

Chemistry and Technology of Lubricants

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Editors

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Third Edition

 Springer

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Preface

The third edition of this book reflects how the chemistry and technology of lubricants have developed since the first edition was published in 1992. Refinery processes have become more precise in defining the physical and chemical properties of higher quality mineral base oils, Part I, Chapters 1 and 2, beneficial with the move away from Gp.I mineral base oils towards Gps.II and III, synthetic base oils such as poly- α -olefins (PAOs), the esters and others. New and existing additives have improved performance through enhanced understanding of their action, Part II, Chapters 3–7. Applications have become more rigorous, Part III, Chapters 8–14. The performance, specification and testing of lubricants has become more focused on higher level requirements, Part IV, Chapters 15–17. The acceleration of performance development in the past 35 years has been as significant as in the previous century. The performance and life between service changes of lubricants have extended dramatically and are expected to extend more, Chapters 9 and 10. Yet more performance will still be required but it will also include the lubricant's ability to 'stay in grade' for efficiency savings and withstand the conditions arising from the use of advanced environmental emission controls, such as for Euro 5 and 6 engines and their North American equivalents.

The physical benefits of having a lubricant film between surfaces in relative motion have been known for several millennia. Dowson [1] found an Egyptian hieroglyph of a large stone block hauled by many slaves. Close inspection shows fluid, presumably water, being poured into the immediate path of the block. Moderately refined vegetable oils and fats were increasingly used to lubricate machines and carriage/wagon bearings; the benefits of reducing the force needed to operate them were a widely received wisdom up to the end of the middle ages, ~1450 AD. Increasing industrialisation after 1600 AD, accelerated during the First Industrial Revolution in Britain after 1760 AD, soon followed by other developed countries, recognised the important contribution that lubricants made in reducing the work required to overcome friction and in extending the working life of machines. The crude technology existed and was effective for its time but it was not understood.

Leonardo da Vinci was the first person recorded to investigate the resistance to motion of two 'smooth' loaded bodies in contact. He set out the Laws of Friction as we now essentially know them [2] but they were not appreciated and nor applied at the time. Whilst Amontons in 1699 [3] and Coulomb in 1785 [4] essentially

re-discovered and extended the Laws of Friction, they concentrated on lubricant effects at the surfaces of two contacting blocks of material in relative motion. They recognised that the surfaces were rough, on a fine scale, and suggested that lubricants held in the crevices and recesses of those surfaces reduced their effective roughness. This concept explained the effects of lubricants for the relatively unsophisticated technology up to the 1850s.

Increased power densities and throughputs placed greater attention upon the lubrication of bearings and both Tower [5] and Petrov [6] separately showed in 1883 that a shaft rotating in a lubricated bearing has a full, coherent film separating the two components. The fluid film thickness was many times that of the surface roughness dimension. Reynolds [7] studied the viscous flow of lubricants in plain bearings in 1886 and his analysis of the results led to the differential equation of pressure within contacts, Eq. (1), that continues as the basis of full fluid film lubrication – hydrodynamic lubrication.

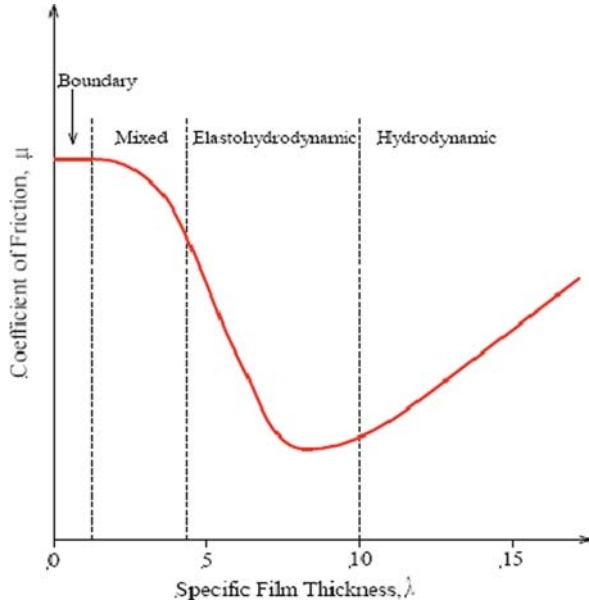
$$\frac{dp}{dx} = 6\eta (U_o + U_h) \left[\frac{h - h_m}{h^3} \right] \quad (1)$$

Equation 1 is an integrated Reynolds equation for the hydrodynamic lubrication of a bearing (for steady state one-dimensional relative motion flow with negligible side leakage (transverse flow) where p is fluid pressure, x the one-dimensional distance into the bearing, h the film thickness and h_m at maximum pressure).

But hydrodynamic lubrication does not always apply. Hardy [8] identified the separate condition of low relative speeds, high loads and low-lubricant viscosities in 1922. Under these conditions the fluid film is not coherent because of the combination of high load and low viscosity and the surfaces are in contact at the tips of the surface roughness, the asperities. In a memorable analogy, Bowden and Tabor [9] described two surfaces in contact as ‘Switzerland inverted upon Austria, with only the mountain peaks in contact’. Deformation of the peaks in contact under load and surface films formed from the lubricant fluid and its constituents determine the friction and wear of these contacting surfaces. Understanding the role of surface films recognised a new mechanism, that of boundary lubrication, separate from hydrodynamic lubrication. Types of additives were developed to modify surface films, either by surface absorption or reaction at the interface, to dramatically reduce friction and wear from the 1950s onwards. Understanding the mechanisms of additive action has been aided by surface analyses and informed molecular synthesis.

Dowson and Higginson [10] completed the range of lubrication mechanisms by demonstrating that under extreme loading between contacts, such as in a rolling element bearing between the roller or ball and its cage, the very high pressures generated within the contact caused a plastic deformation of the contact materials together with a pressure-induced enhanced viscosity of the lubricant. This is elastohydrodynamic lubrication, or EHL, which has been of immense value in understanding and predicting the behaviour of thin films in highly loaded contacts. The relationship of these forms of lubrication is shown in the well-known curve brought together by Stribeck [11] (Fig. 1).

Fig. 1 The Stribeck curve



Lubricants are a component part of a mechanical system and must be developed in parallel with that system, as is seen in the API and ACEA specifications, Chapter 17. When that axiom is not followed, then wear and reliability problems begin to occur as extensive wear and serious machine damage. Thus, steam engines in the 1870s were developing to higher power densities through increased steam temperatures and pressures. ‘Superheating’ of steam removed liquid droplets to produce a homogenous, working fluid at higher temperatures. The natural fats and oils used as lubricants of the time began to break down under the enhanced physical working conditions and their degradation products, particularly the organic acids, corroded steel and particularly non-ferrous metal components. The performance demands of the system had moved ahead of the ability of the lubricants to perform and protect it. Fortunately, just at that time, heavier hydrocarbons from crude petroleum production began to be available for use as lubricants which were able to withstand higher temperatures in high-pressure steam environments.

