

# FTIR CHANGES OF DIFFERENTLY DEGRADED MINERAL AND TURBINE OILS

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This paper investigates the oxidative degradation of mineral-based hydraulic and turbine oils using Fourier Transform Infrared (FTIR) spectrometry and RapidOxy 100 testing. A hydraulic mineral oil and two turbine oils of the same viscosity grade were studied under accelerated thermal and oxidative stress. FTIR analysis monitored additive depletion, base oil degradation (oxidation, nitration, sulfation), and contaminant accumulation (water, soot, fuel, glycol), while RapidOxy 100 tests provided oxidation induction times as a measure of resistance to degradation. Accelerated laboratory aging confirmed a consistent trend of decreasing oxidation stability and increasing oxidation/nitration products across all oils, with hydraulic mineral oil showing the fastest degradation. Results highlight the importance of base oil composition, additive system, and moisture control in determining oxidation stability and service life of lubricants in demanding industrial applications.

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## 1 Introduction

In general, oils suitable for use in hydraulic systems come from two different sources, biological or non-biological, which offer a large number of hydrocarbon compounds. These usually occur as complex mixtures and can be used for many purposes other than lubrication (i.e. controlling wear and friction). The required properties of lubricants in modern industry are on the one hand greater, and on the other hand increasingly diverse. Therefore, the selection and "design" of an appropriate mixture of hydrocarbons for lubrication is a professional and complex process. Most natural oils contain substances that can hinder the lubricating properties, but at the same time they also contain compounds that are essential for the lubrication process. Lubricants derived from natural or mineral oils are partly refined and partly impure. The balance between the impure and refined parts is crucial for the oxidation stability of the oil and is important for the area of application of the lubricant. So, it is necessary to deliberately add additives to the oils in order to improve their properties and to make them more suitable for a certain use under certain operating conditions. These can radically change the properties of the lubricants and are essential for its overall effectiveness. In addition, additives determine the specific properties of the lubricant, such as oxidation stability, protection against wear and friction, corrosion tendency, foaming, hardening, and other properties.

A typical mineral oil consists of 95 % base and 5 % additive. The physicochemical properties of the oil depend on both the additives and the base oil itself. Base oils can be classified into three basic categories: biological, mineral and synthetic, with mineral oils being the most commonly used lubricants in industry.

For long service life under harsher operating conditions, the oxidation stability of a hydraulic fluid is important. The oxidation stability of three different mineral oils will be discussed below, a hydraulic mineral oil and two turbine oils. All three oils are of the same viscosity grade.

## **2 Oxidation stability – a key performance parameter for long-life of hydraulic and turbine oils**

Oxidation stability is a critical property of hydraulic fluids, especially in demanding industrial applications where long service life, system reliability, and cost-efficiency are of paramount importance. Oxidation refers to the chemical reaction between the oil and atmospheric oxygen, which is accelerated at elevated temperatures, in the presence of catalytic metal contaminants (e.g., copper, iron), or in systems where moisture ingress occurs.

This degradation leads to the formation of a variety of harmful by-products, including organic acids, sludge, varnish, and insoluble compounds. These products can cause filter plugging, valve sticking, increased wear, and changes in oil viscosity - ultimately reducing the efficiency and reliability of the turbine or hydraulic system. In severe cases, oxidation-induced deposits may result in unplanned downtime, expensive repairs, and reduced equipment life [1].

The oxidation stability of the oil is strongly influenced by the type and quality of the base oil used, as well as by the formulation and performance of the antioxidant additive system. Carefully selected antioxidants are incorporated into the formulation. Over time, however, antioxidant levels deplete, which is why oxidation stability is also a key indicator of oil life. These improvements extend service intervals, reduce oil change frequency and waste oil generation, and lower maintenance and filter replacement costs. At the same time, system reliability increases and the risk of unplanned downtime decreases. In summary, proper oil selection and an understanding of oxidation stability have a direct impact on the efficiency, safety, and cost-effectiveness of system operation.

### **2.1 ASTM E2412 method**

ASTM E2412 test method [2] is commonly used to monitor the condition of used lubricants using trend analysis based on Fourier Transform Infrared (FTIR) spectrometry. It covers the use of FTIR to monitor additive depletion, contaminant accumulation, and base component degradation in machine lubricants, hydraulic fluids, and other fluids used in normal machine operation.

