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THERMAL TESTS FOR TESTING OF DEGRADATION BEHAVIOUR OF MINERAL BASED HYDRAULIC OILS

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ABSTRACT
The tests concerned with lubricating properties and the service-lives of components usually use techniques and procedures similar to those occurring during the actual component usages. Therefore, testing is performed in approximately identical environmental conditions as during actual use. Particularly important are the temperature conditions, presence of contamination, presence of water and various metals, presence of alternating pressure, stop – start intervals…
These kind of mechanical tests are more suitable for tribological research of used components rather than the durability of used oil and predicting of him remaining useful life. For these purposes it is preferable to use thermal tests. This paper presents these kind tests with its own characteristics, particularities and limitations, and design a special thermal test that allows testing of larger quantities of sample required for a comprehensive analysis of the physical-chemical properties of degraded oil.
Keywords: mineral-oil-based lubricants, dry thermal tests, larger sample

1. MECHANICAL AND THERMAL TESTS FOR OIL DEGRADATION TESTING
The often used s.c. mechanical test for the determining of lubricant degradation, such as FZG test, Vickers pump test, Komatsu 500 hour test, Denison test, etc,, are very time consuming (they can last several weeks or months), they consume a lot of energy, based on a large amounts of fluids, require the use of specified real components, … and do not give an accurate, comprehensive and detailed answer regarding the degree of degradation of the fluid used. They are more appropriate for the tribological investigation of used hydraulic components.
Another group of tests are the tests developed for establishing the lubricant resistance to ageing, where the emphasis of the test is on evaluating the resistance to oil oxidation. Oxidation is the dominant reaction in the process of hydraulic oil use and ageing, and causes many physical-chemical changes evidenced in the viscosity increase, varnish and deposit formation, additive decomposition, "breakdown" of base oil, increased foaming, increased neutralization number and increased corrosion and rusting [1]. The lubricant oxidation is accelerated by increased temperature and presence of well-known contaminants like water, air and metals (catalysts). Accordingly, all these test types can be called the thermal tests, as the temperatures during testing are much higher than in mechanical tests. Oil degradation results in weak organic acids and insoluble oxidation products which gradually gather on the hydraulic component surfaces [2]. Practically all lubricants and oils on the mineral basis contain additives for oil oxidation control. They are called antioxidants and act on the principle of sacrificing mechanisms since they react and oxidize before oxidation of the bas oil expires.

2. A BRIEF OVERVIEW OF COMMON THERMAL TEST

Over the course of time, several standardized thermal oxidation tests have been introduced to establish the oxidation stabilities of fresh and used hydraulic fluids. They are based on exposing the hydraulic fluid to high temperatures and catalysts like air (oxygen), water and metals, copper and iron being the more important. A short survey of tests and the operating conditions is shown in Table 1 [1].

Table 1. Summary of oxidation tests and operating conditions [75].

Oxidation test (ASTM)	Gas	Pressure	Temperature	Catalyst
PDSC (D6186)	O_2	34.5 bar	180 °C	Fe
RPVOT (D2272)	O_2	6.2 bar	150 °C	Cu/Fe
UOT (D6514)	Air	Atmospheric	155 °C	Cu/Fe
UOT (D5846)	Air	Atmospheric	135 °C	Cu/Fe
TOST (D943)	O_2	Atmospheric	95 °C	Cu/Fe/H2O

The more established and more frequently used tests are RPVOT [3] and TOST [4] serving as a basis for our own developed thermal test, appropriate for testing greater oil test-volumes as in previously mentioned standardized thermal tests.

In the **ASTM D2272 – RPVOT** test a pressure vessel with oxygen containing oil in the presence of water and copper is used to determine the oxidation stabilities of fresh and used oils. The weighed oil sample (50 ±0.5 g) is poured into the vessel into which still 5 mL of water and 3 m of copper wire of 1.63 mm diameter are added. After the vessel has been closed, it is to be filled with oxygen up to 6.2 bar at room temperature 25 °C. The vessel is then dipped into the tempering bath (150 °C) at a 30° angle. During the test the vessel content is mixed at 100 rev/min. After the closed vessel has been dipped into the bath, the temperature and pressure in the vessel start to increase. In the course of the test the oil in the vessel reacts with oxygen (and water and copper), therefore the pressure in the system starts to decrease slowly. The test is considered to be completed when a certain quantity of oxygen has reacted with oil, which is detected as pressure drop in the vessel for 1.75 bar of the read initial maximum pressure.

The **ASTM D943 – TOST** test is used to determine the oil oxidation stability in the presence of oxygen, water, copper and iron. Copper wire of 1.63 mm diameter and iron wire of 1.59 mm diameter coiled into a spiral of 225 ±5 mm length are added to the oil sample (300 mL) poured into the oxidation cell. The cell is also provided with an oxygen supply pipe closed by a special cover into which water is added during the test and which functions as a moisture condenser. The cell is then dipped into the tempering bath at 95 °C, the oxygen supply being adjusted to flow 3 ±0.1 L/min. During the test the oil reacts with oxygen in the presence of water and catalysts (copper and iron wire) at 95 °C. The test continues until the oil neutralization number increases to 2.0 mg KOH/g or more. The test result is the elapsed number of hours and can also be called the oxidation lifetime of oil.

3. DEVELOPING A DRY THERMAL TEST FOR A GREATER SAMPLE QUANTITY

During the research regarding the oil degradation mechanisms it was discovered, that the both mentioned tests were not quite adequate for intended task, since they included only a minor oil sample quantity (RPVOT: 50 g and TOST: 300 mL). So, we were compelled to conceive our own thermal test allowing execution of accelerated oil ageing on a greater sample quantity in amount of 1500 mL, which is sufficient for all further extensive laboratory analyses of physic-chemical properties of tested oil. Test is based on the mentioned RPVOT and TOST tests and comprises oil heating on the magnetic heater/mixer in the presence of air blowing-in and in the presence of a copper catalysts.

The test apparatus consists of two glass cups, the smaller (3 L) being located in the larger one (5 L) by means of a special support as shown in Figure 1. The larger 5 L cup contains the rape seed oil serving as a heat transmitter and a heating bath for the smaller 3 L cup. In that way, the strongly increased temperatures at the bottom of the smaller 3 L cup containing the tested oil were avoided. Each of the cups contains also a magnetic tablet serving for oil mixing during execution of the test. The smaller cup contains 15 ±0.1 m of 1.5 mm^2 copper wire wound into a spiral of 10 mm diameter as shown in

Figure 2. Then a measured quantity of the tested oil sample 1500 ±10 mL is poured into the cup. The test and heating is executed in parallel on three magnetic heaters/mixers placed into a chamber with oil vapor suction. The magnetic mixers mix the oil in the tempering bath, as well as the tested oil in the smaller cup, at the rate of 300 ±50 rev/min. During the test the temperature 150 ±0.5 °C of the tested oil is maintained by means of additional temperature regulators (PID) and temperature sensing elements wetted into the tested oil. Prior the start of testing all temperature sensing elements were calibrated by dipping them into the same fluid at 150 °C to ±0.1 °C precision.

Figure 1. Preparation of glassware and copper wire for implementation of the test (left) and preparation of the samples (right).

After the start of the thermal test, when all samples reach 150 °C by means of heating and mixing, the blowing-in of air through a diffuser made of sintered balls starts. Compressed ambient air with 3 ±0.1 L/min flow, previously de-wetted by the use of the preparation group for the pneumatic system is blown-in into the test oil. The test takes place at the atmospheric pressure within a closed purpose made chamber with a built-on system for oil vapor extraction. The chamber allows execution of the test on the individual sample or on all three samples simultaneously as shown in Figure 2.

Figure 2. Preparation of implemented dry thermal test for accelerated oil ageing.

4. IMPLEMENTATION OF THERMAL ACCELERATED OIL AGEING TEST

As distinct from the RPVOT and TOST tests our test has no accurate fixed end-time, as our aim is not only to measure the tested oil oxidation stability but to "record" the accelerated oil ageing process throughout its entire service-life. Therefore, the test was executed on six identical samples of HLP ISO VG46 mineral hydraulic oil exposed to accelerated ageing conditions for different numbers of hours. In that way six differently degraded samples were obtained for executing further analyses.

After completion of the thermal test the samples were further analyzed in the chemical laboratory in accordance with the standardized procedures.
When the testing had been completed, a strong change of color (Figure 3) and an express odor of burnt oil could be detected on the samples. Inscriptions on presented samples represent the test duration in hours, e.g. HL 60 – hydraulic oil after 60 hour of testing.

Figure 3. Samples after completion of the test.

The results of the laboratory sample analysis are summarized in Table 2 and commented on hereinafter. The physical-chemical values allowing prediction of the current oil condition and further estimation of the remaining useful lifetime are gray-shaded in the table.

Table 2. Analysis results of individual samples after accelerated thermal ageing test.

Sample	HLP 0	HLP 40	HLP 50	HLP 60	HLP 70	HLP 90	HLP 110
Test duration [h]	0	40	50	60	70	90	110
Color [-]	2.0	6.0	7.5	> 8.0	> 8.0	> 8.0	> 8.0
Flash point [°C]	220	226	236	220	224	224	218
Viscosity at 40 °C [mm^2/s]	46.45	48.35	49.07	49.30	49.75	51.74	62.18
Viscosity at 100 °C [mm^2/s]	6.91	7.06	7.15	7.18	7.21	7.40	8.08
Viscosity index [-]	104	104	104	104	103	103	96
Neutralization number [mg KOH/g]	0.54	0.65	0.68	0.72	0.77	1.43	1.9
FT-IR Oxidation [-]	0.31	0.43	0.51	0.64	0.76	1.28	2.30
Four ball weld load [kg]	130/140	130/140	130/140	130/140	130/140	130/140	140/150
Four ball wear test [mm]	0.5	0.5	0.5	0.5	0.5	0.55	0.7

Based on the results, it appears the trend of changes in individual parameters that can be used as the basis for predicting the oil remaining useful lifetime – RUL indicator (for more information see [5]).

5. REFERENCES

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