The initial main driving force for the development of the oil industry in the latter half of the 19th century was the supply of lighting, or lamp, oil to augment and then replace animal and vegetable lamp oils. Mineral oil seepages from many natural surface sites had used the lighter components as lamp oils with the heavier components as lubricants and the heaviest components as pitch for caulking and waterproofing. As demand built up for liquid hydrocarbon fuels into the 1920s, the heavier hydrocarbon lubricants became much more readily available for heavy machinery and automotive use. In retrospect, the internal combustion engines of the time had low energy densities and did not stress the simple base oils used as lubricants.

This relatively unchallenged situation was upset in the mid-1930s by Caterpillar introducing new designs of higher power and efficiency engines for their tractors and construction equipment [11]. These characteristically rugged engines were very successful but soon developed problems due to extensive piston deposits resulting from degradation of the lubricants available at that time. Piston rings, stuck in their grooves by adherent carbonaceous deposits, lost their sealing action and engine efficiency declined. Caterpillar responded by developing a lubricant additive to remove and reduce the adherent carbonaceous piston deposits, the first ‘additive’ as would be recognised now. Whilst successful, variable results were found in the field for different base oils and Caterpillar developed a standard test for the effectiveness of lubricants. This is a classic case of machine system development moving ahead of lubricant performance. However, two major developments can be traced from it, first, the additive industry and second, the system of specification and testing of lubricants as now organised by API, ACEA and ILSAC, Chapter 17.

A further step change required for lubricant performance came from the development of the gas turbine in the 1940s. New lubricants were needed to withstand higher operating and lower starting temperatures, for conventional oxidation of unprotected mineral hydrocarbon oils accelerates above 100°C yet their flowpoints are limited to -20°C or so. Synthetic base oils, either as esters derived by reaction from vegetable sources or as synthetic polymers, have been developed initially for the aircraft industry, then aerospace, with wider liquid ranges and superior resistance to thermal and oxidative degradation (Chapter 11 and 12). Their superior performance has now extended into automotive and industrial machinery lubricant formulations.

The reality of machine operation, of whatever form, is related to the regions of the Stribeck curve, Chapter 8. When a machine is operating, with solid surfaces sliding, rotating or reciprocating against each other, then a fluid film of lubricant separates them as the physical effect of hydrodynamic lubrication. A general trend driven by increased efficiencies has increased bearing pressures and reduced lubricant fluid viscosity, giving thinner mean effective film thicknesses. Dowson [12] has demonstrated the thicknesses of fluid film under hydrodynamic and elastohydrodynamic conditions relative to a human hair diameter (Fig. 2).

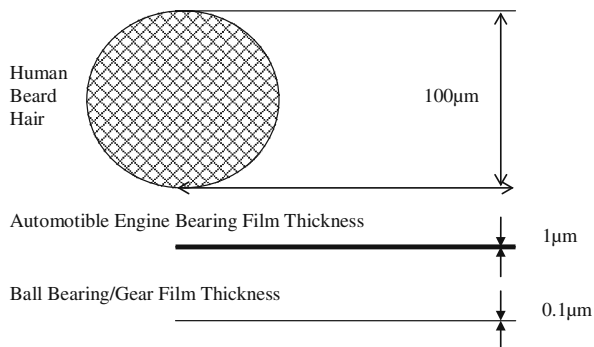


Fig. 2 Relative lubricant film thicknesses (after Dowson [12])

The problem with thin fluid film lubrication occurs when the relative motion of the solid surfaces either stops completely, stops at reversal in reciprocating motion or the dynamic loading of a cam on its follower, one gear tooth on another or on a journal within a bearing such that this lubrication mechanism fails and the surfaces make contact. Under boundary lubrication conditions the role of adsorbed molecular films of protective additives is crucial in protecting against wear.

Anti-wear additives are but one of a number of additive types formulated into base oils – there are also anti-oxidants, Chapter 4, and anti-acid, detergents and dispersants, Chapter 7, lubricity, anti-wear, extreme pressure, pour point depressants, anti-rust and anti-foam additives, Chapter 6. Viscosity index improvers, VIIs, are high-molecular weight polymers which alter the temperature dependence of the base oil viscosity, Chapter 5. Taken altogether, the additive mass percentage of a formulated lubricant can be as high as 15–20%, a veritable ‘chemical soup’ but one which is very carefully formulated and tested. The additives are often multi-functional, thus some VII compounds have a pour point depressant function, Chapters 5 and 6. Some anti-oxidants have anti-wear and also anti-acid functionality, Chapters 4, 6 and 3. Given these cross-interactions, formulation of a final lubricant product is a complex and skilled activity, Chapters 8–13.

Whilst most formulation development work has gone into vehicle automotive lubrication, Chapters 9 and 10, more specialised development has gone to formulate lubricants for specific applications such as gas turbine, Chapter 11, and aerospace lubricants, Chapter 12, the different requirements to cover the marine diesel engine size and power range, Chapter 13, industrial machinery and metal working (both cutting and forming), Chapter 8. The apparently simple, but complex in detail, formulation, manufacturing and performance applications of grease are discussed in Chapter 14.

The environmental implications of lubricant production, use and disposal are discussed in Chapter 15 to show that lubricants have an outstanding environmental record in both extending the use of hydrocarbon resources by longer service intervals and also by extending the life and reliability of machines. However, the requirements to recycle used lubricants will increase. Ensuring the reliability of machines is discussed as ‘Condition Monitoring’ in Chapter 16 and ensuring the fitness for purpose of lubricants is the subject of Chapter 17, ‘The Specification and Testing of Lubricants’.

Looking to the future, it is self-evident that further demands will be made for improved lubricant performance. The service change lifetime of automotive engine lubricants will continue to increase, whereas powertrain lubricants are already close to ‘fill for life’. The limit for engine lubricant service life will possibly be set by other constraints such as the need for annual or biennial vehicle services for all vehicle systems. Thus, North America could readily adjust its lubricant change periods over time to those already used in Europe and save many Mt/base oil each year. Problems to deal with on the way to enhanced service intervals include the effects of bio-fuels on lubricants and their performance, maintaining efficiency gains across the service life of a lubricant charge and the effects of engine modifications for even lower emissions.

