Industrial Lubricants: Applications and Impact of Additives on Performance

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Abstract

The performance and service life-time of industrial lubricants rely strongly on the action of components which are typically present at low fractions: the additives.

These lubricant additives have the following main functions:

- 1. Enhance properties of the base oil itself with additive types such as antioxidants, pour-point depressants and viscosity index improvers.
- 2. Suppress undesirable base oil properties with anti-foam and demulsifying agents.
- 3. Impart new properties either to the base oil itself with detergents or to the base oil metal interface with extreme pressure additives, anti-wear additives, corrosion inhibitors, metal deactivators, friction modifiers and tackiness agents.

Especially the additives acting on phase boundaries often are polar in nature, which promotes directional attraction to other polar phases (metal surfaces, dirt surface, sludge and water). Other additive types within the oil like the antioxidants are sacrificial, their mode of action consumes them. The additive types and their mode of action are comprehensively discussed in several monographsⁱ.

A multitude of lubricant additives are available, and they are selected for use based upon their ability to perform their intended function in the specific industrial oil (hydraulic, gear or turbine oil etc). They are also chosen for their ability to mix easily with the selected base oils, to be compatible with other additives in the formulation, to be cost effective and increasingly often due to their positive effect on sustainability.

Contents

1. INTRODUCTION

2. THE EUROPEAN INDUSTRIAL LUBRICANT MARKET

3. INDUSTRIAL LUBRICANT FORMULATIONS

- 3.1. Lubricant Categories
- 3.2. Base Oils
- 3.3. Additives
 - 3.3.1. Antiwear Additives
 - 3.3.2. Extreme Pressure Additives
 - 3.3.3. Antioxidants
 - 3.3.4. Antifoam
 - 3.3.5. Corrosion inhibitors
 - 3.3.6. Metal deactivators
 - 3.3.7. Dispersants/Detergents
 - 3.3.8. Friction modifiers
 - 3.3.9. Pour Point Depressants
 - 3.3.10. Viscosity Modifiers
- 3.4. Guideline formulations
- 4. CONCLUSIONS/SUMMARY

1. Introduction

The purpose of ATC as a European lubricant additives manufacturer sector group is to ensure communication with all fuel and lubricant industry, customer, original equipment manufacturer and governmental stakeholder groups. This paper intends to provide relevant information of a technical nature to allow focus on future priorities of industrial lubricant technology developments.

Lubricants are fluids or greases which reduce friction between surfaces in mutual contact. This allows machine parts to function under severe conditions and expands their operational range. Adequate lubrication allows smooth, continuous operation of machine elements, reduces the rate of wear, and prevents excessive stresses or seizures at e.g. bearings. The performance and service lifetime of industrial lubricants is strongly improved by the action of components (i. e. the additives) which form only a small fraction (low % rate) of the finished fluid. This leverage effect of small additive fractions enables to formulate finished fluids on a more sustainable basis.

The aim of this document is to show the contribution of additives to lubricant performance.

Different types of additives for industrial lubricants in terms of chemistry and function are presented in the main section.

2. The European Industrial Lubricant Market

Lubricants permeate every aspect of our life and are needed wherever machines are used. The document confines itself to industrial fluids excluding automotive applications like automotive gear and engine oils.

Major groups of industrial lubricants are hydraulic fluids, industrial gear oils, turbine and compressor oils, metalworking fluids and others.

The European lubricant market accounts for ca. 19% of the global 36,1 Mio tonnes per year (in 2017, reported by Lutz Lindemann, Fuchs, June 2018). The industrial oil categories considered in this document refer to typically approximately 25% of this global volume.

The pie chart shows an estimate for the European lubricant breakdown of major applications as discussed with regard to their additive use in the following chapters.



Figure 1: European lubricant market breakdown into major applications

The lubricant markets of Germany, the United Kingdom, France, Italy, and Spain amount to an estimated 3 million metric tonnes in 2019, equivalent to 8% of global lubricant demand (nearly 50% of the total lubricant demand in Europe). In these countries industrial oils and fluids form the majority of lubricants consumed, accounting for almost 60% of total demand. The transportation equipment manufacturing, machinery, primary metals, chemicals, and power generation industries lead the industrial segment in Europeⁱⁱ.





3.1. Lubricant Categories

Lubricants are used in a variety of industries with various requirements.

Differences exist in the loads, temperature ranges, pressures, on equipment uptime and maintenance intervals, etc. These factors determine the requirements with regard to equipment protection, oil life time, reliability expectations and energy efficiency.

Lubricant properties are adjusted by the application of additives in combination with either mineral or synthetic base oils.

Table 1: Overview of additives in selected industrial lubricant applications; HF=Hydraulic Fluid, IGO=Industrial Gear Oil, TO=Turbine Oil, CO=Compressor Oil, MWF=Metal Working Fluid, GR=Grease, x = typically used, (x)= occasionally used

Component	Components for typical formulations in selected application						
Application	HF	IGO	то	со	MWF	MWF (water based)	GR
Thickener							х
Antioxidant	х	х	х	х	(x)	(x)	х
Corrosion inhibitor	х	х	х	х	х	х	х
Antifoam	х	х	х	х	х	х	х
PPD	(x)	х		(x)			х
VI Improver	(x)	(x)		(x)			
AW additives	х	(x)	х	х	х	х	(x)
EP additives		х	(x)	(x)	х	х	х
Metaldeactivator and -passivator	(x)	х	х	x	х	х	х
Friction modifier	(x)	(x)					
Detergent, Dispersant, (D)emulsifier	(x)	(x)	(x)			x (emulsifier)	

3.2. Base oils

At least one base oil is used to blend an industrial lubricant, in many cases more than one type is used.

API group oils can be used together with a wide variety of additives.

The API groups are defined according to the base oils content of saturates, sulfur and the viscosity index.

Table 2: API oil groups and their characteristics

Group	Туре	Saturates [%]	Sulfur [%]	Viscosity Index	
I	Mineral base stock	< 90	>0.03	80 - 119	
П	Mineral base stock	≥ 90	≤ 0.03	80 - 119	
+	Mineral base stock	≥ 90	≥ 0.03	110 - 119	
III	Mineral base stock	≥ 90	≥ 0.03	≥ 120	
+	Mineral or synthetic base stock	≥ 90	≥ 0.03	> 140	
IV	Synthetic base stock Polyalphaolefins (PAO)				
V	all other base stocks not included in Group I - IV				

The base oil properties are measured according to the following test methods.

