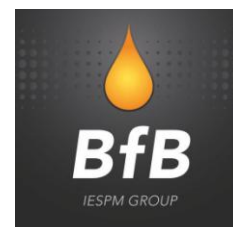


Overview of oxidation laboratory tests on industrial lubricants *part 1*

Vincent Bouillon

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INTRODUCTION

Developments in technologies and industrial material have led the additives and lubricants manufacturers to create higher performance lubricants. One of the key characteristics, besides anti-wear and EP resistance, good surface properties, and anti-corrosion protection is oxidation and thermal stability. There was a need to evaluate the performance of these oils using laboratory bench tests. The thermal and oxidation characteristics can be evaluated by many tests, some of which are detailed below. This paper relates to the following applications:

- Hydraulic fluids
- Turbine oils
- Compressor oils
- Industrial gear oils

Even if the control of fluid degradation including physico-chemical characteristics of the fluid as well as the monitoring of anti-oxidant depletion, the presence of degradations in the products is the key to detecting early stage of degradation and oxidation in order to avoid problems in the future. The very high quality level and performance of the formulated new oil remains essential.

The aim of this article is to give you an overview of existing laboratory oxidation tests and a guide to select the right test related to the application.

OIL COMPOSITION

In an industrial lubrication system, the lubricant is submitted to a lot of constraints like a wide range of temperature, the presence of hot spots, the evaporation and sometimes several top-up; these severe service conditions lead formulators to search for thermo-stable base oils as well as the most efficient additives.

Base Oils

Lubricating base oils are mixtures of a large number of chemical compounds and are therefore characterised by the following

physico-chemical properties: viscosity and viscosity index (indicates viscosity-temperature relationship), specific gravity, cloud and pour point – flash, fire and auto-ignition point, aniline point, composition (content of paraffinic, iso-p., naphthenics, aromatics, saturates, sulphur), carbon residue, volatility, air release value, water separability, thermal stability, eco-toxicity and biodegradability.

API (the American Petroleum Institute) established five base oil categories based on sulphur and saturates concentrations and VI.

Base oil category	Sulfur (%)	Saturates (%)	VI
Group I	>0.03	≤90	80–120
Group II	≤0.03	≥90	80–120
Group III	≤0.03	≥90	≥120
Group IV	All PAOs		
Group V	All others not included in groups I, II, III, and IV		

Group I contains oils that have >0.03% sulphur and ≤90% saturates by mass. These oils have a viscosity index in the range of 80-120. Group II and III oils, on the other hand, have ≤0.03% sulphur and ≥90% saturates by mass. However, they differ from each other in their viscosity index. The viscosity index for Group II oils ranges from 80-120 and viscosity index for Group III oils is ≥120. In general, Group II, Group III, and Group IV oils are low in aromatic and unsaturation. Hence, they oxidise at a slower rate than Group I oils which maintain a higher concentration in such structures. This is because such structures more readily form hydroperoxides and peroxy radicals that constitute the propagation stage of the oxidation process. Synthetic basestocks (Group V oils) have oxidation rates that vary because of varying structures. Alkylaromatics, for example, contain aromatic rings, and hence oxidise faster than ester basestocks, which in turn oxidise faster than olefin oligomers (PAOs) that belong to Group IV.

Vegetable oils oxidise at a fast rate as well because of the presence of unsaturation. New synthetic fully-saturated biobased fluids come into force through recent developments.

Additives

Although base oil quality has considerable impact on formulated oil's performance, additives are able to improve several existing properties of base oils as well as to give some new.

The following properties can be improved by chemical additives:

Viscosity

VI

Pour point

Rheological properties at low and high temperatures

Friction properties

Detergency

Dispersancy

Oxidation stability

Anti-wear

EP

Load-carrying capacity

Foaming

Water separation characteristics

Rusting and corrosion

Industrial oils generally do not need detergents and dispersants, since these do not come in contact with the fuel combustion products, but need anti-rust, anti-foam, and oxidation inhibitor along with anti-wear and EP additives (wherever these properties are required). The nature of these additives and dosage vary according to the equipment requirement and need to be optimised in every grade. Each lubricant specification has been carefully defined to meet these requirements. This is only possible through the use of suitable chemical additives. A careful selection of the additive combination is therefore necessary in a fully formulated lubricant. Additives exhibit both synergistic and antagonistic effects when used in combination, and this requires careful selection and evaluation. The dosage of each additive in a particular product is of great importance, since this decides the cost and performance of the product.