To meet enhanced lubricant performance and service interval life, base oils are already moving upwards, away from Gp.I towards the more highly treated and refined mineral base oils of Gps.II and III and also the synthetic base oils of PAOs and esters. Their relative costs and benefits will determine the base oil mix, Chapters 1 and 2.

Additives have two apparent counteracting pressures. The demands for improved lubricant performance can mean more sophisticated additives, Chapters 3–7, in more complex formulations, Chapters 8–14. On the other hand, there is the pressure of the ‘REACH’ chemicals assessment program in the EU, paralleled elsewhere by a general direction to reduce chemical eco-toxicity on consumer products, for no business wishes to have warning cryptograms of dead fish and dying trees on its products! To meet these requirements, the ‘CHON’ philosophy for additives is being explored, where lubricant additives will only contain carbon, hydrogen, oxygen and nitrogen. This excludes metals such as zinc and molybdenum and the non-metals sulphur and phosphorus because of their environmental effects. This will be a stringent test of research and development.

Finally, at the end of their useful life, lubricants will be regarded as a valuable resource and re-refined/recycled into new lubricant products and fuels. Acceptance of recycled base stocks into new lubricant formulations will take time and require rigorous quality testing but will, and must, inevitably happen.

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Part I
Base Oils

Chapter 1

Base Oils from Petroleum

R.J. Prince

Abstract The source, composition and suitability of crude oils for base oil production are reviewed. The physical and chemical properties of alkanes, naphthenes and aromatics and their characteristics for lubricant applications are examined. Properties and applications of various base oils are defined and specified. Production of conventional mineral oils is described, including the various processes to remove wax and other deleterious substances, followed by increasingly severe hydrogenation to produce base oils of increased quality and performance. The API categorization of mineral base oils, either direct from the refinery or after hydrotreatment of increasing severity, is described, together with sub-categories.

1.1 Introduction

Modern lubricants are formulated from a range of base fluids and chemical additives. The base fluid has several functions but it is primarily the lubricant which provides the fluid layer to separate moving surfaces. It also removes heat and wear particles whilst minimizing friction. Many properties of the lubricant are enhanced or created by the addition of special chemical additives to the base fluid, as described in later chapters. For example, stability to oxidation and degradation in an engine oil is improved by the addition of antioxidants whilst extreme pressure, EP, anti-wear properties needed in gear lubrication are created by the addition of special additives. The base fluid acts as the carrier for these additives and therefore must be able to maintain them in solution under all normal working conditions.

The majority of lubricant base fluids are produced by refining crude oil. Estimates of the total worldwide demand for petroleum base oils were 35 Mt in 1990 and this has remained approximately stable since [1]. The reasons for the predominance of refined petroleum base oils are simple and obvious – performance, availability and price. Large-scale oil refining operations produce base oils with excellent performance in modern lubricant formulations at economic prices. Non-petroleum base fluids are used where special properties are necessary, where petroleum base oils are in short supply or where substitution by natural products is practicable or desirable.

This chapter is concerned with base oils from crude petroleum oil. Crude oil is an extremely complex mixture of organic chemicals ranging in molecular size from simple gases such as methane to very high molecular weight asphaltic components. Only some of these crude oil constituents are desirable in a lubricant base fluid and a series of physical refining steps separate the good from the bad. Other process steps involving chemical reactions are also used to enhance properties of the oil fractions. Different types of base oils are produced at refineries with different viscosities or chemical properties, as needed for different applications.

1.2 Base Oil Composition

Crude oil results from physical and chemical processes acting over many million years on the buried remains of plants and animals. Although crude oil is usually formed in fine-grained source rocks, it can migrate into more permeable reservoir rocks and large accumulations of petroleum, the oilfields, are accessed by drilling. Each oilfield produces a different crude oil which varies in chemical composition and physical properties. Some crude oils, ‘crudes’, have a low sulphur content and flow easily, whereas others may contain wax and flow only when heated, yet others contain very large amounts of very high molecular weight asphalt, Table 1.1. Despite the wide range of hydrocarbons and other organic molecules found in crude oils, the main differences between crudes are not the types of molecules but rather the relative amounts of each type that occur in each crude oil source.

Table 1.1 Variation in crude oil properties between sources

Source	North Sea	Indonesia	Venezuela	Middle East
Sulphur content (%)	0.3	0.2	5.5	2.5
Pour point (°C)	-3	39	9	-15
Viscosity at 40°C (cSt)	4	12	19,000	8

1.2.1 Components of Crude Oil

The components of crude oil can be classified into a few broad categories. Some of these components have properties desirable in a lubricant whilst others have properties which are detrimental.

Hydrocarbons: Hydrocarbons (organic compounds composed exclusively of carbon and hydrogen) predominate in all crude oils and can be further subdivided into the following:

- *alkanes*, known as paraffins, with saturated linear or branched-chain structures,
- *alkenes*, known as olefins, unsaturated molecules, but comparatively rare in crude oils. Certain refining processes produce large amounts of alkenes by cracking or dehydrogenation,

- *alicyclics*, known as naphthenes, are saturated cyclic structures based on five- and six-membered rings,
- *aromatics*, cyclic structures with conjugated double bonds, mainly based on the six-membered benzene ring.

This is a simplified classification because many hydrocarbons can be combinations of these classes, e.g. alkyl-substituted cyclic or mixed polycyclics containing both aromatic and fully saturated rings; examples are shown in Fig. 1.1

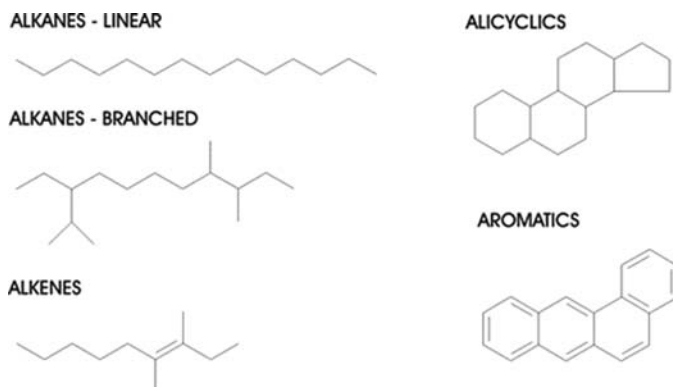


Fig. 1.1 Examples of straight- and branched-chain aliphatic, alkenes, alicyclic and aromatic hydrocarbon structures

Non-hydrocarbons: Many organic compounds in crude oil incorporate other elements, sometimes within ring structures or as functional groups attached to a hydrocarbon structure. Organosulphur compounds are generally much more prevalent than nitrogen- or oxygen-containing molecules, whilst organometallics are usually present as trace compounds. Within the boiling range appropriate to lubricant base oils, almost all organosulphur and organonitrogen compounds are heterocyclic molecules, see Fig. 1.2 for examples. In contrast, the principal oxygen-containing molecules are carboxylic acids as either saturated aliphatic acids or cycloalkanoic acids (naphthenic acids). Traces of phenols and furans may also occur.