Saturates: ASTM D2007 Viscosity Index: ASTM D2270 Sulfur: ASTM D1552, D2622, D3120, D4294, D4927.

3.3. Additives

The following table summarizes chemistry and function of additives in industrial lubricants.

Table 3: Additive classes for industrial lubricants

Additive class	Chemistry examples	Function / Performance	Application/typically used in	
Antioxidants	Sterically hindered phenols, aromatic amines, phosphorous, sulfur	Lifetime extension (via quenching of alkyl radicals or peroxy- radicals)	All industrial lubricants	
Corrosion inhibitors	Surface active structures like e.g. long chain carboxylic acids or their derivatives	Protection of metal surfaces from corrosion, e.g. rust and extension of equipment lifetime (protect against moisture and oxygen	HF, IGO, TO, CO, MWF, GR	
Antifoam	Silicones, acrylates	Quick reduction of foam volume after oil churning (by mechanical destruction of the foam bubbles)	HF, IGO, TO, CO	
PPD	Polyalkylmethacrylates, EVA	Improving flowability at low temperatures and reduction of <i>Pour Point</i>	Depending on base oil choice and for applications at low temperature, e.g. arctic applications	
VII	OCP, Polyalkylmethacrylates, PIB, SBR	Extension of the temperature operating window via reduced temperature dependence of the fluid viscosity	Mainly HF and IGO, where wide operating temperature ranges occur	
AW additives	Organo- phosphorous and sulfur compounds with or without metal, e.g. Zn	Generating protective layers against wear	HF, IGO, TO, CO, MWF, GR	
EP additives	Organo-sulfur compounds	Generating protective layers against welding	IGO, TO, CO, MWF, GR	
Metal- deactivator and -passivator	Complexing agents, triazoles, thiadiazoles	Complexing dissolved copper or forming protective layers on copper surfaces, capturing active sulfur	HF, IGO, TO, CO, MWF, GR	
Friction modifiers	Organic: carboxylic acids and derivatives. Inorganic: layered systems like MoS ₂ or graphite	Reducing friction between moving parts	HF, IGO, TO	
Detergents, Dispersants	Succinimide derivatives, Phenates, Sulfonates	Keep particles in dispersion, avoid sedimentation	IGO, some HF	
Demulsifier	Polyethers	Separate water from oil	TO, HF	

3.3.1. Antiwear Additives

3.3.1.1. Purpose

Antiwear (AW) additives are expanding the working life of a lubricated device by protecting the surfaces. They are added to the lubricating oil to prevent wear under conditions where it comes to direct contact at high spots of the interacting surfaces due to rupture of the elasto-hydrodynamic lubricating film.

3.3.1.2. Chemistry used (examples)





Metal-dialkyldithiophosphate

Ashless dialkyldithiophosphate



Amine-neutralized dialkyldithiophosphate



3.3.1.3. Mode of action

Antiwear additives function in various ways under moderate stress and reduce the rate of continuous wear. Some AW additives deposit multilayer films (of e. g. phosphate glass) thick enough to supplement hydrodynamic films and prevent asperity contact. Some develop easily regenerative monolayer films that reduce the local shear stress between contacting asperities and are preferentially removed in place of surface material. Others bond chemically with the surface and slowly modify surface asperity geometry by controlled surface material removal until conditions conducive to hydrodynamic film generation reappear.



Figure 3: Mechanism of antiwear additives

3.3.1.4. Representative Test Methods

The "Four-Ball Test" was developed to evaluate the AW and EP properties of lubricants. The simple test machine consists of 4 steel balls arranged in the form of an equilateral tetrahedron and measures the protection provided by the lubricant under conditions of high pressures and varying sliding velocities at the balls' surface. The three lower balls are held immovably in a clamping pot, while the fourth ball is made to rotate against them. Test lubricant is added in the test pot, covering the contact area of the test balls. During the test, wear scars are formed on the surfaces of the three stationary balls. The diameter of the scars depends on the load, speed, temperature, duration of run, and type of lubricant. Increasing AW performance is demonstrated by lowering the wear scar diameter in the Four Ball test by addition of different types of antiwear additives.



rigule 4. Example wear scal diameters with different antiwear additives

A broadly used wear test is the Schaeffler FE-8 Axial Roller Bearing test rig according to DIN 51819. The test is generally used for gear oils and circulating oils.

3.3.1.5. Application

Anti-wear additives are widely used in industrial oils (hydraulic-, gear-, compressor oils and metalworking fluids - see table 1).

3.3.2. Extreme Pressure Additives

3.3.2.1 Purpose

Extreme Pressure (EP) additives expand the working load range of a lubricated device by modifying the metal surfaces. They are added to the lubricating oil to prevent cold welding under conditions where it comes to direct contact at high spots of the interacting surfaces due to rupture of the elastohydrodynamic lubricating film.

3.3.2.2. Chemistry used (examples)



Sulfurized synthetic esters (light color)



Sulfurized olefins

Figure 5: Chemical structures of extreme pressure additives

3.3.2.3. Mode of action

EP additives are expected to react rapidly with the metal surface under severe distress (high-speed, high-load, or high-temperature operation) where other protective films (either by absorption or chemical reaction) are removed, and prevent catastrophic modes of failure such as scuffing or seizures caused by metal-metal adhesion or welding. EP additives function by reacting with the metal surface and therefore being consumed to form a metal compound such as iron sulfide. Some EP additives prevent scoring and seizure at high speed and under shock loads; others prevent ridging and rippling in high-torque, low-speed operations. In both cases, EP additives and surface metal are consumed, and a smoother surface is created with an improved chance of hydrodynamic action, resulting in less local distress and lower friction. In the absence of such additives, heavy wear and distress well beyond the scale of surface asperities would occur accompanied by very high friction^{III}.

3.3.2.4. Representative Test Methods

EP properties are measured by increasing load in the "Four Ball Test" (see Anti Wear Additives) up to the level at which the ball movement is blocked due to welding (see figure 6).



Figure 6: Four ball EP welding end of test specimen

The maximum load which does not lead to blockage is called weld load provided by a lubricant.

The following graph shows how the weld load (a force, measured in Newton, N) in the Four Ball test increases with rising concentration of a sulfurized triglyceride (indicated as concentration of sulfur in the oil).



Figure 7: Four ball welding load result versus Sulfur content

3.3.2.5. Application

Extreme Pressure additives are important for metalworking fluids and gear oils, and are also used in greases and optionally in hydraulic, turbine and compressor oils.

Guideline formulations are shown in chapter 3.4.