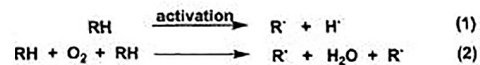
MECHANISM OF LUBRICANT OXIDATION & DEGRADATION

The process of oxidation proceeds in three stages: initiation, propagation, and termination. During the initiation stage, oxygen reacts with the lubricant to form alkyl radicals. During the propagation stage, these radicals react with oxygen and the lubricant to form peroxy radicals and hydroperoxides. As indicated by the oxygen uptake, hydroperoxides are accumulated during the induction period, after which the auto-acceleration of oxidation occurs. Hydroperoxides, either thermally or in the presence of metal, decompose to a variety of additional radicals and oxygen-containing compounds. The oxygen-containing compounds include alcohols, aldehydes, ketones, and carboxylic acids.

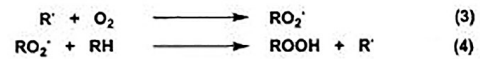
From these compounds, polymers, metal carboxylates can be formed; the latter can increase the rate of oxidation due to their catalytic effect.

Some metal salts, at low concentration, can act as oxidation inhibitors; this is the case for some copper salts.

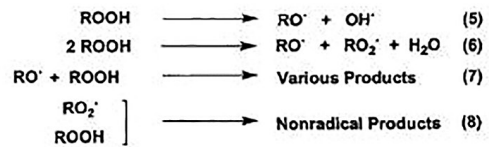
INITIATION



PROPAGATION



Decomposition of Peroxide:

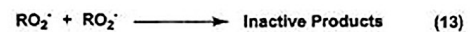


Induced Decomposition of Peroxide:

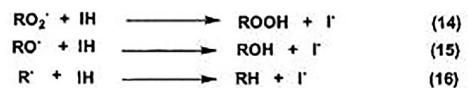


TERMINATION

Self Termination:



Chain-Breaking Termination:



RH = Organic substrate RO_2^\cdot = Peroxy radical ROOH = Hydroperoxide
X, Y, Z = Decomposers of Radicals RO^\cdot = Alkoxy radical R^\cdot = Alkyl radical
M = Metal IH = Chain-breaking inhibitor or inhibitor of free radicals I^\cdot = Inhibitor radical

FIG. 3—Mechanism of oxidation.

From oxidation of lubricants and fuels, ASTM [2]

Some parameters affect the oxidation process profoundly:

- The temperature which is supposed to double the rate of oxidation every ten degree centigrade rise.
- The wear metals ~~esetite pf~~ water

If oxidation is not controlled, lubricant decomposition will lead to oil thickening, sludge formation, and the formation of varnish, resin, deposits, and corrosive acids.

OVERVIEW OF INDUSTRIAL OILS OXIDATION AND THERMAL STABILITY TESTS

General overview

Numbers and various methods are used to evaluate thermal stability and anti-oxidant properties; some are specific for certain types of lubricants. Nevertheless, all these methods attempt to simulate the oxidation phenomena at various operating conditions and in various mechanical components. They are more or less based on the same principle. The oil ageing depends upon :

- The thermal stress: temperature and temperature cycle
- The presence of air or oxygen at a specific flow rate or through a static pressure
- The presence of metal catalysts: this may be massive metals or organometallic compounds
- ~~esetite pf~~ water

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The evaluation of the oxidation stability is determined by the follow-up of some parameters:

- The evolution of the characteristics of the fluid (viscosity, acidity, additives depletion, metals concentration from the catalysts, peak area increase (carbonyl peak by IR spectrometry).
- The volatile acidity.
- The evaluation of the corrosion on metal specimens, including the weight loss.
- The quantification and appearance of insoluble materials coming from oxidation (deposits, sludge, varnish).
- The pressure drop as indication for the induction time.

Considerable efforts have been expended in the development of better methods to evaluate the oxidation resistance of lubricants.