Oxidation stability indicates the resistance of a substance to chemical degradation in the presence of oxygen. It is crucial for fuels, oils and foodstuffs, as oxidation can cause rancidity of fats and oils, the formation of resins and deposits in fuels and shorten the service life of lubricants. Factors affecting the stability of a medium include the presence of air, light, heat, and especially moisture and metal ions, while antioxidants are used to slow down the degradation process.

Oxidation according to ASTM Standard (American Society for Testing and Materials), is used to measure and monitor the oxidation of materials, most commonly lubricants, fuels and other hydrocarbon-based products. These tests evaluate the resistance of a material to oxidation as well as identify and determine oxidation products using techniques such as Fourier Transform Infrared Spectrometry (FTIR) or by monitoring exothermic reactions. Key ASTM Standard include D943 for steam turbine oil oxidation, D2274 for diesel fuel stability and D7414 for monitoring oxidation in lubricants during their use.

In our case, the purpose of ASTM E2412 oxidation tests is to assess the oxidative stability of hydraulic oils. In other words, to determine how well the oil resists degradation when exposed to oxygen and heat, especially in applications where the oil is exposed to more demanding operating conditions such as those typical of hydraulic systems.

The ASTM E2412 test is based on the use of FTIR to track molecular changes in the spectrum of the oil, which can indicate the following three groups of changes that occur:

- Additive depletion: As additives are consumed, their characteristic absorption peaks in the spectrum decrease.
- Base stock degradation, which includes:
  - Oxidation: Formation of new carboxylic acids due to heat and oxygen exposure, a common form of oil degradation.
  - Nitration: Reactions that form nitrous oxides, particularly in high-temperature engines, which can lead to increased oil acidity and viscosity.
  - Sulfation: Formation of sulfuric acid from sulphur compounds and heat, leading to increased acidity and sludge.

In addition, the test also provides the occurrence of the formation and accumulation of the following contaminants:

- Water: A key contaminant and degradation accelerator.
- Soot: As products of incomplete combustion, which can be present in the oil.
- Ethylene glycol: A commonly present coolant that can also contaminate lubricants.
- Fuel: The presence of fuel can be detected by characteristic spectral changes.

Oxidation, nitration and contamination of hydraulic oil (test label H) and two different turbine oil (test labels M and C) were measured by Spectrum Two spectrophotometer with automatic sampler (Figure 1).



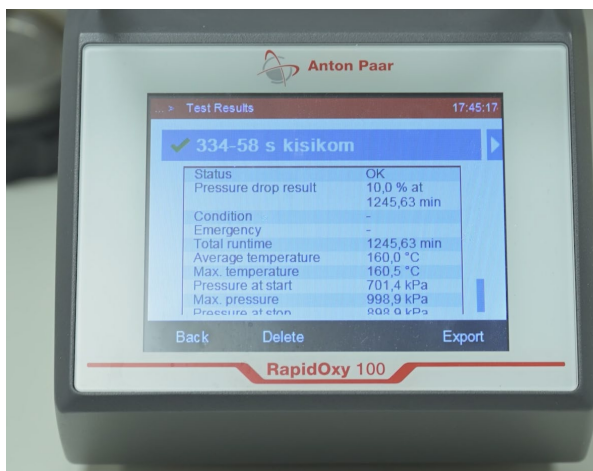
**Figure 1: Spectrum Two spectrophotometer with automatic sampler.**

Source: own

The spectrophotometer measures the transmittance and absorbance of IR light in the spectrum from 7,800 to 370  $\text{cm}^{-1}$ . The IR light source sends rays with the full spectrum through the sample on a ZnSe prism or between two KBr plates. The remaining light on the other side is detected by a sensor, which records the intensity of each wavelength and converts it into a graph. In this way, the presence of various additives, molecules, contaminants, oxidation, soot etc. can be determined [3].

## 2.2 Determining the oxidation stability of hydraulic oils

The oxidation stability of hydraulic oil (test label H) and two different turbine oil (test labels M and C) was evaluated using the RapidOxy 100 instrument (Figure 2).



**Figure 2: RapidOxy 100 instrument for measuring oxidation stability of oils.**

Source: own

RapidOxy 100 is a modern laboratory instrument designed for fast and reproducible determination of the oxidation stability of lubricants, fuels, and other organic materials. It is based on the accelerated aging method under elevated pressure and temperature, in which the pressure drop of oxygen (or synthetic air) in a sealed chamber containing the sample is continuously monitored.

