The Importance of Monitoring Individual Antioxidants in Multi-Component AO Additive Packages

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Abstract

The measurement of the total oxidation resistance of the base oil, plus its anti-oxidant (AO) additives, by RPVOT (ASTM D-2272) may have worked well for single-component type of anti-oxidant additive packages in the past. But more recently, today's oils are using complex and synergistic anti-oxidative additive packages that present some challenges to this measurement of the total oxidation resistance. In a complex AO package, the premature depletion of just one of the components can have a major impact on other physical properties of the oil, other than just the RPVOT value. These properties include individual antioxidants (by RULER™), demulsibility and air release properties, which are closely related to the oxidation and varnish/sludge tendencies of the oil. In this paper we will examine some of the common mistakes that can occur when using RPVOT data alone and ignoring the premature depletion of individual anti-oxidant components as part of a global oil monitoring program.

Introduction

Large manufacturing organizations have new lubricant challenges that need to be addressed in order to compete at a global level. These challenges include ecological – extending drain intervals, reducing CO_2 emissions and minimizing waste streams; performance – lubricants are blended with increasingly complex additive chemistries in order to perform in more demanding applications; more rigorous applications place extreme stress on the lubricant; management – fewer people are available to do higher profile tasks.

In parallel with these drivers, new ASTM and ISO standards have been released and revised over the last years, in order to offer the industry a strong backbone and support structure. Over the last decade, Oil Condition Monitoring (OCM) has moved from being on the periphery of Condition Based Maintenance (CBM) to being the core technology that leads companies' reliability programs. The economic driver behind the OCM program is the organization's accounting of the Total Cost of Ownership for critical lubrication applications.

And as of recently, these oil analysis and condition monitoring practices have been undergoing major phase changes, from off-site oil analysis, on-site Trend Analysis, and fully integrated in-line sensors.

With these three different strategies all having their pros and cons, the question that troubles lubrication professionals most is how the newest developments in lubricants have been addressed by new oil analysis and condition monitoring methods.

One of the major developments and criteria today with modern lubricants is the increase of oxidative stability of a lubricant. As mentioned above, this is a logical consequence from the following operating parameters:

- Operating temperature increase (overall or local temperature) – gas turbines and air compressor bearings are good examples of that
- Longer lubricant lifetime expectations or extended drain intervals
- Increased load factors on new and existing types of rotating equipment

- Extended operational service/maintenance periods (from predictive to proactive maintenance strategies)
- Decreased lube reservoir volume (cost/weight/engineering reasons)

These demanding specifications challenge the new generation of industrial lubricants, such as compressor, turbine and hydraulic fluids, to possess greatly improved capabilities to inhibit oxidation and explains why in the last decade a wide variety of new antioxidant technologies have been emerging to respond to this market need. Until the effects of the new antioxidant technologies on long-term lubricant performance and traditional condition monitoring techniques have been fully established, there will be a need for measuring and trending their concentration in order to make proper Remaining Useful Life evaluations of in-service lubricants.

For this paper, Remaining Useful Life or Remaining Oxidation Stability is the length of time the lubricant can be used before the antioxidant(s) becomes depleted**,** allowing rapid base-oil oxidation signified by increased Acid Number (AN) and viscosity values, lubricant darkening, deposit formation, etc. Using this definition, new oil has 100% remaining useful life and fully depleted oil with elevated AN values, etc., have 0% remaining useful life. The most popular test for measuring oxidation stability, the Rotating Pressurized Vessel Oxidation Test (RPVOT), has been widely used and touted as an industry standard, while at the same time, the test results are often ignored, especially when the results come into conflict with other test data or other operating criteria. Typical examples of this phenomenon are when the RPVOT of the new oil is lower in value than the RPVOT of the in-service oil, or when the in-service oil RPVOT results are far out of range with the new oil data, and yet the oil has only been in service for a short period of time and no other abnormal parameter can be found with the oil. Some real-life RPVOT data are given in this paper in the section titled RPVOT Reproducibility Issues.

1. Antioxidant additives, the #1 lubricant additive, now and in the future

Before explaining in more detail the different classes of antioxidants and their principle of working, it is vital to understand that in many modern lubricants, a mixture of antioxidants is applied. Therefore it will be important to understand the total oxidative health of the lubricant and not just one type of antioxidant.