Base oils

Most of the thermal stability and oxidation tests are dedicated to fully formulated lubricants. Nevertheless, taking into account the wide variety of base oils in terms of chemicals structure and performances, some “soft” tests may be carried out to differentiate the base oils:

IP-306: Determination of Oxidation Stability of Straight Mineral Oils

This method is designed to give an indication of the oxidation stability of straight, unadditised, mineral oil based lubricants under specific conditions; the test time is reduced to 48 hours and either no catalyst or a solid copper catalyst is used. The degree of oxidation is expressed as “total oxidation products” (TOP) percent.

DIN 51554: Test of Susceptibility to Ageing According to Baader

The Baader ageing test is an accelerated oxidation test enabling the probable in-service behaviour of various lubricants to be predicted. The Baader test was developed to evaluate mineral oil based hydraulic fluids. However, today it has found wide acceptance in predicting the performance of biodegradable hydraulic fluids. Both vegetable oil (triglyceride) and synthetic ester based fluids are evaluated.

This is a non-severe oxidation test in which a copper coil stirs air into the lubricant at a rate of 25 cycles/min. The test conditions are 140 h at 110°C for insulating oils and synthetic ester hydraulic fluids. For mineral oil hydraulic fluids and vegetable based hydraulic fluids the conditions are 72 h at 95°C; these may also be applied to base oils. At the end of the ageing period, the viscosity of the aged fluid is determined and compared to the original fluid viscosity. The percentage of viscosity increase at 40°C is reported.

Turbine oils

Oxidation is the most important property of turbine oils, and high oxidation stability means longer lubricant life. Base oils as produced in the refinery do not have sufficient oxidation stability to support turbine oil performance. This property is therefore obtained by the incorporation of anti-oxidant molecules that function by interaction with the free radicals produced during the process of hydrocarbon oxidation. Different base oils respond differently to anti-oxidants and need to be investigated thoroughly before arriving at the turbine oil composition. Oxidation stability of turbine oils is evaluated by the main following methods:

ASTM D 943: Oxidation Characteristics of Inhibited Mineral Oils

This method was developed for and is used to determine the oxidation life of inhibited turbine oils. It is now widely used for predicting the oxidation life of anti-wear hydraulic oils, and R&O oils, as well as turbine oils. The test is designed to simulate the conditions found in a typical steam turbine system. The test oil is heated in the presence of copper and iron catalysts, which are typical of the metallurgy found in a steam turbine. Water is added to simulate steam condensate and finally, oxygen is introduced to accelerate the oxidation process. The degree of oxidation is determined by an increase in the acid number of the lubricant oil. The test is conducted in the following manner: 300 ml of test oil, along with catalyst coils of copper and steel are placed into a large glass test tube and placed into a heated bath, maintained at 95°C. 60 millilitres of distilled water is introduced into the test tube. A water-cooled condenser is used to prevent the loss of water vapour during the test. Oxygen is bubbled through the oil sample at a rate of 3 litres per hour (l/h). Periodic samples of the oil are taken and the acid number is determined. The test is usually concluded when the total acid number (TAN) reaches or increases 2.0 mg KOH/g. The number of hours needed is considered to be the “oxidation lifetime” of the oil.



This test method is widely used for specification purposes like in ISO 8068, DIN 51515 part 1 L-TD, Siemens TLV 901304, Mitsubishi MS4 – MA – CL 001 002 003, General Electric GEK 107395A, and is considered useful in estimating the oxidation stability of lubricants. Uninhibited oils will usually fail within 200 hours, while high quality oils can exceed 5000 h – 10.000 h. However, it should be recognised that the correlation between this test and actual field performance can vary markedly. It is assumed that the longer the oxidation life is in the D 943 test, the longer the lubricant will perform in the field. It should be noted that the D 943 has an upper life limit of 10 000 hours. Values higher than 10 000 hours are considered to be nonstandard extensions of the method.

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D 4310: Determination of the Sludging and Corrosion Tendencies of Inhibited Mineral Oils

This method is a modified alternate to the ASTM D 943 test method, and is used to determine the tendencies of inhibited mineral oils, especially turbine oils, to form sludge during oxidation.

The test conditions described under ASTM D 943 are used. After 1000 hours, the test is stopped. The oil and water layers are separated and filtered. The weight of insoluble material is determined gravimetrically by filtration of the oxidation test tube content through a 5 micron pore size filter. The amount of copper in the oil, water, and sludge phases can be determined according to any suitable methods.

