

# **CHAPTER ONE**

## **INRODUCTION**

### **1.1 General Introduction**

The auto-mobility is an important factor in the socio-economic life of man. Automobile is thus a basic necessity. Adequate lubrication allows smooth continuous operation of the engine and equipment with only mild wear, and without excessive stresses or seizure at the bearing (Kinghom, 1983). While the main function of engine or motor oil is actually to lubricate moving parts, motor oil also cleans, inhibits corrosion, and improves scaling by carrying heat away from moving parts (George, 1980). This then calls for importance of the quality of automobile consumable oils especially for the engine, the gear box and the brake system. This vehicles and machinery needs lubricants, especially for proper functioning of engine parts and to reduce the wearing (Dryoff, 1993). Lubrication as a branch of science reveals the need, cause and effect of the application of oils and grease to machines or engine parts. In engineering practice, the relative motion of two or more metal surfaces in contact with each other often results in friction, heat generation and wear of the rubbing surfaces. This problem has been solved by applying a fluid known as lubricant between the two rubbing surfaces, the process

of which is known as lubrication. (Chilingar *et al.*, 2005). Lubrication is needed for correct operation of mechanical systems pistons pumps, cams, bearings turbines etc. While without lubrication the pressure between the surfaces in close proximity would generate enough heat for rapid surface damage which in coarse condition may literally weld the surfaces together, causing seizure (Robert and Perry, 1997). Mineral lubricating oils are derived from crude oil in the same process that produces gasoline, kerosene and fuel oil. They vary according to the type of crude oil and the refinery methods used (Boyde, 2002). Modern motor engine oils are based upon oil refined from crude petroleum, synthetic oil treated with various compounds, or a mixture of one or two grades of mineral base oil and chemical additives (Saheed, 2012). It was also known that additives which consist of difference chemical substances are included in the oil formula to extend its range of performance. In journal bearing lubrication, a rust and oxidation inhibited additive system is used in the oils. Also antifoam and pour point depressant additives may also be present. The effectiveness of lubricating oil was known to contribute to the life span of an engine. Different lubricating oils are used in various engines because the fuels used differ chemically from one another and hence resulting products of combustion differ (Okoye, 2009). When physicochemical properties of fresh and used engine oils were carried out, it was

found that the degrading trend in the properties depend on the operating temperature of the engine, the distance covered and the age of the engine (Okeola *et al*, 2011). Another test on lubricating oils was the comparable investigation of wear metals in virgin and used lubricating oil. Choosing the best motor oil was observed to come up frequently in discussion between motor heads either on motorcycle or cars. There are various brands and types of automobile oils in the markets, the choice of which is left to the users' discretion. Therefore, the objective of this work is to study some physicochemical qualities of automobile engine oil in the market.

## **1.2 Aim and Objectives**

This work is aimed at assessing the physicochemical properties of locally blended Engine oils within Awka metropolis, and its specific objectives are;

- To collect different eight (8) samples of locally blended engine oil within Awka metropolis and two commercially made engine oil.
- To carry out quality control analyses on the samples.
- Compare the physicochemical properties of the samples.
- Determine if the properties meet specifications, so as to prevent damage on the engines on which they are applied.

### **1.3 Significance of Study**

This work is carried out to comparatively assess the physicochemical properties of locally blended engine oils within Awka metropolis with a standard to ensure good quality of the engine oils in order to minimize or totally prevent engine failure and increase the life span of the engine.

### **1.4 Scope of Study**

This work covers the collection of engine oil samples within Awka metropolis, carrying out quality control analyses on the samples and comparatively assessing the physicochemical properties of the samples with that of a standard.

## **CHAPTER TWO**

### **LITREATURE REVIEW**

#### **2.1 Review of Past Work**

Engine oils being the most effective lubricant in automobiles have attracted researcher's interest. Extensive research has been carried out on lubricants and their performance properties by international research institutes and students in the field of engineering

##### **2.1.1 Measurement and Evaluation of the Properties of Selected Commercially Produced Lubricants**

Robson H. A. B analyzed and evaluated the performance of properties of commercially produced lubricants. The samples analyzed were OANDO SAE 20W50, Total SAE 20W50, Mobil SAE 0W40, Total SAE 40, and AZ SAE 20W50 and the parameters determined were viscosity at different temperatures, flashpoint, pour point, carbon residue and pressure mapping experiment. He concluded that the oils investigated were all good for use in internal combustion engine with the best performing oil being the synthetic oil and followed by the multi grade and then the mono grade.

### **2.1.2 Comparative Assessment of Physicochemical Properties of Common Automobile Consumable Oils**

Okeola *et al.* carried a Physicochemical analysis on some common consumable automobile oils within Ilorin metropolis Kwara State, Nigeria. The analysis was carried out on Five (5) different brands of Engine oil, five (5) different brands of Brake fluid and three (3) different brands of Gear oils and the parameters tested were Kinematic Viscosity, Viscosity Index, Specific gravity and Total Base Number for the Engine oils. They concluded that the properties of the various automobile consumable oils were found to be appropriate to the various working parts.

### **2.2 Definition of lubricating oil**

Lubricating oils are fluids applied between objects or two surfaces in contact in order to reduce or prevent friction or wear. The process of applying this fluid between surfaces is called lubrication. Lubricating oils are one of many fractions or components that can be derived from raw petroleum, which emerges from an oil well as a yellow-black flammable, liquid mixture of thousands of hydrocarbons (Kinghom, 1983).

## **2.3 Lubricant Sources and Composition**

Man's search for effective lubricants is as old as recorded history. Countless materials have been used, discarded and replaced by better ones. However, some lubricants in use today are surprisingly old in origin (Carey, 1971).

Lubricant materials may be broadly classed as gaseous, liquid, semi solids and solids. Gaseous lubrication, with air as the lubricant is in practice today for high-speed and high-load applications ultracentrifuges, high speed grinding spindle, gas circulators for nuclear reactors, etc. Liquid lubricant materials range all the way from liquefied gas to synthetic liquids. The most important are mineral oils made from petroleum, but they also include animal or vegetable oils as well as water (Robert and Perry, 1992). These lubricants sources are known as base oil which serves as the foundation of the lubricant before it is blended with additives or a thickener in the case of grease. Thus, lubricants can be classified by its constituent base oil as Mineral, Synthetic or Vegetable (Robert and Perry, 1992).

### **2.3.1 Base Oil Characteristics**

All base oil has characteristics that determine how they will hold up against a variety of lubrication challenges. For a mineral oil, the goal of the refining process is to optimize the resulting properties to produce a superior lubricant. For

synthetically generated oils, the objective of the various formulations is to create a lubricant with properties that may not be achieved in a mineral oil (Fitch, 2012).

Some of the most important base oil properties include the viscosity limitations and viscosity index, pour point, volatility, oxidation, thermal stability, etc.

### **2.3.2 Base Oil Groups**

The 20<sup>th</sup> century saw a number of improvements in the refining process used for mineral oils along with the introduction of a variety of synthetics. By the early 1990s, the American Petroleum Institute (API) had categorized all base oils into five groups, with the first three groups dedicated to mineral oils and the remaining two groups predominantly synthetic base oils (Rosenbaun, 2015).

#### **2.3.2.1: Groups I, II and III**

These Groups are mineral oils with an increasing severity of the refining process. Group I base oils are created using the solvent-extraction or solvent-refining technology. This technology which has been employed since the early days of mineral oil refining, aims to extract the undesirable components within the oil such as ring structures and aromatics (Fitch, 2012). Solvent extraction starts with a heavy refinery feedstock from a crude distillation unit that's called vacuum gas oil. A very selective solvent removes much of the sulfur, aromatic and nitrogen compounds. The resulting oil stream comes out of the extractor still containing

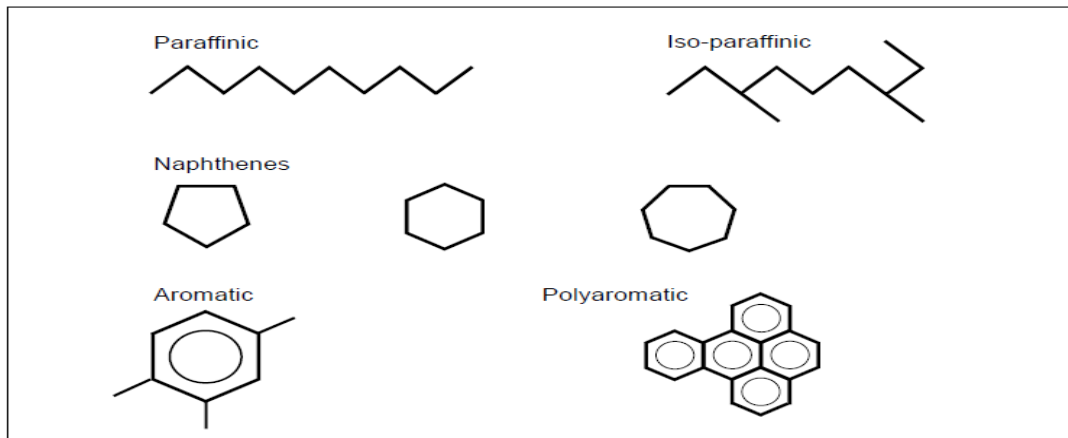


paraffins that need to be removed to make it base oil. A light solvent is added and the mixture is refrigerated to a low temperature at which the paraffins precipitate is filtered out (Rosenbaun, 2015). Thus, Group I base oil stocks contain greater than 0.03% sulfur, less than 90% saturates and has a VI ranging from 80-119 (Rosenbaun, 2015).

Group II and III base oils are both produced using hydrogen gas in a process called hydrogenation or hydro treating. The goal of this process is the same as the solvent-refining, but it is more effective in converting undesirable components like aromatics to desirable hydrocarbon structures (Fitch, 2012). Hydro processing is a method that typically uses three catalytic reactors to produce Group II and Group III base oils. The process begins with the same vacuum gas oil feedstock as Group I, but runs it through a hydrocracker at very high pressures, frequently above 2,000 pounds per square inch. The hydrocracker reshapes the molecules, saturating a lot of the aromatic compounds with hydrogen, which improves their stability. It converts sulfur into hydrogen sulfide gas and nitrogen into ammonia, creating a product called a waxy base oil that's a lot cleaner than the output of a solvent extraction and contains more than 90% saturated compounds. The second reactor is a catalytic dewaxer (also known as a hydroisomerization unit), where the wax molecules are converted to isoparaffins and even more saturation occurs. Finally,

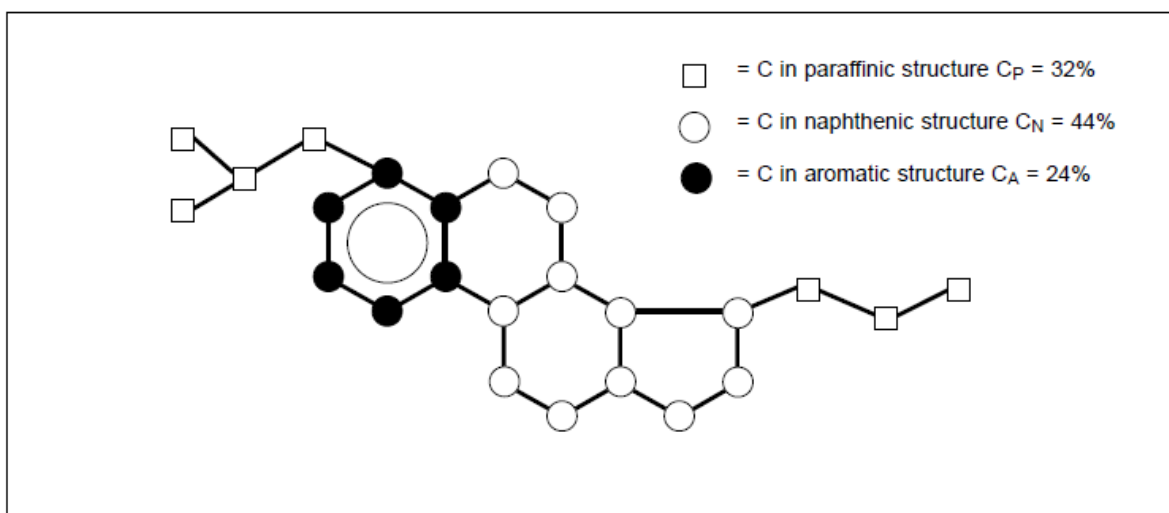
there is a lower temperature but very high pressure finishing step in which the final few percent of residual aromatic compounds are saturated to form Group II base oils. A more severe hydro cracking process is used to produce Group III oils. Groups II and III have comparable purity and typically 99% saturates (Rosenbaun, 2015). The big difference between them is that Group III has a VI of 120 or greater.

Generally, mineral base oil can be classified as Paraffinic, Naphthenic or Aromatic. Paraffinic structures can either be straight or branched chains (Schlosber *et al*, 2001). Waxes are straight type N-alkanes. Thus, Paraffinic structures provide inferior solubility of water and oxidation products, which may lead to precipitated sludge in the oil system. The advantage with paraffinic oils however is their high viscosity index. Naphthenic structures are also called cycloalkanes. They have excellent low temperature properties and better solubility than N-alkanes. The ring structure can have five, six or seven carbons (six being the predominant number). Aromatic molecules are ring structures with alternating double bonds. They are totally different from paraffinic and naphthenic molecules, chemically and physically. Aromates can be present both as monoaromates and polyaromates. Polyaromates have several aromatic rings directly adjacent to each other. These basic hydrocarbon structures in mineral oils are shown below.



**Figure 1: Hydrocarbon structures in mineral oil (Schlosberg *et al*, 2001)**

A typical “oil molecule” containing paraffinic, naphthenic and aromatic structures is shown in the figure below



**Figure 2: A typical oil molecule (Schlosberg *et al*, 2001)**

Atoms in the oil molecule which are not carbons (or hydrogen) are called heteroatoms. These atoms consist of nitrogen, sulfur and oxygen. The physical composition of mineral base oil is shown below.