What is oxidation? Oxidation is the chemical reaction of a lubricant at elevated temperatures between the dissolved atmospheric oxygen and the base-oil (Figure 1). During the oxidation, as hydrocarbon molecules will break down, reaction products will be formed, better known as radicals (very reactive chemical compounds). Subsequent reactions of these radicals lead to the formation of peroxides and must therefore be quenched by the antioxidants to preserve the lubricant integrity, or its Remaining Useful Life, RUL. Most of us relate the oxidation (or aging) of an oil with the formation of weak organic acids, resins, or other nasty chemical compounds, which have to be seen as final reaction compounds, or when it's too late to react. The role of antioxidants is to protect the base oil by either scavenging these radicals or by decomposing hydroperoxides into stable products via the following mechanisms:

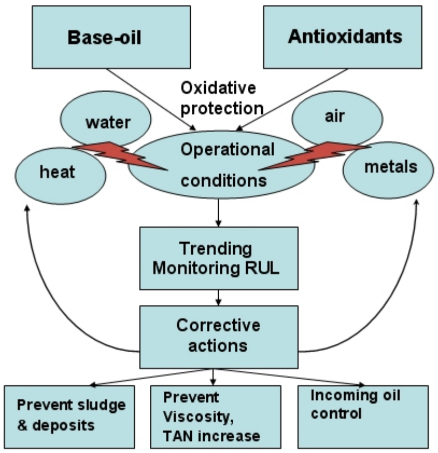
Primary antioxidants - remove the radicals (known as radical scavengers) that initiate the chain reaction that results in accelerated lubricant oxidation. Hindered aromatic amines and phenols are characteristic types of primary antioxidants, widely used in industrial lubricants. Therefore these types of ashless antioxidants go straight to the root of the problem and prevent deposits from forming in the first place.

Secondary antioxidants – react with peroxides (known as hydroperoxide decomposers) and form non-reactive products that do not participate in further oxidation of the lubricant.

Zinc dithiophosphates, better known under the name of ZnDtP or ZDDP, phosphites and phosphonites belongs to this class of antioxidants [1,5].

Mixed antioxidant systems When two or more antioxidants are added to oil, an antioxidant effect is frequently observed in excess of either additive introduced individually. Antioxidants are often used in synergistic mixtures in modern lubricant formulations, to achieve an extended useful life, where one of the antioxidants sacrifices itself in preservation and regeneration of the other. A realistic example is the synergy between amines and phenols [1,2,3 & 8] whereby the hindered phenols give excellent protection at low-temperature regimes (and deplete first), while the amine antioxidants are more effective in extending lubricant life at higher temperature ranges.

Figure 1: Antioxidant Function



In the above Figure 1a, the oxidation accelerators (heat, water, air and metals) play an important role, as they can act separately or in combination, but are very much application dependent (will also be discussed in the practical case studies). The antioxidant (inhibitor) reaction mechanism scavenges the free radicals to stop the formation of oxidation products (carbonyl).The actual mechanism will depend upon the type of antioxidants applied and selected.

With the continuous increase of power plants' efficiency, as well as the overall increasing size of power plants, thermal loads of turbine oils have been rising over the last ten years. This has resulted in a large change in new lubricant specifications for gas and steam turbine lubricants. The gas turbine lubricants inherently work at higher operating temperatures, and are faced with higher oxidative stresses. Other industrial lubricants such as gear and compressor oils do not follow the same temperature trend as for the gas turbines, but have also seen an increase in operating temperature over the years, requiring a higher oxidative stability.

Also with the increases of operating temperatures and equipment availability, industrial lubricating equipment manufacturing companies have started to include new proactive parameters in their maintenance specifications. Not only will these parameters result in a better balance between equipment and oil health monitoring, but also increase the availability of the equipment. This explains why oxidative health monitoring will be of high economic value for in-service oils as well for incoming oil batches.

2. The value of monitoring antioxidants – a key parameter for modern condition monitoring and reliability programs

The answer to this question is found in the basic characteristic of modern maintenance techniques, which require Root Cause Failure analysis. In order to help extend fault free machine operating-life, the trending of oxidative health, or antioxidants concentration, will be required to look at the root causes of lubricant failures.