This method is used primarily for specification purposes. Formation of oil insolubles or metal corrosion products during this test may indicate that oil will form insolubles or corrode metals, or both, during field service. However, correlation with field service has not been established.

D 2272: Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel Oxidation Test (RPVOT)

The RPVOT is a rapid method of comparing the oxidation life of lubricants in similar formulations, in the presence of water and a copper catalyst. This method can be used to evaluate the oxidation characteristics of turbine oils, hydraulic oils, and transformer oils. The test apparatus consists of a pressurised vessel axially rotating at 100 rpm, at an angle of 30° from the horizontal, in a bath maintained at 150°C. Fifty grams of test oil, 5g of distilled water, and a freshly polished copper coil are placed into a glass liner, and inserted into the vessel. The vessel is initially pressurized with oxygen to 600 kPa at room temperature.

At 150°C, the pressure in the vessel increases to approximately 1400 kPa. When oxidation occurs, the pressure drops, and the usual failure point is taken at 175 kPa from the maximum pressure obtained at 150°C. The results are reported as the induction time which is the number of minutes to reach 175 kPa loss.

The RPVOT is favoured as a quality control test because it is rapid. The RPVOT result is useful in controlling the continuity of this property for batch-to-batch acceptance of production lots, having the same composition. The RPVOT is also useful in determining the remaining oxidation life of in-service systems by charting the original RPVOT value versus subsequent samples of that system. It should be noted that the D 2272 test method is dependent on additive chemistry. No correlation has

been established between RPVOT and ASTM D 943 methods neither between actual field service. Mitsubishi specification for heavy-duty turbine oils requires RPVOT (ASTM D 2272) retention value in addition to the ASTM D-943 test.

ASTM D 7873: Determination of oxidation stability and insolubles formation of inhibited turbine oils at 120 °C without the inclusion of water (Dry TOST Method)

A total of six to eight tubes containing 360 ml of sample without water are heated at 120 °C with oxygen in the presence of an iron-copper catalyst. Each tube is removed over time and the sample is analysed by Test Method D2272 and the insolubles are measured until the RPVOT residual ratio reaches below 25 %. The criterion is to maintain less than 100 mg/kg of sludge at a RPVOT value corresponding to 25 percent of the new oil. The 100 mg/kg limit was determined by Mitsubishi Heavy Industries (MHI) based on field experience with their turbines and hydraulic control systems.

IP-280: Determination of Oxidation Stability of Inhibited Mineral Turbine Oils

This method is commonly used for European specifications relating to turbine oils and other hydraulic fluids. This method is technically identical to the CIGRE method 'Turbine Oil Oxidation Stability Test.' The test apparatus consists of a suitable sized test tube containing 30g of test oil, plus copper naphthenate and iron naphthenate as soluble catalysts. The sample test tube is placed in a heated bath, 120°C for 164 h. During the test period, oxygen is bubbled through the oil sample at a rate of 1.0 l/h. Both the test temperature and the oxygen flow rate must be carefully maintained throughout the test period. The volatile acids, soluble acids, and the sludge are used to calculate the "Total Oxidation Products" (TOP).

Because of the relatively short test time, this method is sometimes used as a replacement for the longer running ASTM D 943. However, no correlation between this test and the D 943 exists. Oils showing good results in D-943 test could fail in IP 280 test. Both the tests are antioxidant specific. Hindered phenols in adequate amount would show good result in D-943 test at 95°C but would sublime at 120°C in the IP 280 test and show poor result. The IP-280 test, therefore, require high-temperature antioxidants. It is, however, possible to design turbine oil by using complex mixtures of antioxidants, which will show up good results in both ASTM and IP tests.

To be continued in LUBE 141.

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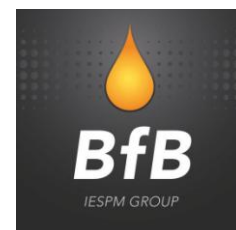
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Overview of oxidation laboratory tests on industrial lubricants *part 2*

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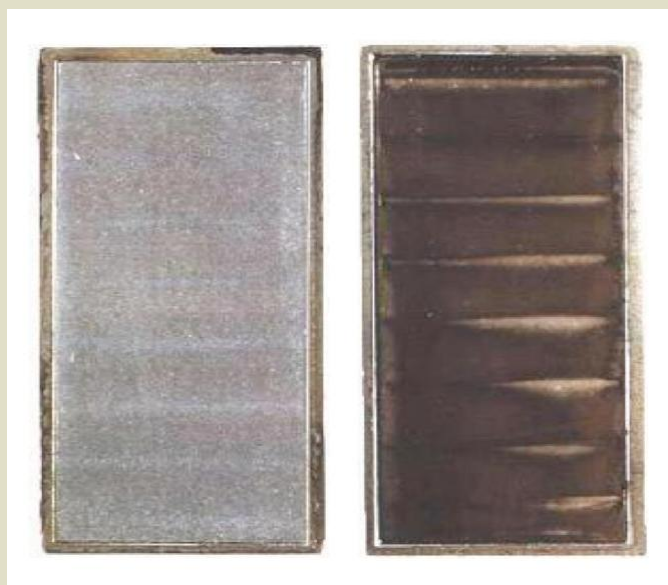


TURBINE OILS

FTM 791A – 3462 Panel Coker Test

The Panel Coker Test is a method for determining the relative stability of lubricants in contact with hot metal surfaces. The test apparatus consists of a rectangular stainless steel reservoir, inclined 25° from horizontal. The test panel (95 mm by 45 mm) is held in place by a heating element, which is fitted with thermocouple probes to control the temperature of the aluminium or steel test panel. A horizontal shaft, fitted with a series of tines, is positioned above the oil and is rotated at 1000 rpm. During rotation of the shaft, the tines sweep through the test lubricant and lubricant droplets are thrown onto the heated test panel.

The test panel is reweighed and the amount of deposit is determined. Weight gain of test panel and the amount of test lubricant consumed during the test are an indication of the lubricant's performance under high temperature conditions.



Many other test methods are dedicated to the evaluation of oxidation stability performance of gas and steam turbine oils as ASTM D 5846, D 6514, "as illustrated by the following examples.

AIRCRAFT TURBINE ENGINE OILS

The aviation environment offers tough conditions for the lubricants such as high temperatures, low pressure, very low temperatures, high load, etc. Therefore, special products are required for these applications. Synthetic oils are usually preferred for severe application. The test method ASTM D4636 is used to give the resistance to oxidation and corrosion tendencies of hydraulic oils, aircraft turbine engine lubricants, and other highly refined oils used in military aircraft and equipment.

ASTM D4636: Standard Test Method for Corrosiveness and Oxidation Stability of Hydraulic Oils, Aircraft Turbine Engine Lubricants and Other Highly Refined Oils.

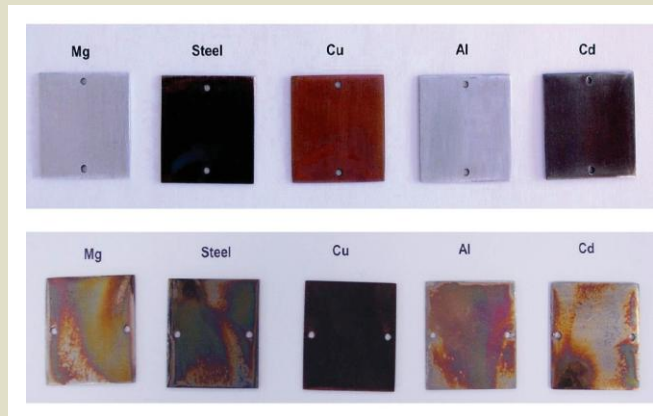
This method is the result of combining Federal test methods 5307.2 and 5308.7.

The test method can be used to evaluate mineral oils as well as synthetic fluids. It can be run using dry or moist air, as well as with or without the metal test specimens. There are two basic versions of this test method. Procedure 1 uses "washer" type metal specimens, which include titanium, magnesium, steel, bronze, silver, and aluminium.

