-lubrication. Failure to re-lubricate with the correct amount of grease can lead to frequent over-greasing.

## 2. Reasons for Analyzing Greases

Grease analysis is the fastest, most inexpensive way to determine its condition. Traditionally, grease analysis has been used as a quality control tool for new greases as larger quantities were required to perform standard testing. But new testing developed over recent years requires a much smaller amount of grease, even milligrams. However, poor reproducibility in the collection of samples remains one of the biggest problems in analyzing greases.

## 3. Sampling In-Service Greases

Proper sampling is vital to obtaining accurate grease analysis. For example, there are two ways to take bearing samples:

- Send the entire component to the laboratory for sampling
- Take the grease sample without disassembling the component

These two problems are quite different. The first can be solved by sending only a representative number of components to the laboratory. The results of these analyses can be extrapolated to represent all like components, which can be very useful in identifying the root cause of repetitive bearing failures. Experience shows that if the lubricated elements are in the same condition for a given application, and if the problem identified is related to lubrication, the root cause will most likely be the same. Once the problem has been found, decisions can then be made concerning grease selection, re-greasing intervals, common wear mechanisms and typical cleanliness levels.

When an actual representative sample is sent to the laboratory, it is of utmost importance to provide as much information about the same and the component as possible. The most representative samples should be taken from areas of contact where there is the greatest evidence of wear particles, contamination and degradation. However, the most representative sample can sometimes be the most difficult to obtain.

## 4. Testing Frequency

During grease changes, consider the following:

- If a different grease is to be used, select one with a thickener that is compatible with the thickener in the grease being replaced.
- Re-lubricate frequently but take care so as to not over-lubricate – it is better to increase re-greasing frequencies and reduce the amount of grease used than to leave a larger amount of grease to lubricate for an excessive period of time.
- Centralized greasing is much more effective than manual greasing manual and ensures that a consistent amount of grease is used.

## 5. Analyzing Used Greases

### 5.1. Routine Grease Analysis

Traditionally, routine grease analysis has required a large sample amount – up to 1 kg – in order to control the state of the grease used. This large amount presents problems when trying to obtain representative as they are often difficult to obtain.

Testing for in-service greases typically includes the following:

1. **Penetration (ASTM D217)** – Measures the change in the consistency of the grease, which is mainly determined by the viscosity of the base oil and thickener. However, the degree of consistency of a

grease in use may vary due to contamination, loss of base oil or mechanical stress.

- 2. Drop Point (ASTM D2265)** – Establishes the temperature at which the grease passes from a solid or semi-solid state to a liquid. The maximum temperature of the grease while in use must be 50°C - 80°C below the experimental drop point. With the help of this method you can establish the suitability of the grease used, or if the grease is suitable for continued service.
- 3. FTIR (DIN 51820E)** - Infrared spectroscopy determines if the correct grease is being used, if greases have been mixed and if there are contaminants present causing degradation. FTIR can also identify the thickener and its concentration as well any oxidation by-products.

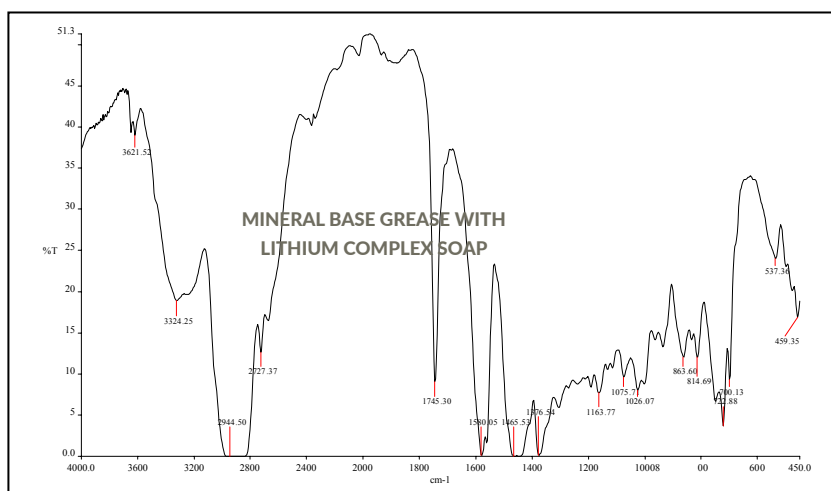


Figure 1 - IR spectrum of a grease with mineral base oil and with a lithium soap complex as a thickener