3.3.3. Antioxidants

3.3.3.1. Purpose

Antioxidants are used to prevent the oxidation of lubricant oils to produce high molecular weight polymers, insoluble and acidic species, which can cause oil darkening and thickening leading to loss of performance. Antioxidants will extend the lifetime of the oil as well as the equipment where it is used.

3.3.3.2. Chemistry used

There are three main classes of Antioxidants: Primary (which react with free radicals), Secondary (which react with peroxides) and Tertiary (which react with metals).

Primary Antioxidants (AO)

Primary AOs react with free radicals formed by the degradation of oil. Examples are Phenolic and Aminic antioxidants.

Phenolic Primary Antioxidants

Typical chemistry for phenolic AOs is a hindered phenol ring with alkyl groups (R) to provide solubility or other properties with some phenolic AOs also having an ester functionality. Phenolic AOs are usually more effective at lower temperatures (e.g., below 120°C).



Figure 8: Chemical structures of phenolic antioxidants

Aminic Primary Antioxidants

Aminic AOs are typically either based on phenyl-alpha naphthylamine or diphenylamine chemistry. Aminic AOs are especially effective at higher temperatures (e.g., above 120°C).



Aminic AO based on diphenylamine



Aminic AO based on phenyl-alpha naphthylamine

Figure 9: Chemical structures of aminic antioxidants

Secondary AOs

Secondary AOs react with free radicals formed by the degradation of oil and typically contain phosphorus (e.g. phosphites) or sulfur (e.g. thioesters).

Some metal-containing lube oil components such as ZDDP antiwear additives that contain sulfur and phosphorus can contribute secondary antioxidancy performance.



Figure 10: Chemical structures of secondary antioxidants

Tertiary AOs

Tertiary AOs are typically metal deactivators which prevent catalyst metals from reacting to degrade oil. Metal deactivators are discussed in Chapter 3.3.6.

3.3.3.3. Mode of action:

Primary, Secondary and Tertiary AOs are effective in different parts of lube oil degradation and oxidation mechanisms.



Figure 11: Mechanisms of antioxidant types

Primary AOs act as free-radical scavengers and react with chain-propagating radicals such as peroxy, alkoxy, and hydroxy radicals in a chain terminating reaction.

Secondary AOs act as peroxide scavengers and decompose hydroperoxides ($ROO \cdot$) into nonreactive products before they decompose into alkoxy and hydroxy radicals.

Tertiary AOs work by inhibiting the formation of catalysts such as metal particles.

The combination of different AO classes (primary/secondary/tertiary) and chemistries within those classes (e.g. aminic and phenolic primary AOs) can be formulated to give synergistic performance in different applications.

3.3.3.4. Representative Test Methods

Some tests methods use the addition of catalytically active metals (e.g. Copper, Iron) to represent the in-use conditions and reduce test duration.

Pressure differential scanning calorimetry (PDSC)

PDSC tests are often used as initial screener tests for antioxidancy, such as ASTM D5483 for greases and ASTM D6186 for lubricants. These tests measure the exotherm caused by oxidation of grease/oil when exposed to elevated temperatures.

Rotating Pressure Vessel Oxidation Test (RPVOT)

The RPVOT (ASTM D2272) is used to evaluate Turbine and Hydraulic oils (both new and in-service oils). A large sample (around 50 g) is rotated within the chamber along with water and metal catalyst. The exotherm caused by oxidation of grease/oil when exposed to a temperature of 150°C is measured.

The RapidOxy test measures the pressure drop caused by the oxidation of Greases.

Turbine Oil Stability Test (TOST)

Turbine oil stability tests are longer term tests for turbine oils measuring total acid number and sludge.

- ASTM D943 and ASTM D4310 both use water, copper and iron as catalysts at 95°C.
- ASTM D943 measures time in hours until total acid number reaches 2.0.
- ASTM D4310 measures total acid number and sludge deposits after 1000 hours.
- In ASTM D7873 (the "dry" TOST) there is no addition of water as catalyst but Iron and Copper as still used. The test temperature is 121°C and it measues time (in hours) to achieve 25% of original perfomance and deposits (mg/kg).

Thermal Stability of Hydraulic Oils (Cincinnati Milacron Thermal Stability)

ASTM D2070 measures the thermal stability of hydraulic oil in the presence of copper and steel rods at 135°C. The rods are the evaluation criteria (weight change, visual appearance) and sludge values are reported for information.

Oxidation Stability of Lubricating Oils Used in Automotive Transmissions by Artificial Ageing This test, known as CEC L-48-00, subjects samples of transmission fluids to heating to a specified temperature (with air bubbling) over time. Results indicate changes in viscosity (vs original sample), oxidation level (PAI) and sludge ratinging.

Oxidation Stability of Extreme Pressure Gear Oils

This method ASTM D2893 is used to measure industrial gear oil oxidation and has two variants: method A which uses temperature of 95°C and method B at 121°C.

3.3.3.5. Application

Antioxidants are used across a wide range of automotive and industrial lubricant applications and well as in greases.

Given the variety of AO used in industrial lubricants, treat rates can vary depending on oil lifetime expectations and operating conditions. Typical treat rate of primary/secondary AO are as follows.

For Turbine oils	0.5 - 1.0%
For Hydraulic oils	0.1 – 0.5%

Guideline formulations are shown in chapter 3.4.

3.3.4. Antifoam Additives

3.3.4.1. Purpose

Anti-foam additives help lubricants to remain a nearly incompressible fluid by releasing air contained in the oil. Air entrainment can impact equipment operating conditions and lead to equipment failure.

3.3.4.2. Chemistry used

There are 2 main categories used for anti-foams:

• Silicon based components: polydimethylsiloxanes for example



Figure 12: Chemical structure of Si-based antifoam additives

• Non Silicon based components: e.g. Polyacrylates.

3.3.4.3. Mode of action

Anti foam are tension active components that are not very oil soluble, they separate from the oil onto the surface of air bubbles and cause them to rupture by reducing the surface tension.

3.3.4.4. Representative test methods

Representative test method for anti-foam properties is ASTM D892.



Figure 13: Schematic of the ASTM D892 foam test

This test method covers the determination of the foaming characteristics of lubricating oils at 24 °C and 93.5 °C.

There are 3 different sequences:

- Sequence I: fresh oil sample & temperature of the bath set at 24°C
- Sequence II: fresh oil sample & temperature of the bath set at 93.5°C
- Sequence III: reuse of sample of Sequence II & temperature of the bath set at 24°C

For each sequence the foam volume in mL is measured at the end of the 5 minutes blowing period and after a 10 minutes settling period.

