A Detailed Study and Comparison of a Variety of Bench Scale ASTM Oxidation Tests for Lubricating Oils

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ABSTRACT

Various ASTM test methods are currently used to test the oxidation properties of lubricating oils. They vary based on the type of lube oil they are attempting to test and the aim is usually to emulate under laboratory conditions, the real world conditions that the oils are subjected to. The primary factors that are usually available in the lab to simulate such a real life condition are the duration of the test, the temperature the sample is subjected to, catalyst that can be used to speed up the oxidation process, the atmosphere and condition under which the sample is tested etc. By changing these conditions for different kinds of oils, various ASTM oxidation tests have been created to study the oxidation and thermal performance of lubricants in the laboratory. Some of the lubricating oils that ASTM accounts for in their oxidation testing include hydraulic, R&O, turbine fuel oils, gear oils, greases, engine oils etc. The test methods include-

ASTM D 943-Oxidation Characteristics of Inhibited Mineral Oils

ASTM D 2893 – Oxidation Characteristics of Extreme Pressure Lubricating Oils

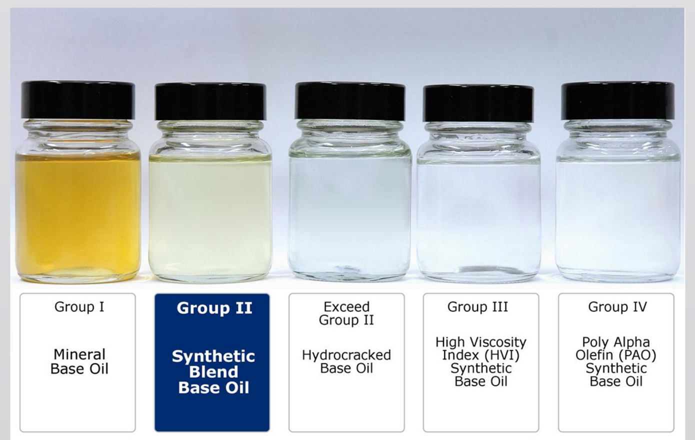
ASTM D 5763 – Oxidation and Thermal Stability Characteristics of Gear Oils Using Universal Glassware

The goal of this study is to look at these various test procedures already developed, and to attempt to create a more universal procedure / apparatus that can be modified by the user in their laboratory to run their own research protocols, while developing newer and longer lasting lube oils. In addition to that the objectives is to evaluate the performance of lubricating oils and additives in certain conditions.

BASE OIL AND ADDITIVES

API (American Petroleum Institute) has established five base oil categories on the basis of percent sulfur, percent saturates and Viscosity Index (VI).

Category	Composition	Sulfur	Viscosity Index
Group I	<90% Saturates	>0.03%	80-120
Group II	≥90% Saturates	≤ 0.03%	80-120
Group III	≥90% Saturates	≤ 0.03%	≥ 120
Group IV	(Poly) alphaolefins (PAO)		
Group V	All others not included above		



Base Oil Categorization [1]

Additives-

- Corrosion protection
- Ageing protection
- Anti-foam
- Anti-wear (AW)
- Extreme pressure (EP)
- Viscosity Index Improver

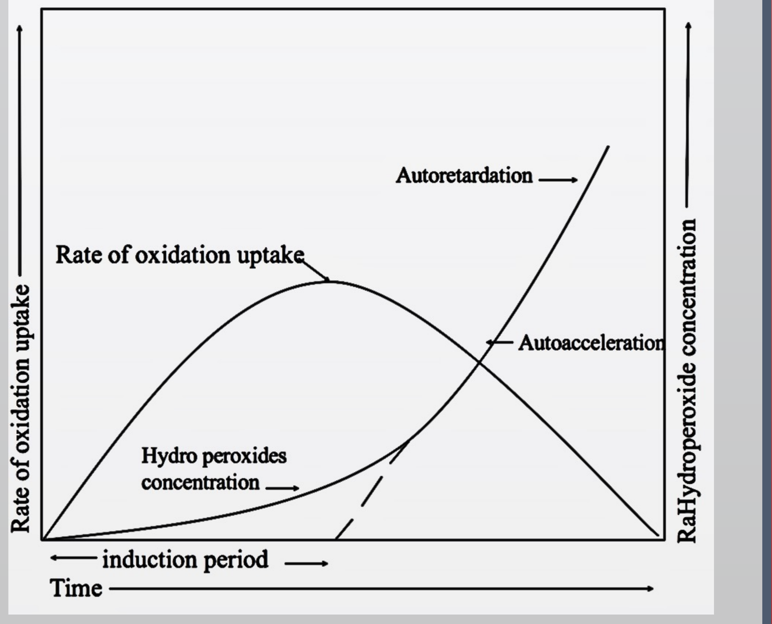
Antioxidants-

- Slow down the breakdown of the base fluid caused by oxygen (air) and heat
- Oxidation is the main cause of lubricant degradation in service

PROCESS OF OXIDATION

Oils used in the petroleum industry are hydrocarbon based that makes them susceptible to water. During the oxidation process, oxygen in the system readily reacts with labile hydrogens of the hydrocarbon structures that make up the lubricant. The oxidation of the hydrocarbons proceeds in the following three stages:

- **Initiation Stage-** the hydrocarbon molecules react with various catalysts leading to the formation of free radicals. Consequently, further initiations of the oxidation sequence may continue as the existing free radicals progress to the propagation stage.
- **Propagation Stage-**Oxygenated compounds (aldehydes, ketones, alcohols, water) and reactions with antioxidants (aromatic amines, phosphates) results in forming alkyl radicals.
- **Termination Stage-** The chain is ended by termination reactions in which free radicals collide and combine their odd electrons to form a new bond. It involves the eventual end of the oxidation process, either positively or negatively. Negative refers to the depletion and continuation of the oxidation process and the positive refers that the antioxidants likely has stunted the oxidation's progression.



