



Sudan University of Science and Technology College of Graduate Studies

A Comparative Study of Base Oil Solvent Neutral SN 150 from Different Sources

دراسة مقارنة لزيت الاساس المذيب المتعادل SN 150 من مصادر مختلفة

A Thesis Submitted in Partial Fulfillment of the Requirements For the Degree of Master of Science

By

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الآية

بسم الله الرحمن الرحيم

قال تعالى: (إِذْ تَمْشِي أُخْتُكَ فَتَقُولُ هَلْ أَدُلُّكُمْ عَلَىٰ مَن يَكْفُلُهُ ﴿ فَرَجَعْنَاكَ إِلَىٰ أُمِّكَ كَيْ تَقَرَّ عَيْنُهَا وَلَا تَحْزَنَ ۚ وَقَتَلْتَ نَفْسًا فَنَجَيْنَاكَ مِنَ الْغَمِّ وَفَتَنَّاكَ فُتُونًا ۚ فَلَبِثْتَ كَيْ تَقَرَّ عَيْنُهَا وَلَا تَحْزَنَ ۚ وَقَتَلْتَ نَفْسًا فَنَجَيْنَاكَ مِنَ الْغَمِّ وَفَتَنَّاكَ فُتُونًا ۚ فَلَبِثْتَ كَيْ تَقَرَّ يَا مُوسَىٰ) سِنِينَ فِي أَهْلِ مَدْيَنَ ثُمَّ جِئْتَ عَلَىٰ قَدَرٍ يَا مُوسَىٰ)

(سورة طه الآية 40)

Dedication

To my parents, brothers and sisters

To my wife and beloved daughter

A cknowledgement

First of all I thank the almighty ALLAH for giving me the strength and support to complete this study.

I would like to express my sincere gratitude to my supervisor Dr.

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I would like to thank and appreciate others not mentioned for helping and supporting me by any way to reach this stage of the study, and I would like to tell them their hands will remain in my memory for good.

Abstract:

This study is designed to compare two samples of base oil solvent neutral (SN 150) from different sources to show which source of base oil is better than the other according to the specifications of the base oil. Some of the results show conformance like: (appearance (clear and bright), odor (pass), density (8819and 8739kg/m3), kinematic viscosity at 40and 100C (31.88, 30.96 and 5.22, 5.12 mm/s), flash point (232and 214C) and viscosity index (92and91)) respectively. On the other hand the remaining tests show very clear differences in (Pour point (-3 and -12 C), color (2.5 and 0.5) and metals determination especially for sulfur (11460 and 2970 ppm)) respectively. The study concluded that sample two is superior than sample one according to the results.

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LIST OF ABBREVIATIONS:

API American Petroleum Institute (USA)

ASTM American Society of Testing Materials (USA)

DMSO Di methyl Sulfoxide

DIN Deutsches Institut for Normung German Institute for Normalization

EPA Environmental Protection Agency (USA)

FCC Fuel Catalytic Cracker

FDA Food and Drug Administration (USA)

GC.MS Gas Chromatography Mass Spectroscopy

GTL Gas To Liquid HC Hydro cracked

HD Heavy Duty

HPLC High Pressure Liquid Chromatography

HVI High Viscosity Index

IARC International Agency for Research on Cancer

IJETT International Journal of Engineer Trends and Technology

ILSAC International Lubricant Standardization and Approval Committee

IP Institute of Petroleum (UK)

ISO International Standard Organization

ISO VG ISO Viscosity Grade
LVI Low Viscosity Index
MLDW Mobil Lube Dewaxing
MS Mass Spectroscopy

MSDW Mobil Selective Dewaxing

N-D-M (Refractive Index) n-Density-Molecular Weight

NMR Nuclear Magnetic Resonance

OEM Original Equipment Manufacturer

PCB Printed Circuit Board

PCMO Passenger Car Motor Oil

PPD Pour Point Depressant

SAE Society of Automotive Engineers (USA)

SAPO Silicon Aluminum Phosphate molecular sieve

SN Solvent Neutral

SUS Saybolt Universal Seconds

TAN Total Acid Number

TBN Total Base Number

UHVI Ultra High Viscosity Index

VGO Vacuum Gas Oil

VHVI Very High Viscosity Index

VI Viscosity Index

VT Viscosity-Temperature

XHVI Extra High Viscosity Index

XRF X-ray Fluorescence spectrometry

Chapter One

Introduction

Base oils are essential component in lubricants. The source of the base oil is petrol after being treated by thermal distillation to produce light products the residue is vacuum distilled to produce base oil which represent 2% from raw petrol e. g one barrel produces one gallon of base oil.

Chemically the base oil has three major types:

- 1- Naphthenic base oils.
- 2- Paraffinic base oils.
- 3- Synthetic base oils.

Petroleum base oil has many classifications one of them are the groups which depend on sulfur content and viscosity index. There are five groups ranked from (I) to (v).

Production of base oils is a very complicated and expensive process, which starts after the thermal distillation of the crude oil. The essential stage of the process is vacuum distillation. This process is considered as the first step at which base oil produced. There are many other steps which vary according to the crude oil chemical characteristics.

This study is concerned with one grade of base oil from two different suppliers. These two samples are subjected to investigation that concerns their specifications and their effects on production of the lubricants. Extensive macro analysis and micro analysis had been carried out for the samples to identify the difference between them.

There are many types and names for the base oil according to the specified name or the utility of the base oil. One of the most important types is the SN (solvent neutral) which includes many grades according to the kinematic viscosities at 40°C. Examples for the grades are SN 90, SN 100, SN 150, and SN 500etc.

The base oil under investigation is (SN 150). The number 150 is an identification number which has an International known specifications according to Saybolt Universal Seconds (SUS). As general every number has it is own specifications and uses, but the major factor is the kinematic viscosity at 40 $\,$ C. For example the kinematic viscosity of SN 150 at 40 $\,$ C is about 30 centi Stock and for SN 500 is about 100 c St so 1 SUS = 5 c St, and 1 c St = 1 mm2/s.

Generally base oil are subjected to many tests such as Thermal tests (flash point, pour point and cloud point) further more visual tests which are appearance and color number. The most important physicochemical tests are the viscosity and specifically the kinematic viscosity, sulfur content and density.

The general requirements of lubricants are:

- High boiling point.
- Low freezing point.
- High viscosity index.
- Thermal stability.
- Hydraulic stability.
- Demulsibility.
- Corrosion, wear and friction prevention.
- High resistance to oxidation.

In terms of volume, base oils are the most important components of lubricants. As a weighted average of all lubricants, they account for more than 95% of lubricant formulations. There are lubricant families (e.g. some hydraulic and compressor oils) in which chemical additives only account for 1% while the remaining 99% are base oils. On the other hand, other lubricants (e.g. some metalworking fluids, greases, or gear lubricants) can contain up to 30% additives.

The origin of the overwhelming quantity of mineral lubricant base oils has led to lubricant being viewed as a part of the petroleum industry and this is underlined by their inclusion in petroleum statistics. Over the last few years, lubricants have increasingly become a separate discipline with clear differences from petroleum mass products. This was caused by the high added value which is generated in this product segment along with the fact that many high-performance lubricants no longer contain petroleum base oils. (Mang and Ming, 2007)

1.1 Base Oils-A Historical Review and Outlook:

Although the most important requirement of base oils in the fifties was the correct viscosity and the absence of acidic components, base oils in the sixties were down graded to solvents or carriers for additives in the euphoria surrounding chemical additives. In the seventies, there was a realization that some synthetic fluids with uniform basic chemical structures offered performance superior to that of mineral base oils. At that time, the considerably high price of these products hindered their market acceptance. In the eighties however, lower-price, quasi-synthetic hydro cracked oils were introduced in Western Europe which closely match the properties of synthetic hydrocarbons (Shell, BP, Fuchs). In the nineties, base oil developments were influenced by the ever-increasing demands on lubricant performance, and by environmental, health and safety criteria. This led to, chemically, more pure oils such as hydro cracked products, poly alpha olefins and esters.

Natural fatty oils, particularly their oleo chemical derivatives, have experienced a renaissance because of their technical characteristics but, above all, because of their rapid bio degradability. The trend towards ever-greater performance and even better environmental compatibility continues in the first decade of the new millennium. The significantly higher price of the new lubricants which will be increasingly characterized by their base oils and less so by their chemical additives, will probably be accepted by users who will benefit from long product life and overall lower costs.

In 2004, approximately 7% (w/w) of base oils were synthetic products (including Group III hydro cracked oils. This segment is forecast to grow to 10% in 2015. In Germany, furthermore, approximately 5% of lubricant base oils were rapidly biodegradable (natural and synthetic) esters in 2005. In addition, gasification of carbon-containing raw materials, for example natural gas, biomass,

and carbon, will enable the production of synthetic lubricant base oils of high quality. Significant industrial-scale production of such base oils will start at the beginning of the second decade of the new millennium. (Downey, 2005)

1.2 Chemical Characterization of Mineral Base Oils:

Characterization of mineral oil fractions, whether crude oil or lubricant base oil fractions, to determine their exact structure is not possible without great expense. Crude oil generally consists of many thousands of single components and these are reflected in the processing of each fraction. It was, therefore, always an objective to describe mineral oil fractions by the comparatively simple expedient of defining their technical properties and quantitatively determine groups of components with similar chemical character. Advanced physicochemical methods are, however, used in routine testing.