The tendency of oils to foam can be a serious problem in systems such as high-speed gearing, high-volume pumping, and splash lubrication. Inadequate lubrication, cavitation, and overflow loss of lubricant can lead to mechanical failure. This test method is used in the evaluation of oils for such operating conditions.

3.3.4.5. Application

Anti-foam additives are used in all industrial applications. It includes (but is not limited to) hydraulic fluids, tractor hydraulic fluids, power transmission fluids, turbine oils, gear oils, etc. Treat rates are typically in the range of 10 – 250 ppm in the finished oil.

3.3.5. Corrosion Inhibitors

3.3.5.1. Purpose

Corrosion inhibitor (CI) additives are expanding the working life of a lubricated device by protecting the surfaces. They are added to the lubricating oil to prevent corrosion, which results from a chemical or electrochemical reaction between a ferrous metal, and its environment that produces a deterioration of the metal and of the lubricant by oxygen and humidity.

3.3.5.2. Chemistry used

Depending on their use, their chemistry and their application different corrosion inhibitors categories are used. Common chemistries are carboxylates, sulfonates, alkyl amines, phosphates, borates^{iv}. The chemical structures of a carboxylate (left) and a phosphate (right) are shown here.



Figure 14: Chemical structures of corrosion inhibitors

All these chemistries have a high polarity and therefore a high affinity towards metallic surfaces and a tendency being adsorbed on the metal surface.

3.3.5.3. Mode of action

The basic mechanism of corrosion inhibitor additives is an adsorption of a monolayer of the inhibitor on the metal surface to form a protective barrier, which is impermeable for water and oxygen, preventing contact with the outside environment. Typically, there is an equilibrium between the additive adsorbed on the metal surface and the additive solubilized in the oil. Typically, the formed monolayer is oriented with the polar head towards the metal surface and the nonpolar tail into the oil. The monolayer has a tight surface packing and is therefore dense. This process works of film formation via a equilibrium adsorption of corrosion inhibitors which are dissolved in the lubricant. The following picture illustrates this process:



Since steel corrosion is associated with an anodic as well with a cathodic reaction and these reactions are interconnected, the dense adsorption layer of corrosion inhibitors disconnects both reactions and hereby inhibits the corrosion process (see figure on the top right). Another strategy to avoid corrosion is to reduce the water-metal-contact e.g. by the use of antiwetting coatings (see figure right bottom).



3.3.5.4. Representative Test Methods

There are a wide variety of corrosion tests available. Some important tests are DIN 51355, DIN 51585 or ISO 7120. Most of the standards require the use of distilled water or synthetic sea water, whereas the standard typically consisits of two parts. The following graph shows the experimental setup for the ISO 7120, part A and part B.



3.3.5.5. Application

Corrosion Inhibitor additives are nearly used in all industrial oils, engine oils metalworking fluids and corrosion protection oils when iron-based metals or metal alloys are used.

Guideline formulations are shown in chapter 3.4.

3.3.6. Metal Deactivator Additives

3.3.6.1. Purpose

In many applications lubricants can be in contact with machine parts made of yellow metals typically copper, or with dissolved metal contaminants. Since copper and its compounds catalytically decomposes peroxides generating radicals, these metal species are very efficient pro-oxidatives (see figure 18) and need to be deactivated. Dissolved copper ions can be deactivated by the use of chelating agents, whereas the metal parts are deactivated with film-forming metal-passivating agents. They prevent the progressive oxidation of the metal surface, which would release copper ions to the fluid. Complexing agents, on the contrary, could even promote the transfer of copper ions from the metal surface into the fluid. A typical example of a chelating agent would be disalycyliden-propylene-diamine, very often used in combination with film-forming agent derivatives of benzotriazole. Typically, copper surfaces are sensitive towards the attack of sulfur, since copper shows a chemical reaction with sulfur, forming copper sulfide which leads to an etching of the copper surface and the transfer of copper into the lubricant (see figure 19) At the same time, the mechanical stability or machine parts and their ability to seal might suffer.

Metals can accelerate the oxidative degradation of lubricants



Figure 19: Dissolution of copper in presence of sulfur species (red circles: Copper, blue circles: Sulfur)

3.3.6.2. Chemistry used (examples)

In principle, three different chemistries are used in order to protect solid or dissolved copper:

- Chelating agents (e.g. salicylidenes)
- Sulfur scavengers (e.g. thiadiazoles)
- Metal deactivators (e.g. triazoles) examples see figure 20



rigure 20. Chemical structures of metal deactiv

3.3.6.3. Mode of action

Chelating agents deactivate dissolved copper by forming a very strong and stable complex with the copper ions which are not reactive anymore. Sulfur scavenger like thiadiazoles capture the active sulfur and therefore prevent the attack of the copper surface by this active sulfur.

MD additives form a protective layer on copper surfaces preventing that copper is attacked by sulfur and at the same time prevents copper ions from being transferred into the lubricant.

Absorption model according to Fang, Olsen und Lynch:

- Chemisorption of the molecule to the copper surface
- Two copper nitrogen bonds
- Hydrogen bridges to stabilize the formed film





CuO surface

Figure 21: Mechanism of yellow metal deactivation vvi

3.3.6.4. Standards and representative Test Methods

There are a few copper corrosion test available. Some important tests are DIN 51811, DIN 51759 or ISO 2160. The following graph shows the experimental setup for the ISO 2160.

- Copper corrosion test ccording to ASTM D 130 :
 - Variation of the temperature: 90°C, 125°C
 - Prolonged reaction times 3 h, 24 h und 48 h
 - Addition of active sulfur: typically 50 ppm (calculated on neat sulfur) of an aggressive sulfur component are added



Figure 22: Schematic of the ISO 2160 copper corrosion test

3.3.6.5. Application

Metal deactivator additives are important for applications using yellow metals in machine parts as for example for metalworking fluids, gear oils, greases and turbine and compressor oils.

Guideline Formulations are shown in chapter 3.4.

3.3.7. Dispersant / Detergent Additives

3.3.7.1. Purpose

Dispersants and detergents additives are expanding the working life of a lubricant and machine by keeping fluids and surfaces clean. They are added to the lubricating oil to prevent, disperse and emulsify deposits, soot, sludge, varnish, lacquer, resins, oxidation products, water, acids, metals and inorganic salt. Detergents have additional the ability to neutralize acids and provide rust and corrosion protection.