Procedure 2 uses "square-shaped" metal specimens, which include copper, steel, aluminium, magnesium, and cadmium. The test is conducted in the following manner: The specified amount of test oil, 100 mL or 200 mL, is placed into a large test tube along with the polished and weighed test specimens. The assembled apparatus is weighed and placed into the constant temperature bath. Test temperature can range from 100°C to 360°C, for a specified amount of time. At the end of the test period the tube assembly is removed from the

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bath and the metal specimens are reweighed to determine weight loss, which is an indication of the oil's corrosiveness. The final viscosity and TAN of the oil is determined. Any sludge remaining in the test tube is determined gravimetrically. This method simulates the environment encountered by fully formulated lubricants in actual service, and uses an accelerated oxidation rate to permit measurable results in a timely manner. Interpretation of results should be done by comparison with data from oils of known field performance.



Specimen examples for Procedure 2 of ASTM D 4636

COMPRESSOR OILS

Low oxidation and carbon deposit formation is the most important property of reciprocating compressor oil since the oil in these compressors is subjected to severe oxidative and vaporising conditions in the air discharge system leading to the deposit formation. Thin film of oil or oil droplets come in direct contact with pressurised air in the presence of metal surfaces at high temperature. The oxidation of oil leads to viscosity increase and ultimately producing oil coke (carbon residue).

The problem of deposit formation in reciprocating compressors needs specific oil formulation to address two main tests provided in DIN 51506 specification. These are carbon residue of oil after aging at 200°C in the presence of iron oxide as catalyst and viscosity and carbon residue of the 20% residue after distillation. In order to meet these two requirements, it is necessary to select base oils with a narrow boiling range (so that heavy oils are avoided) and not to blend the products with very low and very high viscosities. This can be controlled by carbon residue of the distilled residue. The oxidation test requirements at 200°C can be met by selecting high temperature antioxidants based on amines. Mixture of several antioxidants would be useful in meeting oxidation test. Most hindered phenol-based antioxidants do not provide adequate protection at high temperatures.

DIN 51352: Pneurop Oxidation

This test method is designed to evaluate the oxidation stability of compressor oils, and is used to qualify compressor oils in European manufactured equipment. The test is conducted in the following manner: Soluble ferric (III) oxide is used as a catalyst and is measured into the glass test tube. 40 mL of test oil is then added into the tube. The assembled apparatus is placed in an oil bath or aluminium block at 200°C, and air is bubbled through the oil mixture at a rate of 15 litres per hour (l/h). The test is conducted for 24 hours. At the end of the test period, the apparatus is allowed to cool and reweighed to determine any evaporation loss. The oxidised oil is then submitted for determination of carbon residue using either the

Conradson Carbon Method ASTM D 189, or the Ramsbottom Carbon Method ASTM D 524. The amount of carbon residue formed in the oxidised oil is used to predict field performance. The carbon residue should not exceed 2.5% for an ISO 46 oil.

Many tests methods are also applied to compressor oils like DIN 51352/1 (IP 48), ASTM D 943, Thermal stability ASTM D 2070.

HYDRAULIC FLUIDS

Hydraulic systems offer an ideal condition for thermal and oxidative oil degradation due to the presence of air/oxygen, higher temperatures, water, and metals. Oil oxidation can generate harmful acids and sludge leading to system failure. These properties are measured by ASTM D-943 (TOST), ASTM D-2272 (RPVOT), ASTM D 5846 and CM heat tests.

For ester-based HF, the hydrolytic stability according to ASTM D 2619 can be evaluated.

The Baader test (an accelerated oxidation test) may predict the performance of biodegradable hydraulic fluids.

D 2070: Standard Test Method for Thermal Stability of Hydraulic Oils

At elevated temperatures, the long hydrocarbon chains in mineral oils may break apart into shorter hydrocarbon chain lengths (thermal decomposition). While some of the chains may vaporise and escape into the atmosphere, others tend to combine with other chains (polymerisation) to form hard, sticky by-products known as gums, varnish, and other deposits.

Thermal stability is a lubricant's ability to resist breakdown under conditions of high temperatures.