**Table 1: Composition of Mineral Base Oil (Jilner, 1997)**

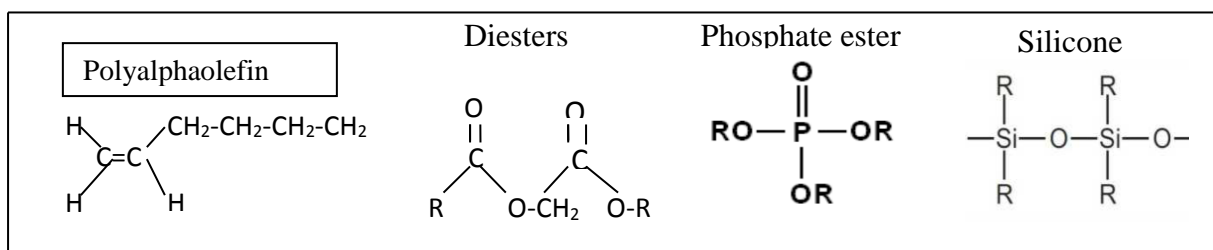
| Characteristics           | 100N             | 150N             | 250N             | 500N             | BRIGHT STOCK     |
|---------------------------|------------------|------------------|------------------|------------------|------------------|
| Kinematic Viscosity (cst) | 3.9-4.3          | 4.9-5.5          | 6.9-7.7          | 8-9              | 29-34            |
| V.I                       | 95               | 95               | 95               | 95               | 95               |
| Flash point (°C)          | 193              | 204              | 210              | 221              | 226              |
| Pour point (°C)           | -9               | -9               | -9               | -9               | -9               |
| Appearance                | Clear and bright | Clear and bright | Clear and bright | Clear and bright | Clear and bright |

### 2.3.2.2 Groups IV and V

Group IV is dedicated to a single type of synthetic called Polyalphaolefin (PAO). It is the most widely used synthetic base oil. PAOs are synthetically generated hydrocarbons with an olefinic tail formed through a polymerization process involving ethylene gas (Hackette, 1999). The result is a structure that looks very much like the purest form of the mineral oil described in Group III. The advantage of PAOs over mineral oil include a higher viscosity index, excellent low- and high-

temperature performance, superior oxidation stability, lubricity, seal shrinkage and film strength (Fitch, 2012). Much like Mineral oils, PAOs are widely employed for lubricating applications and are often the preferred option when higher temperatures are expected.

Group V is assigned to all other base oils not included in Groups I-IV and are particularly synthetic. Some of the most common in this group include Diesters, Polyolesters, Polyalkylene glycols, Phosphate esters and Silicones (Schlosberg *et al*, 2001). Some of the structures are shown below.



**Figure 3: Hydrocarbons in Synthetic Oils**

**Table 2: General Classification of Base Oil (O’conor and Boyd, 1968)**

| GROUPS    | %<br>SATURATES                         | SULFUR<br>CONTENT | VISCOSITY<br>INDEX |
|-----------|--|-------------------|--------------------|
| GROUP I   | <90%                                   | >0.03%            | 80-120             |
| GROUP II  | <99%                                   | <0.03%            | 80-120             |
| GROUP III | <99%                                   | <0.03%            | >120               |
| GROUP IV  | All Polyalpha Olefins (PAOs)           |                   |                    |
| GROUP V   | All stocks not included in Groups I-IV |                   |                    |

## **2.4 Production of Lubricating oils**

Lubricating oil is refined from crude oil. After undergoing a purifying process called Sedimentation, the crude is heated in huge fractionating towers. Within the towers, the thousands of hydrocarbons in crude are separated from each other by a process called fractional distillation. As the vapors rise up through the towers, the various fractions cool, condense, and return to liquid form at different rates determined by their respective boiling points (the lower the boiling point of the fraction, the higher it rises before condensing). Natural gas reaches its boiling point first, followed by gasoline, kerosene, fuel oil, lubricants and tars. After further processing to remove unwanted compounds, the lube oil that has been collected in the fractionating tower is passed through several ultrafine filters, which removes the remaining impurities. The filtered oil is finally mixed with additives to give the desired physical properties (such as ability to withstand temperature) (Saheed, 2012). At this point, the lubricating oil is subjected to variety of quality control test that assess its viscosity, specific gravity, color, flash and fire points. Oil that meets the quality standards is then packaged for sale and distribution (Olayinka, 2006).

## **2.5 Lubricant Additives**

The quality of lubricating oils or lubricants is attained not only through purification and manufacturing processes but also through the addition of certain chemical

compounds or additive agents. Additives are put into lubricating oils for a variety of purposes and do a great deal to improve the lubricating oils which nature and the refiners produce. Without proved chemical additives incorporated in the lubricants, the present high mechanical output and long service life of machines would be impossible (Robert and Perry, 1992).

Lubricant additives are proved chemicals or materials which when incorporated in base lubricating fluids, supplement their natural characteristics and improve their field service performance in existing applications or broaden the area of their utility. They are also referred to as addition agents or improvers (Nehal *et al*, 2011). Modern lubricating oils can be classified under three headings:

- I. Those designed to protect the finished product in service in one way or the other by limiting chemical change or deterioration.
- II. Those that protect the machine either from harmful fuel combustion products deposited in the lubricants or from failure of the fuel and/or lubricant to function properly.
- III. Those which improve existing physical properties or impart new characteristics to it.

### 2.5.1 General Properties of Additives

In addition to specific service requirement, additives must possess certain general properties to allow them to be effectively incorporated in petroleum or synthetic fluids (Robert and Perry, 1992). Among these are:

- **Solubility:** Solubility of most additive must be maintained over the operating temperature range. If this is lacking, complicated blending procedures are required to prepare the finished products and there is also a possibility of additive decomposition if the blending necessitates the use of elevated temperature. Separation in use or storage is of course highly undesirable.
- **Insolubility and lack of reaction in aqueous solution:** Motor oils and gear lubricants should dissolve in oil rather than water. Water diluents in the crank case or gear case tend to leach out water soluble additives. Certain greases require their soap and additive component to be water resistant, lest water contact soften the soap and dispense the additives.
- **Color:** The additive color is important. Clear, highly refined base oils are attractive in appearance, but when adversely colored owing to additives are likely to give unfavorable impression.



- **Volatility:** The additive volatility must be low, otherwise when the blended oil is exposed to high operating temperature, the additive fraction vaporizes and the concentration and effectiveness decreases.
- **Stability:** An additive must remain stable in blending storage and in use. It should resist hydrolysis in aqueous solution and decomposition at elevated temperature.
- **Compatibility:** This is one of the most important characteristics of any additive. Two or more additives in an oil blend are compatible if there is no evidence of reacting together, such as bringing color change to the finished product or causing throw down of insoluble substances. In addition, two additives are compatible only when their individual properties are both beneficial. The additives must be suited to each other and cannot be selected at random. Compatibility may also refer to two or more finished lubricants when mixed together are jointly successful.
- **Odor:** Undesirable odors imparted by some additives to the finished lubricant or resulting from either normal oxidation or decomposition should be corrected or masked with suitable highly odoriferous chemical compounds or synthetic perfumes.

- **Controlled activity:** The additive must be effective in the operating temperature range of the equipment which is being lubricated or protected.

### **2.5.2 Tests for Additive Effectiveness**

Chemical additives are relatively high cost materials and must be used with extreme accuracy, both to avoid loss and to ensure that the accurate proportion is incorporated in the lubricant. The percentage of additive recommended is determined through “machinery-and engine-performance test” as well as in the laboratory. Close tolerance must be set in the formulation of the finished product to maintain minimum predetermined performance requirements. Ultimately, a laboratory test must be made of blends of lubricants and other products containing additives. The quality control problem does not end when the laboratory has approved the blend. In passage from blending tanks to service tank and to point of use, contaminants from other blends may be introduced. To avoid this, all lines should be cleaned of previous unlike blends and samples at least at the start and finish of storage operations. The capabilities of the additive type lubricant must always exceed the demand of the machine by a safe margin (Robert and Perry, 1992).

Additive effectiveness in providing definite service performance characteristics depends on the chemical composition of oil and additives, concentration of additives, design, service and environment of equipment to be lubricated. Among the desirable factors, additive type lubricant must render in any application are minimum coefficient of friction, maximum adhesion to lubricated surfaces, maximum film strength, physical stability at operating temperature and pressure, chemical stability against oxidation and thermal decomposition, freedom from corrosive acids and rusting, resistance to emulsion and foaming, non-volatility, proper fluidity at lowest temperature, minimum consistency, etc (Robert and Perry, 1992).

### **2.5.3 Types of Additives**

Additives can be divided into two general classes

- I. Those that affect some physical characteristics of the lubricating oil such as pour point etc.
- II. Those whose end effect is chemical in nature; which effect usually is measurable through some service performance characteristics such as detergency.

**Table 3: Additives that Affect Physical and Chemical Properties (Robert and Perry, 1992).**

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| <b>Additives that affect chemical properties</b> | <b>Additives that affect physical properties</b> |
|--|--|
| 1. Anti-oxidant                                  | 1. Pour point depressant                         |
| 2. Anti corrosion                                | 2. Viscosity index improver                      |
| 3. Anti-wear                                     | 3. Antifoam                                      |
| 4. Detergent dispersant                          | 4. Emulsifier                                    |
| 5. Alkaline agent                                | 5. Solid filler                                  |
| 6. Anti-rust etc.                                | 6. Color stabilizer                              |

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Each of these two classes of additives may be blended into a multipurpose additive for ease in compounding the finished product.

### **1. Oxidation and Corrosion Inhibitors**

Additives called anti-oxidants decreases oil oxidation with the secondary effect of reducing corrosion of certain types of sensitive bearing materials. If production is allowed to precede unchecked, oxidation will increase lubricant viscosity to the point where oil circulation is seriously impaired (O’conor and Boyd, 1968). The chemical change results in the formation of peroxide and organic acids within the

lubricating oil. The rate of oxidation of lubricating oils depends not only on the chemical composition or the stability of the oil but on the temperature and length of time in operation. The amount of exposure to air or oxygen, design and state of equipment, and operating condition affect the oxidation rate. Many metals, copper especially accelerates the oxidation process, water and foreign particles suspended in the oil do likewise (Robert and Perry, 1992).

Corrosion applies also to the decomposition of nonferrous metals. The preservation of such metals is primarily a matter of resistance to attack from acids formed in unstable oils rather than from water. One type of corrosion inhibitor will protect bearing surface by minimizing formation of organic acids caused by lubricant oxidation. An oxidation inhibitor also minimizes organic acid development and thus acts as a corrosion inhibitor. In addition, the corrosion inhibitor may act to coat certain surfaces with a protective film impervious to acid attack or to neutralize corrosive acids as they appear in the lubricating oil (Robert and Perry, 1992).

Anti-oxidants bearing corrosion inhibitors vary in chemical structure but generally contain sulfur and/or phosphorous compounds. The more common types include zinc dithiophosphate,  $P_2S_5$  (olefin reaction product),  $P_2S_3$  (terpene reaction

product), and sulfurized olefins. It is important to note that excess of sulfur promotes sludging (Robert and Perry, 1992).

## **2. Anti-Wear Improvers**

The most commonly used anti-wear improver is zinc dithiophosphate, others includes tricresyl phosphate, lead naphthenate and fatty oils. These additive compounds acts either as corrosive to copper, lead or act as pro oxidant (Robert and Perry, 1992).

## **3. Rust Inhibitors**

Rust inhibitors are polar in nature and are metallic soaps, esters, ethers, and derivatives of dibasic acids. Barium and calcium sulfonates used as detergent also impact rust inhibiting characteristics (Robert and Perry, 1992).

## **4. Pour Point Depressant**

Lubricating oils containing straight chain hydrocarbons tend to form waxy crystals at moderately low temperature. Other oil components such as branched chain paraffin, naphthenes, and aromatics show less tendency to crystallize. Such wax crystals formed in lubricating oils at winter temperature assume a lattice like structure which traps the liquid like oil and keeps it from pouring or flowing. The temperature at which this thickening takes place is the pour point, a factor to be considered if the oil is to be used in automotive crankcase, gear cases and bearings

under winter conditions or in refrigerating-ice machine lubrication. Low pour point may be achieved by intensively de-waxing the oil during refining (Robert and Perry, 1992).

Pour point depressants are thought to act by retarding the formation of wax crystals or by changing the size and shape of this crystal structures, thus do not interfere with the ability of the oil to flow. Pour point depressants are polymeric materials usually made from specially prepared hydrocarbon materials much like the oil itself. When added to lubricating oils, they lower the pour point without affecting any of the other properties or performance characteristics of the oil (O'conor and Boyd, 1968). Three types are commonly used:

- I. Wax alkylated naphthalene (condensation product of chlorinated paraffin wax and naphthalene) and its polymers.
- II. Wax alkylated phenol and its polymers.
- III. Polymers of methacrylic acid esters (also increases viscosity index)

## **5. Viscosity Index Improvers**

The major consideration for selecting oil for a particular application is viscosity and its variation with temperature. The viscosity temperature relationship in oils is designated by the viscosity index scale.

Viscosity index improvers have been used by the petroleum industry not only when the nature of the oil makes it impractical to obtain a product with the desired high viscosity index by solvent refining. The higher the viscosity index and the lower the pour point, the wider the range over which the lubricant will function effectively. Without viscosity improvers, the multi grade SAE 5W-20 or SAE 10W-30 could not be practically blended to cover viscosity range provided by SAE 30 at 210F and SAE 10W at 0F, likewise the SAE 5W-20 motor oils (O'conor and Boyd, 1968). Two principal types of Viscosity Index Improvers are marketed:

- I. Isobutylene polymers
- II. Acrylate copolymers

The Acrylate copolymer may be 100% Lauryl methacrylate or a copolymer of Lauryl and Butyl methacrylate (Robert and Perry, 1992).

## **6. Color Stabilizers and Dyes**

Greases and lubricating oils sometimes use color stabilizers to control uniformity of color or to prevent the product from turning dark when subjected to light or heat and oxidation. Certain complex amines, such as aliphatic amines and some dicyclohexylamines, stabilize lubricants against color deterioration due to light and heat in storage or in operation. In some applications, it is found desirable to have a



distinctive color for a lubricant, either for identification purposes, for application or for display value.