3.3.7.2. Chemistry used (examples)

Detergents

Three main categories of detergents are in use: Phenates, sulfonates and salicylates, there are also carboxylate and glyoxylate structures. Detergents contain a hydrocarbon tail, which provides the solubility in oil, the polar or acidic group is neutralized with metal oxides or hydroxides, usually from Ca and Mg, but also Na and Ba.

Detergents (metal containing)

Phenates



x = 1-2 n = 0-2 Me: most common Ca and Mg

Sulfonates



Salicylates



Figure 23: Chemical structures of detergent additives

Detergents can be neutral or slightly basic in case of a more stoichiometric reaction of the phenate/sulfonate/salicylate with metal hydroxide. To increase the alkalinity, detergents are often reacted with a surplus of metal hydroxide and additional blown CO_2 into the reaction, so called 'overbased' detergents are formed, which means they contain excess of CaCO3 in a micelle structure. The reserve alkalinity can further neutralize acid components formed by oxidation and protects against rust formation. The overbasing level is indicated by the "Total Base Number" (TBN), measured using potentiometric titration (e.g. ASTM D2896) which expresses the basicity of the detergent in terms of the equivalent number of milligrams of potassium hydroxide per gram of detergent. Detergents can have TBN numbers of up to 500 mg KOH/g.



Dispersants (metal free)

Dispersants are usually based on a polar head, derived from oxygen or nitrogen and a hydrocarbon tail. Backbone of the majority of dispersant technology is PIB, poly isobutylene. Isobutylene is polymerized to PIB and that reacts further with maleic acid anhydride to PIBSA, poly isobutylene succinic anhydride. The PIBSA further reacts either with polyethylene amines to form succinimides (PIBSI = poly isobutylene succinic imide). PIBSA can react further with polyols, TMP (trimethylolpropane) or PE (pentaerythritol) to succinic esters (PIBSE = poly isobutylene succinic ester). The maleic acid anhydride works as a connector between non-polar and polar functional groups. Other common types are the Mannich-dispersants. In that case an alkylphenol reacts with formaldehyde and further with polyethylene amines to alkylphenol-amines. All dispersants are metal free by definition.



Figure 25: Chemical structures of metal free dispersants

3.3.7.3. Mode of action

Main function of dispersants / detergents is to work as a cleaning agent. Dispersants and detergents consist of an oil soluble part in form of a long hydrocarbon tail, and a polar head to suspend soot and solubilize polar contaminants. Main functions are to disperse contaminant particles in the fluid and also to remove and solve deposits from surfaces and to keep them all in suspension.





Clean and remove deposits from metal surfaces

Figure 26: Mechanism of dispersants / detergents

Detergents protect the equipment especially at high temperatures against oil sludge and varnish by removal and dispersion of particles.

Dispersants act similar to detergents and usually provide protection at medium to low temperatures to keep insoluble particles and contaminants in suspension and avoid deposit formation.

Furthermore, detergents, especially sulfonates, work as corrosion inhibitors as they protect the surface in form of protective layers on the surface.



Figure 27: Corrosion protection achieved by use of sulfonate based detergents

In addition 'over-based' (surplus capacity to neutralize acids) detergents can neutralize acids and acidic degradation products in the oil.



3.3.7.4. Representative Test Methods

As dispersants and detergents are by far mostly applied in automotive fluids, there are several specific tests in determing the function and performance of detergents and dispersants in engine oils as: i.e. the VW TDI PV 1452 / CEC L-78-99 – TDI2, designd to determine piston cleanliness, the Mercedes Benz MB M271 Classic Sludge test, designd to determine sludge formation and a common bench test, ASTM D 7899-13, a standard test method for measuring the merit of dispersancy of in-service engine oils with the "Blotter Spot Method".

More common for industrial fluids dispersancy testing are

- DBL 6751-4 Determination of the dirt carrying capacity
 - Used to determine the dispersancy capability of hydraulic fluids
 - A chromatographic method, which measures the ability to disperse the contaminated fluid:



Figure 29: Schematic of the dirt carrying capacity test according to DBL 6751-4 (Daimler inhouse standard)

- Schaeffler FE-8 with focus on residues in step 4 of the 4-step method. Typically applied for paper machine and wind turbine gear oils.
 - Applied for wind turbine and paper machine oils
 - Performed on a FE-8 Axial Roller Bearing test rig (DIN 51819)
 - Special equipement: the oil can be heated in an oil preheating tank in range variable adjustable from 100 to 140°C
 - Optional water can be added in sequence dropwise to the fluid circuit
 - $^-$ Oil will be filtered via a 10 μm filter
 - Test is running for 600 hrs, bearing speed is 750 min-1,
 - Test criteria are beside the bearing wear, the residue formation at the bearings and at the preheating system
- Simple lab tests are:
 - ASTM D 2070 (usually applied for hydraulic fluids)



200 ml for 168 h at 135 °C

- Evaluation:
 - Report color of the steel rod
 - Report color of the copper rod
 - Mass of sludge on filter in mg/100 ml
- SKF Roller Test (often applied for wind turbine gear fluids)



8 weeks at 100°C / 120°C in oven

• (120°C for gear boxes without cooling)

Evaluation:

- Corrosion: Roller attack max 2
- Viscosity change: max 2
- No sludge formation, no incrustion

Figure 30: Oxidation and dispersant testing according to ASTM D2070 and SKF Roller test

3.3.7.5. Application

Dispersants and detergents are mostly used in automotive engine oils and marine lubricants. Smaller volumes and treat rates are also used in industrial oils, as gear, hydraulic and turbine oils. Hydraulic fluids HLPD and HVLPD contain a certain amount of dispersants/detergents to achieve a level of dispersancy of dirt, other contamination and water. In gear oils, dispersants and detergents are used to keep the gears and bearings cleaner and contribute to a longer fluid and machine life time.

In industrial equipment, the stress on the fluid is increasing, at the same time oil reservoirs are getting smaller, that often results in higher temperatures and increasing oxidation.

In industrial fluids the treat rate of dispersants/detergents in the finished fluid can be between 0.1 and 1%. The use of detergents/dispersants in industrial fluids has lot of advantages, as mentioned above, but also disadvantages as dispersants/detergents can attack seals, can also be hydrolytic instable and can thus reduce the ability of the fluid to demulsify.