By monitoring the antioxidants, lubricant operators will detect additive failure in advance of oxidation, acid formation, thickening and varnishing so as to avoid secondary component failure caused by accelerated wear, corrosion, filter plugging and bearing lubricant starvation. And herein lies the major benefit of monitoring the antioxidant concentration or the Remaining Useful Life (RUL), as users will be able to look forward, rather than look backward by being reactive on changes of parameters like viscosity, Acid Number or oxidation by FTIR (FTIR-Ox).

This is why, in contrast to conventional fluid degradation techniques, other techniques are required which can routinely monitor the antioxidant concentration in a predictive/proactive mode. These tests need to be able to trend results well, with easy to recognize data slopes that give sufficient early warning, warning times that are well within the acceptable sample frequency interval.

For each type of equipment, antioxidant depletion rate will reflect the equipment's characteristic operating conditions, enabling operators to look at the root causes for possible abnormal conditions. Experiences [7, 8, 10] have shown that with 20-30% remaining antioxidant concentration, especially with higher temperature applications, large changes in the base oil's physical properties occur, i.e. the useful life of the oil ends. If a lubricant is then used past its end of useful life, excessive basestock degradation can occur, resulting in component wear and eventually equipment/engine malfunction**.** The better known oxidative accelerators can be divided into 3 categories:

Temperature stress - elevated temperature is an important accelerator to oil oxidation. This can be due to local hot spots (local bearing effects, dieseling), or overall high operating temperature. The impact of high temperature on the rate of oil oxidation (rule of Arrhenius – for operating temperatures higher than 100°C/170°F each increase by 10°C/17°F) will double the rate of oxidation or half the oxidative life of the lubricant.

Solid contamination (through wear debris or dirt ingestion) - accelerate the oxidation as being catalysts and decompose hydroperoxides.

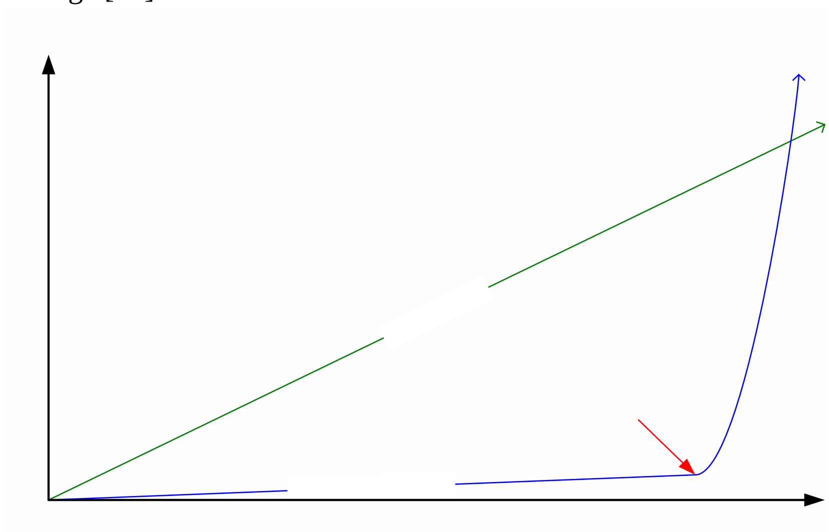
Water contamination acting as oxidative accelerators – Moisture/Water contamination (due to ingestion, condensation, and fresh lubricant top-up) and hence the importance of combining antioxidant trend analysis with predictive maintenance techniques like wear/contamination and water.

These oxidative accelerators will enhance the fluid degradation and will increase the degradation when they work in combination, like water and metals (Figure 1):

- With equipment conditions changing continuously lubricant suppliers are changing the lubricant formulations to meet these demands
- To detect faulty storage conditions of the fluids resulting in fast (auto) depletion of antioxidants
- To assist lubricant operators during normal oil top-off operation and avoid mixing of industrial lubricants