Certain chemical dyes are soluble in oil and have high coloring power. These dyes are the same type used in gasoline but are added in considerable greater quantity. They are usually chemical compounds of alkylated aniline dyes or more common azo- and anthroquinone dyes. Fluorescent dyes which darken oils and give them a very pleasing cast is added in small amounts sometimes as little as 0.25% (Robert and Perry, 1992).

## **8. Odor Control Additives**

When it is desirable to provide lubricants with a distinctive odor, synthetic perfume or highly odoriferous chemicals are used. They may be used to provide a distinctive desirable odor such as pine or cedar oil, or to mask an undesirable odor impacted by some other additives. This odor occurs either originally or after the product reacts at high temperature in use. For this purpose, formaldehyde polymers have been used (Robert and Perry, 1992).

### **2.6 Properties of Engine Oil**

Motor oils are made from a heavier, thicker petroleum hydrocarbon based stock derived from crude oil, with additives to improve certain properties (O'conor and Boyd, 1968). Therefore, the properties of the engine oils must be checked to ensure

optimum performance because they are produced from different base stocks. Some of these properties are;

**1. Viscosity:** This is one of the most important properties of motor oil in maintaining a lubricating film between moving parts. The Viscosity of a liquid can be thought of as its thickness or a measure of its resistance to flow at a specific temperature in relation to time. The viscosity must be high enough to maintain a lubricating film, but low enough that the oil can flow around the engine parts under all conditions (Okoye, 2009). The two most important temperatures for engine oil viscosity are 40<sup>0</sup>C and 100<sup>0</sup>C.

**2. Viscosity Index:** The viscosity index is a measure of how much the oils viscosity changes as temperature changes (change in flow rate with respect to temperature). A higher viscosity index indicates that the viscosity changes less with temperature change than a lower viscosity index (Okoye, 2009). Thus, high Viscosity Index is recommended in engines and systems that operate at elevated temperatures.

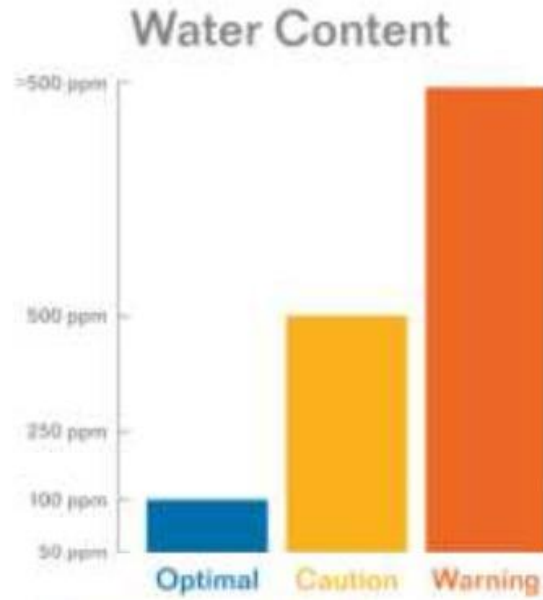
**3. Pour Point:** This is the property of oil in which it must be able to flow adequately at the lowest temperature it is expected to experience in order to minimize metal to metal contact between moving parts upon starting up the engine (Archbutt, 1998).

**4. Flash Point:** Oil is largely composed of hydrocarbons which can burn if ignited. Thus, another important property of motor oil is its flash point, which is the temperature at which the oil gives off vapors which can ignite. It is dangerous for the oil in a motor to ignite and burn, so a high flash point is desirable (Archbutt, 1998).

**5. Total Base Number:** This is a measure of the reverse alkalinity of oil, meaning its ability to neutralize acids. The resulting quantity is determined as mg KOH/gram of lubricant. Analogously, Total Acid Number is a measure of a lubricant's acidity which is the quantity of acid or acid-like components in the lubricant (Archbutt, 1998). Mostly, the Total Base Number is preferred to be analyzed in oil sample but Total Acid Number should also be checked as it is not necessarily zero for new oils since additives can be acidic in nature.

**6: Moisture Content:** This is the amount of water in form of moisture present in engine oil. Water can enter the oil either through evaporation and condensation processes, through oxidation or absorption from moist atmosphere. Though water is typically evaporated by engines at normal operating temperatures, water may remain in the oil when engine temperatures are too low for evaporation to occur. High moisture in oil leads to corrosion, acid formation, high viscosity and engine

overheating which consequently result to engine failure. Thus, the minimum amount of water required in oil is <100ppm as shown in figure 4 below:



**Figure4: Minimum Amount of Water Required in Engine Oil (Iselstaff, <http://www.Iselinc.com/technology/test-oil-viscosity-tan-water-content-metals/>)**

## 2.7 Engine Oil Grades

The Society of Automobile Engineers (SAE) established a numerical code system for grading motor oils according to their viscosity characteristics. SAE viscosity grade numbers refer to two types of measurements-one set which measures cold temperature performance (0W, 5W, 10W, 15W and 20W) (Caines and Haycock, 1996). The W denotes a winter grade from the days when the oils were mono grades only. The second set of measurement is for high temperature performance

(8, 12, 16, 20, 30, 40 and 50). The kinematic viscosity is graded by measuring the time it takes for a standard amount of oil to flow through a standard orifice, at standard temperature. The longer it takes, the higher the viscosity and thus bigger SAE code. Therefore, the engine oil is graded into two, Single grades and Multi grades (Caines and Haycock, 1996).

### **2.7.1 Single grade engine oil**

Single-grade engine oil as defined by SAE J300 cannot use a polymeric viscosity index improver additive. SAE J300 has established eleven viscosity grades, of which six are considered winter grades and given 11 designations. The 11 viscosity grades are 0W, 5W, 10W, 15W, 20W, 25W, 20, 30, 40, 50 and 60. These numbers are often referred to as the ‘weight’ of motor oil and single grade motor oils are often called ‘straight-weight’ oils (O’conor and Boyd, 1968).

For single- grade winter oils, the dynamic viscosity is measured at different cold temperatures, specified in J300 depending on the viscosity grade, in units of mPa.s, or centipoise, using two different test methods which are the Cold Cracking Simulator (ASTMD5293) and the Mini-Rotary Viscometer (ASTMD4684). Based on the coldest temperature the oil passes, that oil is graded as SAE viscosity grade 0W, 5W, 10W, 15W, 20W, or 25W. The lower the viscosity grade, the lower the temperature the oil can pass. For example, if an oil passes at the specifications for

10W and 15W, but fails for 0W, that oil must be labeled as SAE 5W. It cannot be labeled as either 0W or 10W (Caines and Haycock, 1996).

For single non-winter grade oils, the kinematic viscosity is measured at a temperature of 100°C (212°F) in units of mm<sup>2</sup>/s (millimeter square per second) or the equivalent of centistokes (cst). Based on the range of viscosity the oil falls in that temperature, the oil is graded as SAE viscosity grade 20, 30, 40, 50, or 60. The higher the viscosity, the higher the SAE viscosity grade (Caines and Haycock, 1996).

### **2.7.2 Multi Grade Engine Oil**

The temperature range the oil is exposed to in most vehicle can be wide, ranging from cold temperature in the winter, before the oil is started up to hot operating temperatures when the vehicle is fully warmed up in hot summer weather (O'conor and Boyd, 1968). Specific oil will have high viscosity when cold and a lower viscosity at the engines operating temperature. The difference in viscosities for most single-grade oil is too large between the extremes of temperature. To bring the difference in viscosity closer, special polymer additives called viscosity index improvers are added to the oil. These additives are used to make the oil a multi-grade motor oil, though it is possible to have a multi-grade motor oil without the

use of Viscosity index improvers. The idea is to cause the multi-grade oil to have a viscosity of the base grade when cold and the viscosity of the second grade when hot. This enables one type of oil to be used all year.

The SAE designation for multi grade oils includes two viscosity grades; for example, 10W-30 designates common multi-grade oil. The first number '10W' is the viscosity of the oil at cold temperature, and the second number is the viscosity of the oil at 100°C (212°F). The two numbers used are individually defined by SAE J300 for single-grade oils. Therefore, oil labeled as 10W-30 must pass the SAE J300 viscosity grade requirement for both 10W and 30. Other multi-grade oils include 20W-20, 15W-40, 20W-50, etc. (O'conor and Boyd, 1968).

## **2.8 Quality of Engine Oil**

Lubricant marketers may claim superior quality of their Engine Oil but sometimes with no factual evidence. The quality of Engine Oil is proven by reference to famous brands, sporting figures or some professional endorsement. All motor oil labels have mark of outstanding quality or quality additives. In most applications, lubricants are required to have optimum Viscosity required for circulation, high Viscosity Index, Odorless, and Oxidation resistant (Caines and Haycock, 1996). Thus, The American Petroleum Institute (API) service classes have two general

classifications: S for ‘Service/Spark Ignition’ (typical passenger cars and light trucks using gasoline engines) and C for ‘Commercial/Compression Ignition’ (typical diesel engine). Therefore, engine oils which has been tested and met standards may display the API service symbol (also known as the ‘Donut’) with the service designation on containers sold to consumers (Caines and Haycock, 1996).

## **2.9 Uses of Engine Oil**

Engine oil is a lubricant used in internal combustion engines, which powers cars, motorcycles, lawn mowers, engine generators and many other machines. In engines, there are parts which move against each other, and the friction wastes otherwise useful power by converting the kinetic energy to heat. It also wears away those parts which could lead to lower efficiency and degradation of the engine. This increases fuel consumption, decreases power output and can lead to engine failure. Thus, some of the uses of engine oil include;

- Reducing wear by creating a separating film between surfaces of adjacent moving parts to minimize direct contact between them, thus decreasing heat caused by friction.



- Inhibiting oxidation at elevated operating temperature, preventing rust or corrosion by coating the metal parts which keeps them from being exposed to oxygen.
- They also serve as cooling agents when sprayed through a nozzle inside the crankcase onto the piston, cooling specific parts that undergo high temperature strain.

## **2.10 Environmental Effects of Engine oil**

Due to the chemical composition of engine oils, its worldwide dispersion and effects is considered a serious environmental problem. Most current motor oil lubricant contains petroleum base stocks which are toxic to the environment and difficult to dispose after use. One gallon of used oil can create an eight-acre slick on surface water, threatening fish, waterfowls and other aquatic life because films of oil on the surface of water prevents the replenishment of dissolved oxygen, impair photosynthetic processes and block sunlight. Toxic effects of used oil on freshwater and marine organisms vary, but significant long term effects have been found at concentrations of 310ppm in several fresh water fish species and as low as 1ppm in marine life forms (Audibert, 2006).

Motor oil also has an incredibly detrimental effect on the environment, particularly to plants that depend on healthy soil to grow. There are three main ways that motor oil affects plants: contaminating water supplies, contaminating soil and poisoning plants. Used motor oil dumped on land reduces soil productivity while those improperly disposed end up in landfills, sewers, or storm drains where soil, groundwater and drinking water may be contaminated (Audibert, 2006).

## **CHAPTER THREE**

### **MATERIALS AND METHODS**

#### **3.1 Lists of Apparatus**

The apparatus used for the various analyses include; 0.85 range hydrometer, 500ml measuring cylinder, Thermometer, Cleveland Open cup flash point tester, Cannon-Fenske Viscometer, pipette filler, Syringe, Analytical weighing balance, 20ml pipette, 50ml Burette, 250ml Erlenmeyer flask, 500ml Volumetric Flask, Test tube, Pour Point machine, Karl Fischer equipment.

#### **3.2 List of Reagents**

The reagents used include; Titration solvent for moisture content, Karl Fischer reagent, 0.0874N Ethanolic Potassium hydroxide, Titration solvent for Total Acid Number (TAN).

#### **3.3 Preparation of Reagents**

The reagents prepared were Titration solvent for moisture content, Ethanolic potassium hydroxide and the titration solvent for TAN. The Karl Fischer reagent is a stock solution.

##### **3.3.1 Moisture Content Titration Solvent**

The moisture content titration solvent was prepared by mixing Methanol and Chloroform in the ratio of 3:1.

### 3.3.2 TAN Titration Solvent

This was prepared by mixing 500ml of Toluene, 495ml of 2-propanol and 5ml of distilled water.

### 3.3.3 Preparation of 0.0874N Ethanolic Potassium Hydroxide (KOH)

2.45g of Potassium Hydroxide (KOH) was weighed out and dissolved in 500ml of ethanol.

### 3.3.4 The Karl Fisher Reagent

The Karl Fisher reagent is an already prepared stock solution containing Iodine, SO<sub>2</sub> and a base.

## 3.4 Collection of Sample

The samples of locally blended engine oil were selected from eight (8) different areas within Awka metropolis, Anambra State of Nigeria and two commercially made engine oils one mono grade and the other multi grade were also selected. The eight areas were Ifite, Aroma, Okpuno, Unizik Junction, Kwata, Amawbia and Amaenyi while the commercially made ones were Total and Mobil coded A, B, C, D, E, F, G, H, R1 and R2 respectively.



(a)



(b)

**Figure 5: Pictures Showing (a) Locally Blended Oils in one of the Areas during Collection and (b) the Two Commercially Made Engine Oil Samples in the Laboratory**

### **3.5 Experimental Procedure**

The physicochemical properties of the engine oil examined in this study and their test methods in line with American Society for Testing and Material (ASTM) include Kinematic Viscosity (ASTM D445), Flash point (ASTM D87), Total Acid Number (ASTM D974), Specific Gravity (ASTM D1298), Moisture Content (ASTM D6304) and Pour Point (ASTM D7346). Among others, the Kinematic Viscosity is the most important analysis carried out on engine oil. Analysis of Variance (ANOVA) using SPSS Computer package version 20 was also used to statistically analyze the results.

#### **3.5.1 Determination of Kinematic Viscosity (ASTM D445)**

The viscosity water bath was first filled with water, heated to the desired temperature (40°C or 100°C), and maintained at that temperature. The viscometer was filled to the mark with the sample, corked, inserted into the water bath and left for about an hour and 30 minutes to attain thermal equilibrium with the temperature of the water in the bath. When this was achieved, the sample was drawn up to the orifice of the viscometer using pipette filler and the time with which it flows down recorded. This experiment was repeated twice and the average result recorded. This result was then multiplied with the constant of the

viscometers depending on the series and the final results recoded in Centistoke (cst). The results were then used to calculate the Viscosity Index.