Guideline formulations are shown in chapter 3.4

3.3.8. Friction Modifiers

3.3.8.1. Purpose

Controlling friction between surfaces moving relative to each other is fundamental to maximizing operating efficiency, maximizing energy / fuel efficiency and maximizing equipment life-time in mixed and boundary lubrication conditions. By definition the base fluid itself is the primary friction modifier, but to further improve the efficiency of lubricated processes one approach has been to progressively reduce the viscosity of the lubricant to minimize hydrodynamic shear, churning and pumping losses. However, this means that an increased number of components operate under boundary lubrication conditions. Especially in this regime the oil film is not adequate to keep moving parts separated causing friction, wear and noise. This function is taken over by a film of polar molecules strongly absorbed on the metal surface. The drag caused by this boundary lubrication depends on how easily these surfaces slide past one another. One way to reduce the energy losses and maintain a boundary film is by using friction modifiers.

3.3.8.2. Chemistry used (examples)

Friction modifier molecules are generally straight hydrocarbon chains with a polar head group. Typical polar head groups are:

- · Amines, amides of fatty acids and their derivatives
- Carboxylic acids or derivatives, like esters.
- · Phosphoric or phosphonic acids and their derivatives



Polymeric Organic



Molybdenum dithiocarbamate



Glyceryl monooleate



Fatty acid sarcosine



Phosphoric and phosphonic acids and derivatives

Figure 31: Chemical structures of friction modifiers. On the top a generalized structure with polar backbone (black) and non-polar tails (red) is depicted

3.3.8.3. Mode of action

There is generally less friction between two fluid molecules moving over each other than between two surfaces moving across each other. In general lubricants reduce friction.



Friction modifiers (FM) are added to a lubricant to further reduce friction and their mode of action is closely related to antiwear additives. The polar head groups are attracted to the metal surface and form relatively strong bonds whilst the long hydrocarbon tail is left solubilized in the oil. The nature of the polar head group and the structure of the hydrocarbon chain both have a strong impact on the contribution to friction reduction.



Molybdenum compounds on the other hand react on the metal surface to yield molybdenum disulfide which has a structure that allows sliding and shearing to take place. These are predominantly used in automotive applications rather than industrial ones. Therefore, this chemistry will only be briefly discussed.





3.3.8.4. Representative test methods

The effectiveness of a friction modifier is usually stated as the coefficient of friction (CoF) of the lubricant. The CoF is the ratio of the frictional force resisting the motion of two surfaces in contact to the normal force pressing the two surfaces together. It is usually symbolized by the Greek letter mu (μ). Mathematically, $\mu = F/N$, where F is the frictional force and N is the normal force. Because both F and N are measured in units of force (such as newtons or pounds), the coefficient of friction is dimensionless. It can be measured by use of a variety of test devices. Depending on the application and purpose of the investigation either wear tests like HFRR, SRV or 4-ball are used or the MTM device for more scientific and developmental purposes.



Figure 36: Test methods to investigate friction

MTM

Mini traction machine

Industrial Lubricants: Applications and Impact of Additives on Performance

The Mini-Traction Machine (MTM) is a multi-purpose instrument for measuring friction and traction properties of lubricated and unlubricated surface contacts, under a wide range of rolling and sliding conditions. The MTM is very flexible as it allows the investigation of friction and traction using a variety of materials, geometrical contacts, oil temperatures and contact pressures.

One of the main uses of the MTM is to measure friction between lubricated steel surfaces, under conditions typically found in industrial applications. The MTM can also produce a Stribeck curve, which is important in understanding which lubrication regime is taking place. The Stribeck curve (Figure 37) essentially considers three types of lubrication:

- Boundary lubrication
- Mixed lubrication
- (Elasto)-hydrodynamic lubrication



Figure 37: Stribeck curves and the effect of friction modifiers

Additives such as extreme pressure additives, anti-wear additives and friction modifiers can be important components in controlling wear and friction in the boundary regime.

The mixed regime is characterised by both asperity contacts and thin film lubrication and therefore both friction modifiers and base fluids are important in controlling friction.

In the hydrodynamic regime there are no asperity – asperity contacts, in this regime opposing surfaces are separated by a full film of lubricant. In the hydrodynamic regime the chemistry of the base fluid and the viscosity of the lubricant are critical considerations in controlling friction.

Representative test methods for the performance of friction modifiers are:

DIN 51834, ISO 12156, ISO 8295 ASTM D5706, ASTM D5707, ASTM D6425, ASTM D7421, ASTM D7594, ASTM D6079, ASTM D5001, ASTM D6078, ASTM D1894, ASTM D202, ASTM D4918.

3.3.8.5. Application

Industrial applications where friction modifiers are used include (but are not limited to) hydraulic fluids, turbine oils, gear oils, paper machine lubricants and chain oils. Treat rates are typically in the range of 0.1 – 1.5%.

Guideline formulations are shown in chapter 3.4.

3.3.9. Pour Point Depressants

3.3.9.1. Purpose

Pour point depressants (PPD) act to lower the pour point of an oil which is the lowest temperature at which an oil will flow when cooled. The pour point is a common parameter to quantify the cold flow performance of industrial lubricants. Oil flow can be restricted by the solidification of components with high tendency to crystallize.

3.3.9.2. Chemistry used (examples)

A large variety of chemistries is used to control crystallization:

- Poly-ethylene-co-vinyl Acetate
- Poly-vinyl Acetate-co-fumarate Ester
- Alkylated Styrene
- Poly-alkylacrylate
- Poly-styrene-co-acrylate
- Poly-olefin-co-maleic Anhydride Esterified
- Poly-styrene-co-maleic Anhydride Esterified
- Poly-alkylmethacrylate

The most common chemical types of PPD in lubricants are polyalkylacrylates (PAA) and polyalkylmethacrylates (PAMA), also often known as polyacrylates and polymethacrylates.

3.3.9.3. Mode of action

Hydrocarbon-based oil formulations typically contain fractions of normal-paraffins with high cloud and pour points. At low temperatures, these waxy components come out of solution to form crystals, producing a gel-like structure which finally impedes the flow of the lubricant.

PPD do not reduce the amount of wax, however they control the nucleation and crystallization process in a way to inhibit the formation of large interlocking crystal networks.

By addition of a pour point depressant (PPD) the sharp increase in viscosity at low temperatures can be supressed and the temperature operating window (TOW) of an oil can be significantly extended to the low temperature side.



The result of formation of large interlocking wax crystals will be a sharp increase of viscosity at low temperatures.