1.2.1 Rough Chemical Characterization:

1.2.1.1 Viscosity–Gravity Constant (VGC):

This value enables only rough chemical characterization of oils. Values near 0.800 indicate paraffinic character whereas values near to 1.000 point to a majority of aromatic structure (ASTM D 2501-91).

1.2.1.2Aniline Point:

The aniline point is also a help when characterizing the hydrocarbon structure of mineral oils. When aniline and oil are mixed and then cooled to a certain temperature (anilinepoint), two phases form. Because of their good solubility, aromatic structures give the lowest values.

1.2.1.3Carbon Distribution

The most important means of analysis for characterization of mineral oil hydrocarbons was, and remains, the determination of carbon in terms of its three categories of chemical bond–aromatic (CA), naphthenic (CN), and paraffinic (CP).

Refractive index, density, and molecular weight N–D–M analysis uses physicochemical data which are easy to obtain. Molecular weight can be determined by measuring the viscosity at different temperatures (ASTM D 2502-92). Carbon distribution is given in% CA, % CN and% CP (100% in total). N–D–M analysis also determines the average total number of rings per molecule (RT) and the break down into aromatic and naphthenic rings (RN) per molecule (RN = RT – RA).

Brandes created a method of determining carbon distribution according to specific bands in the infrared spectrum. The method has proven itself for lube base oils and can be performed at acceptable expense. Exact determination of aromatic carbon content can be performed by (ASTM D 5292-91), High-resolution nuclear magnetic resonance (NMR).

1.2.1.4 Hydrocarbon Composition:

A further refinement in lubricant characterization of base oils is the determination of molecular families. Chromatography is used first to separate components and the fractions are then subjected to advanced analytical procedures.

To differentiate mineral oils including lube base oils in the boiling range from 200 to 550 °C, high ionizing voltage mass spectrometry (ASTM D 2786-91) is used for saturated fractions and (ASTM D 3239-91) for aromatic fractions. Saturated fractions are separated into alkanes (0-ring), 1-ring, 2-ring, 3-ring, 4-ring and 5-ring naphthenes. The aromatic fractions are subdivided into seven classes: monoaromatics, diaromatics, triaromatics, tetraaromatics, pentaaromatics, thiopheno aromatics, and unidentified aromatics.

1.2.1.5 Polycyclic Aromatics in Base Oils:

Polycyclic aromatics hydrocarbons (PAH) or in general polycyclic aromatics (PCA) are carcinogenic, environmentally harmful substances which are found in crude oils. In general they are not created when a lubricant is used.

PAH formed by the combustion of gasoline can gather in engine oils. They can also gather in quenching oils after long periods of heavy-duty use.

In traditional solvent refining processes, PAH largely remain in the extract. Non solvent extracted distillates contain PAH in line with their boiling point. The carcinogenic characteristics of non- severely treated distillates in the petroleum industry were established by the (International Agency for Research on Cancer) IARC in 1983. This led to considerable limitations in the manufacture and application of naphthenic base oils and the ending of the use of aromatic extracts as lube base oils. Previously, Grimmer and Jacobs, in particular, confirmed the presence of PAH in lubricant base oils analytically and highlighted their carcinogenic characteristics.

The standardization of analytical methods of determining PAH and the setting of threshold values was the subject of lively discussions between 1985 and 1995.

Whereas one group was primarily interested in establishing exact analyses and others wanted benzo (a) pyrene as a reference, another group was pressing a head with the IP 346 method. This was later adopted into national legislation in several countries. PAH can be determined by HPLC with anthracenes or other aromatics as markers or by GC–MS after appropriate sample preparation.

The IP 346 method does not analyze PAH directly but an extract is obtained in DMSO (dimethyl sulfoxide), in which PAH accumulate. As a rule, the extract, which is often wrongly referred to as a PAH concentrate, largely contains naphthenes or mono aromatics. According to IP 346, DMSO extracts contain only 0.1% PAH.

After numerous skin-painting tests on mice, the carcinogenicity of petroleum products corresponds to the percentage of DMSO extract. Criticism of the IP 346 threshold value continues.

The weakness of the value is clear when used gasoline engine oils are evaluated. The PCA enrichment caused by gasoline combustion is not considered by the IP method. For example, a 1000 ppm increase in PCA only increases the DMSO extract of the used' base oil from 1.0(fresh oil) to 1.1 %.

1.2.1.6 Aromatics in White Mineral Oils:

White oils which are used for pharmaceutical, medical or food processing applications should be free from aromatics, or at least low in aromatics, and preferably contain no traces of polycyclic aromatics. Ultraviolet absorption in the range 260 to 350 nm is used. Enrichment by extraction is worthwhile. (ASTM D

2296-99) describes such a process including pre-enrichment with dimethyl sulfoxide extraction. (G. Brandes, Brennstoff- Chem. 1956)

1.3 Refining:

Since the beginning of the petroleum industry, mineral oils have been used for lubricant base oils. The process of converting crude oil into finished base oil is referred to as refining. As far as base oil manufacturing is concerned, the actual refining process begins only after the distillation stages. Refining is thus the term often used to describe all the manufacturing stages after vacuum distillation.

Lubricant refineries are divided into integrate and none integrated plants. Integrated refineries are linked to primary crude oil refineries and are fed with vacuum distillate by pipeline. Non-integrated refineries purchase vacuum distillate on the open market or buy atmospheric residues and perform their own vacuum distillation. Occasionally they perform vacuum distillation on crude oil. (IARC, 1984)

1.3.1 Distillation:

By way of fractional distillation, products are removed from crude oil which approximately meets the viscosity grades ultimately required. Often only four or five cuts suffice to fulfill lubricant requirements. As described later, the viscosity of the primary vacuum distillate is independent of the finished base oils in hydro cracking processes because the hydro cracking process creates new molecule dimensions.

After the corresponding separation of the lighter components from the crude oil by atmospheric distillation, the lubricant components are in the atmospheric residue. (IARC, 1984)

1.3.2 De-asphalting:

Although the vacuum residue still contains highly viscous hydrocarbons which can supply valuable components for lube base oils, distillation cannot separate these from the asphalt which is also present and extraction processes must be used to separate these highly viscous base oils, commonly known as

bright stocks. Bright stocks are produced in lube oil refineries when the use of the asphaltene by-product (hard asphalt) is worthwhile.

The quality of the hard asphalt for the manufacture of high quality bitumen depends on the crude oil.

Extractive separation uses light hydrocarbons (propane to heptane), of which propane is the leading product for de-asphalting. Bright stocks can be manufactured with viscosities of more than 45 mm2 s–1 at 100 °C. (IARC, 1984)

1.3.3 Traditional Refining Processes:

Vacuum distillation cuts principally determine the viscosity and flashpoint of later base oils. The precision of the fraction at the upper and lower boiling limits of a cut are of great importance.

The distillates still contain components which can detrimentally affect aging, viscosity–temperature behavior and flowing characteristics, and components which are hazardous to health. To eliminate these disadvantages several refining methods were developed, of which solvent refining has become the most accepted method over the past few decades. Today, however, new plants increasingly use hydro treatment. (IARC, 1984)

1.3.4 Acid Refining:

Acid refining has become less popular because the acid sludge waste produced is difficult to dispose of and this method has been replaced by solvent extraction. Acid refining is still used to some extent for the re-refining of used lubricating oils and for the production of very light-colored technical or pharmaceutical white oils and petroleum sulfonates as by-products.

When the distillates are treated with concentrated sulfuric acid or fuming sulfuric acid (oleum), substances which accelerate oil aging are removed. Oleum treatment (wet refining) has a greater chemical effect on the structure of aromatics and not only readily removes reactive oil components such as olefins but also reduces the aromatic content, which in turn increases the viscosity index of the product. Reactions with saturated paraffinic structures lead to refining losses. Acid-refined oils require complex neutralization and absorption follow-up treatment to remove all traces of acid and undesirable by-products. Some lubricant

specifications still require base oils to be free from acid even though modern base oils no longer come into contact with acids. (IARC, 1984)

1.3.5 Solvent Extraction:

Whereas acid refining uses chemical reaction to reduce aromatic content and to eliminate reactive, oil-aging accelerators, solvent extraction is based on physicochemical separation. Solvent extraction creates base oils which are known as solvent raffinates or solvent neutrals (SN). Extraction processes using solvents create both base oil and, after evaporation of the solvent, an aromatic-rich extract.

The selectivity of the extraction media for aromatics is an important selection parameter. In particular the selectivity towards polycyclic aromatics with three or more aromatic rings has attracted attention because of the carcinogenicity of these compounds.

Numerous extraction media have been developed, of which furfural, (N-methyl-2-pyrrolidone) NMP, and phenol have become economically significant. Sulfur dioxide (SO2) should also be mentioned because of its historical importance.

Edeleanu introduced extraction to petroleum technology in 1912. In 1999, sulfur dioxide was only used in very few refineries (one in Germany until 1999) for the refining of naphthenic distillates.

In recent years several furfural extraction plants have been converted into NMP plants, and even phenol plants have taken a back seat. NMP is a non-toxic solvent and can be used in a low solvent-to-oil ratio with high selectivity. This generates significant energy savings. NMP in new plants results in physically smaller units and thus lower capital expenditure.