Kinematic viscosity (cst) = time of flow (sec) × Viscometer constant

### **3.5.2 Determination of Specific Gravity (ASTM D1298)**

A 500ml measuring cylinder was rinsed with the sample to avoid contamination by the previous sample analyzed. The sample was poured into the measuring cylinder and a hydrometer of range 0.85-0.95 gently dropped into the sample allowing it to become stable. The density reading was then taken from the point where the sample cuts the hydrometer and the hydrometer removed. The sample was then stirred with a thermometer immediately after removing the hydrometer and the temperature of the sample taken. An international reference standard table was used to correct the density at the observed temperature to Specific Gravity@ 15°C.

### **3.5.3 Determination of Flash Point (ASTM D87)**

The sample cup of the flash point tester was cleaned and dried. The sample was made homogenous by shaking and then filled to the mark of the sample cup. A thermometer was inserted into the cup, the heater turned on, and the test flame lighted and adjusted. The sample was monitored at intervals of 3°C rise in

temperature from 28°C- 30°C below the expected flash point and the observed flash point recorded in Celsius.

#### **3.5.4 Determination of Moisture Content (ASTM D6304)**

The Karl Fischer equipment used for the titration was switched on and 30ml of the titration solvent poured into the titration flask using a pipette. The equipment was then conditioned severally to remove any available moisture which may interfere with the result. A syringe was weighed, used to collect a sample of the engine oil, and both the syringe with the sample weighed. The sample collected was transferred into the titration flask and titrated against the Karl Fischer reagent. The volume of the Karl Fischer reagent used for the titration was recorded as displayed on the screen and the moisture content in ppm calculated.

$$\text{Moisture (ppm)} = \frac{\text{vol. of Karl Fischer reagent} \times 3.5714 \times 1000}{\text{Weight of sample}}$$

$$\text{Normality of Karl Fischer reagent} = 3.5714$$

#### **3.5.5 Determination of Pour Point**

The sample tube was thoroughly rinsed with the sample and filled to the mark with the sample. A thermometer was inserted into the tube and corked. The temperature of the sample was allowed to drop to 27°C before being inserted into the pour point

machine. The sample was monitored at every 3°C drop in temperature until the sample ceases to flow. The observed temperature was recorded in Celsius.

### **3.5.6 Determination of Total Acid Number (TAN)**

20g of the engine oil was weighed in a 250ml Erlenmeyer flask. 100ml of the titration solvent and 0.5ml p-naphtholbenzene indicator was also added to the weighed engine oil. On the addition of p-naphtholbenzene, the solution developed a yellow-orange color. The solution was then swirled to dissolve the sample and titrated immediately at room temperature with standardized acidic potassium hydroxide (KOH). Titration continued till a green-brown color developed and recorded as the end point if the color persists for 15seconds and the TAN calculated.

$$\text{TAN (mg KOH/g sample)} = \frac{(A-B) \times N \times 56.1}{W}$$

Where: A = Titre value

B = Blank value

N = Normality of KOH = 0.0874

W = Weight of sample



## CHAPTER FOUR

### RESULTS AND DISCUSSION

#### 4.1 Presentation of Results

The results of the physicochemical analysis of the oil samples are shown in the tables below. The recorded results are the average of 2-3 tests.

##### 4.1.1 Kinematic Viscosity results and Discussion

**Table 4: Kinematic Viscosity @ 40°C and 100°C Results**

| SAMPL<br>E | K. V @ 40°C    |                       | K. V @ 100°C   |                       |
|------------|----------------|-----------------------|----------------|-----------------------|
|            | MEA<br>N (cst) | STD.<br>DEVIATIO<br>N | MEA<br>N (cst) | STD.<br>DEVIATIO<br>N |
| A          | 157.10         | 0.0014                | 15.22          | 0.0141                |
| B          | 158.34         | 0.0021                | 15.53          | 0.0070                |
| C          | 155.70         | 0.0035                | 15.26          | 0.0212                |
| D          | 157.10         | 0.0042                | 18.12          | 0.0212                |
| E          | 159.11         | 0.0014                | 18.41          | 0.0141                |
| F          | 157.31         | 0.0028                | 17.77          | 0.0070                |
| G          | 143.21         | 0.0007                | 13.30          | 0.0282                |
| H          | 153.33         | 0.0049                | 15.21          | 0.0212                |
| R1         | 154.54         | 0.0028                | 15.26          | 0.0282                |
| R2         | 155.40         | 0.0014                | 18.42          | 0.0141                |

From the results of the Kinematic Viscosity @ 40°C and @ 100°C shown in table 4, it was observed that there was a significant reduction in the viscosity of the oil @ 100°C. This is because as the temperature increases, the time of interaction

between neighboring molecules decreases as a result of the increased average kinetic energy of the molecules, consequently leading to decrease in Viscosity.

The results of the Kinematic Viscosity @ 100°C showed that samples A, B, C and H, were within the range of single grade engine oil which is 9.30-16.30, while samples D, E and F were within the range of multi grade engine oil of 16.30-21.90.

In view of this, samples A, B, C and H, will protect the engine at one temperature, either hot or cold, while samples D, E and F, will protect the engine at more than one temperature, both hot and cold. The Kinematic viscosities of the commercial samples R1 and R2 which are single-grade and multi-grade respectively show that they are well graded. Sample G with viscosity of 13.30 fell below the standard.

This may be as a result of oxidation reaction taking place in the oil or due to high water or moisture content. Thus, this oil when used in motor engines will not lubricate the engine effectively due to low viscosity. From the ANOVA analysis, the probability value for kinematic viscosity @ 40°C which is 0.00 shows that there is significant statistical difference between the kinematic viscosities of the sample while at 100°C, the probability value is 0.388 indicating no significant statistical difference.

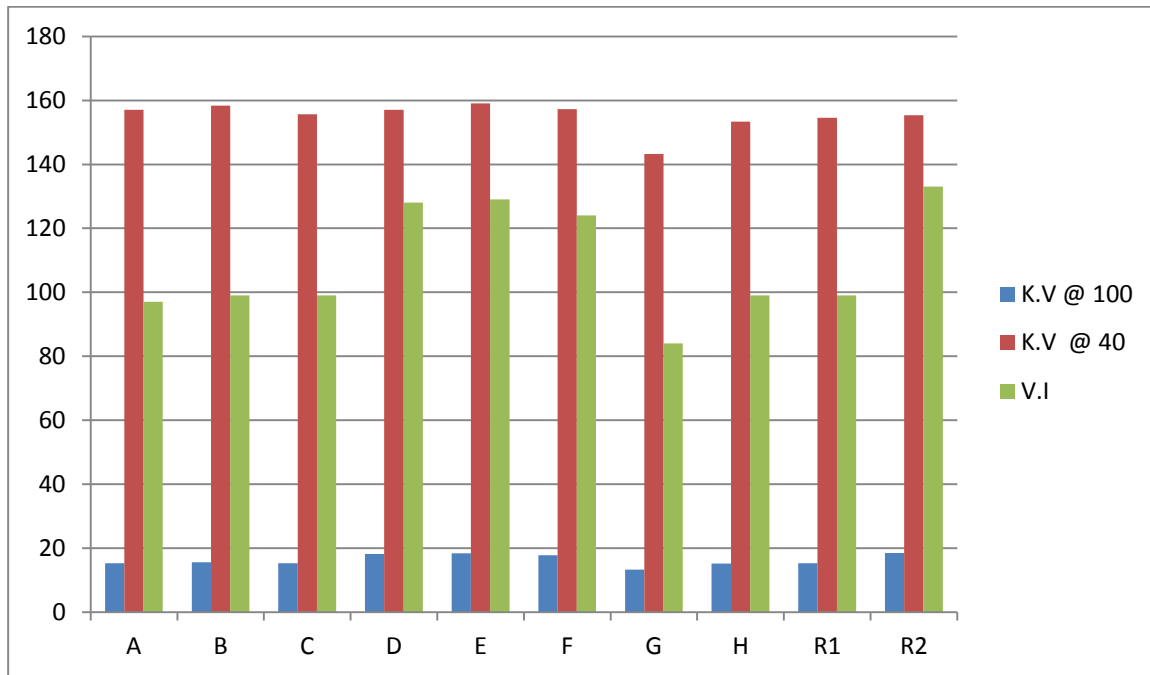
#### 4.1.2 Viscosity Index Results and Discussion

**Table 5: Viscosity Index Results**

| SAMPLE | VISCOSITY INDEX |                   |
|--------|-----------------|-------------------|
|        | MEAN            | STD.<br>DEVIATION |
| A      | 97              | 0.0141            |
| B      | 99              | 0.0212            |
| C      | 99              | 0.0283            |
| D      | 128             | 0.0707            |
| E      | 129             | 0.1414            |
| F      | 124             | 0.0707            |
| G      | 84              | 0.2828            |
| H      | 99              | 0.0070            |
| R1     | 99              | 0.0212            |
| R2     | 133             | 0.2828            |

From table 5 above, the result of Viscosity Index shows that samples A, B, C and H met the API's requirement of 95 (min) for single grade oils while samples D, E and F fell within the range of minimum of 120 for multi grade. The results of samples R1 and R2 show that they are well graded. Thus, these samples A, B, C, D, E, F, R1 and R2 will resist thinning at high temperatures and thickening at low temperatures while sample G with Viscosity Index of 84 which does not meet the requirements for both single grade and multi grade will not resist thinning at high temperatures and thickening at low temperatures due to its low viscosity Index which may be as a result of inadequate addition of viscosity index improvers.

#### 4.1.2.1 Comparison between Viscosities @ 40°C and 100°C with Viscosity Index



**Figure 6: Chart Showing the Different Samples and Their Variation In Viscosities And Viscosity Index**

From figure 5, it was observed that the sample with the lowest kinematic viscosity @ 100°C has the lowest viscosity index while the sample with the highest kinematic viscosity @ 100°C has the highest viscosity index. It is also shown that the kinematic viscosity @ 40°C does not follow this trend. Thus, based on this results, it can be said that the Viscosity index depends mainly on the value of the kinematic viscosity @ 100°C which is the engines operating temperature and

increases as the kinematic viscosity of the engine oil @ 100°C increases and vice versa.

### 4.1.3 Specific Gravity Results and Discussion

**Table 6: Specific Gravity Results**

| SAMPLES | SPECIFIC GRAVITY @ 15°C |                |
|---------|-------------------------|----------------|
|         | MEAN                    | STD. DEVIATION |
| A       | 0.8865                  | 0.0014         |
| B       | 0.8875                  | 0.0007         |
| C       | 0.8971                  | 0.0021         |
| D       | 0.8960                  | 0.0007         |
| E       | 0.8998                  | 0.0028         |
| F       | 0.8960                  | 0.0007         |
| G       | 0.8821                  | 0.0014         |
| H       | 0.8755                  | 0.0021         |
| R1      | 0.8913                  | 0.0007         |
| R2      | 0.8961                  | 0.0042         |

From the API's requirements for specific gravity of engine oil which are minimum of 0.870, 0.880 and 0.890 for SAE30, SAE 40 and SAE 50 respectively which are single grades and minimum of 0.880 and 0.885 for SAE 15W40 and SAE 20W50 respectively which are multi grades, it was observed from table 6 that all the samples met the requirements for either single grade or multi grade engine oil and thus will have optimum pump ability and circulate better throughout the engine. The ANOVA analysis also showed that there is significant statistical difference between the samples.

#### 4.1.4 Flash Point Results and Discussion

**Table 7: Flash Point (<sup>0</sup>C) Results**

| SAMPLES | FLASHPOINT ( <sup>0</sup> C) |                |
|---------|------------------------------|----------------|
|         | MEAN                         | STD. DEVIATION |
| A       | 197                          | 0.7071         |
| B       | 108                          | 0.7071         |
| C       | 125                          | 0.7071         |
| D       | 191                          | 0.7071         |
| E       | 200                          | 0.7071         |
| F       | 202                          | 0.7071         |
| G       | 213                          | 0.7071         |
| H       | 196                          | 0.7071         |
| R1      | 225                          | 0.7071         |
| R2      | 233                          | 0.7071         |

From the results shown in the table 7 above, it was observed that only three samples which are samples G, R1 and R2 met the API's requirements for flashpoint which is minimum of 204-220<sup>0</sup> C while samples A, D, E, F and H are close to the flash point requirement for SAE 30. Samples B and C with flash point of 108<sup>0</sup> C and 125<sup>0</sup> C does not meet the requirement and thus is not advisable in a service environment where a high temperature is expected and in an internal combustion engine where over 90<sup>0</sup>C is expected during abnormal condition of engine overheating. Under normal working condition, samples G, R1 and R2 which satisfies the flash point requirement and samples A, D, E, F and H which are close to the requirement are suitable for use in internal combustion engine.

Between the samples, there is significant statistical difference according to ANOVA analysis.