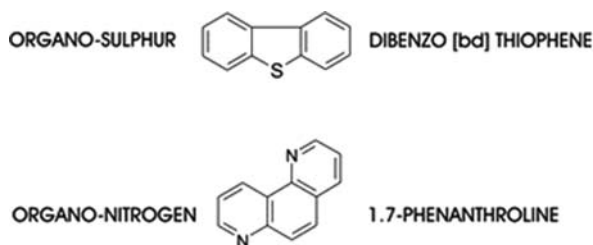


Fig. 1.2 Non-hydrocarbon examples of sulphur- and nitrogen-heterocyclic structures

Finally, there are very high molecular weight resins and asphaltenes which contain a variety of aromatic and heterocyclic structures. Resins are the lower molecular weight, <1000 amu, species, whilst asphaltenes result from linking together many other structures and have exceptionally high molecular weights.

1.2.2 Characteristics of the Hydrocarbons for Lubricant Performance

Only hydrocarbon properties are discussed in this section because most of the non-hydrocarbons are prone to oxidation or degradation and are deleterious to lubricant performance. However, organosulphur molecules are known to act as naturally occurring antioxidants and it is frequently desirable to retain some of these in a refined base oil.

Alkanes, alicyclics and aromatics of the same molecular weight have markedly different physical and chemical characteristics. Physical characteristics affect the viscometrics of the lubricant, and the chemical stability of each class to oxidation and degradation is very important in use.

Alkanes: Of the three main classes, alkanes have relatively low densities and viscosities for their molecular weights and boiling points. They have good viscosity/temperature characteristics, i.e. they show relatively little change in viscosity with change in temperature – see ‘viscosity index’ in Section 1.3.1 – compared to cyclic hydrocarbons. However, there are significant differences between isomers as the degree of alkane chain branching increases, Fig. 1.3.

Linear alkanes, the ‘normal’, or *n*-paraffins in the lubricant boiling range have good viscosity/temperature characteristics but their high melting points cause them to crystallise out of solution as wax. In contrast, highly branched alkanes are not waxy but have less good viscosity/temperature characteristics. There is a compromise region in which acceptable viscosity index, VI, and acceptable low-temperature properties are achieved simultaneously. In general, alkanes also have good viscosity/pressure characteristics, are reasonably resistant to oxidation and have particularly good response to oxidation inhibitors.

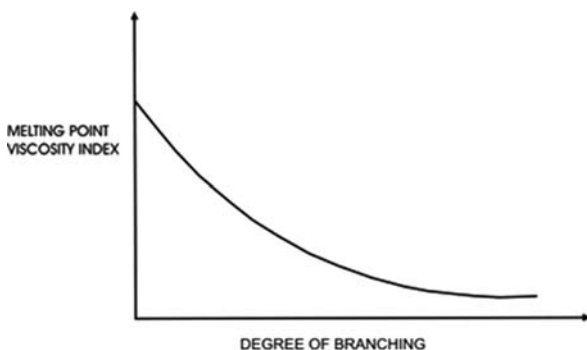


Fig. 1.3 Variation in properties of alkane isomers

Alicyclics have rather higher densities and viscosities for their molecular weights compared to alkanes. An advantage of alicyclics over alkanes is that they tend to have low melting points and so do not contribute to wax. However, one disadvantage is that alicyclics have inferior viscosity/temperature characteristics. Single-ring alicyclics with long alkyl side chains, however, share many properties with branched alkanes and can be highly desirable components for lubricant base oils. Alicyclics tend to have better solvency power for additives than pure alkanes but their stability to oxidative processes is inferior.

Aromatics have densities and viscosities which are yet still higher. Viscosity/temperature characteristics are in general rather poor but melting points are low. Although they have the best solvency power for additives, their stability to oxidation is poor. As for alicyclics, single-ring aromatics with long side chains, alkylbenzenes, may be very desirable base oil components.

1.2.3 Crude Oil Selection for Base Oil Manufacture

Different crude oils contain different proportions of these classes of organic components and also vary in the boiling range distribution of their components. The main factors affecting crude oil selection for the manufacture of base oils are the following:

- content of material of a suitable boiling range for lubricants,
- yield of base oil after manufacturing processes,
- base oil product properties, both physical and chemical.

The manufacturing process at a base oil refinery consists of a series of steps to separate the desirable lube components from the bulk of the crude oil, described in detail in Section 1.4, but briefly, their aims are as follows:

Distillation: removes both the components of too low boiling point and too high boiling point, leaving the lubricant boiling range distillates.

Aromatics removal: leaves an oil that is high in saturated hydrocarbons and improves VI and stability.

De-waxing: removes wax and controls low-temperature properties of the base oil.

Finishing: removes traces of polar components and improves the colour and stability of the base oil.

The yield of base oil after these processes depends on the amount of desirable components in the lubricant boiling range. Lubricant distillates from different crudes can have radically different properties, Table 1.2. Both the Forties and Arabian distillates have relatively high VI and high pour point because they are rich in alkanes and are examples of paraffinic crude oils. Paraffinic crudes are preferred for manufacturing base oils where viscosity/temperature characteristics are important, e.g. for automotive lubricants for operation over a wide temperature range. However,

there is a big difference in sulphur content between these two crude oils and this has an effect on base oil composition and its chemical properties, especially natural oxidation stability. Careful control of the manufacturing processes can minimise some of these differences.

Table 1.2 Comparison of lubricant distillates from a range of crude oils

Crude source	North Sea (Forties)	Middle East (Arabian)	Nigeria (Forcados)	Venezuela Field (Tia Juana)
Viscosity at 40°C (cSt)	16	14	18	23
Pour point (°C)	25	19	18	-48
Viscosity index	92	70	42	10
Sulphur content (wt%)	0.3	2.6	0.3	1.6
Aromatics (wt%)	20	18.5	28	21

The Nigerian and Venezuelan distillates are examples of naphthenic products because they are relatively low in alkane content. In particular, the Venezuelan distillate is wax-free and a de-waxing step is not required. Although naphthenic products have inferior viscosity/temperature characteristics, they have other beneficial properties which are particularly useful in industrial applications.