As a rule, extraction plants are the first refining step for vacuum distillates because subsequent solvent dewaxing is the more complex refining process in terms of capital expenditure and operating costs. This route means that the extract part does not undergo unnecessary dewaxing.

Depending on the crude, the extract part of paraffinic oils can be 30 to 50 %. As the standard requirement of solvent neutral oils is a viscosity index (VI) of at least95, the extraction severity is matched to this demand. A higher proportion of aromatic and naphthenic hydrocarbons in the distillates require greater

extraction severity and thus a larger quantity of extract. The percentage share of the extract is a major economic factor in conventional lube refining.

In general, extracts can only be used as products which are of lower value than the finished base oils. The large quantities are used as cracker feed and only a comparatively small proportion can be used as process oil. Their use as plasticizers has been severely limited in recent years because their carcinogenicity. The same applies to their use as lubricant base oils. Aromatic extracts have a very high viscosity—pressure coefficient and in the past were often used in metal-forming operations, such as cold extrusion.

A 1-point increase in viscosity index as a result of greater extraction severity creates, on average, 1% more extract. Some refineries these days produce so-called semi-raffinates with VIs of between 70 and 80. It is cheaper to adjust the VI later with VI improvers than to produce 15 or 20% more extract. These half-raffinates, however, have disadvantages resulting from the lower extraction severity— e.g. lower oxidation stability, possibly higher amounts of sulfur or higher amounts of polycyclic aromatics.

Solvent extraction is generally only economic to a minimum VI of 50 of the vacuum gas oil. It must, however, be remembered that subsequent solvent dewaxing further cuts the yield by approx. 20 %. (IARC, 1984)

1.3.6 Solvent Dewaxing:

In traditional refining processes, solvent extraction is followed by solvent dewaxing.

Long-chain, high melting point paraffins negatively affect the cold flow properties of lube oil distillates and lead to a high pour point. This is caused by the crystallization of waxy substances at low temperatures which results in turbidity and an increase in viscosity. Their removal has, therefore, been an important consideration since the beginning of crude oil refining.

Dewaxing by crystallization of paraffins at low temperatures and separation by filtration are the principal processes in traditional refining. Compared with catalytic dewaxing with hydrogen, urea-dewaxing to separate n-paraffins is of relatively minor importance in lube oil refining. Crystallization

methods involve mixing the solvent with the oil; this improves filtration, as a result of dilution, and promotes the growth of large crystal formations.

The important solvents are ketones and chlorinated hydrocarbons. Dewaxing with ketones (dimethyl ketone, methyl ethyl ketone, MEK) is normally used for pour points down to -12°C. For lower pour points, the Di–Me (dichloroethane–dichloromethane mixtures) method is used. This also enables the manufacture of hard and soft waxes. As for the extracts created by solvent extraction, the by-product situation is again problematic.

The paraffins are only worthwhile if their quality and processing is good and they can then be sold as candle wax, for coatings, and for other purposes. They are often used as cracker feed in fuel catalytic crackers (FCC) and the cracker yield is then assessed by use of special formulas. (IARC, 1984)

1.3.7 Finishing:

A finishing stage often follows the two refining stages of extraction and dewaxing.

In the past, methods which employed absorbents were often used but these days the processes almost all use hydrogen (hydro finishing). Finishing should improve the color of the product and remove surface-active substances which can negatively affect the air-release or demulsifying properties of lubricating oil.

Depending on the temperature, pressure, catalyst, and space velocity of the Hydro finishing, a more or less severe hydrogenating process can be initiated. In general, the hydro finishing process is referred to as mild hydro treating and focuses on improving color, odor, and ultra-violet stability. Ferro fining (BP) has achieved major economic importance as a finishing process. The process parameters do not generally lead to de-sulfurization. More severe processes with pressures in excess of 100 bars can bring about significant de-sulfurization and some de-aromatization.

Additional finishing with absorbents (bleaching clays, bauxite) is sometimes used for the manufacture of refrigerator, transformer, or turbine oils. The subsequent filtration process in filter presses or other filtering equipment represents additional complexity and the disposal of the filter residue is an increasing problem. (IARC, 1984)

1.4 Lube Crudes:

Crude oils which yield high-grade base oils are preferred for the production of lube base oils. Of importance to the vacuum gas oils (VGO) which are derived from the crude oil and which are the direct feeds for lube refining are, particularly, VI, wax content and sulfur content. A high VGO VI leads to low extraction losses in solvent extraction processes and low hydrogen consumption during hydrogenating processes.

High wax contents increase production costs, as a result of the high operating costs of the dewaxing plant, as well as reducing base oil yield. In a base oil production unit using a wax isomerization process, high wax content can also contribute to a higher VI. The finished base oils have high sulfur content, especially after conventional solvent refining. Hydrogenating processes almost fully eliminate sulfur but desulfurization consumes hydrogen. (IARC, 1984)

1.5 Base Oil Manufacturing by Hydrogenation and Hydro cracking:

Traditional solvent refining is the separation of unwanted components from vacuum distillates. Hydrogenation and hydro cracking in the manufacture of lubricant base oils significantly influence the chemical structures of mineral oil molecules. On one hand, unstable molecules are chemically stabilized by the removal of the hetero atoms (sulfur, oxygen, nitrogen) and on the other, severe hydrogenation can convert aromatics into saturated naphthenic or paraffinic structures. In addition to the hydrogenation process, hydro cracking breaks-down or cracks larger molecules into smaller ones. Larger molecular structures can reform from small fragments.

The principal process criteria are temperature, pressure, and the catalyst and space velocity. If special conditions are met, a focal point of the process is the isomerization of paraffinic structures. Besides the saturation of aromatics, opening of the naphthenes rings can occur.

It is clear that lubricant base oils can be much more easily tailored using these processes than is possible with simple solvent refining separation. The future of lube base oil production thus lies with hydrogenation and hydro cracking. An additional advantage of advanced hydro cracking is the lower dependence on the quality of the crude oil. Although the economic boundaries of solvent refining are set by yield (extract and paraffin quantities), altering hydro cracking process parameters can compensate for varying crude oil qualities.

The roots of manufacturing lubricant base oils by hydrogenation lie in previous attempts to liquefy coal by high-pressure hydrogenation. On the basis of the results of Bergius (1913, Hanover, Germany), the first technical plant began operations in1921 in Mannheim–Rheinau, Germany. Lubricating oil cuts were, however, inferior to those obtained from petroleum. The combination of results with the above and experience from ammonia synthesis of the high pressure behavior of hydrogen (BASF Ludwigshafen, Germany, 200 bar, 1923) formed the basis of the catalytic high-pressure hydrogenation of coal, tars, and petroleum products. Key contracts between the German IG and the American Standard Oil Co. of New Jersey for the hydrogenation of petroleum in 1927 and 1929 led to the construction of two large-scale hydrogenation plants in Baton Rouge, Louisiana and Baybay, New York in 1930 and 1931. Apart from light products, _Essolube' lubricant base oils were thus created.

Disregarding finishing processes and white oil production, high-pressure hydrogenation processes to create lubricant base oils from petroleum became fashionable again only in the early seventies. In the German Democratic Republic, lubricant base oils were manufactured by the high-pressure hydrogenation of lignite tars up to the early nineties.

While the processes mentioned are generally replacing solvent extraction for the manufacture of base oils, there are hydrogenation processes which either crack long-chain paraffins with specific catalysts into light products and remove them from the base oil or convert them into iso paraffins with good low-temperature characteristics without a significant loss in yield. If one combines the previously mentioned hydrogenation processes with this type of catalytic hydrogenating dewaxing, this is called the _all-hydrogen route'. (Grimmer. etal, 1981)

1.6 Manufacturing Naphthenic Base Oils by Hydrogenation:

Only about 10% of the petroleum base oils used in lubricants is naphthenic. Before 1980, naphthenic oils were significantly more important (USA 25 %). In 1983, a publication by the IARC (International Agency for Research on Cancer) defined a number of mineral oil products as carcinogenic. None of these was a severely treated distillate. Naphthenic oil, which normally has good natural lowtemperature properties, did not need to be dewaxed and it was possible to convert the vacuum distillates into low viscosity index (LVI) and moderately good aging behavior lubricant base oils by simple refining (acid refining or hydrofinishing). In the past these base oils were much cheaper than paraffinic solvates and this also explained their popularity. Although the IARC publication and subsequent legislation and classification requirements led to the disappearance of this type of oil from important markets, naphthenic base oils were still required for some lubricant applications (greases, metalworking oils, and refrigerator oils) because of their outstanding solubility and these are now manufactured with severe hydrogenation which produces non-carcinogenic base oils. Solvent extraction (e.g. the Edeleanu process) with its high proportion of extracts and low yields is now less advantageous. (Grimmer. etal, 1981)