Figure 39: Oil viscosity as a function of temperature with and without wax crystallization. The use of a proper PPD will shift the curve closer to the blue line and avoid rapid viscosity increase caused by crystallization

3.3.9.4. Representative test methods

The Pour Point is measured according to ISO 3016 (ASTM D97 or ASTM D5950).

The ASTM D5950 method is described in the picture. The oil is cooled down in 3°C steps and flowability is automatically checked by tilting. The lowest temperature with flowability is defined as the Pour Point.



Figure 40: Description of the Pour Point test according to the automatic tilt method ASTM D5950 with improved repeatability and reproduceability relative to ASTM D97 $^{\times}$

Other important cold flow tests are viscosity measurements down to low temperatures, e.g. Brookfield Viscosity according to ISO 9262 (ASTM D2983) at e.g. -20, -26, or even down to -40 °C.

Other important cold flow tests are viscosity measurements down to low temperatures, e.g. Brookfield Viscosity according to ISO 9262 (ASTM D2983) at e.g. -20, -26, or even down to -40 °C.

3.3.9.5. Application

A low pour point is particularly important for proper performance of all kinds of lubricants in cold climates and for operation over a wide temperature operating window.

PPD's are used at relatively low treat rates of typically well below 1 %. There is an individual optimum treat rate for PPD and overtreatment can lead to a so-called PP reversion.

The use of a PPD is an economic way to upgrade the properties of mineral based fluid formulations and allow to reduce cost and energy to produce lubricants of defined performance.

Guideline formulations are shown in chapter 3.4

3.3.10. Viscosity Modifiers / Viscosity Index Improvers

3.3.10.1. Purpose

Viscosity modifiers (VM) are polymeric thickeners that are used to adjust the viscosity of an oil formulation to the desired viscosity grade as defined by the kinematic viscosity at 40 °C.

In order to keep the viscosity more constant over temperature, which is described by the viscosity index, specially designed polymers called viscosity index improvers (VII) can be added to the formulation. Viscosity index improvers can be understood as viscosity modifiers that lift the viscosity at higher temperatures more pronounced than at low temperatures.

3.3.10.2. Chemistry

Medium to high molecular weight polymers (from 10,000 to 500,000 g/mol) of various chemical types are used as viscosity modifiers for lubricants. The main chemical families are Olefin-Copolymers (OCP), Polyisobutenes (PIB), hydrogenated styrene-diene copolymers (HSD) and Polyalkylmethacrylates (PAMA). These co-polymers are composed of more or less oil soluble units, e.g. methacrylic esters with a variety of different alcohol chain lengths. The preferred molecular structure greatly depends on the application requirements and can be adjusted to oil solvency power and the desired low-temperature performance.

Polyalkylmethacrylates (PAMA) are the commonly used VI improvers in industrial lubricants. PAMA chemistry offers the highest VI lift and excellent low temperature behaviour in a variety of base oils and viscosity grades, e.g. in hydraulic fluids of viscosity grades 32 to 68.





PIB, Polyisobutene

OCP, e.g Ethylene-Propylene copolymers





HSD, e.g. Hydrogenated Styrene-Isoprene copolymers



PAMA, Polyalkylmethacrylates

HSD, e.g. Hydrogenated Styrene-Butadiene copolymers

Figure 41: Chemical structures of Viscosity Modifiers

3.3.10.3. Mode of action

A viscosity modifier has relatively high molecular weight and a significantly higher viscosity than the used base oils and acts as a thickener.

The more pronounced thickening at higher temperatures depends on the combination and resulting interaction of VI improver and base oils.

Two things happen by addition of a viscosity index improver to a base oil:

- 1. Viscosity goes up,
- 2. Viscosity goes up more at higher temperatures.

Consequently the viscosity over temperature correlation becomes "flatter", and a flatter line is equivalent to a "better" lubricant as it covers a wider temperature range within the upper and lower viscosity limits.



Figure 42: Double logarithmic plot of kinematic viscosity as function of temperature for a base oil (black) and a base oil with viscosity index improver (green). Thickening at high temperatures is more pronounced than at lower temperatures and the line becomes flatter in the double logarithmic graph

To understand the VII mechanism, it is important to know that polymers dissolved in a solvent, such as in an industrial lubricant base oil, adopt nanoscale solvated coil structures, having an ellipsoid or spherical shape, known as hydrodynamic spheres. These spheres increase the fluid resistance to moving (= viscosity) as they increase the friction forces between the internal fluid layers. The greater the expansion in the size of the coil, the stronger the sum of its intermolecular forces, and the higher the viscosity of the fluid. The size of the hydrodynamic spheres depends on many parameters, such as their affinity for the oil matrix, the polymer chain length, structure and composition, the shear rate prevailing in the fluid and the fluid temperature and pressure.

3.3.10.4. Representative test methods

The viscosity index (VI) of a fluid is used to describe the change of viscosity with temperature. It is calculated from kinematic viscosities at 40 and 100 °C according to ISO 2909 (ASTM D2270).

In addition to the fresh oil VI it is of importance to investigate the viscosity of oils in use.

Therefor three methods for measuring shear losses of lubricants are common, depending on the shear stress in a given application.

- 1. The Bosch Injector Test (DIN 51382, ASTM D6278) run for 250 cycles at 2550 psi. This is considered the least severe shear test applied to hydraulic fluids.
- 2. The Sonic Shear Test (ASTM D5621) shears the sample fluid for 40 minutes in a sonic oscillator. This test corresponds to real life stress in many applications.
- 3. The KRL Tapered Roller Bearing Test (ISO 26422) requires the test oil to run in a fitted tapered roller bearing for 20 hours or even 100 hours under the design load. This is a harsh test and correlates to stress under extreme hydraulic conditions at high pressures, temperatures and shear rates.

3.3.10.5. Application

Typical treat rates viscosity modifiers or viscosity index improvers for industrial fluids strongly depend on the targeted viscosity index and the performance level in terms of shear stability and low temperature viscosity. The higher the VI, the wider the temperature operating window (TOW). This is illustrated in figure 43, in which the TOW and the optimum temperature range for the high VI fluid is significantly larger than that of the monograde fluid with a VI of approximately 100.



Figure 43: Kinematic viscosity as a function of temperature for two ISO VG46 fluids with different viscosity index

Temperature is the one dominating parameter affecting the viscosity of the fluid in operation. In many applications, VII's have to work and perform over a wide range of temperatures, sometimes experiencing conditions ranging from - 40° C to + 100° C. High performance VII's must be carefully designed to address these extreme conditions.