#### 4.1.5 Pour Point Results and Discussion

**Table 8: Pour Point (°C) Results**

| SAMPLES | POUR POINT (°C) |                |
|---------|-----------------|----------------|
|         | MEAN            | STD. DEVIATION |
| A       | -15             | 0.7071         |
| B       | -16             | 0.7071         |
| C       | -14             | 0.7071         |
| D       | -11             | 0.0000         |
| E       | -12             | 0.0000         |
| F       | -10             | 0.7071         |
| G       | -18             | 0.7071         |
| H       | -16             | 0.0000         |
| R1      | -11             | 0.7071         |
| R2      | -12             | 0.7071         |

The results of the pour point as shown in table 8 shows that sample G met the requirement of pour point for SAE 30 which is maximum of -18 while the remaining samples were within the range of the requirement for SAE 40 and 50 which are maximum -9 and -21 respectively. The sample R2 which was branded as multi grade oil met the requirement for single grade instead of multi grade which it was graded. This high pour point of sample R2 may be due to inadequate addition of pour point depressant or due to the presence of straight chain alkanes. Thus,

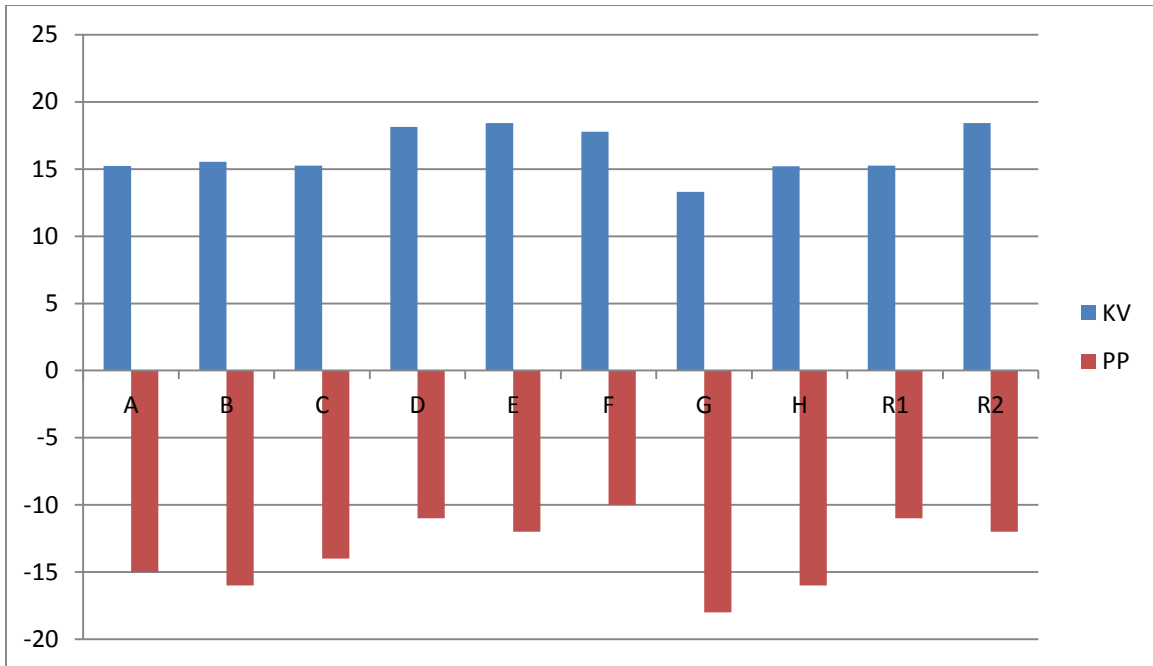
sample G with the lowest pour point of  $-18$  will show better performance in a very cold environment, followed by samples B, A and others in increasing order of pour point values with samples E and F having the highest pour point values and thus low performance in a very cold environment w. However, in Nigeria all the oil samples analyzed are suitable for use in internal combustion engine based on the Pour Point Test results and weather condition. On the basis of ANOVA analysis on the results, there was a significant statistical difference between the samples.

#### 4.1.5.1 Comparison between the Viscosity @ $100^{\circ}\text{C}$ and Pour Point

**Table 9: Results of Viscosity @  $100^{\circ}\text{C}$  and Pour Point ( $^{\circ}\text{C}$ )**

| SAMPLE | K. V @ $100^{\circ}\text{C}$ | Pour Point ( $^{\circ}\text{C}$ ) |
|--------|------------------------------|-----------------------------------|
| A      | 15.22                        | $-15$                             |
| B      | 15.53                        | $-16$                             |
| C      | 15.26                        | $-14$                             |
| D      | 18.12                        | $-11$                             |
| E      | 18.41                        | $-12$                             |
| F      | 17.77                        | $-10$                             |
| G      | 13.30                        | $-18$                             |
| H      | 15.21                        | $-16$                             |
| R1     | 15.26                        | $-11$                             |
| R2     | 18.42                        | $-12$                             |





**Figure 7: Comparison between Kinematic Viscosity @ 100°C and Pour Point (°C)**

From figure 6 above, it was observed that the sample G with the lowest kinematic viscosity had the lowest pour point. Other samples that followed this trend include samples A, B, C, D, E, H and R2 while samples F and R1 showed fluctuation in this trend. Thus, based on these results and neglecting the fluctuations in samples F and R1, the pour point of the oil samples decreased with decreasing kinematic viscosity determined at 100°C.

#### 4.1.6 Total Acid Number (TAN) Results and Discussion

**Table 10: Results of Total Acid Number (mgKOH/g)**

| SAMPLES | TOTAL ACID NUMBER<br>(mgKOH/g) |                   |
|---------|--------------------------------|-------------------|
|         | MEAN                           | STD.<br>DEVIATION |
| A       | 0.099                          | 0.0007            |
| B       | 0.147                          | 0.0000            |
| C       | 0.074                          | 0.0000            |
| D       | 0.074                          | 0.0003            |
| E       | 0.209                          | 0.0000            |
| F       | 0.098                          | 0.0000            |
| G       | 0.049                          | 0.0007            |
| H       | 0.072                          | 0.0000            |
| R1      | Nil                            | .                 |
| R2      | Nil                            | .                 |

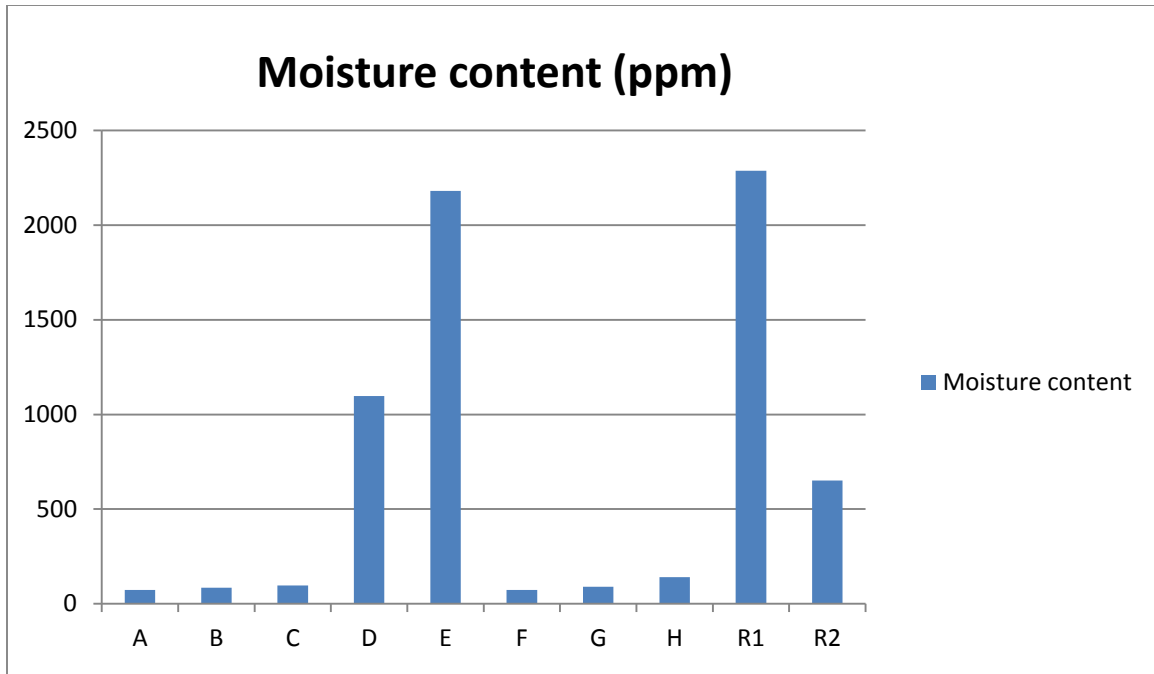
From table 10, the results of the total acid number showed that samples A-H (0.049-0.209mgKOH/g) met the requirement of 1.0mgKOH/g (max) for engine oils. Thus according to API, the acid levels in these samples are negligible. The results of Samples R1 and R2 which are commercially made oils shows that no acid was present in the oils. These samples which have little or no amount of acid will help prevent corrosion of metallic components, reduce oxidation, reduce oil thickening and additive depletion. Therefore, they are suitable for use. From the results of the Total Acid Number obtained, The Total Base Number which is the amount of base required to neutralize the acid formed in engine oil can also be

known. Thus, it is shown in the results that the engine oil contains reasonable amount of base to neutralize the acids but not completely for samples A, B, C, D, E, F, G and H while samples R1 and R2 contains the amount of base sufficient to neutralize all the acids formed in the oil. Therefore, the commercially made samples contain more base to neutralize the acid than the locally blended oils.

#### 4.1.7 Moisture Content Results and Discussion

**Table 11: Moisture Content Results**

| SAMPLES | MOISTURE (ppm) |                |
|---------|----------------|----------------|
|         | MEAN           | STD. DEVIATION |
| A       | 71.99          | 0.0014         |
| B       | 83.81          | 0.0007         |
| C       | 96.88          | 0.0021         |
| D       | 1096.94        | 0.0007         |
| E       | 2181.33        | 0.0028         |
| F       | 72.69          | 0.0007         |
| G       | 90.29          | 0.0014         |
| H       | 141.09         | 0.0021         |
| R1      | 2287.56        | 0.0007         |
| R2      | 651.62         | 0.0042         |



**Figure 8: Chart Showing the Variations in Moisture Content of the Samples**

From the results and chart obtained as shown in table 11 and figure 7, it was observed that the moisture content of samples A, B, C, F and G meets the requirement for moisture in engine oil which is maximum of 100ppm while sample H is slightly above the optimal amount and is referred as the caution level (101-500ppm). Samples D, E, R1 and R2 are above the caution level which is referred as the warning level (>500ppm). At this warning level, it is required that the oil be changed because high moisture in oil leads to corrosion, acid formation, high viscosity, poor lubrication and engine overheating which consequently results to engine failure. In view of this, samples A, B, C, E and F will preserve the engine life more effectively than sample H followed by samples D, G, R1 and R2 with

high moisture. The result of ANOVA analysis also showed that the engine oils have significant statistical differences.

## CHAPTER FIVE

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

Summarily, this study showed that only samples A and G met all the requirements for single grade, SAE 40 precisely. Other samples were found not up to the standard in one or more of the following properties: Flash Point, Pour Point and Moisture Content. These samples especially those with low flash point are not advisable for use as it is dangerous if the engine is operating at a very high temperature. The results of the commercially produced engine oil samples R1 and R2 which were already graded, showed that its moisture content falls below standard. This might be due to evaporation and condensation processes as a result of the oil being in an air tight container, the weather condition as at the period of these experiments or direct absorption of moisture from the atmosphere. Thus, having met other requirements showed that these oils were well graded with R1 AND R2 being single grade (SAE 40) and multi grade (SAE 20W50) respectively and has all requirements to lubricate effectively and prevent engine failure.

In accordance with the Society of Automobile Engineers (SAE) for grading engine oil on the basis on viscosity and based on the results obtained, the engine oil

samples can conclusively be graded as single grade (SAE 40) for samples A, B, C, G and H and multi grade (SAE 20W50) for samples D, E and F.

## **5.2 Recommendation**

In view of the above results, although some of the imperfections can be managed by motorists, it is recommended that consumers should purchase engine oils with service designation required for their engines from registered companies or fuel stations since some of these locally blended oils gotten from black markets were suspected to be simply base oils, adulterated oils or oils of inferior production to make it cheaper. It is also recommended that extensive care be taken in blending of the oils during production as some of the additives react to give an undesirable product.

## REFERENCES

- American Society for Testing and Materials, (1997), Petroleum Products, Lubricants and Fossil Fuel, Annual Book of ASTM Standards, 05(04), pp. 1544- 1552.
- Archbutt, L., (1998), Lubricants and Lubrications: A Treatise on the Theory and Practice of Lubrication and On the Nature, Properties and Testing of Lubricants, C. Griffin and Company Ltd., London, pp. 244-249.
- Audibert, F., (2006), Waste Engine Oil Refining and Energy Recovery, Elsevier Science Publishers, Amsterdam, pp. 77-79.
- Avilino, S., (1994), Lubricant Base Oil and Wax Processing, Marcel Dekker Inc., Newyork, pp. 17- 21.
- Bartz, W. J., (1992), Engine Oil and Automotive Lubrication, Taylor and Francis Inc., New York, pp. 462-469.
- Boyde, S., (2002), Green Lubricants: Environmental Benefits and Impacts of Lubrication, Green Chemistry, Pp. 4, 293-307.
- Bull, S. and Marmin, A., (1979), Lube Manufacture by Severe Hydro Treatment, World Petroleum Congress, Buchare, pp. 88-92.
- Caines, A. and Haycock, R., (1996), Automotive Lubricant Reference Book, Society of Automotive Engineers (SAE) Inc., pp. 355-370.
- Carrey, D., (1971), How It Works: The Motor Car, Lady Bird Books Ltd, London, pp.177-181.
- Chilingar, G. V., Buryakovsky, L. A. and Eremenko, N. A., (2005), Geology And Geochemistry Of Oil And Gas, 1<sup>st</sup> Edition, Elsevier Science Publishers, Amsterdam, p. 78.



- Cocks, L.V. and Van, C., (1996), Laboratory Handbook for Oil and Fat Analysis, 3<sup>rd</sup> Edition, Academic Press Publishers, London, p. 96.
- Dryoff, V. G., (1993), Manual on Significance of Tests for Petroleum Products, 2<sup>nd</sup> Edition, ASTM Publications, Philadelphia, pp. 82-91.
- Edewor and Afolabi, (2005), Formulation and Characterization of Printing Ink from Used Engine Oil. *Science Focus*, 10(3): 155-159.
- Fitch, J., (2012), Understanding the Difference in Base Oil Groups, <http://www.machinerylubrication.com/Read/29113/base-oil-groups> (Accessed 03/05/2017)
- George, J. W., (1980), Lubrication Fundamentals, CRC Press Book, Florida, pp. 544-546.
- Hackette, E.D., (1999), ‘More Than You Ever Wanted to Know about Motor Oil’, <http://micapeck.com/info/oiled>, (Accessed 23/01/2017).
- Hamid, R. T., (2012), Base Oil Classification, <http://www.otusholding.com/base-oil-classificationphp>. (Accessed 27/12/2016).
- Hawley, J.G., Banister. C. J., Akehurst, S., Pegg, I. And Avery, M.R., (2010), The Effect of Engine and Transmission Oil Viscosities on Vehicle Fuel Consumption. *Journal of Society of Automobile Engineering*, 4(6): 1213-1222.
- Hobson, G.D. And Pohl, W., (1973), Modern Petroleum Technology, 4<sup>th</sup> Edition, John Willey & Sons Ltd., New York, pp. 675-679.
- Institute of Petroleum, (1987), Methods of Analysis and Testing of Petroleum Related Products, John Willey and Sons Ltd., London, pp.734-742.
- Jilner, D. A., (1997), Method of Analysis and Testing of Petroleum Related Products. *Journal of Material Processing Technology*, 7(4): 15-19.