Compliant with ASTM D8206 and DIN 51466 Standards, the instrument measures the oxidation induction time (OIT) - defined as the time it takes for the pressure in the chamber to drop by a defined percentage (typically 10 %) from its initial value. This parameter is a reliable indicator of oxidation resistance and, by extension, the expected service life of the oil. The measurement procedure includes the following steps:

- A small sample (approximately 5 mL) is carefully measured and placed into the steel pressure chamber of the instrument.

- The chamber is sealed and pressurized with pure oxygen or synthetic air to about 700 kPa.
- The sample is heated to a defined temperature (most commonly 140 °C), while the instrument continuously monitors the pressure drop.
- The test ends when the pressure has decreased by 10 %; the elapsed time is recorded as the test result (OIT in minutes).

Advantages of the RapidOxy method include [4]:

- Speed: the complete test is significantly faster than traditional methods (e.g., RPVOT, TFOOT), typically requiring only 1 to 3 hours.
- Low sample volume: suitable for R&D and high-value products where sample availability may be limited.
- Versatility: applicable to various types of oils (turbine, hydraulic, engine, compressor) and additive formulations.
- Excellent reproducibility and sensitivity: allows for reliable comparison of different formulations or production batches.

Due to its efficiency and robustness, RapidOxy 100 has become an essential tool in the development and quality control of high-performance lubricants intended for demanding industrial applications, where extended service intervals and high oxidation resistance are critical performance requirements.

### **3 Initial condition of the tested oils**

When monitoring the condition of hydraulic oils, it is important not only to know the current condition of the oil, but also to know the initial values of the observed parameters, i.e. the baseline values of the material properties of the oil. Knowing the baseline values is crucial for all further analyses and for monitoring the trend of changes in individual parameters. Table 1 gives the baseline values for the fresh oils under consideration.

The oxidation properties of hydraulic mineral oil HL type (test label H) and two different turbine oil (test labels M and C) is discussed. Turbine oil with the M designation, is a medium-high performance circulating lubricant specifically designed for steam and water turbine systems and other industrial applications. In

case of turbine oil labelled with C, is a turbine oil formulated with highly refined mineral base stocks (Group II base oils) and a complex additive package, including rust and oxidation inhibitors. It has no anti-wear additives and is designed for the lubrication of steam and industrial gas turbines, especially those with high local temperatures, due to its excellent resistance to foaming, air release and water separation properties.

Table 1 gives the experimental values obtained according to ASTM E2412 Standard. In our case study, oxidation stability and oxidation are in the foreground, as well as moisture (water) content and the content of AW additives. The other parameters listed are less important for our discussion. For example, it is immediately clear that turbine oil C has a very high oxidation stability, especially compared to mineral hydraulic oil H, and the lowest oxidation tendency value. The moisture content and the already mentioned AW additives present (typical for C) are also low.

**Table 1: Baseline values of monitored parameters for three types of oils**

Parameter	Label	H0	M0	C0
Oxidation stability (OS) (160 °C, 700 kPa, 10 % O <sub>2</sub> ) [min]		378.1	907.3	1353.6
Water (ASTM E2412)		6.9470	4.3432	3.7144
Soot (ASTM E2412)		3.2736	3.222	3.2467
Oxidation (ASTM E2412)		4.6453	2.7543	1.8388
Nitration (ASTM E2412)		3.0719	1.9743	1.5966
AW additive (ASTM E2412)		13.039	10.525	9.6119
Gasoline (ASTM E2412)		-0.0190	0.0117	0.01944
Diesel fuel (ASTM E2412)		261.36	207.997	200.157
Sulphates (ASTM E2412)		14.6555	11.5042	11.2905
Ethylene glycol (ASTM E2412)		0.4110	0.3059	0.4681

Oxidation stability (OS) is a substance's resistance to chemical breakdown and degradation caused by exposure to oxygen over time, which is measured by the duration it remains stable before undergoing undesirable changes. Data on oxidation, nitration and sulfation by-products indicate the degree of fluid degradation. Oxidation testing performed by FTIR measures the breakdown of a lubricant due to age and operating conditions and is reported in abs/cm (absorbance units per centimetre). By observing specific absorption, FTIR testing detects the presence of carbonyl groups (C=O), such as ketones, esters, and carboxylic acids, that result from oxidation in the oil. Nitration testing is also performed using FTIR (ASTM E2412 method), which indicates the presence of nitric acid, which speeds



up oxidation. Nitrates exhibit peaks in the infrared spectrum, allowing FTIR to identify their presence in the oil.