3. Existing techniques to monitor oxidative health of industrial lubricants

Today typically oxidative health is monitored by Acid Number (AN) tests which have a very low proactive value in CBM programs. Especially with new Group II and III base oils (highly refined and purified base-oils), the AN values are showing very low-slope rates-of-change, and tend to show completely normal results until the very latter stages of the oils life. When critical oxidative depletion finally starts to take place in the oil, these tests take on a rapid rate- of-change or slope, but they are usually not noticed because the fluid is not sampled frequently enough to detect the initial rapid rate of change. In addition to that, an AN increase of just 0.1 AN, from 0.1 AN to 0.2 AN, can be significant, yet the AN test has accuracy problems in this range.[19]



<u>Figure 2:</u> Acid Number increase vs. time for Group I, II and III turbine oils [19]

Viscosity increase, which is a direct result from the polymerization (chain formation) between hydrocarbon (base-oil) chains and enhanced by the oxidation products is a second indicator or signal that heavy lubricant degradation exists.

For modern industrial oil oxidation assessment, as part of oil condition monitoring practices, different techniques are applied, two of which will be evaluated in this paper : RPVOT (Rotating Pressure Vessel Oxidation test as per ASTM D-2272), and Voltammetric techniques (RULER as per ASTM D-6971).

3.1 RPVOT test method (per ASTM D-2272)

In today's industrial applications a common test for turbine oxidative life measurement is the rotating pressure vessel oxidation test (RPVOT) as per ASTM D-2272. During the RPVOT test, the oil's ability to resist oxidation degrades as a result of stress-induced antioxidant depletion, to the point where the base oil starts to react with the oxygen as the oil molecules begin to oxidize. At that point the pressure drop in the pressure vessel starts to accelerate and when the pressure-drop reaches a value of 25 psi, this will be known as the end-point of the RPVOT test. The time in minutes is reported as the oil's RPVOT value, and should be directly linear to the depletion of the antioxidant additive package which is degrading during operation. Consequently the number of minutes required to reach the RPVOT end point decreases as an oil begins to age in-service and indicates a loss of the RUL.
Initially the RPVOT test was used on turbine oil formulation using phenolic mixtures resulting in RPVOT test times between 300 and 600 minutes. Over the last decade, turbine

generator sets have been characterized by a significant increase in operating temperature, as well as life time, which resulted in the introduction of mixtures of aromatic amines and phenols. With these synergetic mixtures, the RPVOT values have significantly increased to values varying between 800 and 3000 minutes.

To test the Precision and Bias Statement of ASTM D 2272 (the RPVOT test), it was decided to conduct blind and non-blind testing of new and used turbine oils in four different laboratories in four different parts of the world. Samples of new gas turbine oil containing a phenols/amines mixture antioxidant package with a posted RPVOT value of 1400 and two (2) similar formulation used gas turbine oils were sent to four (4) well established petroleum testing laboratories in different regions. Only one of the laboratories was told the brand and type of oil, and which sample was new oil and which samples were used oils. The results of this testing are given in Table 1 below. [11]

Table 1 – Reproducibility Results for RPVOT Year 2004

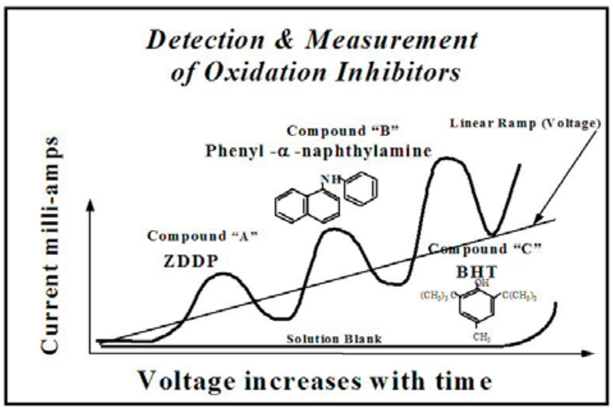
Same Sample to Four Different Labs in Four Different Parts of the World			
Lab & Area	**New Oil**	**GT No 1**	**GT No 2**
Lab 1 - SE Asia	750		
Lab 2 - USA	806		
Lab 3 - Europe	920		
Lab 4 - Australia	1490		
Lab 1 - SE Asia		818	
Lab 2 - USA		588	
Lab 3 - Europe		650	
Lab 4 - Australia		1440	
Lab 1 - SE Asia			1009
Lab 2 - USA			596
Lab 3 - Europe			950
Lab 4 - Australia			1385
New Oil Avg	992		
Oil No 1 Avg		874	
Oil No 2 Avg			985
** Note Laboratories 1 and 3 reported higher values for the used oils than the new oil.*			

It is interesting to note that the only laboratory that was able to successfully report the results in the correct sequence of new vs. used oils with the expected RPVOT ranges was the one laboratory that was told the oil type and which samples were new and used. A conclusion from this data could be that in true blind situations, the RPVOT test is not capable of detecting new oil from used on that particular type of oil.