Cincinnati Machine (formerly Cincinnati Milacron), a leading manufacturer of machine tools, originally developed this test

method to assess the thermal stability of the zinc dialkyl dithiophosphates containing oils that were being used in their equipment. The motivation behind this test was the high cost of warranty claims this manufacturer experienced. The test apparatus consists of a beaker, a copper and steel test rod, and an electric convection oven capable of maintaining 135°C for 168 h. The copper and steel test rods are polished, weighed, and placed into a beaker of test oil. The rods are arranged in an "X" pattern with a single contact point. The assembled apparatus is placed in the test oven. This method does not involve the use of air or oxygen blowing, nor is any agitation involved. At the end of the test period, the test rods are compared to a reference chart to determine the degree of chemical attack. Ideally, the rods should show little evidence of any discoloration. The oil is evaluated to determine any changes in viscosity, to measure any increase in acid number, and to determine the amount of sludge.

This method is widely used for approval purposes and is useful in evaluating the thermal stability of lubricants. It is used primarily for hydraulic oils, but it can also be used to evaluate other industrial fluids.



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Examples of oxidation laboratory tests - BfB

D 2619: Hydrolytic Stability of Hydraulic Fluids (Beverage Bottle Method)

This test method covers the determination of the hydrolytic stability of petroleum or synthetic-based hydraulic fluids. A copper test specimen and 75 g of test fluid plus 25 g of water (or 100 g of a water-containing fluid) are sealed in a pressure-type beverage bottle (Cola bottle which can resist a high pressure). The bottle is rotated, end for end, for 48 hours in an oven at 93°C. This causes the layers to separate and the weight change of the copper specimen is measured. The acid number change of the fluid and acidity of the water layer are determined.

This test method differentiates the relative stability of hydraulic fluids in the presence of water under the conditions of the test. Hydrolytically unstable hydraulic fluids form acidic and insoluble contaminants which can cause hydraulic system malfunctions due to corrosion, valve sticking, or change in viscosity of the fluid. The degree of correlation between this test method and service performance has not been fully determined.

INDUSTRIAL GEAR OILS

Oxidation of oil at higher temperatures can lead to the formation of sludge and deposits. Viscosity will also increase due to degradation. Sludge and deposits clog oil passages, change heat transfer rates, and deplete additives' contents. Poor oxidation may thus reduce oil and equipment life. ASTM D-2893 method is used to evaluate oxidation stability of industrial gear oils.

The method ASTM D 5763: Oxidation and Thermal Stability Characteristics of Gear Oils Using Universal Glassware is a modification of D 2893.

D 2893: Oxidation Characteristics of Extreme Pressure Lubricating Oils

This test was developed to measure the high temperature oxidation stability of industrial gear oils. It is important to note that D 2893 was designed to run at 95°C. However, to meet the requirements of U.S. Steel, and AGMA 9005 (American Gear Manufacturers Association), the temperature is elevated to