- 4. Water Content by Karl Fischer (ASTM D 6304)** – Determines the amount of water present in ppm.
- 5. ICP (ASTM D5185) Atomic Emission Spectroscopy by ICP (inductively-coupled plasma)** - Identifies both the type and concentration of wear, contaminant and additive elements present to determine what machine components are wearing and whether or not additive levels are sufficient for providing adequate metal protection.

## 5.2. Advanced Grease Analysis

The development of new technologies has allowed for more advanced grease testing. This testing includes the following:

**Rheology Viscometry** – Measures both grease consistency and viscosity – the deformation and flow of matter when subject to pressure, temperature and time. This technique is ideal in that it requires only a few grams of sample and yields much more information than the individual cone penetration and viscosity tests.

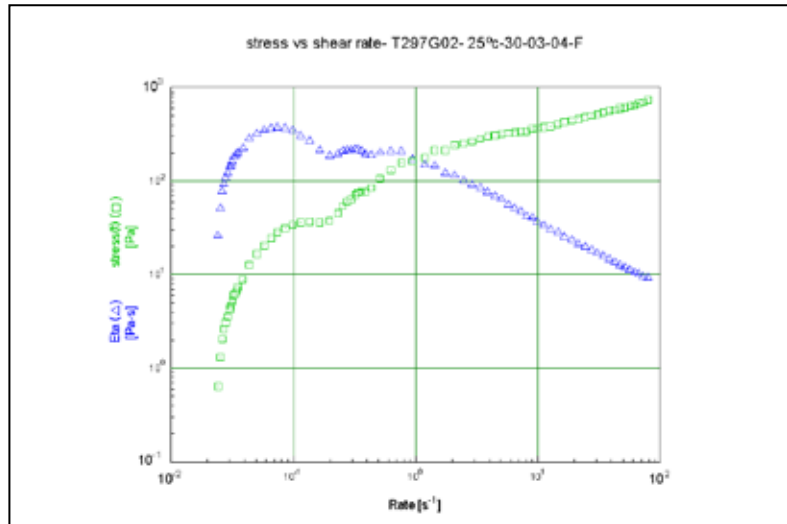


Figure 2 - Flow curve Viscosity and effort versus speed of formation

- 6. Minitest FFK (DIN 51805)** – Determines the flow properties of a grease according to Kesternich (DIN 51805) up to temperatures as low as -60°C. It determines the pressure needed to pass a grease through a nozzle at a given temperature.
- 7. FTIR (DIN 51820E) Infrared Spectroscopy** - Determines if the correct grease is being used, if greases have been mixed and if there are contaminants present causing degradation. It can also identify the thickener in use and oxidation by-products.
- 8. Oxidation Stability** – Measures anti-oxidant additives to determine the remaining useful life of the grease. It is simpler to perform than the RULER and requires a smaller amount of sample.

**DSC (ASTM D5483)** – Identifies at what point in time oxidation began, therefore determining the grease's stability to oxidation. By comparing this point in time with the oxidation stability of the grease when new it, the remaining useful life of the grease can be determined. You can estimate the useful remaining life of the grease. A minimal amount of sample (<1g) is introduced to a cell that is then heated and pressurized with oxygen. The induction time, which is the point at which an exothermic reaction occurs as the grease begins to degrade, is then compared to the induction time of the grease when new.

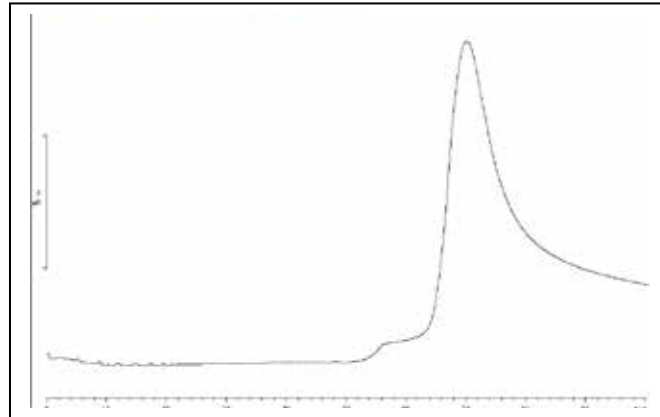


Figure 3 - Thermogram of a grease used by the DSC

- RULER** – Linear scanning Voltammetry determines the anti-oxidant concentration of a grease in use. Less than 1g of sample is dissolved in a polar solvent to extract the antioxidants for measurement. This concentration is then compared to anti-oxidant concentration of the grease when new to determine its remaining useful life (%RUL) and then its concentration is determined. Comparing it to that of new grease, it is estimated that percentage of remaining useful life of the oil (% RUL). It is a proactive tool for determining the condition of the grease before significant changes in grease properties occur.

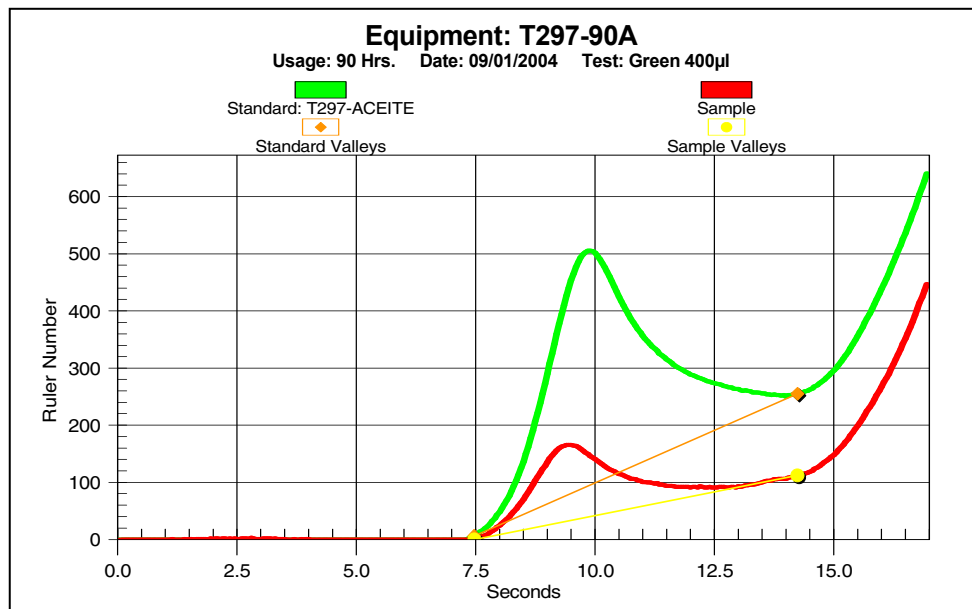


Figure 4 - Voltammogram of a used grease (red) compared to the new one (green) made with the RULER

- Base Oil Extraction** – Determining the state of the base oil in use is useful in determining its kinematic viscosity, acidity index and IR spectrum and provides for a clearer interpretation of the results.