3.2 RULER techniques (ASTM D-6971) for the measurement of total antioxidants

The working principle of the RULER™ method is based on voltammetric analysis (6,7,8,12) in which the oil sample is mixed with an electrolyte and a solvent, and placed in an electrolytic cell to detect the electrochemical (antioxidant activity). The oil samples (max. 400µl) are diluted in a prepared RULER™ Green test solution vial, enhancing the extraction of the antioxidants (AO[s]) into the solvent phase. When performing a voltammetric analysis, the potential across the electrodes varies linearly with time, and the resulting current is recorded as a function of the potential. With increased voltage to the sample in the cell, the various additive species under investigation in the oil oxidize electrochemically. A typical current-potential curve produced during the practice of the voltammetric test is illustrated in

Figure 3. Initially the applied potential produces an electrochemical reaction with a rate so slow that virtually no current flows through the cell. As the voltage is increased (Fig 3), the electro-active species (such as antioxidants) begin to oxidize at the microelectrode surface, producing an anodic rise in the current. As the potential is increased (from 0 to 1.7 V at a rate of 0.1 V/second), the decreases in the electro-active species concentration at the electrode surface and the exponential increase of the oxidation rate lead to a maximum in the current-potential curve (Fig. 3); this is the oxidation wave. The data recorded during this oxidation reaction can then be used to predict the remaining useful life of the lubricant, or used to evaluate the remaining antioxidant additives of the used samples. The peak of a zinc dialkyl dithio phosphate (ZDDP) additive is followed by an amine (PANA), and then by a hindered phenol (BHT) (see figure below).



<u>Figure 3</u>: Voltammetry as a technique for monitoring antioxidants in oils – Voltage vs. Current for different antioxidants e.g. ZDDP, amine and phenols.

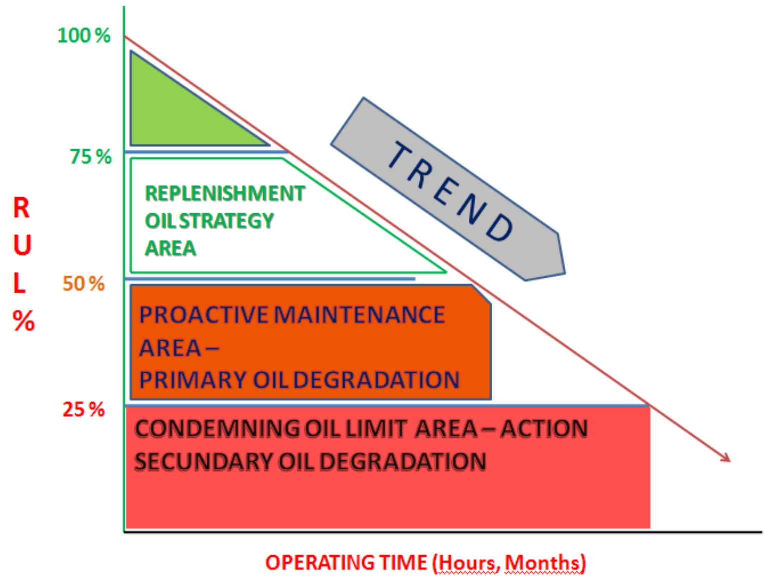
How is a RULER test performed?

- Dispense 400µl of the oil sample inside the vial, containing the electrolytic solution
- Shake vial for 10 seconds and let the solution settle for about 2 minutes
- Perform RULER test. The fresh lubricant is used as the 100% standard and the measurements of the used lubricant samples were expressed as percentage remaining additives While ASTM D-6810 [12] specifically covers the measurement of phenolic inhibitors in turbine oils, ASTM has approved a second standard, ASTM D-6971 [15], to measure the concentration of phenolic and aromatic amine antioxidants in non-zinc containing turbine oils. Voltammetric test practices are also part of ASTM practices for steam and gas turbine lubricant monitoring (ASTM D 4378-03 and D 6224-02) [12,13,14] The RULER instrument is also perfectly capable of measuring the oxidation stability provided by ZDDP and similar antioxidant/antiwear additives [7].