Test name	Method	Oil (ml / g)	T°	Duration	Gas	Flow or Speed	Catalyst & water	Analyses	Application
Cincinnati Milacron	ASTM D 2070	200 ml	135°C	168 hours			Cu rod - Steel rod	Cu and Steel rods Evaluation. Total sludge (mg/100ml)	Hydraulic - Industrial Fluids (Turbine)
RPVOT	ASTM D 2272	50 g	150°C	Variable	Oxygen	620kPa - 100 rpm at 30° from horizontal	Cu catalyst coil - 5 ml water in oil & 5 ml water in the vessel body	Minutes for a 175 kPa pressure drop - determination of induction type	Turbine Oils (new and in service) -
Oxidation Characteristics of EP Lubrication Oils	ASTM D 2893	300 ml	95° / 121°C	312 hours	Air	10 l/h		Viscosity - Precipitation number	EP lubricants - Gear oil - Mineral oil
Sludging and Corrosion Tendencies	ASTM D 4310	300 ml	95°C	1000 hours	Oxygen	3 l/h	60 ml water - Iron & copper coil	Proc. A : Insoluble material - copper content in oil / water / Sludge, "AN" Proc. B : Insoluble material only, "AN"	Turbine (steam) - hydraulic - R&O
Oxidation Stability and Insolubles Formation of Inhibited Turbine Oils at 120°C Without the Inclusion of Water	ASTM D 7873 (Dry-TOST)	360 ml	120°C	Variable - Time to reach 25% RPVOT	Oxygen	3 l/h	No water - Iron & copper coil	Insolubles mass (mg/kg) is plotted against RPVOT Insolubles mass (mg/kg) at 25% RPVOT (or specific time) : Sludging tendencies	Steam & Gas Turbine oil - Industrial oils (circulating oils)
TOST	ASTM D 943 (TOST)	300 ml	95°C	Variable	Oxygen	3 l/h	60 ml water - Iron & copper coil	Acid number - oxidation lifetime	Turbine (steam) - hydraulic - R&O
Cincinnati Milacron	Cincinnati CM 10-SP-7989	200 ml	135°C	168 hours			Cu rod - Steel rod	Cu and Steel rods Evaluation, Weight loss & Deposits of Cu & Steel rods (CM). Total sludge (mg/100ml)	Hydraulic - Industrial Fluids (Turbine)
Baader ageing	DIN 51554	60 ml	95°C	72 hours	Air	By copper wire up and down 25X/min	Cu wire	Viscosity at 40°C increase - Acidity	Base oil - hydraulic - R&O
Panel Cocker Test (PCT)	FTM 791a-3462	300	315°C	8 hours		1000 rpm	Al (Steel) plate	Coking value (weight of coke in mg) Appearance of the Panel	Turbine - Compressor
Oxidation Stability of Inhibited Mineral Turbine Oils (CIGRE)	IP 280	30 g	120°C	164 hours	Oxygen	1 l/h	Fe naphtenate - Cu naphtenate - 20 mg/kg Cu & Fe	TOP - TS/SA/VA - If required Induction time based on VA	Turbine oil Hydraulic Fluid
Oxidation Characteristics of Lubricating Oils	IP 48	40 ml	200°C	2 x 6 hours	Air	15 l/h		Viscosity - CRR (% mass)	Compressor
TOST	ISO 4263	300 ml	95°C	Variable	Air	3 l/h	60 ml water - Fe and Cu wire	Acidity	Turbine (steam) - hydraulic - R&O
Ageing Characteristics	Pneurop	40 ml	200°C	24 hours	Air	15 l/h	0,4 Fe-oxide	% evaporation - CCR	Compressor

Examples of oxidation laboratory tests - BfB

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121°C. The other test conditions remain unchanged. The test method is designed to measure resistance to oxidation by determining the change in viscosity. In this test, the same glassware required by the D 943 test is used. 300 ml of the test lubricant is measured into the tube. The glass inlet tube from D 943 is used to introduce the air into the test lubricant. The entire assembly is placed in an oil bath at 95°C, (121°C). Dry air is bubbled through the sample at 10 l/h for a period of 312 hours. At the end of the test the final viscosity and precipitation number are determined. The appearance of the test tube and oil may be inspected for evidence of oxidation.

Qualifying industrial gear oils give 0-6% viscosity increase in test. Though unspecified, cleanliness of the glassware is very important for today's industrial gear oils. It is felt that glassware cleanliness is an indication of the lubricant's thermal stability.

SUMMARY

The purpose of oxidation testing is to study and evaluate the oxidation and thermal performance of formulated lubricants under severe simulated operating conditions in order to predict the performance of those lubricants in real world applications. There are two essential ways to test lubricants: bench testing and field testing.

Bench testing studies the performance of lubricants under simulated conditions and field testing predicts lubricant performance in actual equipment. Because of the high costs that are often associated with field testing, bench testing becomes the choice to study and evaluate lubricants. Bench testing can be used as a cost-effective way to evaluate the performance of experimental or new additives and formulated lubricants. It can be used to compare the relative performance of different commercially available lubricants, and finally it can be used to assess the remaining, useful oxidation life of lubricants in service.

Field testing can be used to prove the performance of lubricants that have been evaluated in bench tests and have successfully met the bench test criteria.

Even if the correlation between oxidation stability test results obtained by bench testing and field testing have not been systematically established, many oxidation and thermal stability tests have been developed by ASTM International; IP, The Institute of Petroleum; DIN, Deutsches Institute for Normurs; ISO, International Standards Organisation, and other organisations. These tests are designed to evaluate performance of additives and lubricant systems including industrial lubricants.

National and international specifications like ISO, DIN, ASTM, SAE as well as OEM specifications, Denison, US Steel, Cincinnati Milacron, Poclain, Vickers, Siemens, Mitsubishi, and General Electric give limits which are a great help as a guideline in the interpretation of test results.

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