An OS value expressed (in minutes), indicates how long a substance, such as oil, can resist degradation from oxygen. A higher OS value signifies greater resistance to oxidation, meaning the product will have a longer useful life and perform better under stress before producing undesirable gums, sediments, or rancid odours. A lower value indicates a substance is more prone to oxidation and has a shorter shelf life.

An oil oxidation value indicates the extent of chemical aging and degradation in an oil due to exposure to heat, oxygen, water, and metals. A low value generally signifies good oil quality and longevity, while a high value signals that the oil is breaking down, forming sludge and varnish, and needs to be changed to prevent equipment damage. For mineral oils, an oxidation value above 30 or 40 may indicate the end of the oil's life.

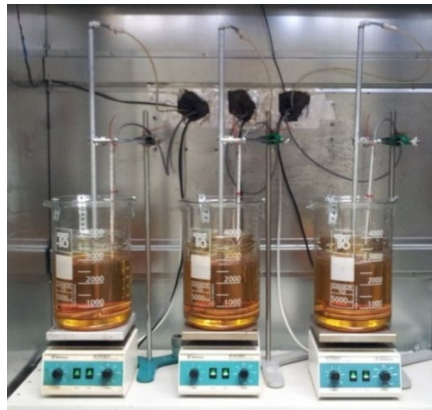
The table clearly shows that both turbine oils have much better oxidation resistance than mineral hydraulic oil. This is especially true for turbine oil with the C designation.

#### **4 Accelerated degradation process of hydraulic oils samples**

For the purposes of accelerated oil aging, a self-developed laboratory dry thermal test was used, with which accelerated oil aging can be performed on a larger sample volume (1500 mL), which enables a wide range of laboratory analyses to be performed. It basically consists of two glass beakers, placed one inside the other, so that the smaller 3-liter one is placed inside the larger 5-liter one.

The larger one contains rapeseed oil as a heating (tempering) fluid, which, with constant stirring, ensures constant temperature conditions of the tested fluid. A copper wire with a cross section of 1.5 mm<sup>2</sup>, a length of 15 ± 0.1 m, twisted into a spiral with a diameter of 10 mm, is inserted into the smaller beaker, which serves as a catalyst (accelerating the degradation of the tested fluid). Oxygen is known to be a further accelerator of oil aging. Therefore, compressed ambient air with a flow rate of 3 ± 0.1 L/min is blown into the test oil, which is previously dehumidified using a ready-made group for pneumatic systems. The test is carried out at atmospheric

pressure in a closed test chamber. The temperature of the tested oil is maintained during the test, in our case  $160 \pm 0.5$  °C. The test setup is shown in Figure 3.



**Figure 3: Accelerated aging test of hydraulic oil – baseline condition.**

source: own

## 5 Results

As an example, Figure 4 shows the change in colour of a sample of C turbine oil over the course of thermal loading, briefly described in the previous chapter (more on this can be found in [5], [6] and [7]). For example, the C60 designation means that it is a C oil sample after 60 hours of thermal stress at 160 °C. The image clearly shows the colour changes in the sample and also the occurrence of varnishing and sludge formation. The colour change is more or less similar for the other two samples, another turbine oil (sample M) and a conventional hydraulic oil (sample H).



**Figure 4: Colour changes as a result of thermal stress in case of sample C.**

source: own

The measurement results for aged turbine oil sample C are presented in Table 2. All values refer to the mentioned ASTM E2412 Standard and oxidation stability measurements with RapidOxy 100. Values are rounded to two decimal places.

RapidOxy 100 and FTIR are two completely different methods for measuring the same phenomenon; namely, oil degradation as a result of chemical reactions, primarily oxidation and nitration. Both techniques have also already been standardized through various (mainly) ASTM standards. It is impossible to claim that one method is the “correct” one and the other is not. These are complex processes that are not easy to monitor and are essentially detected indirectly.

In our case, the RapidOxy 100 device was used to expose the sample to an accelerated oxidation process. The results of the two methods are not directly comparable, but it is possible to meaningfully track trends in the results obtained by both methods. The tables 2, 3 and 4 clearly show that the trend of the results is similar and meaningful.