Besides temperature changes, pressure and shear also affect viscosity. For example, advances in hydraulic system technology have led to an increase in operating pressure and power density. This results in a high fluid pressure gradient across the solvated polymer hydrodynamic volume that can result in "shear" distortion or breakage of the polymer coils.

This "shear" manifests itself as a reduction in the fluid viscosity and can be either temporary or permanent.

Measuring the tendency of a fluid to lose viscosity on shearing is an important indicator of the fluids fit for purpose. This is considered in the leading OEM approvals for e.g. hydraulic fluids.

The same holds for other applications like industrial gear or compressor oils.

Typical treat rates in high VI fluids are ranging from 2 to 10 % depending on the application and specific performance targets.

Guideline formulations are shown in chapter 3.4

3.4. Guideline formulations

Base oils and additives are combined to provide a lubricant with appropriate viscosity and performance for a given application.

The requirements in industrial applications can be very different, depending on the purpose of the oil in the application, e.g. wear protection, heat removal, corrosion protection or the transmission of power like in hydraulics. Some fluids are frequently exchanged or used over long periods of time up to fill for life.

The operating conditions can also be quite different, and we need to distinguish between in-door, typically stationary equipment and out-door, typically mobile equipment. A significant proportion of stationary equipment is operated 24/7 whereas mobile equipment like excavators is started up in the morning and shutdown in the evening. Oil temperatures are on either pseudo-stationary levels or cover a wide range.

Three different oils were selected to give an indication for "typical" treat rates of additives in combination with base oils.

A large fraction of industrial oils belongs to hydraulic fluids.

They are categorized in ISO 6743-4 according to their base oil type into mineral based, environmentally friendly, and fireresistant fluids covering synthetic and water-based formulations. Only the five types of mineral oil based hydraulic fluids are covered in the ISO 11158 international standard with a detailed description of minimum requirements in categories HH, HL, HM, HV and HG. Hydraulic viscosity grades rank from ISO VG 10 to ISO VG 150 while the majority of hydraulic fluids belongs to VG32, VG46 and VG68.

A fully additised hydraulic fluid is well characterized in terms of defined performance tests as described by the minimum requirements for HVLP fluids according to DIN 51524-3 and other specifications including OEM requirements.

Table 4 shows the formulation of an HVLP 32 with VI of 160 as used in demanding mobile and stationary hydraulic applications like excavators and injection moulding equipment.

Another large share of industrial fluids belongs to industrial gear oils (IGO). Top tier IGO are based on synthetic base fluids. The additive package is treated at higher levels than in hydraulic fluids, because loads are higher and applications like wind turbine gears call for long oil drain intervals. Oil performance is defined by DIN 51517 and other specifications including OEM requirements.

The third example for a "representative" industrial oil formulation is selected from air compressor oils. These oils have multiple tasks and are in contact with fresh air that they need to release quickly at the discharge side. Air compressor oils are defined by DIN 51506 depending on the air discharge temperature.

Some additive groups are essential to provide an oil with good performance, others like viscosity index improvers can be added on demand to extend the temperature operating window or to improve energy efficiency.

Table 4: Exemplary formulations for high tier hydraulic fluid, industrial gear oil and air compressor oil. Additive selection is based on input from references ^{vii} and ^{ix}

Application	Hydraulic fluid	Industrial gear oil	Air Compressor Oil
Complies with DIN	51524-3	51517-3	51506 VDL and SAE MS1003
Formulation details			
ISO VG	32	220	46
KV40	32	220	46
KV100	6.5	26.9	7.7
VI	ca.160	ca.160	ca.135
Base oil	Grp II	Grp III, IV, V	Grp III, IV, V
Total additive treat rate excl. PPD and VII which are counted separate	0,5 – 1 %	1,5 – 5 %	0,5 – 6 %
Antioxidants (typically up to 1%)	Х	Х	x
Corrosion inhibitors (typically up to 0,2%)	Х	Х	Х
Antifoam (typically up to 0,025%)	Х	Х	x
PPD (typically up to 0,7%)	Х	optional	optional
VI Improver (typically up to 10%)	Х	-	optional
AW additives (typically up to 1.5 %)	х	optional	Х
EP additives (typically up to 1%)	optional	Х	-
Metaldeactivators and -passivator (typically up to 0,1%)	optional	Х	x
Friction modifiers (typically up to 1,5%)	optional	х	-
Detergents, Dispersants (typically up to 1%)	optional	optional	optional
Demulsifier (typically up to 0,1%)	optional	Х	x

4. Summary and conclusions

All major additive classes for industrial lubricants were discussed in terms of chemistry, mode of action and application.

The performance and lifetime of industrial lubricants rely strongly on the action of components which are typically present at low fractions: the additives.

These additives have the following main functions:

- 1. Enhance properties of the base oil itself with additive types such as antioxidants, pour-point depressants and viscosity index (VI) improvers.
- 2. Suppress undesirable base oil properties with anti-foam agents and demulsifying agents.
- 3. Impart new properties either to the base oil itself or to the base oil metal interface with extreme pressure (EP), anti-wear (AW) additives, corrosion inhibitors, metal deactivators, friction modifiers and tackiness agents.

Especially the additives acting on phase boundaries often are polar in nature, which promotes directional attraction to other polar phases (metal surfaces, dirt surface, sludge and water).

Other additives types which act in the body of the oil like the antioxidants are sacrificial, their mode of action consumes them.

A multitude of lubricant additives are available, and they are selected for use based upon their ability to perform their intended function in the specific industrial oil (hydraulic, gear or turbine oil etc). They are also chosen for their ability to mix easily with the selected base oils, to be compatible with other additives in the formulation, to be cost effective and increasingly often due to their sustainability.

The use of appropriate additives makes industrial lubricants more powerful and efficient. This is of benefit for the end user and serves the increasing demand for sustainable solutions by our technically oriented society.

Glossary of terms

AO Antioxidant Anti Wear AW Coordinating European Council (Development of Performance Tests for Fuels, Lubricants and Other Fluids) CEC CI Corrosion Inhibitor CO Compressor Oil DIN Deutsches Institut für Normung EP **Extreme Pressure** GR Grease HF Hydraulic Fluid Industrial Gear Oil IGO MD Metal Deactivator MWF Metal Working Fluid OCP **Olefin Copolymer** PIB Poly isobutene PPD Pour Point Depressant SBR Styrene Butadiene Rubber **Turbine Oil** TO TOW **Temperature Operating Window** VI Viscosity Index VII Viscosity Index Improver ZDDP Zink-Dialkyldithiophosphate

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