1.7 Production of White Oils:

The large-scale production of white oils by high- pressure hydrogenation is a part of the first steps in the manufacture of lubricant base oils by hydrogenation. White oils – light-colored, odorless, low-aromatic or aromatic-free mineral oil raffinates are used in a number of applications as medical and pharmaceutical white oils or technical white oils in, above all, the food and beverage industries as food-grade lubricants. BASF Germany was the first to develop white oil manufacturing from the high-pressure hydrogenation technology used to liquefy coal. Up to that point, white oils were manufactured exclusively by acid treatment. The feeds for hydrogenated white oil manufacturing are generally naphthenic or paraffinic solvent-treated vacuum distillates or untreated vacuum distillates. In the BASF process hydrogenation takes place in two stages: the first at 300–380 °C

and 80–200 bar with a Ni Mo catalyst and the second at 200–300 °C and 100–200 bar with Ni catalyst. The first stage produces the technical qualities (according to the US Food and Drug Administration) while the second stage produces the medical grades (e.g. German Pharmacopoeia). (Grimmer, etal, 1981)

1.8 Lube Hydro cracking:

The principal elements of the lube hydro cracking process are the cracking of low-VI components and the saturation of aromatics. Hydro cracking as a major method of future mineral base oil manufacture can be performed in two types of refinery. The first is a lube oil refinery which operates a hydrocracker with the principal objective of producing lubricating oils and the second is a refinery which operates a fuel hydrocracker to convert vacuum gas oil into high-grade fuels. Fuel hydrocracker residues are excellent feeds for the manufacture of lubricant base oils and hydrocrackers used to make olefin feeds for steam crackers can supply a premium quality feed for lube base oils. The severity has the most important effect on the quality of the base oil produced. High severity (e.g. 80% light products) generates a high- VI and low-evaporation oil. While hydro cracking refineries for base oils with normal Vis and normal Noack evaporation (HVI oils, Group II oils.) at moderate severity are being built in the USA and the Far East, based mainly on Chevron Technology, Western European hydro cracking plants were designed for low-evaporation base oils (Shell, Petit Couronne, France, 1972; Union FUCHS, Wesseling, Germany, 1986and BP, Lavera, France). These produce Group III oils, on one hand from waxes and on the other from vacuum gas oils with hydrocracker residues.

Hydro cracked base oils differ from solvent-extracted oils by their extremely low aromatic content and their chemical purity, i.e. only traces of hetero atoms such as sulfur, nitrogen, or oxygen. Furthermore, Group III oils which were manufactured at high severity or from waxes have Noack evaporation characteristics which are about 50% down on equi viscous, solvent-extracted vacuum distillates. If catalytic dewaxing is performed, even lower pour points can be achieved than with SN oils.

Since 1995, more than half of all new base oil manufacturing plants have been built or are planned using hydro cracking technology.

Apart from high-value base oils, sufficiently large hydro cracking plants offer favorable operating costs and greater crude oil flexibility, despite high initial investment costs. A lube oil hydro cracking plant which operates at approx. 50% vacuum distillate severity generates just as many high-quality light products as lube base oils while, at present, just as many low-value extracts and paraffin waxes are generated by solvent extraction.

To increase VI from 100 to 125, the hydro cracking severity has to be increased so much that up to one half of the base oil yield is lost. The increasing number of fuel hydrocrackers, which are a response to increasing demands for low-sulfur oils (diesel fuels with < 50 ppm sulfur), represents a growing source of hydro cracked feeds. Fuel hydrocracker residue can be processed into high-quality, hydro cracked base oils by distillation and subsequent dewaxing (wax isomerization) at acceptable cost.

During the hydro cracking process, especially if vacuum gas oils are severely treated to create very high viscosity (VHVI) oils, polycyclic aromatics can be formed along with saturated structures (aromatics to naphthenes or paraffins and isoparaffins) under some process conditions. These have to be removed by subsequent high pressure hydrogenation or by extraction if hybrid methods are used. This subsequent hydrogenation can also be manipulated so that it also produces a significant increase in VI. (Grimmer. etal, 1981)

1.9 Catalytic Dewaxing:

The most complex stage of traditional base oil refining is solvent dewaxing. In addition to the high capital expenditure and, above all, operating cost, the solvent-related limits to the achievable pour point are a disadvantage.

In recent years various methods have been developed which can remove un branched, long-chain or short-chain paraffins, less branched paraffins, and some other petroleum components by catalytic and hydrogenation reactions or convert them into such components which improve the low-temperature characteristics of the base oil.

The first technologies were based on the catalytic cracking of these abovementioned substances. In 1979, Mobil introduced the _Mobil Lube Dewaxing Process' (MLDW). The development of catalysts since then has led to strong hydro isomerization activity processes (Mobil selective dewaxing, MSDW).

While the MLDW process mainly involves the cracking of long-chain paraffins and the production of larger quantities of light by-products and poor base oil yields, MSDW processes create high-yield, non-waxy iso paraffins from the waxy parts of the feed.

At the beginning of the nineties, Chevron successfully introduced its iso dewaxing process. In 1999 it is the most important technology for catalytic dewaxing.

The process combines relatively high yields with low pour points and high VIs. The method uses a hydrogenation component as an intermediate pore-silico aluminum phosphate molecular sieve (e.g. Pt on SAPO-11). In terms of process economy, high base oil yields but also the creation of mainly high-value C5+ liquids as by- products are of significance. In particular, the formation of low-value by-products such as propane is avoided.

In the development of new dewaxing catalysts, it is important to achieve the right balance between hydrogenation activity and acid activity. Increasing hydrogenating properties usually leads to a reduction in isomerization and thus a worsening of the pour point.

Higher acid activity increases cracking and thus yields losses. Sulfur and Nitrogen in the feed play important roles. Nitrogen is detrimental to acid activity and sulfur is poison for catalysts for metal hydrogenation components.

Although iso dewaxing enables manufacture of lubricant base oils with pour points below – 45°C, with catalytic dewaxing processes one must never forget that dewaxing to very low pour points (depending on process conditions and catalysts) leads to VI losses.

A major success of this new dewaxing process is the molecular sieve catalysts –zeolites with exactly defined mesh sizes. (Grimmer. etal, 1981)

1.9.1 Wax Isomerization:

VHVI oils have been manufactured from waxes since the early seventies. Feeds were wax cuts from solvent extraction processes. The conversion of long-chain normal paraffins or less branched paraffinic components into isomerized

products with good low-temperature characteristics and high VIs succeeded, with suitable catalysts, in isomerizing hydro cracking processes. Because the input feed is already aromatic and naphthene-free, the catalysts can be fully optimized to the conversion of the paraffinic material. At relatively high yield losses, the process creates high-quality but comparatively expensive hydro cracked oils (Shell XHVI, Exxon Exxyn, Mobil MWI-2 catalyst). (Grimmer. etal, 1981)

1.10 Hybrid Lube Oil Processing:

The combination of traditional solvent refining with severe hydro treating and hydro cracking processes is known as hybrid processing. On one hand, such processes should represent an extension of existing refineries operations, and on the other, exploit the favorable properties of hydrogen processes. To create higher VIs (> 105) and to reduce sulfur content, hydro treating can follow solvent extraction (e.g. in the manufacture of turbine oils).

The combination of a mild furfural extraction with hydro cracking can produce hydro cracked oils by low-hydro cracking conversion in small hydro cracking reactors.

The low hydrogen consumption adds to the economy of such processes given that the extraction plant exists and the hydrocracker can be integrated into the infrastructure of a lube oil refinery. The introduction of a hydro cracking stage into a conventional solvent refinery offers the attractive possibilities of debottlenecking if the corresponding dewaxing capacity is available.

Catalytic dewaxing can, on the other hand, follow solvent refining. This presupposes that the catalytic dewaxing catalysts can withstand, and are not poisoned by, the sulfur and nitrogen components in the solvent raffinate. In the Mobil processes, this is more likely with MLDW catalysts than with MSDW catalysts.

In connection with the isomerization of slack waxes from petroleum distillates, several other high-paraffin components should be considered as future base oil feeds. These could include high-wax (> 70 %) natural distillates and Fischer–Tropsch waxes or synthetic fluids from natural gas. Significant future importance is given to this latter group of products. (Grimmer. etal, 1981)

1.11 Hystart:

The quality of low-quality lube feeds vacuum gas oils (VGO) can be improved by hydrogenation before solvent extraction. This process is called Hystarting or Hystart. (Grimmer. etal, 1981)

1.12 All-Hydrogen Route:

The production of base oils by hydro cracking and catalytic, solvent-free dewaxing is called the all-hydrogen route. Depending on the severity of the hydrocracker, Group II HVI or Group III VHVI oils can be produced (HC-I and HC-2 oils). Leading examples for HC-I oils are the Chevron refinery in Richmond, California and the Conoco–Pennzoil refinery in Lake Charles (Exel Paralubes). Some HC-I refineries have been prepared to produce HC-2 oils by the all-hydrogen route by increasing the severity of the hydrocracker.

Table 1-1 shows the API and ATIEL classification of the base oil according to sulfur concentration wt/wt %, saturates percentage and the viscosity index.