4. Experimental with Antioxidant Trending Analysis

With purpose of developing a balanced formulation between base oil and additives (in this case, more specifically the antioxidants) we can make an assumption that an industrial lubricant will truly be oxidized up to, or beyond, the critical point when antioxidants have been consumed, and the base oil will be undergoing secondary degradation (due to the lack of antioxidants to neutralize the C-radicals). It is well accepted that beyond this point, the AN- and viscosity oil values will show a significant increase over their new oil values.

In order to detect the critical point of antioxidants depletion, it is vital to establish a trending curve per individual equipment, showing the individual antioxidant depletion vs. the operating hours of the equipment (Fig. 4). By keeping the oil with a RUL% higher than 50%, via oil replenishment strategies, the oil's life will be extended significantly without any severe consequences or constraints for the lubrication system. The value of this trending curve is high in order to avoid (nearly) total exhaust of antioxidants resulting in excessive base-oil degradation, and the formation of irreversible oxidation products. Earlier research papers [19, 20, 21, 22] have also indicated that varnish formation reactions have a direct relationship between antioxidant depletion (phenols) and the formation of insoluble, submicron, soft contaminants (often referred to as varnish).



<u>Fig 4:</u> Trend analysis strategies as part of Root Cause Failure analysis

In addition to the RUL% trending, as part of Root Cause Failure Analysis, once a lubricant professional detects a significant change in RUL%-depletion (by comparing the trend incline over the time), it is advised to combine specialized laboratory testing such as FTIR, HPLC, or foam/air testing (at a specialized external laboratory). This combination of the on-site trend analysis, in combination with an external specialized laboratory will lead to a timely detection of abnormal oil condition, and consequently avoid the secondary oil degradation (or base-oil degradation).

4.1 Case Study 1 – Steam Turbine Monitoring

Attached is one of our recent case histories showing the advantages of individual antioxidant monitoring (using RULER technology) versus the overall anti-oxidant strength.

- The customer is a power plant in Asia Pacific using a new steam turbine, approximately two years old, using a high quality R&O oil with Zinc-based and Phenol-based anti-oxidant additives (peaks #1 at around 8 seconds and peaks # 2 at around 14.5 seconds). See Figure 4 below, where Zero hours is the new oil.
- The turbine had experienced several massive water floodings of the oil reservoir, which were successfully cleaned up each time with centrifugal separation.
- Our first analysis highlighted the fact that the phenol additive was nearly depleted at a very premature point in time of the oils' life (about two years). We felt this additive was "water-washed." Our FTIR analysis confirmed the Ruler results.
- The local agent of the oil company took a sample and paid for local RPVOT analysis and said the oil was fine, since it had more than 50% remaining life according to RPVOT.

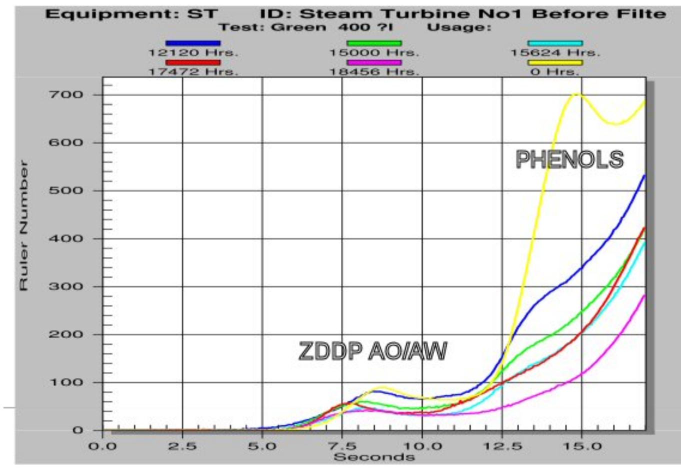
- The customer had faith in the RULER analysis and took our warnings seriously, took more samples, whereupon we finally said enough is enough, and recommended an oil change due to the total phenol depletion from water-washing of the additive.