The examples given are all crude oils regularly used to make base oils but many other crudes do not contain sufficient useful lubricant components and cannot be economically used for conventional base oil production. However, in Section 1.5, a modern catalytic process is described which upgrades distillates of less suitable origin and so creates desirable lubricant components.

1.3 Products and Specifications

1.3.1 Introduction

Lubricants are formulated by blending base oils and additives to meet a series of performance specifications, Chapter 17. These specifications relate to the chemical and physical properties of the formulated oil when it is new and also ensure that the oil continues to function and protect the engine or machinery in service. Self-evidently, lubricant performance is determined by the base oils and the additives used in the formulation.

A range of properties can be measured and used to predict performance when selecting an appropriate base oil for use in formulation. Many of these properties are used as quality control checks in the manufacturing process to ensure uniformity of product quality. Although many of these properties are modified or enhanced by the use of additives, knowledge of the base oil characteristics, especially any limitations, is vital for the effective formulation of any lubricant.

The complexity of the chemical composition of the base oils requires that most measurements are of overall, bulk, physical or chemical properties which indicate

the average performance of all the molecular types in the base oil. Many tests are empirically based and are used to predict, or correlate with, the real-field performance of the lubricant. Although not rigorously scientific, the importance of such tests should not be underestimated.

A wide range of tests was developed by different companies and different countries in the early days of the oil industry. Many tests are now standardized and controlled on an international basis by organisations such as the following:

USA	American Society for Testing and Materials, ASTM,
UK	Institute of Petroleum, IP (now the Energy Institute),
Germany	Deutsches Institut für Normung, DIN,
Europe	Association des Constructeurs Européens d'Automobiles, ACEA,
Japan	Japanese Automotive Standards Organization, JASO,
International	International Organisation for Standards, ISO.

1.3.2 Physical Properties – Viscosity

Viscosity measures the internal friction within a liquid, reflecting the way molecules interact to resist motion. It is a vital lubricant property, influencing the ability of the oil to form a lubricating film or to minimise friction and reduce wear.

Newton defined the absolute viscosity of a liquid as the ratio between the applied shear stress and the resulting shear rate. If two plates of equal area A are considered as separated by a liquid film of thickness D , as in Fig. 1.4, the shear stress is the force F applied to the top plate causing it to move relative to the bottom plate divided by the area of the plate A . The shear rate is the velocity V of the top plate divided by the separation distance D .

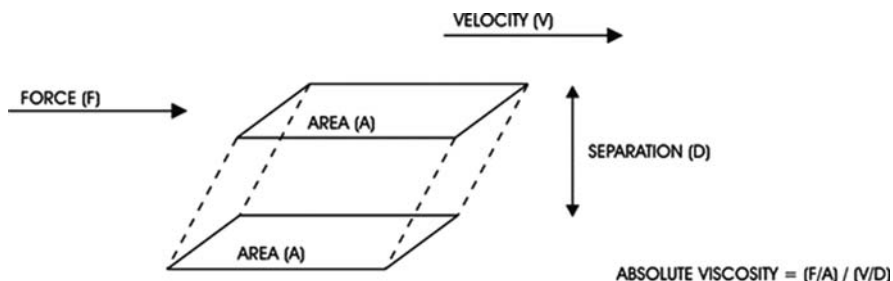


Fig. 1.4 Definition of absolute viscosity

The unit of absolute viscosity is the pascal second (Pa.s), but centipoise (cP) is generally used as the alternative unit, where $1 \text{ Pa.s} = 10^3 \text{ cP}$. Absolute viscosity is usually measured with rotary viscometers where a rotor spins in a container of the fluid to be measured and the resistance to rotation, torque, is measured. Absolute viscosity is an important measurement for the lubricating properties of oils used

in gears and bearings. However, it cannot be measured with the same degree of simplicity and precision as kinematic viscosity, defined as the measurement of liquid flow rate through a capillary tube under the constant influence of force of gravity. Kinematic and absolute viscosities are related by Equation (1.1):

$$\text{Kinematic viscosity} = (\text{Absolute viscosity})/(\text{Liquid density}) \quad (\text{Eqn. 1.1})$$

The unit of kinematic viscosity is m^2/s but for practical reasons it is more common to use the centistoke, cSt, where $1 \text{ cSt} = 10^{-6} \text{ m}^2/\text{s}$. It is routinely measured with ease and great precision in capillary viscometers suspended in constant temperature baths. Standard methods are ASTM D445, IP 71 and several standard temperatures are used. Measuring the kinematic viscosity of a liquid at several temperatures allows its viscosity/temperature relationship to be determined, see immediately below this subsection.

There are other, empirical, scales in use such as SUS (Saybolt Universal Seconds) or the Redwood scales, and conversion scales are available. Base oil grades are sometimes referred to by their SUS viscosities.

Viscosity/temperature relationship – the viscosity index: The most frequently used method for comparing the variation of viscosity with temperature between different oils calculates a dimensionless number, the viscosity index, VI. The kinematic viscosity of the sample oil is measured at two different temperatures, 40 and 100°C, and the viscosity change is compared with an empirical reference scale. The original reference scale was based on two sets of lubricant oils derived from separate crude oils – a Pennsylvania crude, arbitrarily assigned a VI of 100, and a Texas Gulf crude, assigned a VI of 0 [2]. The higher the VI number, the less the effect of temperature on the viscosity of the sample. Full definitions of the calculation methods are given in the ASTM 2270 or IP 226 manuals, summarized in Fig. 1.5. In this figure, L is the viscosity at 40°C of an oil of 0 VI which has the same viscosity at

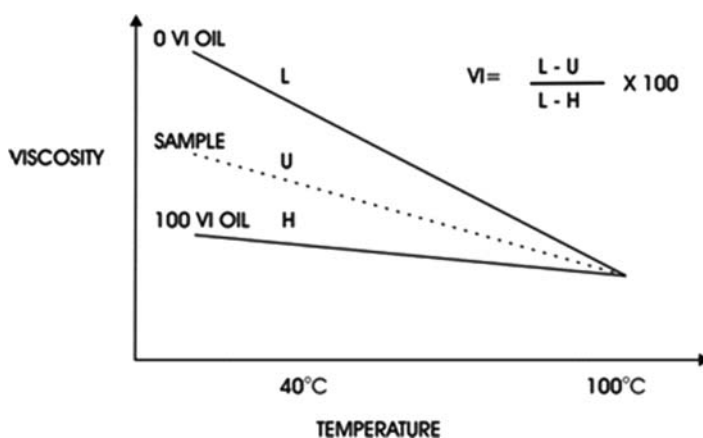


Fig. 1.5 Definition of viscosity index

100°C as the sample under test; H is the viscosity at 40°C of an oil of 100 VI which has the same viscosity at 100°C as the sample under test; and U is the viscosity at 40°C of the oil sample. L and H are obtained from standard tables. A modified procedure applies to oils of VI above 100 or to oils of high viscosity.