Table 1.1

Group	Sulfur (% w/w)	Saturates (% w/w)	Viscosity index	
I	> 0.03 and /or	< 90	80–120	
II	≤0.03 and	≥90	80–120	
III	\leq 0.03 and	≥90	> 120	
IV		All polyalpha	olefins (PAO)	
V		All base oils not included in Groups I-		
		IV or VI		
VI		All polyinternalolefins (PiO)		

The use of a fuel hydrocracker for the production of base oils using the all-hydrogen route was first realized by the SK Corporation (Ulsan, Korea) in 1995. By recycling the hydrocracker bottom and the special integration of the fuel

hydrocracker in the lube oil process, SK also developed a specific method (UCO process). (Grimmer.G etal, 1981)

1.13 Gases-to-Liquids Conversion Technology

As a result of efforts to increase the value of natural gas in logistically favorable locations, the chemical liquefaction of natural gas (also the chemical reaction route) was developed (on the basis of the Fischer–Tropsch process). This process creates high-quality liquid products and paraffin wax. High-quality UHVI oils can then be obtained from natural gas by part oxidation, polymerization, and isomerization.

The base oil market could undergo dramatic changes if Fischer-Tropsch waxes, which are part of gas-to-liquids technology, become more generally available.

The 80-year-old Fischer-Tropsch technology has attracted considerable attention in the last few years. The focus of this attention is the better utilization of natural gas. Syngas (CO and H2) is made from methane, oxygen (air) and water vapor and this, in turn, is made into fluid and solid hydrocarbons in the Fischer-Tropsch reactor. The solid hydrocarbon waxes (> 99% paraffins) are hydrocracked, hydro-isomerizated and iso-dewaxed into super-clean base oils. Shell has used such waxes from its Bintulu plant in Malaysia for its XHVI's. New technologies for smaller, efficient plants have been developed by Rentech and Syntroleum in the last years.

It is, nevertheless, most likely that major oil companies will be the first to operate large-scale gas-to-liquid (GTL) plants. ExxonMobil, Shell, and Sasol Chevron have each announced GTL projects including base-oil-production units in Qatar. These plants start production between 2009 and 2012.

GTL base oils will have premium characteristics, including very high viscosity indices, essentially no sulfur and nitrogen, very low evaporative losses, and almost no aromatic content. They will most probably be classified as Group III+ base oils, because their characteristics will vary between UHVI base oils (Group III) and poly alphaolefins (Group IV).

Besides natural gas, all carbon containing materials can, in principle, be used for production of liquid products and paraffin wax by Fischer–Tropsch technology.

Because of the limited availability of crude oil, gasification and liquefaction of carbon, biomass, and even oil sands are of increasing interest. (Grimmer. etal, 1981)

1.14 Boiling and Evaporation Behavior of Base Oils:

In traditional solvent refining, the boiling point distribution of base oils is largely determined during vacuum distillation. Three, four or five (including bright stock) cuts are taken whose boiling point distribution is reflected in the finished base oil.

Lighter products can only be created during hydro finishing as the final refining stage. Components which negatively affect the evaporation behavior of the base oil can remain if these light components are not fully stripped.

In hydro cracking processes, the decisive distillation stages take place after cracking or after catalytic dewaxing. The function of the stripper or fractional distillation columns plays an important role whenever light cracked products are created. The target of base oil distillation is the viscosity desired at 40 and 100°C. The same distillation cut (same boiling point distribution) with different chemical structures leads to different viscosities. A highly naphthenic cut produces a higher viscosity than a paraffinic cut. In other words, equiviscous cuts of different chemical structures have different boiling-point distributions.

In practice, ISO viscosity grades or other required viscosities are created by blending different cuts. If the boiling points of both cuts are too far apart, the flashpoint drops significantly and evaporation increases. Flashpoint and evaporation are generally determined by the base oil components with the lowest boiling points.

Apart from the use of a variety of laboratory distillation apparatus to determine the boiling point distribution of base oil, gas chromatographic methods of determining boiling point have gained popularity over the last few years. The use of gas chromatography to determine the boiling range has developed continuously, especially as a result of improvements in columns and software, and

has found its way into various standards. Both non-polar packed and capillary gas chromatographic columns can be used. The injection port temperature is between 360 and 390°C depending on the length of the column. The initial column temperature can be as low as –50°C, the final column temperature is between 360 and 390°C and the programming rate is usually below 100 min–1. The maximum temperatures stated are the decomposition thresholds of the fractions being tested or of the column material.

In recent years, the evaporation characteristics of lubricants have become increasingly important quality criteria. The reasons for this are the emissions created when a lubricant evaporates, and the accompanying change to the lubricant's composition.

The topic of evaporation behavior has become increasingly important in line with the trend towards lower viscosity oils for most applications (energy-saving oils).

Evaporation is dependent on the vapor pressure of the base oil components at a given temperature and the ambient conditions (such as atmospheric pressure, turbulence boiling point \mathbb{C} .

Mathematical models have been created for complex hydrocarbon mixtures. As a simple laboratory method, Noack evaporation (1 h at 250C) has become the established method of characterizing evaporation behavior in lubricant specifications with the evaporation losses being given in w\w%. Gas chromatographic methods are also used but these can produce somewhat deviating results.

This led to a simulated Noack evaporation method using gas chromatography. In the USA, a gas chromatographic process was developed to determine engine oil volatility (ASTM D5480). In this case, evaporation takes place at 371C (700 F) and the evaporating components are measured to ca. n-C22. Although this process is easily reproducible, the values obtained are not comparable with those from the Noack.

Base Oil Categories and Evaluation of Various Petroleum Base Oils

The American Petroleum Institute (API) and the Association Technique de
l'Industrie Europenne des Lubrifiants (ATIEL) have classified base oils according
to their chemical composition. Initially there were four groups; after the

introduction of VHVI oils in Europe this was increased to five. The most important reason for these groups was the necessity to regulate base oil interchangeability for engine oils.

The classification of petroleum base oils (Groups I to III) considers Three parameters: saturates content, sulfur content, and viscosity index. Table 1.2 shows this classification. Accordingly, Group I oils are solvent extracted HVI oils (SN oils), Group II oils are hydrogenated or hydro cracked oils (as the sulfur content of < 300 ppm shows) and Group III products are VHVI oils manufactured by severe hydro cracking and or wax isomerization (VI > 120, sulfur < 300 ppm).

The table below1-2 shows typical data of various hydrocracked base oils (HC-oils), in comparison with solvent refined oils and polyalphaolefins. Typical data for 4 mm2 s-1 base oils-HC oils- in comparison with conventional solvent refined oils and polyalphaolefins. API group I II III IV.

Although there are several technical intermediate possibilities, severely hydro cracked Group III oils have become well-established on the market.

Table 1.2

	Solvent Refined	HC-1	HC-2	HC-3	Poly alpha olefin
Viscosity at 100 C	4	4	4	4	4
Viscosity Index	100	105	125	130	125
Volatility, Noack evaporation loss(%w/w)	23	18	14	13	12
Pour point	-15	-15	-18	-20	-65
API Group	I	Ц	Ш	Ш	IV

(T. Mang, 1989)

High quality base oil definitely produces a lubricant of a high quality, which would reduce the cost of the enhancements by using low levels of the additives, further more additives are not replacing the quality of the base oil so the base oil choosing by formulators is a critical issue for lubrication industry.

Table 1.3. shows the general properties of SN 150.

Table 1.3

	Unit		Specifications	
Oil property		Test Method	Min	Max
Kinematic Viscosity at 100C	mm2\s or cSt	ASTM D445	5.00	6.50
Flash Point	C	ASTM D92	210	
Pour Point	C	ASTM D97		-6
SpecificGravity at15.6C	-	ASTM D1298	870	
Sulfur Content	Wt%	ASTM D2622	0.3 Typical	
Color	-	ASTM D1500		1.5
Noack	Wt%	ASTM D5800	10 Typical	
Carbon Residue	Wt%	ASTM D189	0.05	

Table: 1.4. 2005 world lubricant consumption by region in million metric tons (Rizvi, 2009)
Table 1.4

Region	Estimated Use (mmt)
North America	9.48
Asia Pacific, Near/Middle East, Africa	14.02
Central and Eastern Europe	4.80
Russia	1.67
Western Europe	4.69
Latin America	3.00
Others	0.24
Total	37.9

1.15 Theoretical back ground for the test methods:

Fundamental tests for the base oils are applied for the two samples. They are including (Visual tests, Thermal tests, physical and chemical tests).

Chemical reagents which were used in this research are of analytical reagent grade.

All the apparatus and equipments used are valid with certificates of calibration.

1.15.1 Appearance:

This test is a visual test to identify the base oil out look, generally the oil should be clear and bright without any suspending matters, and further more it should look homogeneous.

This method is established primarily for liquid raw materials used and finished products handled in the lubricants business including greases. For qualitative determination of appearance in base stocks, process oils, and white oils, follow AM-S 1718.

This method covers visual observation of liquid raw materials kept in clear transparent containers as defined in the method, as well as visual observation of grease samples smeared to a thickness of no more than 1/2-inch.

Color or other parameters may be specified in the specification for this test but the assumption is the requirement is "Bright and Clear" or "Clear and Bright" as well as the color or other parameter stated unless otherwise specified.

Definitions/Terminology:

- 1. **Bright and Clear** A condition in which the sample is free of haze or cloudiness, with no visible excess free water droplets or particulates. Also may be referred to as Clear and Bright.
- 2. **Haze or Cloudiness** The sample clarity is less than transparent caused by wax crystals, moisture, or particulates suspended in the material. Individual wax crystals, moisture, or particulates must be smaller than the visible range by the naked eye.
- 3. **Particulates** Small solid or semisolid particles that may or may not be suspended in the sample and visible by the naked eye.
- 4. **Free water** Visible water droplets in the sample.