The customer acquired more detailed testing information, other than just RPVOT analysis, and the results showed that the demulsibility and foaming characteristics of the oil started to really fall around June/July 06. (see Table 2 below)

Sample date	26 FEB 07	12 DEC 06	12 JUN 06	16 FEB 06	10 NOV '05
Demulsibility o/w/s/t	0/34/46 (30)	1/35/44 (30)	0/18/62 (30)	42/38/0 (25)	43/37/1 (20)

<u>Table 2:</u> Demulsibility and Foaming Characteristics

An Outage was scheduled for the upcoming low-peak demand season and the oil was changed



<u>Figure 4</u> Steam turbine lube oil with individual antioxidant depletion, antioxidant #1, ZDDP and antioxidant #2, phenolic type of antioxidant

<u>4.2 Case Study 2 – Gas turbine oil monitoring program</u>

Case Study 2 describes a case study from a large power plant, where oil analysis was performed locally through an oil laboratory, to report monthly Acid Number, viscosity, flash point and water content until massive deposits on the axial bearing pads of a gas turbine were reported.

<u>Figure 5</u>: Gas turbine axial bearing pad deposit

The analysis reports indicated fluctuations of data between 45.2 and 47.5 cStokes, and Acid Number increased from 0.17 to 0.26 mg KOH/g.

Oil cleanliness via particle counting indicated a 22/20 cleanliness according to ISO 4406, and Acid Number increased at that time to 0.30 mg KOH/g.
RULER testing for individual antioxidants was performed at that moment (Figure 6), with 15% of aromatic amines and 0% of remaining phenols for the respective oil sample. The tail from the graph also indicated a typical pattern when a high amount of soluble and polar oxidation products were present in the turbine oil.
The OEM recommended to the power plant to immediately change the oil, in addition to carefully cleaning the oil system, as a large amount of oxidation products were anticipated to be present in the lubricating oil system.
A more regular and adequate oil analysis program resulted from this case study, in order to avoid future gas turbine bearing deposits. It is also recommended to monitor the individual antioxidants, and certainly the phenolic antioxidants.

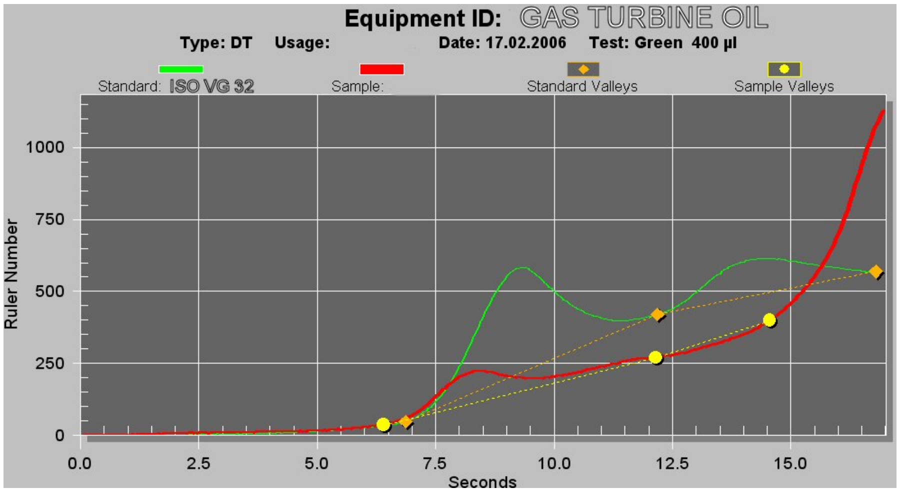
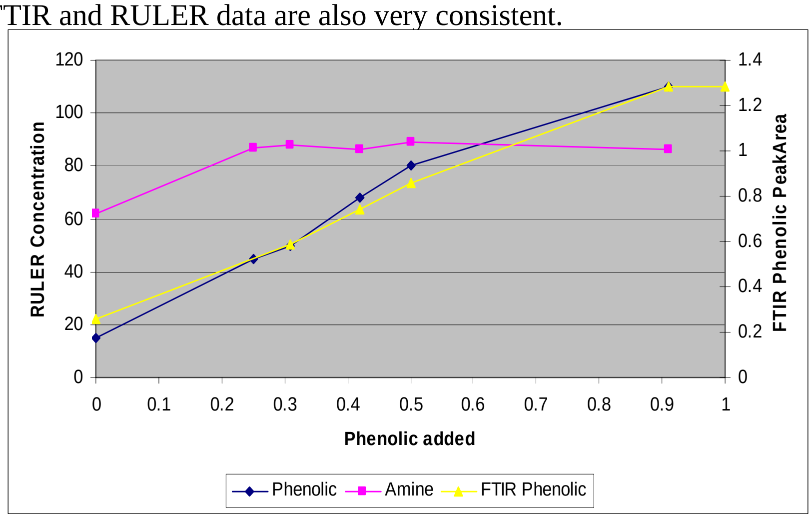


Fig 5: RULER graph from gas turbine oil before oil change out

4.3 Case Study 3: Additive replenishment strategies as part of Fluid Maintenance Procedures

It is suggested from the data in earlier research paper [19,20,21,22] that problems start to occur once the phenolic additive chemistry depletes. Research is underway to investigate the feasibility of replenishing phenolic additives back into used in-service turbine oils. Figure 6 shows one such project that was undertaken. It is interesting to note that the addition of phenols increased the amount of amines in the fluid by regenerating some of the degraded species. FTIR and RULER data are also very consistent.



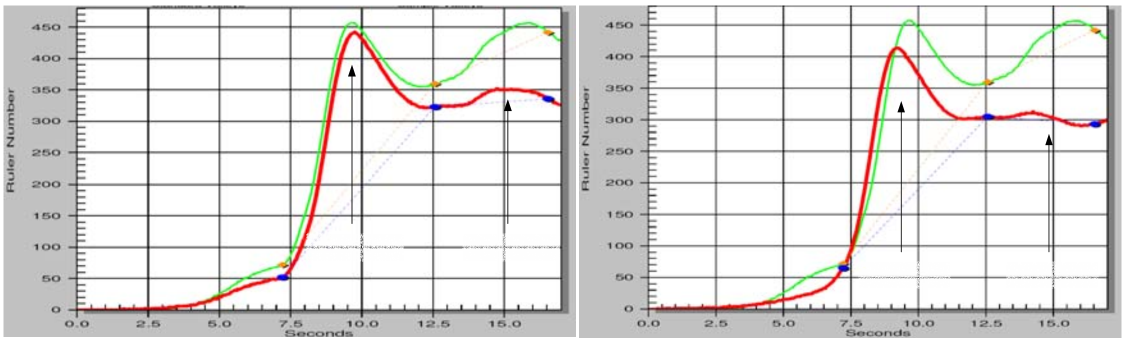


Fig 6: RULER and FTIR measurements of additive replenishment. Source: EPT, Inc.

Additive replenishment is technically feasible, and if carefully done, low risk. This practice has the potential of increasing the life of turbine oils by several orders of magnitude.

Summary

Antioxidant chemistry is playing an important role in meeting the longer-life and higher temperature performance criteria of modern generation industrial lubricants. The careful assessment of the antioxidant package, in combination with regular parameters such as cleanliness or varnish tendency have their impact as important parameters as part of today's industrial oil diagnostic services, where methods such as AN and RPVOT are not able to alert industrial lubricant users about the onset of (base-oil) oxidation and auto-degradation. The following points further summarize the findings in this paper:

- Traditional oil analysis methods cannot determine a lubricant's oxidative potential as part of proactive and condition based maintenance strategies
- Important condition monitoring tests of modern industrial oils include RULER, FTIR and MPC [21]. These tests monitor the condition of the antioxidant package and the creation of soft contaminants [22, 23], besides the traditional ones (AN, Viscosity, ISO particle count, etc).
- Electrostatic oil cleaning is an important technology to employ to remove insoluble degradation by-products that lead to varnish. When coupled with Ion Charge Bonding, which removes the soluble degradation products, one has a complete contamination control system.
- As a final note, a quick word on the process of additive replenishment has been technically proven, although this practice is not widely accepted. Future industrial lubricants, such as turbine lube oils operation, will likely incorporate an aspect of additive replenishment into their fluid maintenance programs to extend the life and performance of their fluids.

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