The VI scale is a useful tool in comparing base oils, but it is vital to recognise its arbitrary base and limitations. Extrapolation outside the measured temperature range of 40–100°C may lead to false conclusions, especially as wax crystals form at low temperatures. VI is also used as a convenient measure of the degree of aromatics removal during the base oil manufacturing process. But comparison of VIs of different oil samples is realistic only if they are derived from the same distillate feed stock. Therefore, great care should be used in applying VI measurements as indicators of base oil quality.

Low-temperature properties: When a sample of oil is cooled, its viscosity increases predictably until wax crystals start to form. The matrix of wax crystals becomes sufficiently dense with further cooling to cause apparent solidification of the oil. But this is not a true phase change in the sense that a pure compound, such as water, freezes to form ice. Although the ‘solidified’ oil will not pour under the influence of gravity, it can be moved if sufficient force is applied, e.g. by applying torque to a rotor suspended in the oil. Further decrease in temperature causes more wax formation, increasing the complexity of the wax/oil matrix and requiring still more torque to turn the rotor. Many lubricating oils have to be capable of flow at low temperatures and a number of properties should be measured.

Cloud point is the temperature at which the first signs of wax formation can be detected. A sample of oil is warmed sufficiently to be fluid and clear. It is then cooled at a specified rate. The temperature at which haziness is first observed is recorded as the *cloud point*, the ASTM D2500/IP 219 test. The oil sample must be free of water because it interferes with the test.

Pour point is the lowest temperature at which an oil sample will flow by gravity alone. The oil is warmed and then cooled at a specified rate. The test jar is removed from the cooling bath at intervals to see if the sample is still mobile. The procedure is repeated until movement of the oil does not occur, ASTM D97/IP 15. The *pour point* is the last temperature *before* movement ceases, not the temperature at which solidification occurs. This is an important property of diesel fuels as well as lubricant base oils. High-viscosity oils may cease to flow at low temperatures because their viscosity becomes too high rather than because of wax formation. In these cases, the pour point will be higher than the cloud point.

The cold crank simulator test, ASTM D2602/IP 383, measures the apparent viscosity of an oil sample at low temperatures and high shear rates, related to the cold starting characteristics of engine oils, which should be as low as possible. The oil sample fills the space between the rotor and the stator of an electric motor, and when the equipment has been cooled to the test temperature, the motor is started. The increased viscosity of the oil will reduce the speed of rotation of the motor and indicates the apparent viscosity of the oil. The test is comparative for different oil samples rather than an accurate prediction of the absolute performance of an oil in a specific engine.

The *Brookfield viscosity* test measures the low-temperature viscosity of gear oils and hydraulic fluids under low shear conditions. Brookfield viscosities are measured in centipoise units using a motor-driven spindle immersed in the cooled oil sample, ASTM D2983.

High-temperature properties of a base oil are governed by its distillation or boiling range characteristics. *Volatility* is important because it indicates the tendency of oil loss in service by vapourisation, e.g. in a hot engine. Several methods are used to characterise volatility, including the following:

- the *distillation curve*, measured by vacuum distillation, ASTM D1160, or simulated by gas chromatography, ASTM D2887,
- *thermogravimetric analysis*,
- *Noack volatility*, where the sample is heated for 1 hour at 250°C and the weight loss is measured, DIN 51581.

Flash Point: The *flash point* of an oil is an important safety property because it is the lowest temperature at which auto-ignition of the vapour occurs above the heated oil sample. Different methods are used, ASTM D92, D93, and it is essential to know which equipment has been used when comparing results.

Other physical properties: Various other physical properties may be measured, most of them relating to specialised lubricant applications. A list of the more important measurements includes the following:

Density: important, because oils may be formulated by weight but measured by volume,

Demulsification: the ability of oil and water to separate,

Foam characteristics: the tendency to foam formation and the stability of the foam that results,

Pressure/viscosity characteristics: the change of viscosity with applied pressure,

Thermal conductivity: important for heat transfer fluids,

Electrical properties: resistivity, dielectric constant,

Surface properties: surface tension, air separation.

1.3.3 Chemical Properties – Oxidation

Degradation of lubricants by oxidative mechanisms is potentially a very serious problem. Although the formulated lubricant may have many desirable properties when new, oxidation can lead to a dramatic loss of performance in service by reactions such as:

- corrosion due to the formation of organic acids,
- formation of polymers leading to sludge and resins,
- viscosity changes,
- loss of electrical resistivity.

A variety of different stability tests have been devised to measure resistance to oxidation under different conditions which correlate with different service uses of lubricants. Since oxidation inhibitors are frequently added to base oils, response of the base oil to standard inhibitors is an important measurement and therefore some tests are carried out in the presence of standard doses of antioxidants, see Chapter 4. Other tests include catalysts to cause accelerated ageing of the oil and reduce the duration of testing to manageable periods. The sulphur content of base oils is often regarded as a useful indicator of natural oxidation resistance. This is because many naturally occurring organosulphur compounds in crude oil are moderately effective in destroying organic peroxide intermediates and breaking the oxidation chain mechanism. However, the effectiveness of these natural inhibitors is usually rather inferior to synthesized additives which can be much more specific in their action.

Corrosion: A lubricant base oil must not contain components which corrode metal parts of an engine or a machine. The problems of oxidation products leading to corrosion have been mentioned above and corrosion tests usually involve bringing the base oil sample into contact with a metal surface (copper and silver are often used) under controlled conditions. Discolouration of the metal, changes in surface condition or weight loss may be used to measure the corrosion tendency of the oil.

Other tests have been devised to measure corrosion protection properties of the oil under adverse conditions, e.g. in the presence of water, brine or acids formed as combustion products; however, these tests are more applicable to formulated lubricants rather than base oils.

Carbon residue: A test used to measure the tendency of a base oil to form carbonaceous deposits at elevated temperatures. The Conradson carbon residue test, ASTM D189, determines the residue which remains after pyrolytic removal of volatile compounds in the absence of air.