Apparatus:

1. The sample must be presented into a clean and transparent container for examination. Typically a clear one-liter bottle should be used for examination of liquids. If sample clarity cannot be assessed due to the one-liter container dimensions, smaller diameter (and capacity) clear and transparent sample

containers can be used. Grease samples are typically collected in wide-mouthed sealed containers.

- 2. The light source should be significant enough to easily see through the container. Typically the available light should be at least 200 lux for viewing the sample.
- 3. Sonicator A sonic bath using a frequency in the range of about 40-50 kHz, a power output of about 100 watts per liter of reservoir volume and a reservoir volume of at least one liter.

1.15.2 Odor:

This test is performed by expertise personnel to check the odor of the base oil, some oils are not well treated and have undesired odor for the customers. So this test is important for the newly received oil.

If a sample does not exhibit an atypical odor, it is reported as "Pass," whereas if an atypical odor is detected, it is reported as "Fail." Respiratory ailments or any other condition that inhibits an analyst's olfactory sense may impact an analyst's ability to perform this test.

Apparatus

Wide-mouth containers those are capable of holding quantities of test samples and reference samples required by the method. 100-mL low-form (Griffin) beakers have been found suitable to use.

Reagents and Materials:

1. Acceptable Odor Reference Samples - Prepare one of more samples that exhibit typical odors (i.e., "Pass") for the product type of interest to use in comparative testing. Ensure the reference sample is properly sealed during storage.

Suitable reference samples include a laboratory hand-blend of the product to be tested made with the same components as the sample to be tested or a retained sample of a previously acceptable batch. NOTE 1: If an acceptable odor reference sample exhibits an atypical odor during storage, it needs to be replaced immediately. Acceptable odor reference samples can be used in comparative testing for up to one year from the date of manufacture before being replaced by another acceptable odor reference sample.

2. Atypical Odor Reference Samples - It is recommended that one or more samples be prepared that exhibit atypical odors (i.e., "Fail") for the product type of interest to be used as a suitable reference in comparative testing. As examples, suitable reference samples may involve intentionally imparting a burned odor smell or leaving out a component that results in a different smell than if prepared properly. Ensure the reference sample is properly sealed during storage.

1.15.3 Density ASTM - D4052-11

This test method covers the determination of the density, relative density, and API Gravity of petroleum distillates and viscous oils that can be handled in a normal fashion as liquids at the temperature of test, utilizing either manual or automated sample injection equipment. Its application is restricted to liquids with total vapor pressures (see Test Method D5191)typically below 100 kPa and viscosities (see Test Method D445or D7042) typically below about 15 000 mm2/s at the temperature of test. The total vapor pressure limitation however can be extended to>100 kPa provided that it is first ascertained that no bubbles form in the U-shaped, oscillating tube, which can affect the density determination. Some examples of products that may be tested by this procedure include: gasoline and gasoline-oxygenate blends, diesel, jet, base stocks, waxes, and lubricating oils.

Significance and Use

- 1. Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and petroleum products.
- 2. Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperature of 15°C.

Apparatus

- 1. Digital Density Analyzer— a digital analyzer consisting of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display. The analyzer shall accommodate the accurate measurement of the sample temperature during measurement or shall control the sample temperature. The instrument shall be capable of meeting the precision requirements described in this test method.
- 2. Circulating Constant-Temperature Bath, (optional), capable of maintaining the temperature of the circulating liquid constant to 60.05°C in the desired range. Temperature control can be maintained as part of the density analyzer instrument package.
- 3. Syringes, for use primarily in manual injections, at least2 mL in volume with a tip or an adapter tip that will fit the opening of the oscillating tube.
- 4. Flow-Through or Pressure Adapter, for use as an alternative means of introducing the sample into the density analyzer either by a pump, by pressure, or by vacuum.
- NOTE 1— it is highly recommended that a vacuum not be applied to samples prone to light-end loss, as it can easily lead to the formation of bubbles. It is recommended to fabricate a special cap or stopper for sample containers so that air, such as from a squeeze pump, is used to displace at set specimen to the U-tube measuring cell by the flow-through method.
- 5. Auto sampler, required for use in automated injection analyses. The auto sampler shall be designed to ensure the integrity of the test specimen prior to and during the analysis and be equipped to transfer a representative portion of test specimen to the digital density analyzer.
- 6. Temperature Sensing Device (TSD), capable of monitoring the observed test temperature to within accuracy of 0.05°C. If a liquid-in-glass thermometer is used as the TSD, it shall be calibrated and graduated to 0.1°C, and have a holder that can be attached to the instrument for setting and observing the test temperature. In calibrating the thermometer, the ice point and bore connections should be estimated to the nearest0.05°C. For non-mercurial thermometers, the TSD device shall be calibrated at least annually against a certified and traceable standard.

7. Ultrasonic Bath, Unheated, (optional), of suitable dimensions to hold container(s) placed inside of bath, for use ineffectively dissipating and removing air or gas bubbles.

Reagents and Materials

1. Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, may be entrained in viscous sample types prior to analysis.

1.15.4 Kinematic Viscosity ASTM D445-12

This test method specifies a procedure for the determination of the kinematic viscosity, n, of liquid petroleum product, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, h, can be obtained by multiplying the kinematic viscosity, n, by the density, r, of the liquid.

The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm²/s.

The values stated in SI units are to be regarded as standard. The SI unit used in this test method for kinematic viscosity is mm2/s, and the SI unit used in this test method for dynamic viscosity is mPa·s. For user reference, 1 mm2/s = 10^{-} 6m2/s = 1 cSt and 1 mPa·s = 1 cP = 0.001 Pa·s

Significance and Use

Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

Apparatus

1. Viscometers—only calibrated viscometers of the glass capillary type have been used, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

Reagents and Materials

Toluene & distilled water& acetone and samples.

1.15.5 Pour Point ASTM D97 -12

This test method covers and intended for use on any petroleum product. Several ASTM test methods offering alternative procedures for determining pour points using automatic apparatus are available. None of them share the same designation number as Test Method D97.When an automatic instrument is used, the ASTM test method designation number specific to the technique shall be reported with the results.

Significance and Use

The pour point of a petroleum specimen is an index of the lowest temperature of its utility for certain application.

Apparatus

1. Test Jar: Cylindrical, of clear glass, flat bottom, 33.2 to 34.8-mm out side diameter, and 115 to 125 mm in height. The in side diameter of the jar can range from 30.0 to 32.4 mm, with in the constraint that the wall thickness be no greater

than 1.6 mm. The jar shall have a line to indicate a sample height 54 ± 3 mm above the in side bottom.

Table 1.5 shows thermometers: Having the following ranges and conforming to the requirements for this test method.

Table 1.5

	Temperature range	Thermometer Number	
		ASTM	IP
High cloud and pour	-38to +50 C	5C	1C
Low cloud and pour	-80 to +20 C	6C	2C
Melting point	+ 32 to + 127 C	61C	63C

1.15.6 Flash Point ASTM D93 -12

This test method covers the determination of flash point of petroleum products in the temperature range from 40 to360° C by a manual Pensky-Martens closed cup apparatus, and the determination of flash point of biodiesel in the temperature range of 60 to 190° C by an automated Pensky-Martens closed apparatus.

Significance and Use

The flash point temperature is one of the tendencies of test specimen to form a flammable mixture with air under controlled laboratory conditions. It is only of a number of properties which must be considered in assessing the over all flammability hazard of a material.

Flash point is used in shipping and safety regulation to define flammable and combustible materials. One should consult the particular regulation involved for precise definition of these classifications.

Apparatus

1. Pensky-Martens Closed cup apparatus (manual).

This apparatus consist of the test cup, test cover and shutter, stirring device, heating source device, ignition source device, air bath and top plate.

2. Temperature Measuring Device

Thermometer having a range as shown as follows and conforming to the requirements prescribed below.

Table 1.6 shows the ASTM and IP, thermometers numbers and their ranges conforming to ASTM D93.

Table 1.6

Temperature range	Thermometer Number	Thermometer Number	
	ASTM	IP	
-5 to + 110 °C	9 C (9F)	15 C	
+ 10 to200 ℃	88 C (88 F)	101 C	
+90 to 370 ℃(200	10C (10 F)	16 C	
to700°F)			

3. Ignition Source

Natural gas flame and bottled gas flame have been found acceptable for use as the ignition source.

Reagents and Materials: Cleaning solvents capable of cleaning out the specimen from the test cup and drying the test cup and cover. Toluene and Acetone are suitable for this purpose.

1.15.7 Standard Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF

This test method covers the quantitative determination of additive elements in unused lubricating oils and additive packages, as shown in Table 1.7 The pooled limit of quantitation of this test method as obtained by statistical analysis of inter laboratory test results is 0.02% for magnesium, 0.003% for phosphorus, 0.002% for sulfur, 0.001% for chlorine, 0.003% for calcium, 0.001% for zinc, and 0.002% for molybdenum.