**Table 2: Results for aged turbine oil C over testing time**

Parameter \ Label	C0	C60	C75	C90	C105	C150
Oxidation stability [min]	1353.6	1280.7	1191.7	39.0	37.7	61.0
Water	3.71	3.69	3.70	10.04	30.95	60.49
Soot	3.25	3.22	3.21	3.57	4.62	6.11
Oxidation	1.84	2.20	2.26	23.61	79.68	151.85
Nitration	1.59	1.48	1.47	3.10	8.68	16.91
AW additive	9.61	9.54	9.54	13.28	21.44	30.76
Gasoline	0.02	0.02	0.02	0.02	0.02	0.02
Diesel fuel	200.16	200.40	200.50	199.63	198.78	198.54
Sulphates	11.29	10.82	10.79	17.05	32.70	52.15
Ethylene glycol	0.47	0.64	0.64	0.849	0.883	0.39

The data show that the oxidation stability value gradually decreases from the beginning of the test until 75 hours of testing under the same conditions, while the oxidation value increases. However, at longer times of thermal stress under the test conditions, i.e. at 90, 105 and 150 hours, both values change significantly. At these testing times, a large drop in the OS value is visible, because these samples were exposed to moisture after testing. Thus, the interdependence of the moisture present on the OS is clearly given.

Similarly, Table 3 shows values for turbine oil (M), and Table 4 shows values for hydraulic oil (H).

**Table 3: Results for aged turbine oil labelled with M.**

Parameter \ Label	M0	M60	M90	M120
Oxidation stability [min]	907.3	1030.2	726.6	146.3
Water	4.34	4.08	4.06	12.94
Soot	3.22	3.23	3.24	3.73
Oxidation	2.75	2.65	3.60	29.59
Nitration	1.97	1.70	1.72	4.11
AW additive	10.53	10.40	10.46	14.00
Gasoline	0.01	0.01	0.01	0.01
Diesel fuel	207.99	207.00	205.76	203.01
Sulphates	11.50	10.85	11.04	18.30
Ethylene glycol	0.31	0.36	0.41	0.31

**Table 4: Results for aged hydraulic oil labelled with H.**

Parameter \ Label	H0	H60	H90	H110
Oxidation stability [min]	378.1	273.9	129.1	98.4
Water	6.95	6.50	10.35	12.91
Soot	3.27	3.34	3.77	4.07
Oxidation	4.65	6.11	14.16	19.39
Nitration	3.07	3.32	4.41	5.22
AW additive	13.04	12.62	14.55	15.63
Gasoline	-0.02	-0.03	-0.03	-0.03
Diesel fuel	261.36	259.01	254.49	252.30
Sulphates	14.65	14.35	16.76	18.10
Ethylene glycol	0.41	0.86	1.06	0.84

In all three cases it is evident that the oxidation stability decreases with aging. On the other hand, with oil aging, nitration and oxidation increase.

## 6 Summary

The study focused on understanding oxidation stability as a key performance parameter for long-life lubricants, especially of turbine oils. Oxidation is a primary cause of oil degradation, leading to sludge, varnish, viscosity changes, and reduced system reliability. Three mineral oils were evaluated: hydraulic oil H, turbine oil M, and turbine oil C.

- Baseline analysis: Turbine oil C had the highest oxidation stability (1353.6 min), followed by M (907.3 min) and H (378.1 min). FTIR revealed lower oxidation and nitration tendencies in turbine oils compared to hydraulic oil.
- Aging experiments: A self-developed accelerated aging test exposed oils to high temperature, oxygen, and catalytic copper. Results showed that with aging, all oils exhibited reduced oxidation stability and increased oxidation/nitration by-products. Moisture significantly accelerated degradation.
- Comparative results: Turbine oils, particularly oil C, demonstrated superior resistance to oxidation compared to hydraulic oil. RapidOxy and FTIR produced consistent trends, proving reliable for monitoring degradation though values are not directly comparable.

Overall, the findings confirm that turbine oils with higher-quality base stocks and antioxidant systems significantly outperform hydraulic mineral oils in oxidative stability. Moisture ingress and additive depletion are critical factors reducing oil lifetime. Proper lubricant selection and monitoring by FTIR and RapidOxy 100 are essential for extending service intervals, minimizing maintenance, and improving system reliability.

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