- Kinghom, R. R. F., (1983), An Introduction to The Physics and Chemistry of Petroleum, 1<sup>st</sup> Edition, John Willey and Sons Ltd., New York, pp. 188-192.
- Nehal, S. A. and Amal, M. N., (2011), Lubricating Oil Additives, INTECH Publisher, Croatia, pp. 122-126.
- O'connar, J. J., Boyd, J. and Auallane, E. A. (1968), Standard Handbook of Lubrication Engineering, Mc-Graw Hill, New York, pp. 87-91.
- Ofofu, J. C. and Anosike, C., (1993), Variation in Rheological Properties of Engine Oils with Usage. *Global Journal of Engineering Research*, 2(2): 95-10.
- Okeola, F. O., Odebunmi, E. O. and Aremu, J., (2011), Comparative Assessment of Physicochemical Properties of Common Automobile Consumable Oils. *Advances in Environmental Biology*, 5(1): 1-2.
- Okoye, I. P., (2009), Fundamentals of Petroleum and Hydrocarbon Chemistry, University of Portharcourt Press, Portharcourt, pp. 111-115.
- Olayinka, O., (2006), An Introduction to Modern Petroleum Science, University of Ibadan Press, Ibadan, pp. 143-144.
- Pawlak, Z., (2003), Tribochemistry of Lubricating Oils, Elsevier Science Publishers, Amsterdam, pp. 56-58.
- Pirro, D. M. and Wessol, A. A., (2001), Lubrication Fundamentals, Marcel Dekker Inc., New York, pp.155-159.
- Robert, H. P. and Perry, W. G., (1997), Chemical Engineering Handbook, 7<sup>th</sup> Edition, Mc-Graw Hill, New York, pp.866-871.
- Robert, B., (1996), Bosch Automotive Handbook, 4<sup>th</sup> Edition, Bentley Publishers Ltd., Cambridge, pp. 241-243.

- Robson, H. A. B., (2014), Measurement and Evaluation of the Properties of Selected Commercially Produced Lubricants, Ahmadu Bello University, Zaria, pp. 3-5
- Rosenbaun, J., (2015), Base Oil Groups: I-V, Manufacturing, Properties and Performance, Werbiners, Barkely, pp. 3-5
- Rundrick, L. R., (2013), Synthetics, Minerals and Bio-Based Lubricants Chemistry and Technology, 2<sup>nd</sup> Edition, Taylor and Francis Group, United States, pp. 195-197.
- Saheed, W. K., (2012), Lube Oil Blending Plant, [Http://Saadappliedianengineer.Blogspot.Com.Ng/](http://Saadappliedianengineer.Blogspot.Com.Ng/), (Accessed 15/05/17).
- Schlosberg, R. H., Chu, J. W., Knudsen, G. A., Suciu, E. N. and Aldrich, H.S., (2001), High Stability Esters for Synthetic Lubricant Applications, Mc-Graw Hill, New York, pp.21-26.
- Sharma, B. K., (2006), Industrial Chemistry Including Chemical Engineering, Karishna Prakashan Media Publishers Ltd., Meruut, pp. 1235-1238.
- Theo Mang, W. D., (2007), Lubricants and Lubrication, John Wiley & Sons, Ney York, pp. 541-544.
- Thomsen, T. C., (1997), The Practice of Lubrication, McGrawhill Inc., Newyork, Pp. 154-157.
- Troyer, D., (2002), Understanding Absolute and Kinematic Viscosity. *Practical Oil Analysis Magazine*, 4(5): 4-8.

## APPENDICES

### Appendix 1: ANOVA tables for Kinematic Viscosity @40°C

#### Descriptive

KV @40°C

|       | N  | Mean       | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum  | Maximum  |
|-------|----|------------|----------------|------------|----------------------------------|-------------|----------|----------|
|       |    |            |                |            | Lower Bound                      | Upper Bound |          |          |
|       |    |            |                |            | A                                | 2           |          |          |
| B     | 2  | 158.341500 | .0021213       | .0015000   | 158.322441                       | 158.360559  | 158.3400 | 158.3430 |
| C     | 2  | 155.702500 | .0035355       | .0025000   | 155.670734                       | 155.734266  | 155.7000 | 155.7050 |
| D     | 2  | 157.103000 | .0042426       | .0030000   | 157.064881                       | 157.141119  | 157.1000 | 157.1060 |
| E     | 2  | 159.111000 | .0014142       | .0010000   | 159.098294                       | 159.123706  | 159.1100 | 159.1120 |
| F     | 2  | 157.312000 | .0028284       | .0020000   | 157.286588                       | 157.337412  | 157.3100 | 157.3140 |
| G     | 2  | 143.210500 | .0007071       | .0005000   | 143.204147                       | 143.216853  | 143.2100 | 143.2110 |
| H     | 2  | 153.333500 | .0049497       | .0035000   | 153.289028                       | 153.377972  | 153.3300 | 153.3370 |
| R1    | 2  | 154.542000 | .0028284       | .0020000   | 154.516588                       | 154.567412  | 154.5400 | 154.5440 |
| R2    | 2  | 155.401000 | .0014142       | .0010000   | 155.388294                       | 155.413706  | 155.4000 | 155.4020 |
| Total | 20 | 155.115800 | 4.4098890      | .9860811   | 153.051908                       | 157.179692  | 143.2100 | 159.1120 |

#### ANOVA

KV @40°C

|                | Sum of Squares | df | Mean Square | F           | Sig. |
|----------------|----------------|----|-------------|-------------|------|
| Between Groups | 369.495        | 9  | 41.055      | 5006710.152 | .000 |
| Within Groups  | .000           | 10 | .000        |             |      |
| Total          | 369.495        | 19 |             |             |      |

KV @40°C

Duncan Multiple Range Test

| Samples | N | Subset for alpha = 0.05 |           |           |           |           |           |            |           |           |
|---------|---|-------------------------|-----------|-----------|-----------|-----------|-----------|------------|-----------|-----------|
|         |   | 1                       | 2         | 3         | 4         | 5         | 6         | 7          | 8         | 9         |
| G       | 2 | 143.21050<br>0          |           |           |           |           |           |            |           |           |
| H       | 2 |                         | 153.33350 |           |           |           |           |            |           |           |
| R1      | 2 |                         |           | 154.54200 |           |           |           |            |           |           |
| R2      | 2 |                         |           |           | 155.40100 |           |           |            |           |           |
| C       | 2 |                         |           |           |           | 155.70250 |           |            |           |           |
| A       | 2 |                         |           |           |           |           | 157.10100 |            |           |           |
| D       | 2 |                         |           |           |           |           | 157.10300 |            |           |           |
| F       | 2 |                         |           |           |           |           |           | 157.312000 |           |           |
| B       | 2 |                         |           |           |           |           |           |            | 158.34150 |           |
| E       | 2 |                         |           |           |           |           |           |            |           | 159.11100 |
| Sig.    |   | 1.000                   | 1.000     | 1.000     | 1.000     | 1.000     | .501      | 1.000      | 1.000     | 1.000     |

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.000.

## Appendix 2: ANOVA tables for Kinematic Viscosity @100°C

### Descriptives

KV @100°C

|            | N  | Mean    | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|------------|----|---------|----------------|------------|----------------------------------|-------------|---------|---------|
|            |    |         |                |            | Lower Bound                      | Upper Bound |         |         |
|            |    |         |                |            | A                                | 2           |         |         |
| B          | 2  | 15.5250 | .00707         | .00500     | 15.4615                          | 15.5885     | 15.52   | 15.53   |
| C          | 2  | 15.2550 | .02121         | .01500     | 15.0644                          | 15.4456     | 15.24   | 15.27   |
| D          | 2  | 18.1150 | .02121         | .01500     | 17.9244                          | 18.3056     | 18.10   | 18.13   |
| E          | 2  | 18.4100 | .01414         | .01000     | 18.2829                          | 18.5371     | 18.40   | 18.42   |
| F          | 2  | 17.7650 | .00707         | .00500     | 17.7015                          | 17.8285     | 17.76   | 17.77   |
| G          | 2  | 13.3000 | .02828         | .02000     | 13.0459                          | 13.5541     | 13.28   | 13.32   |
| H          | 2  | 15.2150 | .02121         | .01500     | 15.0244                          | 15.4056     | 15.20   | 15.23   |
| R1         | 2  | 15.2600 | .02828         | .02000     | 15.0059                          | 15.5141     | 15.24   | 15.28   |
| R2         | 2  | 18.4200 | .01414         | .01000     | 18.2929                          | 18.5471     | 18.41   | 18.43   |
| Monograde  | 6  | 16.9000 | 4.22942        | 1.72665    | 12.4615                          | 21.3385     | 12.50   | 21.90   |
| Multigrade | 4  | 19.1000 | 3.23316        | 1.61658    | 13.9553                          | 24.2447     | 16.30   | 21.90   |
| Total      | 30 | 16.7590 | 2.65884        | .48543     | 15.7662                          | 17.7518     | 12.50   | 21.90   |

### ANOVA

KV @100° C

|                | Sum of Squares | df | Mean Square | F     | Sig. |
|----------------|----------------|----|-------------|-------|------|
| Between Groups | 84.209         | 11 | 7.655       | 1.141 | .388 |
| Within Groups  | 120.804        | 18 | 6.711       |       |      |
| Total          | 205.013        | 29 |             |       |      |

**KV @100°C**

Duncan Multiple Range Test

| Samples    | N | Subset for alpha<br>= 0.05 |
|------------|---|----------------------------|
|            |   | 1                          |
| G          | 2 | 13.3000                    |
| H          | 2 | 15.2150                    |
| A          | 2 | 15.2200                    |
| C          | 2 | 15.2550                    |
| R1         | 2 | 15.2600                    |
| B          | 2 | 15.5250                    |
| Monograde  | 6 | 16.9000                    |
| F          | 2 | 17.7650                    |
| D          | 2 | 18.1150                    |
| E          | 2 | 18.4100                    |
| R2         | 2 | 18.4200                    |
| Multigrade | 4 | 19.1000                    |
| Sig.       |   | .057                       |

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.215.

b. The group sizes are unequal. The harmonic mean of the group sizes is used.

Type I error levels are not guaranteed.

### Appendix 3: ANOVA tables for Viscosity Index

#### Descriptive

##### VISCOSITY INDEX

|            | N  | Mean     | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|------------|----|----------|----------------|------------|----------------------------------|-------------|---------|---------|
|            |    |          |                |            | Lower Bound                      | Upper Bound |         |         |
|            |    |          |                |            | A                                | 2           |         |         |
| B          | 2  | 99.0150  | .02121         | .01500     | 98.8244                          | 99.2056     | 99.00   | 99.03   |
| C          | 2  | 99.0200  | .02828         | .02000     | 98.7659                          | 99.2741     | 99.00   | 99.04   |
| D          | 2  | 127.9500 | .07071         | .05000     | 127.3147                         | 128.5853    | 127.90  | 128.00  |
| E          | 2  | 128.9000 | .14142         | .10000     | 127.6294                         | 130.1706    | 128.80  | 129.00  |
| F          | 2  | 123.9500 | .07071         | .05000     | 123.3147                         | 124.5853    | 123.90  | 124.00  |
| G          | 2  | 84.2000  | .28284         | .20000     | 81.6588                          | 86.7412     | 84.00   | 84.40   |
| H          | 2  | 99.0050  | .00707         | .00500     | 98.9415                          | 99.0685     | 99.00   | 99.01   |
| R1         | 2  | 99.0150  | .02121         | .01500     | 98.8244                          | 99.2056     | 99.00   | 99.03   |
| R2         | 2  | 133.2000 | .28284         | .20000     | 130.6588                         | 135.7412    | 133.00  | 133.40  |
| Monograde  | 6  | 95.0333  | .31411         | .12824     | 94.7037                          | 95.3630     | 94.50   | 95.40   |
| Multigrade | 4  | 119.9500 | .33166         | .16583     | 119.4223                         | 120.4777    | 119.50  | 120.30  |
| Total      | 30 | 107.7510 | 15.58972       | 2.84628    | 101.9297                         | 113.5723    | 84.00   | 133.40  |



## Appendix 4: ANOVA tables for Specific Gravity

### Descriptives

Specific gravity @15°C

|            | N  | Mean    | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|------------|----|---------|----------------|------------|----------------------------------|-------------|---------|---------|
|            |    |         |                |            | Lower Bound                      | Upper Bound |         |         |
|            |    |         |                |            | A                                | 2           |         |         |
| B          | 2  | .887500 | .0007071       | .0005000   | .881147                          | .893853     | .8870   | .8880   |
| C          | 2  | .892100 | .0021213       | .0015000   | .873041                          | .911159     | .8906   | .8936   |
| D          | 2  | .896000 | .0007071       | .0005000   | .889647                          | .902353     | .8955   | .8965   |
| E          | 2  | .899800 | .0028284       | .0020000   | .874388                          | .925212     | .8978   | .9018   |
| F          | 2  | .897100 | .0007071       | .0005000   | .890747                          | .903453     | .8966   | .8976   |
| G          | 2  | .865400 | .0014142       | .0010000   | .852694                          | .878106     | .8644   | .8664   |
| H          | 2  | .875500 | .0021213       | .0015000   | .856441                          | .894559     | .8740   | .8770   |
| R1         | 2  | .891300 | .0007071       | .0005000   | .884947                          | .897653     | .8908   | .8918   |
| R2         | 2  | .896100 | .0042426       | .0030000   | .857981                          | .934219     | .8931   | .8991   |
| Monograde  | 6  | .880000 | .0089443       | .0036515   | .870614                          | .889386     | .8700   | .8900   |
| Multigrade | 4  | .882500 | .0028868       | .0014434   | .877907                          | .887093     | .8800   | .8850   |
| Total      | 30 | .886153 | .0101139       | .0018465   | .882377                          | .889930     | .8644   | .9018   |

### ANOVA

Specific gravity @15 °C

|                | Sum of Squares | Df | Mean Square | F     | Sig. |
|----------------|----------------|----|-------------|-------|------|
| Between Groups | .003           | 11 | .000        | 8.780 | .000 |
| Within Groups  | .000           | 18 | .000        |       |      |
| Total          | .003           | 29 |             |       |      |

**Specific gravity @15°C**

Duncan Multiple Range Test

| Samples    | N | Subset for alpha = 0.05 |         |         |         |         |         |
|------------|---|-------------------------|---------|---------|---------|---------|---------|
|            |   | 1                       | 2       | 3       | 4       | 5       | 6       |
| G          | 2 | .865400                 |         |         |         |         |         |
| H          | 2 | .875500                 | .875500 |         |         |         |         |
| Monograde  | 6 |                         | .880000 | .880000 |         |         |         |
| Multigrade | 4 |                         | .882500 | .882500 | .882500 |         |         |
| A          | 2 |                         |         | .886500 | .886500 | .886500 |         |
| B          | 2 |                         |         | .887500 | .887500 | .887500 |         |
| R1         | 2 |                         |         |         | .891300 | .891300 | .891300 |
| C          | 2 |                         |         |         | .892100 | .892100 | .892100 |
| D          | 2 |                         |         |         |         | .896000 | .896000 |
| R2         | 2 |                         |         |         |         | .896100 | .896100 |
| F          | 2 |                         |         |         |         | .897100 | .897100 |
| E          | 2 |                         |         |         |         |         | .899800 |
| Sig.       |   | .051                    | .187    | .171    | .089    | .068    | .134    |

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.215.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

## Appendix 5: ANOVA tables for Flash Point

### Descriptive

Flash point (°C)

|            | N  | Mean     | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|------------|----|----------|----------------|------------|----------------------------------|-------------|---------|---------|
|            |    |          |                |            | Lower Bound                      | Upper Bound |         |         |
|            |    |          |                |            | A                                | 2           |         |         |
| B          | 2  | 107.5000 | .70711         | .50000     | 101.1469                         | 113.8531    | 107.00  | 108.00  |
| C          | 2  | 124.5000 | .70711         | .50000     | 118.1469                         | 130.8531    | 124.00  | 125.00  |
| D          | 2  | 190.5000 | .70711         | .50000     | 184.1469                         | 196.8531    | 190.00  | 191.00  |
| E          | 2  | 199.5000 | .70711         | .50000     | 193.1469                         | 205.8531    | 199.00  | 200.00  |
| F          | 2  | 201.5000 | .70711         | .50000     | 195.1469                         | 207.8531    | 201.00  | 202.00  |
| G          | 2  | 212.5000 | .70711         | .50000     | 206.1469                         | 218.8531    | 212.00  | 213.00  |
| H          | 2  | 195.5000 | .70711         | .50000     | 189.1469                         | 201.8531    | 195.00  | 196.00  |
| R1         | 2  | 224.5000 | .70711         | .50000     | 218.1469                         | 230.8531    | 224.00  | 225.00  |
| R2         | 2  | 232.5000 | .70711         | .50000     | 226.1469                         | 238.8531    | 232.00  | 233.00  |
| Monograde  | 6  | 214.6667 | 8.26236        | 3.37310    | 205.9958                         | 223.3375    | 204.00  | 220.00  |
| Multigrade | 4  | 220.0000 | .00000         | .00000     | 220.0000                         | 220.0000    | 220.00  | 220.00  |
| Total      | 30 | 197.9333 | 34.98663       | 6.38766    | 184.8691                         | 210.9976    | 107.00  | 233.00  |

### ANOVA

Flash point

|                | Sum of Squares | df | Mean Square | F       | Sig. |
|----------------|----------------|----|-------------|---------|------|
| Between Groups | 35151.533      | 11 | 3195.594    | 166.085 | .000 |
| Within Groups  | 346.333        | 18 | 19.241      |         |      |
| Total          | 35497.867      | 29 |             |         |      |

**Flash point**

Duncan Multiple Range Test

| Samples    | N | Subset for alpha = 0.05 |          |          |          |          |          |          |
|------------|---|-------------------------|----------|----------|----------|----------|----------|----------|
|            |   | 1                       | 2        | 3        | 4        | 5        | 6        | 7        |
| B          | 2 | 107.5000                |          |          |          |          |          |          |
| C          | 2 |                         | 124.5000 |          |          |          |          |          |
| D          | 2 |                         |          | 190.5000 |          |          |          |          |
| H          | 2 |                         |          | 195.5000 | 195.5000 |          |          |          |
| A          | 2 |                         |          | 196.5000 | 196.5000 |          |          |          |
| E          | 2 |                         |          | 199.5000 | 199.5000 |          |          |          |
| F          | 2 |                         |          |          | 201.5000 |          |          |          |
| G          | 2 |                         |          |          |          | 212.5000 |          |          |
| Monograde  | 6 |                         |          |          |          | 214.6667 |          |          |
| Multigrade | 4 |                         |          |          |          | 220.0000 | 220.0000 |          |
| R1         | 2 |                         |          |          |          |          | 224.5000 | 224.5000 |
| R2         | 2 |                         |          |          |          |          |          | 232.5000 |
| Sig.       |   | 1.000                   | 1.000    | .061     | .202     | .104     | .295     | .071     |

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.215.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

## Appendix 6: ANOVA tables for Pour Point

### Descriptive

Pour point (°C)

|            | N  | Mean     | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|------------|----|----------|----------------|------------|----------------------------------|-------------|---------|---------|
|            |    |          |                |            | Lower Bound                      | Upper Bound |         |         |
|            |    |          |                |            | A                                | 2           |         |         |
| B          | 2  | -15.5000 | .70711         | .50000     | -21.8531                         | -9.1469     | -16.00  | -15.00  |
| C          | 2  | -12.5000 | .70711         | .50000     | -18.8531                         | -6.1469     | -13.00  | -12.00  |
| D          | 2  | -11.0000 | .00000         | .00000     | -11.0000                         | -11.0000    | -11.00  | -11.00  |
| E          | 2  | -10.0000 | .00000         | .00000     | -10.0000                         | -10.0000    | -10.00  | -10.00  |
| F          | 2  | -10.5000 | .70711         | .50000     | -16.8531                         | -4.1469     | -11.00  | -10.00  |
| G          | 2  | -18.5000 | .70711         | .50000     | -24.8531                         | -12.1469    | -19.00  | -18.00  |
| H          | 2  | -16.0000 | .00000         | .00000     | -16.0000                         | -16.0000    | -16.00  | -16.00  |
| R1         | 2  | -11.5000 | .70711         | .50000     | -17.8531                         | -5.1469     | -12.00  | -11.00  |
| R2         | 2  | -12.5000 | .70711         | .50000     | -18.8531                         | -6.1469     | -13.00  | -12.00  |
| Monograde  | 6  | -12.0000 | 4.64758        | 1.89737    | -16.8773                         | -7.1227     | -18.00  | -9.00   |
| Multigrade | 4  | -21.0000 | .00000         | .00000     | -21.0000                         | -21.0000    | -21.00  | -21.00  |
| Total      | 30 | -14.0333 | 4.07248        | .74353     | -15.5540                         | -12.5126    | -21.00  | -9.00   |

### ANOVA

Pour point (°C)

|                | Sum of Squares | df | Mean Square | F     | Sig. |
|----------------|----------------|----|-------------|-------|------|
| Between Groups | 369.467        | 11 | 33.588      | 5.422 | .001 |
| Within Groups  | 111.500        | 18 | 6.194       |       |      |
| Total          | 480.967        | 29 |             |       |      |

**Pour point (°C)**

Duncan Multiple range Test

| Samples    | N | Subset for alpha = 0.05 |          |          |          |
|------------|---|-------------------------|----------|----------|----------|
|            |   | 1                       | 2        | 3        | 4        |
| Multigrade | 4 | -21.0000                |          |          |          |
| G          | 2 | -18.5000                | -18.5000 |          |          |
| H          | 2 | -16.0000                | -16.0000 | -16.0000 |          |
| B          | 2 |                         | -15.5000 | -15.5000 | -15.5000 |
| A          | 2 |                         | -14.5000 | -14.5000 | -14.5000 |
| C          | 2 |                         |          | -12.5000 | -12.5000 |
| R2         | 2 |                         |          | -12.5000 | -12.5000 |
| Monograde  | 6 |                         |          | -12.0000 | -12.0000 |
| R1         | 2 |                         |          | -11.5000 | -11.5000 |
| D          | 2 |                         |          | -11.0000 | -11.0000 |
| F          | 2 |                         |          | -10.5000 | -10.5000 |
| E          | 2 |                         |          |          | -10.0000 |
| Sig.       |   | .059                    | .137     | .057     | .057     |

Means for groups in homogeneous subsets are displayed.

- a. Uses Harmonic Mean Sample Size = 2.215.
- b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

## Appendix 7: Calculation of Total Acid Number (TAN)

$$\text{TAN (mg KOH/g sample)} = \frac{(A-B) \times N \times 56.1}{W}$$

Where: A = Titre value

B = Blank value

N = Normality of KOH = 0.5N

W = Weight of sample = 20

### BLANCK

Titre value

Final 16.50

Initial  $\frac{16.40}{0.10}$

### SAMPLE A

Titre value

Final 16.00

Initial  $\frac{15.50}{0.50}$

$$\text{TAN (mg KOH/g sample)} = \frac{(0.50 - 0.10) \times 0.0874 \times 56.1}{20} = 0.098 \text{mgKOH/g sample}$$

### SAMPLE B

Titre value

Final 26.70

Initial  $\frac{26.00}{0.70}$

$$\text{TAN (mg KOH/g sample)} = \frac{(0.70 - 0.10) \times 0.0874 \times 56.1}{20} = 0.147 \text{mgKOH/g sample}$$

**SAMPLE C**

Titre value

|         |              |
|---------|--------------|
| Final   | 16.40        |
| Initial | <u>16.00</u> |
|         | 0.40         |

$$\text{TAN (mg KOH/g sample)} = \frac{(0.40 - 0.10) \times 0.0874 \times 56.1}{20} = 0.074\text{mgKOH/g sample}$$

**SAMPLE D**

Titre value

|         |              |
|---------|--------------|
| Final   | 15.50        |
| Initial | <u>15.10</u> |
|         | 0.40         |

$$\text{TAN (mg KOH/g sample)} = \frac{(0.40 - 0.10) \times 0.0874 \times 56.1}{20} = 0.074\text{mgKOH/g sample}$$

**SAMPLE E**

Titre value

|         |              |
|---------|--------------|
| Final   | 30.10        |
| Initial | <u>28.90</u> |
|         | 1.20         |

$$\text{TAN (mg KOH/g sample)} = \frac{(1.20 - 0.10) \times 0.0874 \times 56.1}{20} = 0.269\text{mgKOH/g sample}$$

**SAMPLE F**

Titre value

|         |              |
|---------|--------------|
| Final   | 36.00        |
| Initial | <u>35.50</u> |
|         | 0.50         |

$$\text{TAN (mg KOH/g sample)} = \frac{(0.50 - 0.10) \times 0.0874 \times 56.1}{20} = 0.098\text{mgKOH/g sample}$$



**SAMPLE G**

Titre value

|         |              |
|---------|--------------|
| Final   | 27.50        |
| Initial | <u>27.20</u> |
|         | 0.30         |

$$\text{TAN (mg KOH/g sample)} = \frac{(0.30 - 0.10) \times 0.0874 \times 56.1}{20} = 0.049 \text{mgKOH/g sample}$$

**SAMPLE H**

Titre value

|         |              |
|---------|--------------|
| Final   | 27.10        |
| Initial | <u>26.70</u> |
|         | 0.40         |

$$\text{TAN (mg KOH/g sample)} = \frac{(0.40 - 0.10) \times 0.0874 \times 56.1}{20} = 0.074 \text{mgKOH/g sample}$$

## Appendix 7.1 ANOVA tables for Total Acid Number (TAN)

### Descriptive

TAN

|            | N  | Mean   | Std. Deviation | Std. Error | 95% Confidence Interval for |             | Minimum | Maximum |
|------------|----|--------|----------------|------------|-----------------------------|-------------|---------|---------|
|            |    |        |                |            | Mean                        |             |         |         |
|            |    |        |                |            | Lower Bound                 | Upper Bound |         |         |
| A          | 2  | .0985  | .00071         | .00050     | .0921                       | .1049       | .10     | .10     |
| B          | 2  | .1470  | .00000         | .00000     | .1470                       | .1470       | .15     | .15     |
| C          | 2  | .0740  | .00000         | .00000     | .0740                       | .0740       | .07     | .07     |
| D          | 2  | .0738  | .00035         | .00025     | .0706                       | .0769       | .07     | .07     |
| E          | 2  | .2090  | .00000         | .00000     | .2090                       | .2090       | .21     | .21     |
| F          | 2  | .0980  | .00000         | .00000     | .0980                       | .0980       | .10     | .10     |
| G          | 2  | .0485  | .00071         | .00050     | .0421                       | .0549       | .05     | .05     |
| H          | 2  | .0740  | .00000         | .00000     | .0740                       | .0740       | .07     | .07     |
| R1         | 1  | .0000  | .              | .          | .                           | .           | .00     | .00     |
| R2         | 1  | .0000  | .              | .          | .                           | .           | .00     | .00     |
| Monograde  | 6  | 1.0000 | .00000         | .00000     | 1.5000                      | 1.5000      | 1.50    | 1.50    |
| Multigrade | 4  | 1.0000 | .00000         | .00000     | 1.5000                      | 1.5000      | 1.50    | 1.50    |
| Total      | 28 | .5945  | .68883         | .13018     | .3274                       | .8616       | .00     | 1.50    |

### ANOVA

TAN

|                | Sum of Squares | df | Mean Square | F            | Sig. |
|----------------|----------------|----|-------------|--------------|------|
| Between Groups | 12.811         | 11 | 1.165       | 16564010.170 | .000 |
| Within Groups  | .000           | 16 | .000        |              |      |
| Total          | 12.811         | 27 |             |              |      |

## Appendix 8: Calculation of Moisture Content (ppm)

$$\text{Moisture (ppm)} = \frac{\text{vol. of Karl Fischer reagent} \times 3.5714 \times 1000}{\text{Weight of sample}}$$

### SAMPLE A

#### First analysis

$$\text{Weight of sample} = \text{weight of syringe + oil} - \text{weight of syringe alone}$$

$$\text{Weight of syringe + oil} = 9.49$$

$$\text{Weight of syringe alone} = \frac{5.00}{4.49}$$

$$\text{Vol. of Karl Fischer reagent} = 0.089$$

$$\text{Moisture (ppm)} = \frac{0.089 \times 3.5714 \times 1000}{4.49} = 70.79 \text{ ppm}$$

#### Second analysis

$$\text{Weight of syringe + oil} = 9.51$$

$$\text{Weight of syringe alone} = \frac{5.02}{4.49}$$

$$\text{Vol. of Karl Fischer reagent} = 0.092$$

$$\text{Moisture (ppm)} = \frac{0.092 \times 3.5714 \times 1000}{4.49} = 73.18 \text{ ppm}$$

$$\text{Average} = \frac{70.79 + 73.18}{2} = 71.99 \text{ ppm}$$

### SAMPLE B

#### First analysis

$$\text{Weight of sample} = \text{weight of syringe + oil} - \text{weight of syringe alone}$$

$$\text{Weight of syringe + oil} = 9.21$$

$$\text{Weight of syringe alone} = \frac{4.70}{4.51}$$

$$\text{Vol. of Karl Fischer reagent} = 0.104$$

$$\text{Moisture (ppm)} = \frac{0.104 \times 3.5714 \times 1000}{4.51} = 82.35 \text{ ppm}$$

**Second analysis**

$$\text{Weight of syringe + oil} = 9.51$$

$$\text{Weight of syringe alone} = \frac{4.71}{4.44}$$

$$\text{Vol. of Karl Fischer reagent} = 0.106$$

$$\text{Moisture (ppm)} = \frac{0.106 \times 3.5714 \times 1000}{4.44} = 85.26\text{ppm}$$

$$\text{Average} = \frac{82.35 + 85.26}{2} = 83.81\text{ppm}$$

**SAMPLE C****First analysis**

$$\text{Weight of sample} = \text{weight of syringe + oil} - \text{weight of syringe alone}$$

$$\text{Weight of syringe + oil} = 9.31$$

$$\text{Weight of syringe alone} = \frac{5.03}{4.28}$$

$$\text{Vol. of Karl Fischer reagent} = 0.114$$

$$\text{Moisture (ppm)} = \frac{0.114 \times 3.5714 \times 1000}{4.28} = 95.13\text{ppm}$$

**Second analysis**

$$\text{Weight of syringe + oil} = 9.51$$

$$\text{Weight of syringe alone} = \frac{5.02}{4.49}$$

$$\text{Vol. of Karl Fischer reagent} = 0.124$$

$$\text{Moisture (ppm)} = \frac{0.092 \times 3.5714 \times 1000}{4.49} = 98.63\text{ppm}$$

$$\text{Average} = \frac{95.13 + 98.63}{2} = 96.88\text{ppm}$$

## **SAMPLE D**

### **First analysis**

Weight of sample = weight of syringe + oil – weight of syringe alone

Weight of syringe + oil = 9.21

Weight of syringe alone = 4.71

4.50

Vol. of Karl Fischer reagent = 1.380

Moisture (ppm) =  $\frac{1.380 \times 3.5714 \times 1000}{4.50} = 1095.23\text{ppm}$

### **Second analysis**

Weight of syringe + oil = 9.40

Weight of syringe alone = 4.94

4.46

Vol. of Karl Fischer reagent = 1.372

Moisture (ppm) =  $\frac{1.372 \times 3.5714 \times 1000}{4.46} = 1098.24\text{ppm}$

Average =  $\frac{1095.23 + 1098.24}{2} = 1096.74\text{ppm}$

## **SAMPLE E**

### **First analysis**

Weight of sample = weight of syringe + oil – weight of syringe alone

Weight of syringe + oil = 9.17

Weight of syringe alone = 4.71

4.46

Vol. of Karl Fischer reagent = 2.722

Weight of syringe alone = 4.71

4.46

Vol. of Karl Fischer reagent = 2.722

Moisture (ppm) =  $\frac{2.722 \times 3.5714 \times 1000}{4.46} = 2179.67\text{ppm}$

**Second analysis**

$$\text{Weight of syringe + oil} = 9.20$$

$$\text{Weight of syringe alone} = \frac{4.93}{4.27}$$

$$\text{Vol. of Karl Fischer reagent} = 2.610$$

$$\text{Moisture (ppm)} = \frac{2.610 \times 3.5714 \times 1000}{4.27} = 2182.99\text{ppm}$$

$$\text{Average} = \frac{2179.67 + 2182.99}{2} = 2181.33\text{ppm}$$

**SAMPLE F****First analysis**

$$\text{Weight of sample} = \text{weight of syringe + oil} - \text{weight of syringe alone}$$

$$\text{Weight of syringe + oil} = 9.30$$

$$\text{Weight of syringe alone} = \frac{4.99}{4.31}$$

$$\text{Vol. of Karl Fischer reagent} = 0.090$$

$$\text{Moisture (ppm)} = \frac{0.090 \times 3.5714 \times 1000}{4.31} = 74.58\text{ppm}$$

**Second analysis**

$$\text{Weight of syringe + oil} = 9.42$$

$$\text{Weight of syringe alone} = \frac{4.98}{4.44}$$

$$\text{Vol. of Karl Fischer reagent} = 0.092$$

$$\text{Moisture (ppm)} = \frac{0.088 \times 3.5714 \times 1000}{4.49} = 70.79\text{ppm}$$

$$\text{Average} = \frac{74.58 + 70.79}{2} = 72.69\text{ppm}$$

## **SAMPLE G**

### **First analysis**

Weight of sample = weight of syringe + oil – weight of syringe alone

Weight of syringe + oil = 9.51

Weight of syringe alone = 5.00

4.51

Vol. of Karl Fischer reagent = 0.112

Moisture (ppm) =  $\frac{0.112 \times 3.5714 \times 1000}{4.51} = 88.69\text{ppm}$

### **Second analysis**

Weight of syringe + oil = 9.43

Weight of syringe alone = 5.00

4.43

Vol. of Karl Fischer reagent = 0.114

Moisture (ppm) =  $\frac{0.114 \times 3.5714 \times 1000}{4.43} = 91.90\text{ppm}$

Average =  $\frac{88.69 + 91.90}{2} = 90.29\text{ppm}$

## **SAMPLE H**

### **First analysis**

Weight of sample = weight of syringe + oil – weight of syringe alone

Weight of syringe + oil = 9.06

Weight of syringe alone = 4.72

4.36

Vol. of Karl Fischer reagent = 0.174

Moisture (ppm) =  $\frac{0.174 \times 3.5714 \times 1000}{4.36} = 142.53\text{ppm}$

### **Second analysis**

Weight of syringe + oil = 9.17

Weight of syringe alone = 4.72

4.45

Vol. of Karl Fischer reagent = 0.174

$$\text{Moisture (ppm)} = \frac{0.174 \times 3.5714 \times 1000}{4.45} = 139.65\text{ppm}$$

$$\text{Average} = \frac{142.53 + 139.65}{2} = 141.09\text{ppm}$$

## **SAMPLE R1**

### **First analysis**

Weight of sample = weight of syringe + oil – weight of syringe alone

Weight of syringe + oil = 9.43

Weight of syringe alone =  $\frac{4.99}{4.44}$

Vol. of Karl Fischer reagent = 2.842

$$\text{Moisture (ppm)} = \frac{2.842 \times 3.5714 \times 1000}{4.44} = 2286.02\text{ppm}$$

### **Second analysis**

Weight of syringe + oil = 9.37

Weight of syringe alone =  $\frac{5.00}{4.37}$

Vol. of Karl Fischer reagent = 2.802

$$\text{Moisture (ppm)} = \frac{0.092 \times 3.5714 \times 1000}{4.37} = 2289.10\text{ppm}$$

$$\text{Average} = \frac{2286.02 + 2289.10}{2} = 2287.56\text{ppm}$$

## **SAMPLE R2**

### **First analysis**

Weight of sample = weight of syringe + oil – weight of syringe alone

Weight of syringe + oil = 9.49

Weight of syringe alone =  $\frac{4.99}{4.40}$



Vol. of Karl Fischer reagent = 0.804

$$\text{Moisture (ppm)} = \frac{0.804 \times 3.5714 \times 1000}{4.40} = 652.59\text{ppm}$$

**Second analysis**

Weight of syringe + oil = 7.46

Weight of syringe alone =  $\frac{4.97}{4.49}$

Vol. of Karl Fischer reagent = 0.818

$$\text{Moisture (ppm)} = \frac{0.818 \times 3.5714 \times 1000}{4.49} = 650.65\text{ppm}$$

$$\text{Average} = \frac{652.59 + 650.65}{2} = 651.62\text{ppm}$$

## Appendix 8.1: ANOVA tables for Moisture Content (ppm)

### Descriptives

Moisture (ppm)

|       | N  | Mean      | Std. Deviation | Std. Error | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|-------|----|-----------|----------------|------------|----------------------------------|-------------|---------|---------|
|       |    |           |                |            | Lower Bound                      | Upper Bound |         |         |
|       |    |           |                |            | A                                | 2           |         |         |
| B     | 2  | 83.8050   | 2.05768        | 1.45500    | 65.3175                          | 102.2925    | 82.35   | 85.26   |
| C     | 2  | 96.8800   | 2.47487        | 1.75000    | 74.6441                          | 119.1159    | 95.13   | 98.63   |
| D     | 2  | 1096.9350 | 2.41123        | 1.70500    | 1075.2709                        | 1118.5991   | 1095.23 | 1098.64 |
| E     | 2  | 90.2950   | 2.26981        | 1.60500    | 69.9015                          | 110.6885    | 88.69   | 91.90   |
| F     | 2  | 72.6850   | 2.67993        | 1.89500    | 48.6067                          | 96.7633     | 70.79   | 74.58   |
| G     | 2  | 2181.3300 | 2.34759        | 1.66000    | 2160.2377                        | 2202.4223   | 2179.67 | 2182.99 |
| H     | 2  | 141.0900  | 2.03647        | 1.44000    | 122.7931                         | 159.3869    | 139.65  | 142.53  |
| R1    | 2  | 2287.5600 | 2.17789        | 1.54000    | 2267.9924                        | 2307.1276   | 2286.02 | 2289.10 |
| R2    | 2  | 651.6200  | 1.37179        | .97000     | 639.2950                         | 663.9450    | 650.65  | 652.59  |
| Total | 20 | 677.4185  | 863.59137      | 193.10490  | 273.2453                         | 1081.5917   | 70.79   | 2289.10 |

### ANOVA

Moisture (ppm)

|                | Sum of Squares | df | Mean Square | F          | Sig. |
|----------------|----------------|----|-------------|------------|------|
| Between Groups | 14169963.273   | 9  | 1574440.364 | 330440.936 | .000 |
| Within Groups  | 47.647         | 10 | 4.765       |            |      |
| Total          | 14170010.920   | 19 |             |            |      |

**Moisture (ppm)**

Duncan

| Sample<br>s | N | Subset for alpha = 0.05 |         |         |         |          |          |           |           |           |
|-------------|---|-------------------------|---------|---------|---------|----------|----------|-----------|-----------|-----------|
|             |   | 1                       | 2       | 3       | 4       | 5        | 6        | 7         | 8         | 9         |
| A           | 2 | 71.9850                 |         |         |         |          |          |           |           |           |
| F           | 2 | 72.6850                 |         |         |         |          |          |           |           |           |
| B           | 2 |                         | 83.8050 |         |         |          |          |           |           |           |
| E           | 2 |                         |         | 90.2950 |         |          |          |           |           |           |
| C           | 2 |                         |         |         | 96.8800 |          |          |           |           |           |
| H           | 2 |                         |         |         |         | 141.0900 |          |           |           |           |
| R2          | 2 |                         |         |         |         |          | 651.6200 |           |           |           |
| D           | 2 |                         |         |         |         |          |          | 1096.9350 |           |           |
| G           | 2 |                         |         |         |         |          |          |           | 2181.3300 |           |
| R1          | 2 |                         |         |         |         |          |          |           |           | 2287.5600 |
| Sig.        |   | .755                    | 1.000   | 1.000   | 1.000   | 1.000    | 1.000    | 1.000     | 1.000     | 1.000     |

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 2.000.

## Appendix 9: American Petroleum Institute (API) Requirement for Engine Lubricating Oils

| CHARACTERISTICS                                     | SPECIFICATION  |                |                |                |                |
|---|----------------|----------------|----------------|----------------|----------------|
|   | MONO GRADE     |                |                | MULTI GRADE    |                |
|   | SAE 30         | SAE 40         | SAE 50         | 15W40          | 20W 50         |
| Appearance  | Bright & clear | Bright & clear | Bright & clear | Bright & clear | Bright & clear |
| ASTM Colour   | Report         | Report         | Report         | Report         | Report         |
| Specific Gravity @15 <sup>0</sup> C (Min)           | 0.870          | 0.880          | 0.890          | 0.880          | 0.885          |
| Kinematic Viscosity @40 <sup>0</sup> C (cst)        | Report         | Report         | Report         | Report         | Report         |
| Kinematic Viscosity @100 <sup>0</sup> C (cst) (Min) | 9.30-12.50     | 12.50-16.30    | 16.30-21.90    | 12.50-16.30    | 16.30-21.90    |
| Viscosity index (VI) (Min)                          | 95             | 95             | 95             | 120            | 120            |
| Total Base Number (mgKOH/g) (Min)                   | 5.0            | 5.0            | 5.0            | 6.5            | 6.5            |
| Total Acid Number (mgKOH/g) (Max)                   | 1.5            | 1.5            | 1.5            | 1.5            | 1.5            |
| Moisture Content (ppm)                              | <100           | <100           | <100           | <100           | <100           |
| Flash Point <sup>0</sup> C (Min)                    | 204            | 220            | 220            | 220            | 220            |
| Pour Point <sup>0</sup> C (Max)                     | -18            | -9             | -9             | -21            | -21            |