Additive packages require dilution with a contamination free diluent (base oil) prior to analysis. The dilution factor has to be calculated from the expected concentrations to bring the concentrations for all elements into the ranges listed in Table 1.7

Some lubrication oils will contain higher concentrations than the maximum concentrations listed in Table1.7These samples require dilution with a contamination free diluent (base oil) prior to analysis. The dilution factor has to be calculated from the expected concentrations to bring the concentrations for all elements into the ranges listed in Table 1.7

This test method is limited to the use of energy dispersive X-ray fluorescence (EDXRF) spectrometers employing an X-ray tube for excitation in conjunction with the ability to separate the signals of adjacent elements by using a high resolution semiconductor detector.

This test method uses inter-element correction factors calculated from a fundamental parameters (FP) approach or from another matrix correction method.

The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

The preferred concentration units are mg/kg or mass %.

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Concentrations of the elements of interest are determined by comparison of these intensities against a calibration curve using a fundamental parameters (FP) approach, possibly combined with corrections from backscatter. The FP

approach uses the physical processes forming the basis of X-ray fluorescence emission in order to provide a theoretical model for the correction of matrix effects. The correction term is calculated from first principle expressions derived from basic physical principles and contain physical constants and parameters that include absorption coefficients, fluorescence yield, primary spectral distribution and spectrometry geometry. The calculation of concentrations in samples is based on making successively better estimates of composition by an iteration procedure. NOTE 1— the algorithm used for the procedure is usually implemented in the instrument manufacturer's software.

The EDXRF spectrometer is initially calibrated using a set of standards to collect the necessary intensity data. Each calibration line and any correction coefficient are obtained by a regression of this data, using the program supplied with the spectrometer.

(Warning—Exposure to excessive quantities of X-radiation is injurious to health. The operator needs to take appropriate actions to avoid exposing any part of their body, not only to primary X-rays, but also to secondary or scattered radiation that might be present. The X-ray spectrometer should be operated in accordance with the regulations governing the use of ionizing radiation.)

Terminology

Definitions: Energy dispersive X-ray spectrometry,n—XRF spectrometry applying energy dispersive selection of radiation.

Significance and Use:

Lubricating oils are formulated with organo-metallic additives, which act, for example, as detergents, antioxidants, antifoaming, or antiwear agents, or a combination thereof. Some of these additives contain one or more of the following elements: magnesium, phosphorus, sulfur, chlorine, calcium, zinc, and molybdenum. This test method provides a means of determining the concentrations of these elements, which in turn provides an indication of the additive content of these oils.

Additive packages are the concentrates that are used to blend lubricating oils.

This test method is primarily intended to be used for the monitoring of additive elements in lubricating oils.

If this test method is applied to lubricating oils with matrices significantly different from the calibration materials specified in this test method, the cautions and recommendations for this case should be taken.

Interferences

The additive elements found in lubricating oils will affect the measured intensities from the elements of interest to a varying degree. In general the X-radiation emitted by the element of interest can be absorbed by itself (self-absorption) or by the other elements present in the sample matrix. Also the X-radiation emitted from one element can further excite (enhance) another element. These inter-element effects are significant at concentrations varying from 0.03 mass %, due to the higher atomic number elements (for example, molybdenum), to 1 mass %, for the lower atomic number elements (for example, sulfur). If an element is present at significant concentrations and an inter-element correction for that element is not employed, the results can be low due to absorption or high due to enhancement.

Absorption and enhancements effects will be corrected by corrections from the FP approach or by other matrix correction models.

There can be spectral overlap of one element onto another, and the instrument must include correction procedures for any such overlaps.

Apparatus

Energy Dispersive X-ray Fluorescent Spectrometer—

Any energy dispersive X-ray fluorescence spectrometer can be used if its design incorporates at least the following features:

Source of X-ray Excitation—X-ray tube with palladium, silver, rhodium, or tungsten target. Other targets may be suitable as well. The voltage of the X-ray tube shall be programmable between 4 and at least 30 kV for preferential excitation of elements or groups of elements.

X-ray Detector—Semiconductor detector with high sensitivity and a spectral resolution value not to exceed 175 eV at 5.9 keV.

Primary Beam Filters (Optional)—To make the excitation more selective and to reduce the intensity of background radiation.

Secondary or Polarization Targets, or Both

(Optional)—To make the excitation more selective and to improve peak-to-background ratio.

Signal Conditioning and Data Handling Electronics— that include the functions of X-ray intensity counting, spectra handling by background variation correction, overlap corrections, inter-elements effects corrections, and conversion of X-ray intensity into concentration.

TABLE 1.7 Elements and Range of Applicability ASTM D7751 – 12 Table 1.7

Element	Concentration Range in mass %		
Magnesium	0.02 to 0.4		
Phosphorous	0.003 to 0.25		
Sulfur	0.002 to 1.5		
Chlorine	0.001 to 0.4		
Calcium	0.003 to 1.0		
Zinc	0.001 to 0.25		
Molybdenum	0.002 to 0.05		

1.16 Solvent Neutral Production Evaluation versus Group (II) and (III):

SN mineral base oils are prepared from crude oil derivatives according to the following processes: Distillation to adjust the viscosity and flash point, Refining to improve viscosity temperature characteristics e.g. viscosity index (solvent extraction with furfural), Dewaxing to improve low temperature properties (M.E.K) dewaxing, Hydro finishing to remove undesirable impurities from petroleum distillates such as sulfur and Nitrogen compounds and olefins. From the results we notice that sample one has well distillate and refined, but the dewaxing process for it is very poor according to very low pour point, further

more the hydro finishing for sample one is very bad, the high concentration of sulfur explain that, on the other hand sample two as general is well treated at all the production processes referring to the results. Base stocks obtained after the above listed operations are called Solvent Neutral base oils (SN).

To investigate between the two samples we had to know the characteristics of group II and group III base oils. Group II and group III base oils are high quality oils containing less than 10% aromatics and less than 300 ppm sulfur as defined by API publication 1509. Group II and III base oils made using hydro cracking\ ISO DEWAXING\hydro finishing catalyst combination typically have about 1% aromatics or less and almost undetectable amounts of sulfur and nitrogen. The virtual absence of hetero atoms containing compounds coupled with inherently low aromatics content gives them superior oxidation stability relative to group (i) base oils. The primary factor that influences the oxidation stability of group II and III oils is the aromatic concentration. But in case of group II as aromatics concentration is so less approaches zero, oxidation stability is influenced more by saturates composition, particularly the paraffin and poly cyclic naphthenes content and distribution. The importance of oxidation stability is that the more the resistance to oxidation, the less tendency to form deposits, sludge and corrosive by products in engine oil, grease and industrial oil applications, and the more resistant it is to undesirable viscosity increase during use.

Many base oils studies have described how an oil is molecular composition influences it is physical and chemical properties, particularly it is oxidation stability. In general most of the studies have shown that nitrogen hetero cycles and aromatic compounds have a negative influence on base oil stability.

On the other hand some sulfur containing compounds are viewed as desirable since they inhibit hydro carbon auto catalytical oxidation through free radical chain termination path way. Of different saturated hydro carbons found in mineral oils, paraffins are considered more stable than cyclo paraffins (naphthenes) to wards oxidation and there fore more desirable.

Understanding how the various classes of aromatics and saturates impact oxidation stability helps us to focus on appropriate product quality targets and refining strategies to best meet the future needs of the customers.

Compositional (and hence stability) differences between base oils has become an increasingly important issue for customers in recent years as changing engine and industrial oils applications demand higher quality, longer lasting lubricants. For example OEM requirements translate into new ILSAC and API categories that call for extended drain intervals in PCMO\ HDMO applications with lower viscosity and lower volatility requirements. Automatic transmission fluids are also being designed with fill- for –life applications in mind. Greases must perform at higher loads, higher bearing speeds and higher temperature. In the industrial oil area, new EPA requirements for PCB stable paper- machine oils and enhanced thermal stability for ammonia refrigeration oils demands that low aromatics catalytically processed mineral oils be used in place of the traditional solvent refined oils.

The potential health hazards of base oil are also dependent on the concentration of aromatic compounds, particularly poly cyclic aromatic compounds (PCAs). Base oils with low concentration of PCA do not cause skin cancer in chronic mouse studies. (Kramer, etal. 1999)

This work aims to:

- 1- Compare the quality of the base oil from different suppliers according to international specifications.
- 2- To give an over view on the quality of the raw materials for an important type of industry in Sudan.

Chapter Two

Materials and Method

2.1 Apparatus and Chemicals:

- 1- Two transparent containers of one liter.
- 2- Two beakers 100 ml.
- 3- Anton Par density meter SN: 81242597
- 4- Viscometer. Serial 150, No. H240. Coefficient at 40 **C**: 0.03529 Coefficient at 100 **C**: 0.03512 Liquid filling (approximate): 6.9 ml
- 5- Viscometer. Serial 150, No. H 293 Coefficient at 40 **C**: 0.03265 Coefficient at 100 C: 0.03248 Liquid filling (approximate): 7.0 ml
- 6- Viscometer. Serial 200, No. H 254 Coefficient at 40 C: 0.1060 ,Coefficient at 100 C: 0.1055 Liquid filling (approximate): 6.7 ml
- 7- Viscometer. Serial 200, No. H 271 Coefficient at 40 **C**: 0.1123,Coefficient at 100 **C**:0.1118 Liquid filling (approximate):6.1 ml
- 8- Cloud and Pour Point Cabinet. Serial Number: 100072004
- 9- Two High cloud and pour thermometer: Serial\ Repair ser No:124768 and Serial\ Repair ser No: 124778 respectively -38to +50 C ASTM 5-86C / IP1C
- 10-Low cloud and pour thermometer: -8to +20 C ASTM 6C IP2C
- 11-Melting Point thermometer +32to +127 C ASTM 61C IP63C
- 12- Petro test Serial Number: 0746054303
- 13-Thermometer Model\Repair Item: ASTM 10C-86\ IP 16C Range: +90 to 370 C Serial\ Repair ser No: 1222943
- 14-X-supreme 8000SN: X13021, filling cups and poly film.
- 15-Toluene, distilled water, acetone and samples

All the apparatus (devices and equipments) used in this research were with valid certificates of calibration, and all the chemicals and reagents were of analytical grades.

2.2 Procedures:

Appearance

Samples of the material were collected in two containers those are clean and transparent. The materials were carefully examined for the presence of any matter suspended and/or settled in the sample. Sonication may be required to remove air bubbles from the sample.

Odor

The samples odors were detected by an expert analyst. If the analyst detects a typical or atypical odor during the course of normal sample handling, the test is reported on that basis. If the analyst is not certain, the sample is compared to a reference sample.

Density

A small volume (approximately 1 to 2 ml) of liquid sample from each was introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density, relative density, or API Gravity of the sample. Both manual and automated injection techniques are described.

Kinematic Viscosity

The times were measured for a fixed volume of liquid to flow under gravity through the capillary of calibrated viscometers under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity (determined value) was the product of the measured flow time and the calibration constant of the viscometer. Two such determinations were collected

from which to calculate a kinematic viscosity results those were the average of two acceptable determined values for each sample.

Pour Point

After preliminary heating the samples were cooled at a specified rate and examined at intervals of -3 **C** for flow characteristics. The lowest temperature at which movement of the specimen was observed was recorded as the pour point.

Flash Point

A brass test cup of specified dimensions, filled to the mark with test specimen and fitted with a cover of specified dimensions, then it was heated and the specimen stirred at specified rates using the suitable rate. An ignition source is directed in to the test cup at regular intervals 2 **C** with simultaneous interruption of stirring, until the flash points were detected for both samples respectively.

Elemental Determination

Specimens of the two samples were placed in the X-ray beam, and the appropriate regions of its spectrum were measured to give the fluorescent intensities of magnesium, phosphorus, sulfur, chlorine, calcium, zinc, and molybdenum. Other regions of the spectrum are measured to compensate for matrix variation. To optimize the sensitivity for each element or group of elements, a combination of optimized excitation and detection conditions may be used (no more than two conditions should be used in order to keep the analysis time as short as possible, typically under ten minutes). There may be a correction of measured intensities for spectral overlap.

Chapter Three

Results and Discussion

Table 3.1 shows the typical results of the two samples of SN 150 under investigation.

Table 3.1 Results:

SPECIFICATION	UNIT	SAMPLE1	SAMPLE2	ASTM
Appearance		C&B	C&B	
Color		2.5	0.5	D-1500
Odor		Acceptable&	Acceptable&	
		marketable	marketable	
Density at 15C	Kg\m3	8819	8739	D-4052
Kinematic viscosity	cSt or	31.88	30.96	D-445
at40 C	mm2\s			
Kinematic viscosity	cSt or	5.22	5.12	D-445
at 100 C	mm2\s			
Viscosity index		92	91	D-2270
Pour point	C	-3	-12	D-97
Flash point	C	232	214	D-92
Metals	Wt%	S=1.146	S=0.297	D-7751
	Wt%	Cl=0.002	Cl=Nil	D-7751
	Wt%	Ca=0.005	Ca=Nil	D-7751
	Wt%	Zn=0.006	Zn=0.006	D-7751
	Wt%	Mo=Nil	Mo=Nil	

Note: The concentrations in the discussion are expressed in ppm, where in the test method D-7751 in wt%, so 1/100*1/10000 = 1/1000000 = ppm.

Discussion:

Both appearance and odor results give acceptable results, where no suspending particulates and the samples are clean and bright.

The ASTM 1500 for color number show a variation in results, since it is for sample one is 2.5 which is considered a high value for this type of base oil, further more it is an indicator for the bad refining process specially the hydrogen treatment because the color is due to aromatics or unsaturated hydrocarbons, on the other hand sample two give an excellent result 0.5 in the same test which is with in the limits of the API for this base oil grade referring to table 1.3.

Density of sample one is 8819 and for sample two is 8739kg\m3 at 15C. These values influence the volumes of the base oil received, as the density increase the volumes received decrease, more over the density play a vital and important role in the shipment and delivery processes and eventually it involved in the blending percentages.

Kinematic viscosity at 40 and 100C results are approximately the same and they are within the limits of the API specifications (31.88and 30.96) and (5.22 and 5.12) centi stock respectively. The importance of the kinematic viscosity is that the desired viscosity of the lubricant is calculated depending on the base oil is viscosity. Viscosity is a very important specification for the formulation of any type of lubricant, where it is the criteria that differentiate the lubricants of the same family from each other what ever the oil is e.g. (engine, hydraulic, gear ...etc).

Pour point results are different, even though both samples are with in the limits of the API -3 and -12C respectively, but sample two may not need any enhancements for the pour point depressant PPD, while sample one need to be adjusted with an additive to give the target pour point for the produced lubricant to meet the specifications. More over, we see that sample one has a bad treatment in it is production and exactly bad dewaxing since it is pour point is very low (minimum limit for the pour point).

The results of flash point are acceptable for both samples, since the API set 190**C** as a minimum temperature for SN 150. They are 232 and 214**C** respectively for both samples; these results are obvious since sample one is considered to contain more wax.

The most important variation appear in the Energy Dispersive X-Ray Fluorescence (EDXRF), this micro analysis show a very strict different in the samples. Sulfur concentration is a very important specification, since it is concentration is involved in the groups classifications \geq 300ppm considered as group I and \leq 300ppm considered as group II & III according to API.

Generally the differences between the two samples are due to different methods employed in the processing of the base oils, the differences are as follow:

- Color: All the 2- stages hydro treated base oils are clear and colorless.
- Carbon residue is lower for hydro treated base oils.
- Total acid number (TAN) IS lower for hydro treated.
- Demulsibility of hydro treated oil will shed water better than solvent refined oils.
- Oxidation resistance for hydro treated base oils at equal levels of anti oxidant treat give superior resistance to oxidation.
- High temperature stability is better for hydro treated than the refined. (IJETT)- June 2016.

Sulfur concentration for samples one and two are 11460 and 2970 ppm respectively which will affect the performance of the lubricants those include it in their formulations, since drain intervals will increase as the sulfur content increases further more sulfur has a negative influence in the lubricants as general where it enhances (corrosion, seals damage, wear and friction) and accordingly spare parts consumption (economics) specially sample one, but sample two with in the market range referring to table 1.3.

Conclusion:

Both samples are classified as Group (I) since the sulfur content is greater than 300 ppm, but sample two show better results in the investigation

The source of sample two is a good choice than one.

The refining of sample one is proper, since the viscosity and flash point are with in the specifications, but the hydro treating and the dewaxing are very bad according to the results.

The EDXRF technique is a powerful and of great importance in this field of business.

Recommendations:

Intensive studies must be performed in this area, since all the base oils are imported to Sudan.

The future is promised to apply this industry in Sudan since the wax is treated through Hydrogenation route.

Group (II) is a good choice for our market due to acceptable specifications (\leq 90% saturates, less than 300 ppm sulfur and high viscosity index)

Sample two can be used in old API engine lubricants, such as SL and CF, but not in new grades like SN and CJ4, where the latter two needed to be formulated by group (II) base oil.

Sample one must not involved in the blending of engine oil, since it is sulfur concentration is very high. It can be used in industrial gear oils or some grease for light pressure and slow bearing.

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AM-S 1738-07* EXXONMOBIL RESEARCH AND ENGINEERING COMPANY January 2007 600 BILLINGSPORT ROAD, PAULSBORO, NEW JERSEY 08801 COPYRIGHT © 2007 Qualitative Determination of Appearance in Lubricant Raw Materials, Finished Lubricants, and Greases

AM-S 1695-08 EXXONMOBIL RESEARCH AND ENGINEERING COMPANY September 2008 600 BILLINGSPORT ROAD, PAULSBORO, NEW JERSEY 08066 COPYRIGHT © 2008 Characteristic Odor of Lubricant Oils and Greases

ASTM D1500 Standard Test Method for Determination of Color Number

ASTM D4052-11 Standard Test Method for Density, Relative Density and API Gravity of liquids by digital Density Meter

.

ASTM D445- 12 Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity).

ASTM D2270 Standard Test Method for Viscosity Index Calculation

ASTM D97 Standard Test Method for Pour point

ASTM D93-12 Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester.

ASTM D7751-12 Standard Test Method for Determination of Additives Elements in Lubricating Oils by EDXRF Analysis.

D.C. Kramer, J.N. Ziemer, M.T. Cheng, C.E. Fry, R.N. Reynolds, B.K. Lok, and M.L. Sztenderowicz, R.R. Krug Influence of Group II & III Base Oil Composition On VI and Oxidation Stability Chevron Global Lubricants
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