Seal compatibility: Lubricants come into contact with rubber or plastic seals in machines. The strength and degree of 'swell' of these seals may be affected by interaction with the oil. Various tests measure the effects of base oils on different seals and under different test conditions.

1.3.4 Base Oil Categories: Paraffinics

Paraffinic base oils are produced from crude oils of relatively high alkane content; typical crudes are from the Middle East, North Sea and US mid-continent. This is not an exclusive list, nor does it follow that all North Sea crudes, for example, are suitable for production of paraffinic base oils. The manufacturing process requires aromatics removal (usually by solvent extraction) and de-waxing.

Paraffinic base oils are characterised by good viscosity/temperature characteristics, i.e. high viscosity index, adequate low-temperature properties and good stability. In oil industry terminology, they are frequently called solvent neutrals, SN, where 'solvent' means that the base oil has been solvent refined and 'neutral' means

that the oil is of neutral pH. An alternative designation is high viscosity index, HVI, base oil. Most base oils produced in the world are paraffinics and are available over a full range of viscosities, from light spindle oils to viscous bright stock; examples of a range of paraffinic base oils from typical refinery production are given in Table 1.3.

Table 1.3 Paraffinic base oils – typical properties (Arabian crude)

Grade	Spindle	150SN	500SN	Bright stock
Density at 20°C (kg l ⁻¹)	0.85	0.87	0.89	0.91
Viscosity at 40°C (cSt)	12.7	27.3	95.5	550
Viscosity at 100°C (cSt)	3.1	5.0	10.8	33
Viscosity index	100	103	97	92
Pour point (°C)	-15	-12	-9	-9
Sulphur content (wt%)	0.4	0.9	1.1	1.5

Paraffinic base oils of very high viscosity index are also manufactured by severe hydrotreatment or hydrocracking processes, Section 1.5, in which isoalkanes are created by chemical reaction and their crude oil origin is of reduced importance.

Naphthenics are made from a more limited range of crude oils than paraffinics, and in smaller quantities, at a restricted number of refineries. Important characteristics of naphthenic base oils are their naturally low pour points, because they are wax-free, and excellent solvency powers. Their viscosity/temperature characteristics are inferior to paraffinics, i.e. they have low/medium VI, but they are used in a wide range of applications where this is not a problem. Since naphthenic crudes are free of wax, no de-waxing step is needed but solvent extraction or hydrotreatment is often used now to reduce aromatic content and especially to remove polycyclic aromatics which may present a health hazard in untreated oils. The main producers of naphthenics are in North and South America because most of the world's supply of naphthenic lubricant crudes are found there.

Other base oil categories: Base oil refineries produce a range of other products besides their main output of paraffinic or naphthenic base oils. These products are either by-products or speciality products made by additional process steps or by more severe processing; the main types are the following:

White oils: These are highly refined oils which consist entirely of saturated components, all aromatics being removed by treatment with fuming sulphuric acid or by selective hydrogenation. Their name reflects the facts that they are virtually colourless and the most highly refined. White oils are used in medical products and the food industry.

Electrical oils: Oils used in industrial transformers for electrical insulation and heat transfer must have low viscosity and very good low-temperature properties. They are produced either from naphthenic crudes or by urea/catalytic de-waxing from paraffinic crudes.

Process oils: Lightly refined base oils or highly aromatic by-product extracts from oil manufacture are used in various industrial products, e.g. plasticisers in automotive tyres, in printing inks and in mould release oils.

1.3.5 Safety of Petroleum Base Oils

Several studies have shown that certain categories of poorly or untreated petroleum base oils can cause cancer in humans. The principal molecular types believed to be responsible are the three- to seven-ring polycyclic aromatics. The IP 346 test method selectively extracts these materials from a sample of the base oil and enables their concentration to be estimated, fully described in a CONCAWE report [3]. Base oils are now classified according to this test method for their carcinogenic potential and the labelling of finished lubricant products must now comply with these rules.

1.4 Conventional Base Oil Manufacturing Methods

1.4.1 Historic Methods

Very early lubricants were made by the simple distillation of petroleum to recover the lower boiling gasoline and kerosene fractions to give a residue useable as a lubricant. Lubricant quality could be improved by very simple additional processing to remove some of the less desirable components such as asphalt, wax and aromatics. Lubricants of this era relied on the inherent properties of the base oil because virtually no additives were used.

Vacuum distillation separated lubricant distillates from crude oil, leaving the asphalt behind in the distillation residue. Wax used to be removed by chilling the lube distillate and filtering in plate and frame presses. Aromatics were reduced by treating the oil with sulphuric acid and separating the acid tar phase. Finally, finishing treatments such as adsorption of acid residues and impurities by activated clays gave further improvement in product quality. These processes were mainly batch operations, labour intensive and characterised by their hazardous nature. They were unsuitable for the great expansion in production capacity which the industry was called upon to supply.

New technology developed continuous operations so that plants became much larger and could make more consistent quality products at lower cost. These new process methods were based on the use of solvents: continuous selective solvent extraction for aromatic removal was the process which replaced acid treatment and continuous solvent de-waxing replaced the very labour-intensive cold-pressing technique. Technology has developed further in the last 40 years. Catalytic hydrogenation processes have become the normal method for finishing base oils and a more severe form is used as an alternative to solvent extraction to control aromatics content.

With the exception of these newer hydrotreatment processes, all other processes used in modern base oil plants are physical separation techniques, i.e. all the essential constituents of the finished base oil were present in the original crude oil and processing methods are used to concentrate the desirable components by removing the less desirable components as by-products.

1.4.2 Base Oil Manufacture in a Modern Refinery

Most base oil plants are integrated with mainstream oil refineries which produce a range of transportation and heating fuel products. Overall production capacity for lubricant base oils is a very small part of total refinery throughputs, amounting to less than 1.3% in America [4].