Effect of Hydroperoxide Concentration on the Rate of Oxidation [2]

TEST METHODS

Hydraulic Oil

High Temperature Convertible Oxidation Bath

Cigre Bath

Corrosiveness and Oxidation stability Test Apparatus

Gear Oils

L-60-1 Performance Test Apparatus

Engine Oils

RPVOT Oxidation Bath

- **ASTM D943 – Oxidation Characteristics of Inhibited Mineral Oils**

This test method is used to determine the oxidation life of inhibited turbine oils. This method is also used for testing other oils, such as hydraulic oils and circulating oils having a specific gravity less than that of water and containing rust and oxidation inhibitors.

- **ASTM D2274 – Oxidation Stability of Distillate Fuel Oil (Accelerated Method)**

Measures the inherent stability of middle distillate petroleum fuels under specified oxidizing conditions at 95ºC. This method provides a basis for the estimation of the storage stability of middle distillate fuels such as No.2 fuel oil.

- **ASTM D4310 – Determination of the Sludging and Corrosion Tendencies of Inhibited Mineral Oils**

Determining the ability of turbine oils to form sludge during oxidation. This method evaluates the tendency of inhibited mineral oil based steam turbine lubricants and mineral oil based anti-hydraulic oils to corrode copper catalyst metal to form sludges in the presence of oxygen, water, and copper and iron metals at an elevated temperature.

- **ASTM D5704 – Evaluation of the Thermal and Oxidative Stability of Lubricating Oils Used for Manual Transmissions and Final Drive Axels**

Determines the corrosion of gear lubricants under sever thermal oxidation conditions. It covers the oil thickening, insoluble-formation, and deposit-formation characteristics of automotive manual transmission and final drive axle lubricating oils when subjected to high temperature oxidation conditions.

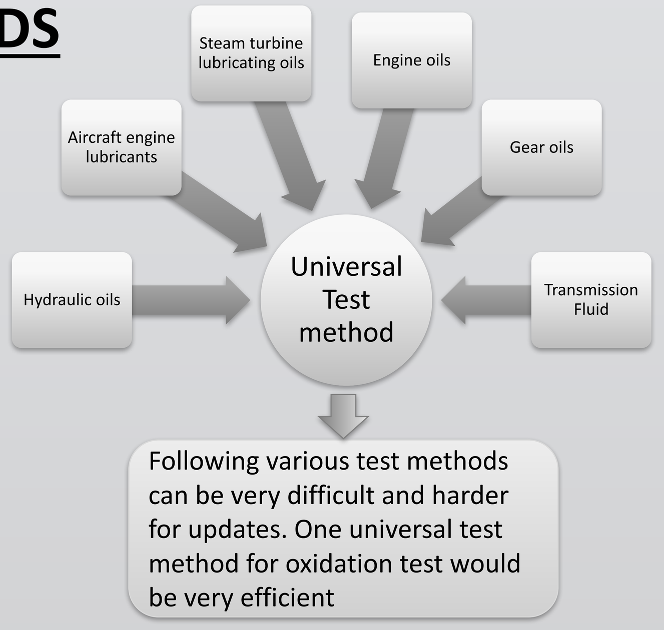
- **ASTM D4742 – Thin Film Oxygen Uptake Test (TFOUT)**

Evaluates the oxidation stability of engine oils for gasoline automotive engines. This test, run at 160 degrees C, utilizes a high pressure reactor pressurized with oxygen along with a metal catalyst package, a fuel catalyst, and water in a partial simulation of the conditions to which an oil may be subjected in a gasoline combustion engine.

SIGNIFICANCE OF TEST METHODS

These test methods are very important for the following reasons:

- Identifying insoluble materials formed in oils that are subjected to oxidizing conditions
- Measuring the tendency of automotive manual transmission and final drive lubricants to deteriorate under high temperature conditions
- Testing oxidation stability of lubricating base oils with additives in the presence of different peroxides
- Understanding the correlation between results of different test methods.
- Estimating oxidation stability of lubricants that are prone to water contamination



FUTURE WORK

The future work of this study includes studying and evaluating the oxidation and thermal performance of formulated lubricants under several simulated operating conditions that may be faster and economical as well.

REFERENCES

1. Energy, Royal Global. "Base Oil." *Royal Global Energy*, Royal Global Energy, 11 Jan. 2018, www.royalglobalenergy.com/Text?id=38.
2. Totten, G. E., Westbrook, S. R., & Shah, R. J. (2006). *Fuels and lubricants handbook: technology, properties, performance, and testing*. West Conshohocken, PA: ASTM International.
3. Fitch, Bennett. "Identifying the Stages of Oil Oxidation." *Machinery Lubrication*, Noria Corporation, 15 June 2015, www.machinerylubrication.com/Read/30165/oil-oxidation-stages.
4. ASTM Standards D943, D2274, D4310, D5704, D4742

Acknowledgements

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