11. **Viscosity (ASTM D445)** - Typically, the viscosity indicated for a new grease is that of its base oil. The method most commonly used to measure viscosity is ASTM D445. Therefore, the base oil of a grease should be extracted and its viscosity measured to determine the viscosity of the grease in service.
12. **Acid Number (ASTM D974)** - Determining the acidic value of a grease will identify the amount of oxidation compounds that have formed. Water, an oxidizing atmosphere, working temperature and wear metal catalysts all contribute to increased acidity.
13. **Water Content (ASTM D6304)** - The preferred method for determining low amounts of water (ppm) is Karl Fischer. Using an oven, water present evaporates and is retained in a reaction cell for valuation.
14. **Filtration (DIN 51813)** - Dissolution of the grease in a solvent to retain the solid particles in a 1 micron membrane for microscopic analysis of the residue amount by weight.
15. **ICP (ASTM D5185)** - The use of Atomic Emission Spectroscopy by ICP (inductively-coupled plasma), to determine the type and concentration of wear, contaminant and additive elements present in the machine.
16. **Ferrography** - Ironographic analysis of wear mechanisms and their severity - determines wear particle size, shape, metal type and wear type.



Figure 5 - 400 micron slip wear particle



## 6. Summary of Recommended Analytical Programs

### 6.1. Routine Analysis Program

- Metal content (ASTM D 5185)
- FTIR
- Water Content by Karl Fischer (ASTM D6304)
- Filtration (and visual particle inspection)
- RULER

\*Minimum amount of sample - 25 grams

### 6.2. Advanced Analysis Program

- ICP (ASTM D5185)
- Water Content by Karl Fischer (ASTM D6304)
- Filtration (and visual particle inspection)
- RULER or DSC
- Penetration
- Acid Number
- Viscosity
- Ferrography
- Drop Point

\*Minimum amount of sample – 500 grams

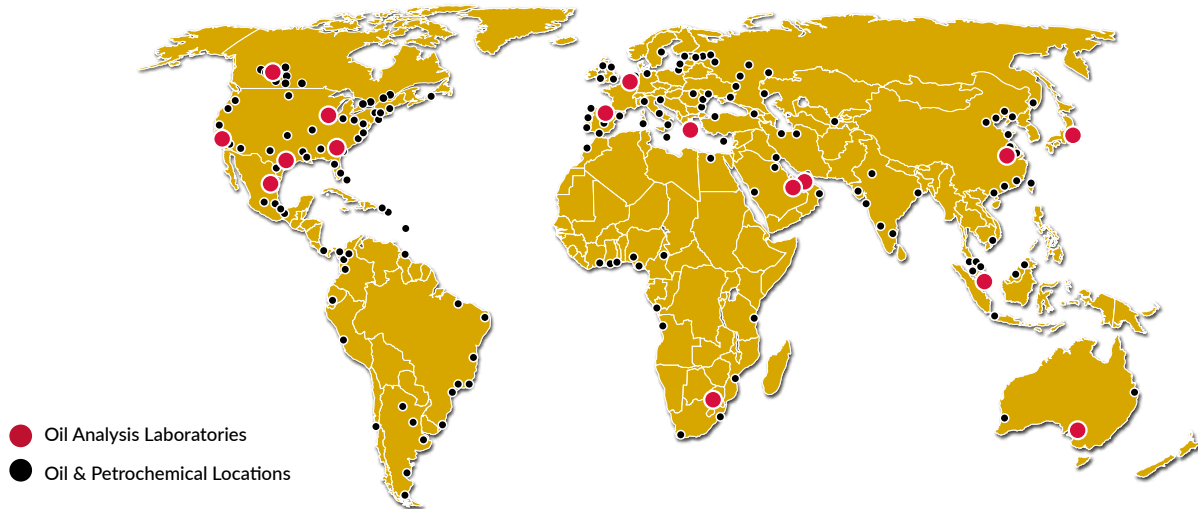
## 7. Conclusions

In the use of greases, careful attention to grease selection, the amount of grease applied and the re-lubrication method used.

In recent years, different analytical techniques have been developed that call for a minimal amount of sample, making it possible to carry out even further, more informative analysis. In turn, these advances have also improved the diagnosis of potential component problems.

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## Oil Analysis



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