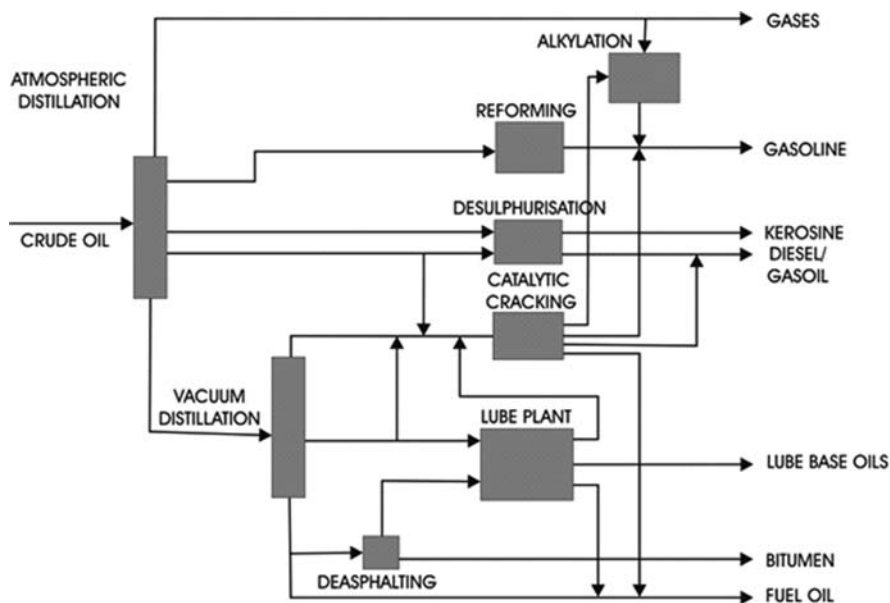


Fig. 1.6 Simplified refinery flow scheme

Figure 1.6 indicates where a lubricant base oil plant fits into the process flow scheme of a 'typical refinery' – if ever there is such a thing. Although the scheme is simplified, the inter-relationship between the base oil plant and other process units and product streams is evident. In a sense, the base oil plant and the fuel-upgrading plant, such as the catalytic cracker, compete for feedstock from vacuum distillation. These interactions are very important to the logistics and production economics of producing base oils.

Base oil manufacture produces large quantities of by-products, the unwanted components of the crude oil. Figure 1.7 is a typical base oil production flow scheme

where the numbers indicate the relative amounts of intermediate and final product throughout the manufacturing process. The basis for the scheme given is processing the residue from the atmospheric distillation of a good-quality Middle East crude. Starting with 100 parts of residue, which in itself represents only about 50% of the original crude oil, even when the maximum possible amount of each base oil is produced, only 24 parts of base oil result. In practice, the demand for grades of different base oils is unlikely to match the possible output of each grade and surplus distillate and residue is returned to the main fuel production part of the refinery. It is quite normal for the actual output of base oil to be less than 10% of the crude oil purchased for making base oil.

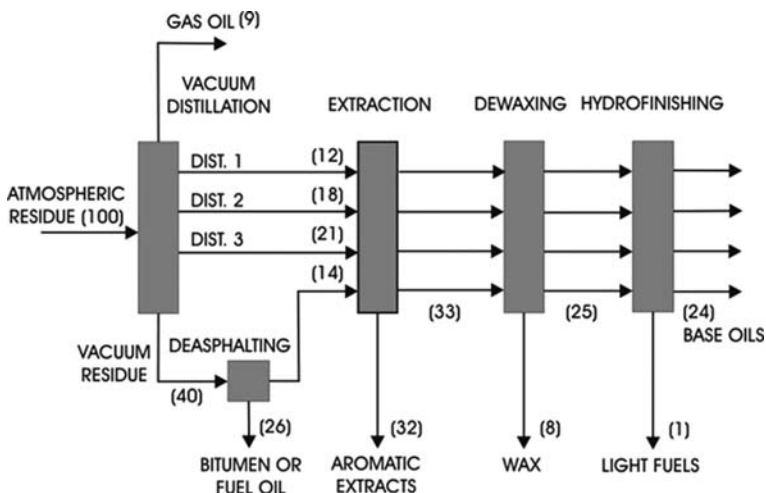


Fig. 1.7 A typical base oil production unit flow scheme

Since the choice of crude oil is restricted when making base oils, the production of relatively small volumes of base oil makes a large imposition on the crude purchasing requirements of a refinery. If suitable crudes are only available at a premium price, then there is an economic penalty for the refinery. Consequently, in recent years refining companies have given considerable effort to expand the portfolio of crude oil which they can use to make satisfactory base oils, giving more flexibility in crude oil purchasing.

1.4.3 Base Oil Production Economics

Each oil refinery is different, with different process units and different relative production capacities arranged in different schemes to make different product ranges. Thus, any view of production economics must, of necessity, be generalised. Production costs can be divided into several categories:

- net feedstock or hydrocarbon cost of making base oil,
- variable operating costs (e.g. energy, chemicals),
- fixed operating costs (e.g. wages, maintenance, overheads),
- costs of capital (e.g. depreciation, interest).

Production costs per tonne of base oil are calculated by dividing the total annual costs by the total annual production of base oils. Net feedstock cost can be calculated in several ways, but it will not necessarily be identical to the cost of crude oil. As the base oil plant in a sense competes with fuel production units for feedstock, the basic feedstock cost to the lubricant base oil complex should be determined by the alternative value of that feedstock if it were used to make mainstream fuels products. The by-products of base oil manufacture also have values for blending into fuel streams or in some cases for direct sale as speciality products, such as waxes and bitumen. Credit must be given for these products so that the net value of the hydrocarbon content of the base oil can be calculated. Refineries use sophisticated linear programming computer models to optimise refinery operations based on different crude oil input, process yields, market prices, production targets, etc.

Variable and fixed operating costs are usually well defined but when these costs are divided by the relatively small output of base oil, they are seen to be significant. If the base oil plant operates below maximum capacity, then the fixed costs have to be shared over an even smaller volume and overall production costs rise in proportion. Energy costs are high because of the number of process steps needed and the energy-intensive nature of equipment such as refrigeration plant and solvent recovery systems. Energy use will vary between refineries, but consumptions as high as 0.4 tonnes fuel oil equivalent per tonne of base oil product are not uncommon.

The costs of capital tend to relate to the age of the base oil plant. A brand new plant has to be financed and since base oil production plant is very expensive to build, depreciation and interest charges will be considerable. Much present day base oil plant is at least 25 years old and so, by now, is almost depreciated. Therefore, for many base oil refineries the cost of making base oil is limited to the hydrocarbon value and operating costs, which make it generally a profitable activity.

1.4.4 Distillation

The primary process for separating the useful fractions for making lubricant base oils from crude oil is distillation. Crude oil is distilled at atmospheric pressure into components of gases, naphtha, kerosene and gas oil, essentially those boiling below 350°C, and a residue containing lubricant base oil boiling range components.

Thermal decomposition increasingly occurs at higher temperatures and further separation by distillation of the atmospheric residue into lubricant base oil is carried out in a vacuum unit, Fig. 1.8. Atmospheric residue feedstock is injected with steam and pre-heated in a furnace before entering the lower part of the vacuum column. Inside the column, a variety of different mechanical arrangements are used to assist separation of different boiling range fractions: