



PROJECT REPORT No. OS18

**THE PHYSICAL
CHARACTERISATION OF
RAPESEED OIL BASED
LUBRICANTS**

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by

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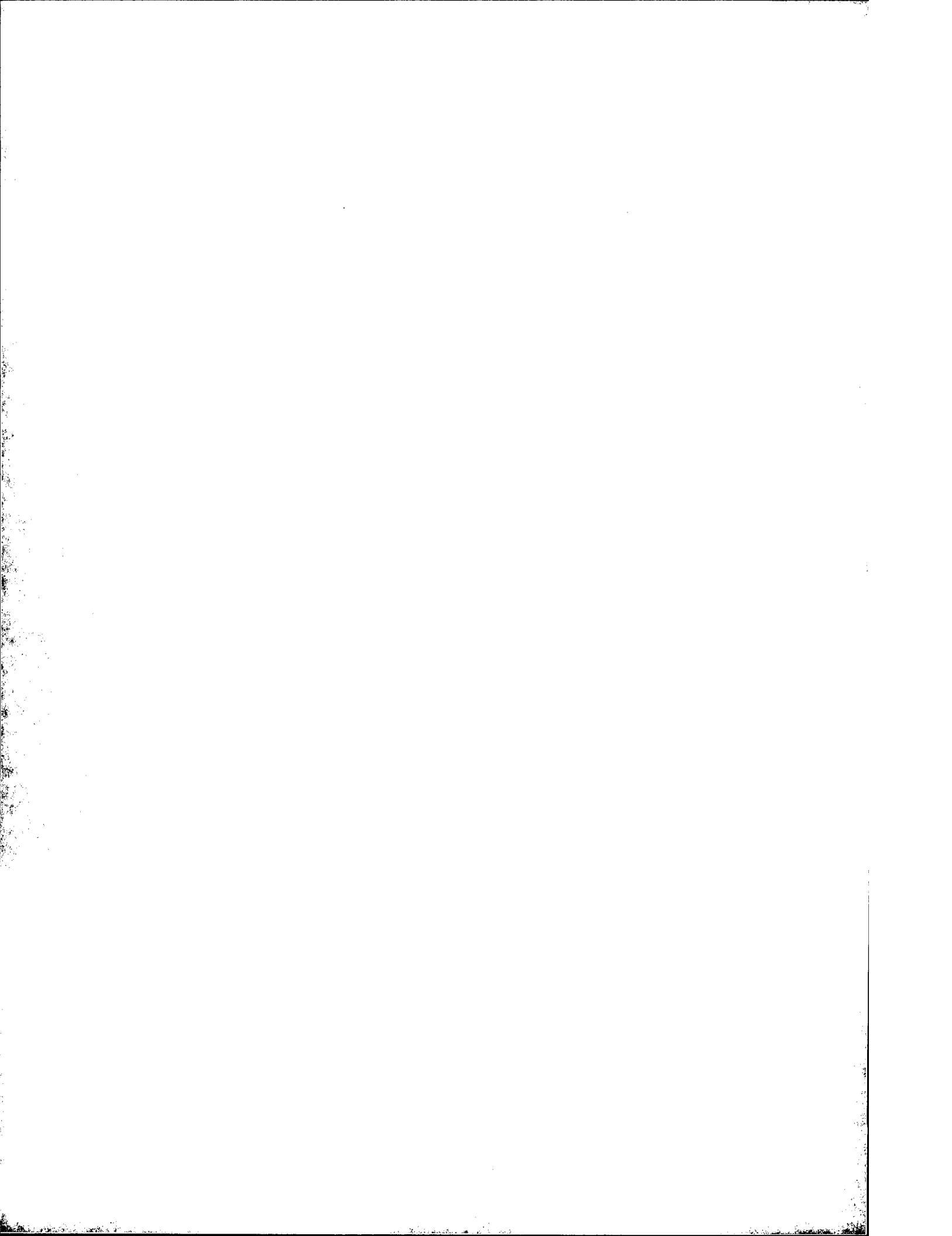
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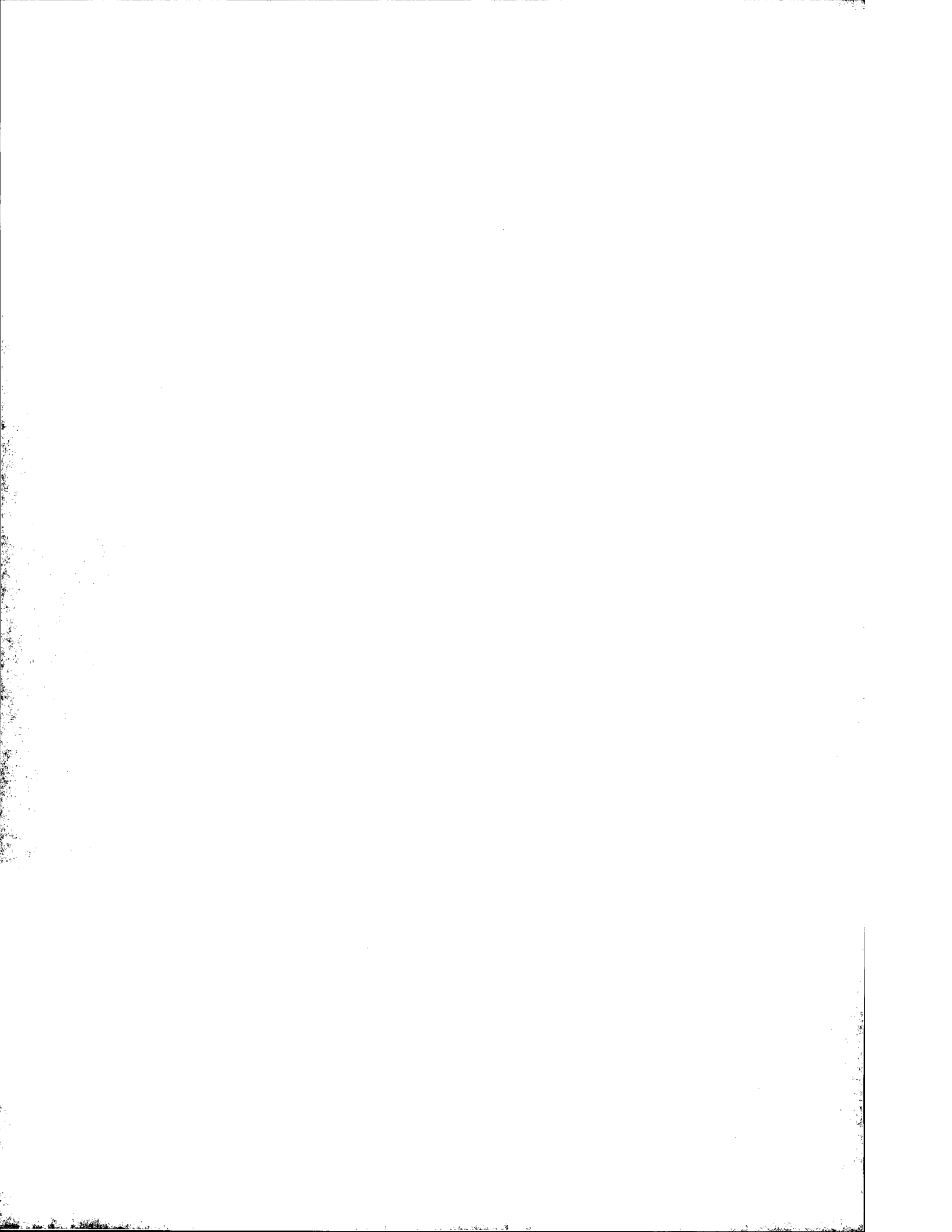
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CONTENTS

	Page
CONTENTS	i
EXECUTIVE SUMMARY	1
ACKNOWLEDGEMENTS	2
1 AIMS & OBJECTIVES	3
2 INTRODUCTION	4
3 BACKGROUND	6
3.1 Biodegradable Lubricant Base Oils	7
3.2 Lubricant Specifications	8
4 PRODUCT DESCRIPTION	12
4.1 Materials	12
4.2 Procedures	12
4.3 Product Profiles	13
4.3.1 The Biolube Range	13
4.3.2 The Envirolube Range	13
4.3.3 Greases: Biogrease/Envirogrease	13
5 RESULTS AND DISCUSSION	14
5.1 Viscometry	14
5.2 Low Temperature Characteristics (Pour Points)	15
5.3 Flash Point	16
5.4 Corrosion Prevention	17
5.5 Foaming Characteristics	18
5.6 Demulsification Characteristics	19
5.7 Anti-Wear Properties	20
5.8 Oxidation Stability/Ageing Properties	21
5.8.1 Addition of Antioxidant	23
5.8.2 Extended Oxidation Test	25
5.9 Hydrolytic Stability	26
5.10 Greases	27



6	FIELD TRIAL/COMMERCIAL POTENTIAL	29
6.1	Biolube 100 Chainsaw Oil	29
6.2	Biogrease	29
6.3	Biolube	29
7	SUMMARY	30
8	REFERENCES	31



EXECUTIVE SUMMARY

The aim of this study was to determine the physical properties of a range of rapeseed oil based lubricating oils and greases.

An increasing environmental awareness has prompted interest in the use of renewable and biodegradable lubricants. Rapeseed oil offers an attractive combination of good low temperature properties with reasonable high temperature stability. It is also compatible with lubricant additives which greatly enhance its performance characteristics.

Using internationally accepted lubricant standards as the required specifications, two series of lubricant base oils and greases were prepared and characterised, ie. the Biolube and Envirolube ranges. These oils were studied in accordance with the test criteria described in the aforementioned lubricant specifications and were found able to meet the necessary standards of viscosity, flash point, pour point, anti-foaming and demulsification; and, on the addition of a relevant additive, also to meet the corrosion prevention and oxidation stability requirements.

After completion of the product development phase of the project, a trial was conducted during which the ability of Biolube 100 to lubricate a chainsaw effectively was assessed. Its lubrication properties were also compared to a commercially available chainsaw oil. Biolube 100 was found to function well under these conditions and gave wear rates, anti-sticking and consumption levels similar to the commercial product. Biogrease is also currently on trial, and this product has shown the ability to withstand the effects of organic solvents.

In summary, therefore, rapeseed oil derived lubricant base stocks have been developed and characterised. These products meet many of the internationally accepted lubricant specifications, and hence could be used as the base oils in a wide range of finished lubricants thus finding application in a very broad range of industries.



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Lubricant samples were provided by Smallman Lubricants Ltd, West Bromwich, and Forest Enterprise Ltd, Edinburgh. Forest Enterprise Ltd also conducted a field trial, and the author is duly grateful to both of these organisations for their support.



1 AIMS AND OBJECTIVES

The aim of this research project was to develop and characterise a series of lubricant base stocks comprising both oils and greases, derived from UK grown, zero erucic acid rapeseed oil.

The principal research objectives, based on both experimental work and literature review, were:

- (i) to formulate products with precise viscometric properties;
- (ii) to minimise the oxidative instability of rapeseed oil;
- (iii) to investigate means of improving the low temperature properties of rapeseed oil;
- (iv) to produce products which meet internationally accepted lubricant specifications;
- (v) not to compromise the ecological benefits offered by rapeseed oil;
- (vi) to initiate new market opportunities for UK grown rapeseed oil.

2 INTRODUCTION

Within Europe, and indeed the wider world community, there is increasing concern for the environmental impact of petroleum-based oleochemicals. Considering lubricants alone, which have a European market of some 4.5 million tonnes/year (1) (a UK contribution of 741,000 tonnes/year (2)), it has been estimated that 13%, ie. over 580,000 tonnes is unaccounted for after use, and presumed 'lost' into the environment. For hydraulic oils the loss is 8%, and for engine oils approaches 34% (3). The European Environmental Agency has calculated that some 260,000 tonnes of oil enter the North Sea each year; 90% of which originates from land sources (4).

These statistics, and others, have prompted several European industrial nations, primarily Germany, Austria and the Scandinavian countries to consider and, in some cases, implement environmental legislation covering the selection, use, labelling and disposal of lubricants (5). In Germany, for example, the contamination of land by the accidental spillage of mineral oil can result in the offender being charged between 1000 and 2000 DM for each cubic metre of land requiring remedial treatment (6).

In response to this legislation, several lubricant companies have developed formulations which are more environmentally benign. These products generally comprise base oils which are more biodegradable than the traditional mineral oil based materials, and can be derived from synthetic esters, eg. adipates, azelates and sebacates or, alternatively, natural esters, eg. vegetable oils.

These ecologically acceptable (EA) fluids are commercially available within the European market (7) and find application in a range of product types, including:

- pump and compressor oils;
- cutting oils;
- chainsaw oils;
- hydraulic oils;
- tractor oils;
- greases;
- two-stroke oils;
- drilling oils.

Sales of these EA products throughout the wider European community remain limited however, and objections such as cost and technical performance, when compared to the traditional mineral oil based materials, have been raised.

On the cost aspect, it has been reported that synthetic esters are between 4 to 15 times more expensive than refined mineral oils. Vegetable oils are more economically competitive, but currently remain between 1.5 to 2 times more expensive than their mineral oil equivalents (2).

On the question of performance, several disadvantages have been highlighted. These drawbacks are found predominantly with the vegetable oil based products, not with the synthetics, and include:

- oxidative instability;
- poorer low temperature properties;
- reduced hydrolytic stability.

The use of lubricant additive technology, eg. antioxidants, pour point depressants, co-solvents (6) and acid scavengers (8) can help to reduce the effects of these shortcomings, and also new varieties of oilseed crops, including a rapeseed variety, with increased levels of oleic acid, have shown improved oxidative and hydrolytic stabilities (9).

Conventional vegetable oils do, however, exhibit certain physical characteristics which make them attractive as lubricant base stock, and excluding their non-toxicity, biodegradability and renewability, include:

- good lubricity;
- a high viscosity index (>200), and therefore retain their viscosity over a wide temperature range;
- high flash point (>300°C);
- low volatility;
- excellent health and safety characteristics;
- the ability to suspend sludge and dirt;
- good seal compatibility.

3 BACKGROUND

The use of vegetable oils as lubricants is by no means a new concept, and has been recorded from as early as 1650BC when the Egyptians used olive oil on wooden planks to facilitate the sliding of large stones, statues and other building materials. Other natural oils such as rapeseed, castor, palm, whale, lard/tallow and lanolin have also been used from this time (10). These materials are very susceptible to chemical/biological oxidation with concomitant deterioration, and in 1791 the French chemist Chaptal wrote: "Oil combines easily with oxygene. This combination is either slow or rapid. In the first case rancidity is the consequence, in the second inflammation" (11). Fortunately, in the case of lubricants, the combination was of the slow variety. Rancidity however, resulted in the lubricants becoming acidic and corrosive to the metal components they were designed to protect.

The Industrial Revolution of the late 18th and 19th centuries stimulated a requirement for cheap, stable lubricants. These were initially a blend of mineral and natural oils, but by the late 19th century, and in particular with the introduction of the petrol powered automobile in 1893, were predominantly mineral oil based.

During the early decades of the 20th century, lubricating oils were simply distillation cuts of crude oil. The expansion of the motor industry in the 1920s and 1930s, however, resulted in developments in lubricant technology. Lubricant additives, ie. chemicals designed to enhance the performance of the base oil, were developed. These were initially wax modifiers, ie. they prevented the crystallisation of wax particles in the oil; by the mid 1940s, however, additives such as detergents, corrosion inhibitors and viscosity index improvers became available. Soon afterwards came friction modifiers, oxidation and foam inhibitors and pour point depressants. The combination of these additives in a mineral based oil, ie. the finished formulation, gave a lubrication performance which was vastly superior to the plain distillation cut which had been the 'high technology' at the beginning of the century (1, 12).

The next significant step in lubricant evolution was the development of man-made alternatives to mineral oils, ie. the so-called synthetics. These now include esters, polyglycols, ethers, phosphate esters, silicones, silicates, chlorofluorocarbons and synthetic hydrocarbons (13).

Although alternatives now exist, mineral oils still remain the most common base oil and are found in over 97% of lubricants (14).

Over the previous 100 years of lubricant technology however, vegetable oils, or, as they are sometimes described, fixed oils (due to their low volatility), have found niche applications. Although seldom used alone, they have been blended into both water based and mineral oil based products to promote lubricity eg. in metal cutting oils. Castor and blown rapeseed oils are the most commonly used vegetable oils in this respect (15).

The increasing environmental awareness of the late 20th century has resulted in a revival in the interest of vegetable oils as lubricant base oils (16). Attempts to gain a market foot hold for such products in environmentally high profile applications have unfortunately met with very limited success. The exception to this slow uptake, however, has been where local authority regulations demand the use of environmentally benign products such as Lake Constance on the Swiss/German border.

Potential areas of application for vegetable oil based lubricants include:

- forestry;
- drilling (on and offshore);
- construction;
- farming;
- dockyards;
- waterways;
- railroads;
- mining;
- construction.

While a slow growth in the demand for biodegradable lubricants was observed for the period 1990-1995, early indications suggest that greater growth should be expected for the future.

3.1 Biodegradable Lubricant Base Oils

Chemical alternatives to mineral oils have been developed over many years in response to specific situations and requirements, eg. oil supply shortages, improved temperature range operation, enhanced fire resistance and environmental concerns. The different chemical categories of lubricant base oils, along with available biodegradability data, are shown in Table 1 (5, 17).

Table 1 Chemical Nature and Biodegradability of Lubricant Base Oils

Chemical Type	Biodegradability (CECL-33-A-93)
Mineral Oil	15-35%
Highly Refined Mineral Oil	24-45%
Synthetic Hydrocarbon (PAO)	5-30%
Natural Esters (including vegetable oils)	75-99%
Synthetic Esters	70-95%
Polyglycols	50-80%
Phosphates	not listed
Silicones	not listed
Water	not applicable

The environmental acceptability of any base oil can be judged on its toxicity, persistence and biodegradability. By current, ecological standards, four of the above base fluids are considered capable of satisfying these demands. These are:

- Synthetic Esters;
- Natural Esters (including Vegetable Oils);
- Polyglycols;
- Water.

It must be stressed, however, that to function effectively as lubricants these base oils require additives which might alter their ecological profile.

In 1990, the total lubricant base oil market in the United States was around 9.5 million litres (18). Currently, world-wide, approximately 80 billion litres of vegetable oil are produced annually (16). Of this renewable resource, 80% is eaten by

humans and a further 6% goes into animal feeds (14). The global vegetable oil capacity is, at first glance therefore, well able to satisfy the demands of the lubricant industry. When the physico-chemical properties of the individual oil types are considered however, the situation is not quite so simple.

A lubricant should be capable of functioning over a broad temperature range and therefore must remain fluid at low temperatures, i.e. sub-zero. Highly saturated fats, eg. coconut and palm kernel oil are solid at room temperature and therefore of limited value as lubricant base stocks. At high temperatures, oxidation processes; potentially leading to a catastrophic increase in viscosity (polymerisation) and the production of acidic residues, must be minimised. Highly polyunsaturated oils, such as soybean and traditional sunflower, are prone to rapid oxidation and deterioration at elevated temperatures. This is a consequence of the rapid oxidation of linoleic and linolenic fatty acids (which comprise 60-70% of the glycerides present in these oils). It has been reported that polyunsaturated fatty acids, eg. linolenic acid, can oxidise more than 70 times faster than mono-unsaturated fatty acids, eg. oleic acid (14).

Olive oil is highly mono-unsaturated (65-85% oleic acid), and hence exhibits good high temperature stability. With a saturated fat content of 10-14% however, it has a solidification point of 0°C. This latter feature, and also because of its relatively high price, have prevented it being developed as a lubricant base oil.

Of the remaining traditional vegetable oils, rapeseed oil offers a good compromise of acceptable low temperature performance and high temperature stability. Although there is variation within the crop, rapeseed oil is approximately 58% mono-unsaturated, 36% polyunsaturated and 6% saturated. World production of rapeseed oil is currently 10.7 million litres annually (19).

A great deal of work has also focused on the development of genetically modified rapeseed and sunflower oils with reduced levels of polyunsaturation and increased levels of mono-unsaturation.

A high oleic sunflower oil is now available which is 87% mono-unsaturated, 4.1% polyunsaturated and 7% saturated. These modified oils, although losing some of their low temperature performance when compared to standard rapeseed oil, exhibit improved high temperature stability and also respond well to antioxidants. They also appear to give better hydrolytic stability than the standard oils (9). These higher performance oils do however, carry a cost premium. Attempts to ascertain the current price of these oils have never been satisfactorily concluded, however, a 1990 price for a high oleic sunflower oil (80% oleic acid) was approximately £1000 per tonne.

3.2 Lubricant Specifications

Lubricants find application in every industry. During operation they provide:

- a reduction in friction;
- cooling;
- cleaning;
- power transmission;
- a reduction in wear;
- an extension of equipment lifetime;
- a reduction in energy requirements.

Lubricants are found in the most delicate, the most robust, the most vital, and the most expensive of mechanical devices, and as such their properties have been optimised to meet the specific performance criteria for any application. All lubricants, irrespective of their composition, can claim some environmental friendliness; since it is generally less ecologically damaging to change a lubricant than to change an entire piece of machinery (6).

Lubricant specifications have been developed by individual nations, equipment manufacturers, armed forces and professional associations, and describe the performance and physical characteristics required for different applications.

Lubricants can be grouped into two broad categories, ie. engine and non-engine. Further breakdown of these two categories gives:

Engine Lubricants

- (i) petrol engine oils;
- (ii) diesel engine oils, ie. automobiles, trains, marine and stationary engines;
- (iii) aviation engines;
- (iv) two-stroke engines.

Non-Engine Lubricants

- (i) transmission fluids, ie. automatic, manual and power;
- (ii) power steering fluids;
- (iii) shock absorber fluids;
- (iv) gear oils, ie. automobiles and industrial;
- (v) hydraulic and general industrial oils;
- (vi) tractor oils;
- (vii) metalworking fluids;
- (viii) greases.

Specifications describe characteristics such as: viscometrics; pour point; flash point; corrosion prevention; foaming inhibition; demulsifiability; load carrying capacity; ageing properties (oxidation tendency); wear prevention and seal compatibility etc.

An example of a lubricant specification is shown Table 2. These particular criteria are often used as the European standard for antiwear hydraulic oils and are taken from the German specification; DIN 51524 Part 2.

Table 2 European Specifications for Antiwear Hydraulic Oils (DIN 51524 Part 2)

	HLP10	HLP22	HLP32	HLP46	HLP68x	HLP100
ISO Viscosity Grade	VG10	VG22	VG32	VG46	VG68	VG100
Viscosity @ 0°C (-20°C), mm ² /s, max.	90(600)	300	420	780	1400	2560
Viscosity @ 100°C, mm ² /s, min.	2.4	4.1	5.0	6.1	7.8	9.9
Pour Point, 0°C, max.	-30	-21	-18	-15	-12	-12
Flash Point (COC), 0°C, min.	125	165	175	185	195	205
Steel Corrosion, max. (DIN 51585)	Class 0 - Method A					
Copper Corrosion, max. (DIN 51759)	Class 2 - 100°C for 3 hours					
Air Release, 50°C, mins, max. (DIN 51381)	5		10		14	
Demulsibility, 54°C, mins, max. (DIN 51599/ASTM D1401)	30	40			60	
FZG A/8.3/90: Load Stage Fail, min.	-		10			
Vane Pump Wear, mg, max. (DIN 51389/2)						
Ring	-		120			
Vane	-		30			
Ageing properties (Maximum increase in Neutralisation number after 1000h) in mg KOH/g	2.0					
Behaviour towards the SRE-NBR 1 sealant specified in DIN 53538 Part 1, after 7 days ±2h @ (100±1)°C	Relative change % in volume	0 to 18	0 to 15	0 to 12	0 to 10	0 to 10
	Change in Shore A hardness	0 to -10	0 to 8	0 to -7	0 to -6	0 to -6
Foam Volume, in ml.						
@ 25°C	150/0					
@ 95°C	75/0					
@ 25°C (after test @ 95°C)	150/0					

The requirements of vegetable oil based lubricants are based on the above specifications but with some noteworthy alterations. The oxidation test has been replaced by a water-free procedure, ie. the Baader oxidation test. The temperature of the seal compatibility test has been reduced from 100°C to 80°C but the duration has been increased from 168 hours to 1000 hours. This change was made because it was assumed that 80°C would be the upper temperature limit for vegetable oils and also because extended seal compatibility tests indicated some adverse effects (5).

The full vegetable oil specification is shown in Table 3.

Table 3 **VDMA 24568 - Vegetable Oil Requirements**

		Requirements			
		HETG 22	HETG 32	HETG 46	HETG 68
ISO Viscosity Class		ISO VG 22	ISO VG 32	ISO VG 46	ISO VG 68
Kinematic viscosity		shall be given by the supplier			
	at -20°C				
	at 0°C max.	300	420	780	1400
	max.	24.2	35.2	50.6	74.8
	mm ² /s at 40°C				
	min.	19.8	28.8	41.4	61.2
	at 100°C min.	4.1	5	6.4	7.8
Low temperature fluidity after 7 days	°C	shall be given by the supplier			
Pour point	°C	shall be given by the supplier			
Flash point	°C	165	175	185	195
Insolubles contents	%	below limit of quantification			
Water content	max. mg/kg	1000			
Steel corrosion test	max.	Degree of corrosion 0 - A			
Copper corrosion test	max.	Degree of corrosion 2-100A3			
Baader oxidation test	95°C/72h				
Increase of viscosity at 40°C	%	<20			
Behaviour towards sealing materials after 1000h	Temperature °C	80			
		HNBR, FOM, NBR1, AU			
HNBR	Change in shore A hardness	±10			
FPM	Relative volume change max. %	-3/+10			
NBR1	Elongation max. %	30			
AU	Force max. %	30			
Air release at 50°C	max.	7		10	
Foam test ml	at 25 °C max.	150/0			
	at 95°C max.	75/0			
	at 25°C max.	150/0			
Demulsification in min.	at 54°C	shall be given by the supplier			
FZG gear test load stage fall		7		10	
Mechanical testing in the vane pump test (mg wear)	ring max. vane max.	120 30			
Density at 15°C	kg/m ³	shall be given by the supplier			
Ash content (oxides)	%	shall be given by the supplier			
Neutralisation value	mgKOH/g	shall be given by the supplier			

The properties of the rapeseed oil based products formulated during this study were based on the specifications described in Tables 2 and 3.

4 PRODUCT DESCRIPTION

4.1 Materials

Rapeseed oil - The oil used throughout the experimental procedures was a zero erucic acid variety. It was purchased locally and comprised 62% mono-unsaturates, 32% polyunsaturates and 6% saturates.

High oleic sunflower oil - This was supplied by an oilseed refiner and comprised 87% mono-unsaturates, 4.1% polyunsaturates and 7% saturates.

The synthetic ester di-2-ethylhexyl adipate was obtained from ICI Chemicals.

White oil, ie. a highly refined mineral oil, was supplied by Pinewood Laboratories Ltd, Clonmel, Ireland.

Lubricant additives, including antioxidants, pour point depressants, viscosity index improvers, anti-rust and anti-wear chemicals, were obtained from several speciality chemical companies.

Commercially available ester/vegetable oil based lubricants were provided by Forest Enterprise Ltd, Edinburgh, and Smallman Lubricants Ltd, West Bromwich.

4.2 Procedures

The bulk of the experimental procedures employed were those described in the above lubricant specifications.

The test procedures used were the following:

• Kinematic viscosity	IP71/ASTM D445
• Pour point	IP15/ASTM D97
• Grease penetration (60 stokes)	IP50/ASTM D217
• Dropping point	IP132/ASTM D566
• Flash point (COC)	IP36/ASTM D92
• Foaming characteristics	IP146/ASTM D892
• Emulsion characteristics	ASTM D1401
• Rust prevention characteristics	IP135/ASTM D665 A
• Copper strip corrosion	IP154/ASTM D130
• Oxidation characteristics	ASTM D2893 (modified)
• Hydrolytic stability	ASTM D2619
• Four-Ball wear	ASTM D4172
• Water washout	ASTM 1264

IP - Institute of Petroleum
ASTM - American Society for Testing and Materials

4.3 Product Profiles

During the course of the project three distinct product groups were developed. The general profile of each of these is described below.

4.3.1 The Biolube Range

This is a series of viscosity enhanced oils within the kinematic viscosity range 35cSt to 250cSt as measured at 40°C. There is, however, scope to increase the upper viscosity limit to over 2000cSt if required. Each individual product contains in excess of 95%v/v rapeseed oil, and is formulated using materials which meet the very highest standards in health and safety, ie. they are food, pharmaceutical and cosmetic industry safe.

4.3.2 The Envirolube Range

This is a family of viscosity modified oils within the kinematic viscosity range 24cSt to 46cSt (40°C). The products have a minimum rapeseed oil content of 80%v/v. Each oil exhibits a viscosity index in excess of 200 (a neat mineral oil is typically 90), and therefore maintains its viscosity over a wide temperature range. Their oxidation stability is generally superior to standard, low erucic rapeseed oil, and is similar to the speciality high oleic oils. These products could find application as the base oils in a range of industrial applications. eg. hydraulic oils or cutting oils.

4.3.3 Greases: Biogrease/Envirogrease

Two greases have been prepared and characterised.

- (i) Biogrease: A general purpose product capable of maintaining its grease texture to temperatures greater than 300°C. It contains in excess of 90% rapeseed oil and is safe for use in food, pharmaceutical or cosmetic industry applications.
- (ii) Envirogrease: A general purpose product with good wear prevention properties and *excellent* water repellency characteristics. This material comprises in excess of 90% rapeseed oil.

The principal objective of the project was to fully characterise the properties of the above base products.

A fully formulated lubricant generally comprises a base oil plus additives, ie. corrosion prevention materials, anti-wear compounds and pour point depressants, etc. In the course of the experimental work, the compatibility and effectiveness of commercial lubricant additives with the above products was examined. In general, no problems were observed with any of the additive combinations used, and this is largely consistent with the findings of others, where only minor incompatibilities have been observed (6).

5 RESULTS AND DISCUSSION

5.1 Viscometry

The importance of viscosity in lubrication cannot be overstated, and is the fundamental measurement which categorises the grade of any product.

Kinematic viscosities are generally determined at 40°C and 100°C. The two data points can be mathematically combined, as described in IP226/ASTM D2770, to give a viscosity index, which is a measure of a product's resistance to viscometric change as its temperature rises.

Low temperature viscosity is also important, for example a product's 'Brookfield Viscosity' (ASTM D2983) which examines viscosity at temperatures as low as -40°C. Such measurements were not carried out during this examination.

National and International bodies, such as the Society of Automotive Engineers (SAE), and the International Standards Organisation (ISO), have established precise viscometric profiles/tolerances for lubricants.

During the formulation of the Biolube and Envirolube oils, the viscosity criteria described in Tables 2 and 3 were used as the required viscometric characteristics for these products.

The kinematic viscosities of the Biolube and Envirolube oils, as determined using the method described in IP71/ASTM D445, along with the viscosity criteria specified in Tables 2 and 3, are shown in Table 4.

Table 4 Biolube/Envirolube Viscosity Data

Base Oil	Test Temperature (°C)	Viscosity (cSt)	Specification (cSt)
Biolube 32	40°C	35.1	28.8 - 35.2
	100°C	7.9	5.0 min
	0°C	210.5	420 max
Biolube 46	40°C	48.6	41.4 - 50.6
	100°C	8.1	6.1 min
	0°C	322.5	780 max
Biolube 68	40°C	64.7	61.2 - 74.8
	100°C	8.2	7.8 min
	0°C	461.7	1440 max
Biolube 100	40°C	92.0	90 - 110
	100°C	8.3	9.9 min
	0°C	882.2	2560 max
Envirolube 22	40°C	24.0	19.8 - 24.2
	100°C	6.0	4.1 min
	0°C	131.7	300 max
Envirolube 32	40°C	35.1	29 - 35.2
	100°C	7.9	5.0 min
	0°C	210.5	420 max
Envirolube 46	40°C	45.8	41.4 - 50.6
	100°C	9.2	6.1 min
	0°C	375.2	780 max

As the above data indicate, the base oils themselves, ie. with no added viscosity modifiers, eg. polymethyl methacrylates, generally meet the required viscosity criteria. The one exception is the Biolube 100 sample at 100°C, where the viscosity is marginally low. This deficit could be easily overcome, however, by the addition of a small quantity of viscosity index improver.

5.2 Low Temperature Characteristics (Pour Points)

The low temperature characteristics of a lubricant, along with, for example, its Brookfield viscosity, define the lower working temperature limits of a product.

The pour point of an oil is the temperature below which it stops flowing. Pour point depressants and co-solvents can be used to reduce these values, and the compatibility of the Biolube and Envirolube oils with a commercial additive was investigated.

Pour points, determined using the method described in IP15/ASTM D97, are shown in Table 5. Relevant specifications are also listed.

Table 5 Biolube/Envirolube Pour Points

Base Oil	Pour Point (°C)	Specification (°C)
Biolube 32 Biolube 32 plus additive	-24 -27	-18 max
Biolube 46 Biolube 46 plus additive	-21 -27	-15 max
Biolube 68 Biolube 68 plus additive	-18 -27	-12 max
Biolube 100 Biolube 100 plus additive	-15 -27	-12 max
Envirolube 22 Envirolube 22 plus additive	-21 -27	-21 max
Envirolube 32 Envirolube 32 plus additive	-27 -30	-18 max
Envirolube 46 Envirolube 46 plus additive	-21 -27	-15 max

It has been well established that vegetable oils generally show poorer low temperature properties, and respond less well to pour point depressants, than either mineral oils or synthetic esters. As the data in Table 5 clearly demonstrates, however, vegetable oils can satisfy low temperature specifications, and on the addition of pour point depressants can meet the criteria with ease.

A cautionary note however is that a prolonged 'cold soak' of a vegetable oil, ie. 7 days at -20°C, generally results in its solidification. This problem can be overcome but

does require a relatively large addition of pour point depressant and a co-solvent, both of which increase the cost of the finished product.

The pour points of several commercially available lubricants (both mineral oil and ester based)* were determined, and are shown in Table 6. For comparison, the pour point of pure rapeseed oil is also shown.

Table 6 Pour Points of Commercially Available Lubricants

Sample	*Base Oil Chemistry	Viscosity Grade (ISO)	Pour Point (°C)
Hydraulic oil	Mineral oil	32	-15
Chain oil	Ester	46	<-30
Chainsaw oil	Mineral oil	150	-12
Chainsaw oil	Mineral oil	100	-12
Chainsaw oil	Ester	68	<-30
Chainsaw oil	Ester	68	-18
Chainsaw oil	Ester	68	-21
Two-stroke oil	Mineral	68	-24
Rapeseed oil	Triglyceride	32	-21

* No information was available as to the nature of the esters found in these products, ie. whether they were triglycerides, transesterified vegetable oils or synthetics.

The data shown in Table 6 clearly indicate that commercial products are available which exhibit higher pour points than neat rapeseed oil, and hence the perceived low temperature disadvantages of vegetable oils are perhaps not of such great concern.

5.3 Flash Points

The flash point of a lubricant gives information on two features: the volatility of the product and any associated fire risk.

There are several techniques available for determining an oil's flash point, and in this study the method described as the Cleveland Open Cup, ie. IP36/ASTM D92 was employed. The test oil is slowly heated, and the lowest temperature at which application of a flame results in the vapour igniting is taken as the flash point.

Flash point data for the Biolube and Envirolube oils are given in Table 7, relevant specifications are also shown.

Table 7 Biolube/Envirolube Flash Points

Sample	Flash Point (°C)	Specification (°C) minimum
Biolube 32	>320	175
Biolube 46	>320	185
Biolube 68	330	195
Biolube 100	330	205
Envirolube 22	268	165
Envirolube 32	296	175
Envirolube 46	296	185

The high molecular weight and low volatility of rapeseed oil results in these high flash points. As the data in Table 7 clearly demonstrate, these base oils easily meet the required specifications.

5.4 Corrosion Prevention

Contamination of an oil with water can result in the rusting of ferrous metal components. An effective lubricant should be capable of inhibiting rust and this is generally achieved by the addition of rust inhibiting chemicals.

Pumps and bearings often contain copper alloys in their component parts. As well as inhibiting rusting of ferrous metals, an effective lubricant should also retard the corrosion of copper.

Standard tests have been developed to assess the corrosion prevention properties of lubricants to both ferrous and copper metals and these are described in IP135/ASTM D665 and IP154/ASTM D130 respectively.

Within these test regimes there is a number of options available. In the rust test, Method A, which uses distilled water, or Method B, which utilises synthetic sea water, can be chosen. In the copper test the duration is generally 3 hours, but the test temperature can be varied, for example, 100°C, 121°C or 150°C. The final make up of the test programme will generally depend on the specific application of interest.

In this study, Method A was selected for assessing rust prevention characteristics, and a test temperature of 100°C was used for copper corrosion testing.

The results obtained are shown in Table 8.

Table 8 Biolube/Envirolube Corrosion Prevention

Sample	Rust Prevention (A)	Copper Corrosion* (100°C)	Specification	
			Rust	Copper
Biolube 32 Biolube 32 plus additive	Fail - Moderate Pass	1a	Pass	2
Biolube 46 Biolube 46 plus additive	Fail - Moderate Pass	1a	Pass	2
Biolube 68 Biolube 68 plus additive	Fail - Moderate Pass	1a	Pass	2
Biolube 100 Biolube 100 plus additive	Fail - Moderate Pass	1a	Pass	2
Envirolube 22 Envirolube 22 plus additive	Fail - Moderate Pass	1a	Pass	2
Envirolube 32 Envirolube 32 plus additive	Fail - Moderate Pass	1a	Pass	2
Envirolube 46 Envirolube 46 plus additive	Fail - Moderate Pass	1a	Pass	2

- * 1 a, b - slight tarnish
 2 a, b, c, d, e - moderate tarnish
 3 a, b - dark tarnish
 4 a, b, c - corrosion

The uninhibited samples all gave a moderate rusting (less than 5% of metal surface) of the test species. On the addition of a rust prevention agent however, all the samples were able to pass the test.

The copper corrosion study indicated a negligible change in the appearance of the copper strip test species. The test method does not however permit a 'no change' result, and hence the samples were rated at 1a.

5.5 Foaming Characteristics

The foaming characteristics of a lubricating oil can be empirically rated using the test method described in IP140/ASTM D842. This procedure determines both the foaming tendency of an oil and, by virtue of allowing the foamed oil to stand (generally for 10 minutes), also the foam stability.

The test is run at two temperatures, ie. 25°C and 95°C, and uses initially a fresh oil sample at 25°C, a second fresh oil sample at 95°C, and then a repeat of the test at 25°C but using the oil previously tested at 95°C.

The data are reported as Sequence I, Sequence II and Sequence III, with the foam tendency result, and the foam stability figure separated by an oblique line eg. 150/0. This result would imply an initial foam volume (foam tendency) of 150ml which had collapsed to 0ml after 10 minutes standing at the test temperature.

The results obtained for the Biolube and Envirolube base oils are given in Table 9. Relevant vegetable oil specifications (taken from Table 3), are also shown.

Table 9 Biolube/Envirolube Foaming Characteristics

Sample	Sequence	Tendency/Stability (ml)	Specification (ml)
Biolube 32	I 25°C	5/0	150/0
Biolube 32	II 95°C	5/0	75/0
Biolube 32	III 25°C	5/0	150/0
Biolube 46	I 25°C	5/0	150/0
Biolube 46	II 95°C	5/0	75/0
Biolube 46	III 25°C	5/0	150/0
Biolube 68	I 25°C	5/0	150/0
Biolube 68	II 95°C	5/0	75/0
Biolube 68	III 25°C	5/0	150/0
Biolube 100*	I 25°C	5/0	150/0
Biolube 100*	II 95°C	5/0	75/0
Biolube 100*	III 25°C	5/0	150/0
Envirolube 22	I 25°C	5/0	150/0
Envirolube 22	II 95°C	5/0	75/0
Envirolube 22	III 25°C	5/0	150/0
Envirolube 32	I 25°C	5/0	150/0
Envirolube 32	II 95°C	5/0	75/0
Envirolube 32	III 25°C	5/0	150/0
Envirolube 46	I 25°C	5/0	150/0
Envirolube 46	II 95°C	5/0	75/0
Envirolube 46	III 25°C	5/0	150/0

* Specifications shown in Table 2

The foaming tendency of these base oils gave no cause for concern, minimum foam was produced which rapidly collapsed.

5.6 Demulsification Characteristics

The scope of this test is to evaluate the ability of a lubricating oil to separate from water. Briefly, the test involves stirring 40ml of oil with 40ml of water for 5 minutes in a graduated cylinder. The quantities of 'free' oil, 'free' water, and emulsion, are then recorded every 5 minutes until the volume of emulsion has reduced to 3ml or less. The complete procedure is given in the standard method ASTM D 1401.

The data obtained for the Biolube and Envirolube base oils are shown in Table 10. The relevant specification is also shown.

Table 10 Biolube/Envirolobe Demulsification Characteristics

Sample	Time for Emulsion to Reduce to 3ml or less (minutes)	Specification (minutes) maximum
Biolube 32	5	40
Biolube 46	5	40
Biolube 68	5	60
Biolube 100	5	60
Envirolobe 22	5	40
Envirolobe 32	5	40
Envirolobe 46	5	40

Both the Biolube and Envirolobe base oils proved to be very hydrophobic and rapidly separated from the water to leave a minimum emulsion.

5.7 Anti-Wear Properties

A major role of a lubricant is to minimise the wear rate of the piece of equipment it lubricates. There is a large range of test procedures available to assess the anti-wear performance of a lubricant. In this instance, the procedure used was the 'Four-Ball Wear Test'. This test is non-application specific but is widely used to make a preliminary evaluation of a product.

The test procedure is described in ASTM D 4172, but, briefly, consists of clamping three 12.7mm diameter steel balls together and then covering them with the test lubricant. A fourth ball is pressed down with a known force (147N or 392N) (15kg or 40kg) onto the three fixed balls and rotated at 1200rpm for 60 minutes. The test temperature is generally 75°C. At the end of the test period the average sizes of the scar diameters worn on the three fixed balls are recorded.

Currently, data are only available on two products, ie Biolube 32 and Envirolobe 46, the results are shown in Table 11.

Table 11 Biolube 32/Envirolobe 46 Four-Ball Wear

Sample	Scar Diameter (mm) 40kg, 75°C, 60 minutes
Biolube 32	0.80
Envirolobe 46	0.69

No relevant specification describing base oil Four-Ball Wear performance was available. Literature data describing the anti-wear characteristics of speciality vegetable oils (viscosity grade 46, cf Envirolobe 46) were however available (20) and gave scar diameters of 0.75-0.78mm. The result of 0.69mm for Envirolobe 46 is therefore very encouraging and an outcome of 0.80mm for Biolube 32 is also promising.

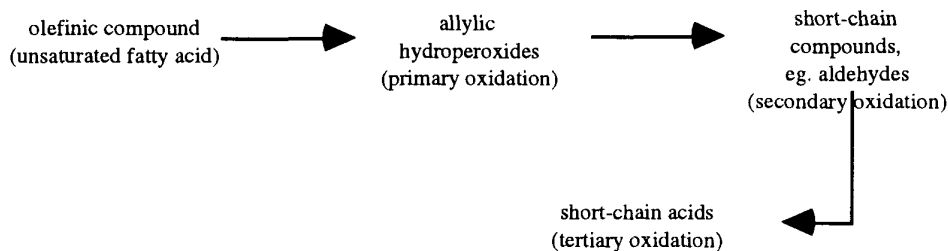
Gear oil and tractor oil specifications are in the range 0.35-0.5mm which is obviously significantly less than the speciality oils or the Biolube/EnviroLube results. However these speciality vegetable oils, with the addition of appropriate additives (1, 19), can meet these more rigorous specifications and there is every reason to assume the same will be true of the Biolube and EnviroLube oils.

5.8 Oxidation Stability /Ageing-Properties

The oxidative instability of vegetable oils has been referred to in previous chapters, and is a consequence of their chemical unsaturation.

In common with all other olefinic compounds oils and fats react with oxygen. This phenomenon is well known in the food industry, where the oxidation processes result in the undesirable tastes and odours which characterise fat rancidity. In some applications however, for example, putty, mastics, inks and paints etc. rapid oxidation and the concomitant formation of a dry, hard surface is desirable. Oils which undergo rapid oxidation are generally described as drying oils and are rich in polyunsaturated fatty acids, eg. linseed oil. Oils which undergo a 'slower' oxidation are described as semi-drying oils and normally comprise di-unsaturated fatty acids, eg. sunflower oil. Oils which undergo a slow oxidation are described as non-drying, and are found to contain predominantly mono-unsaturated or saturated fatty acid glycerides. Rapeseed oil falls into this final category.

The entire chemical mechanism of unsaturated fatty acid oxidation is complex, but generally goes via the route (14):



The latter process, tertiary oxidation, is generally only found when the fat/oil has been subjected to elevated temperatures, for example during cooking, or in lubricant application.

The control/reduction of oxidation during use is probably the major challenge to those groups wishing to develop vegetable oil based lubricants. Additive technology and the use of highly mono-unsaturated oils, for example high oleic sunflower oil, have indicated that order-of-magnitude improvements can be made in the oxidation stability of vegetable oils, and, depending on the test method used, eg. ASTM D 2272, oxidation stabilities comparable to inhibited mineral oils can be achieved (1).

The oxidation of a lubricant during use can result in changes in viscosity, acidity and sludge levels etc. and can thus have a profound effect on the performance characteristics of the product. It is, therefore, vital that the oxidation stability of any lubricant be known.

There are several standard oxidation tests available for the evaluation of lubricants, all of which follow a general theme, ie. they are carried out at elevated temperature, will usually include the use of an air/oxygen stream and may utilise the catalytic nature of

metals, eg. copper. Examination of the lubricant, such as viscosity increase, precipitation number, sludge accumulation, volatile and soluble acidity levels, along with any measured changes in the catalyst, are used to determine the oxidation stability of the product.

A widely used test is that described in ASTM D943. This involves the use of 300ml of oil, 60ml of water, a metal catalyst and an oxygen stream of 3 litres/hour. The test is carried out at 95°C and is continued until the acid number of the oil is 2.0mgKOH/g or greater.

Under these conditions vegetable oils 'fail' after ca 24-40 hours. Additives can increase their lifetime to ca 300 hours, but this is still well short of the 3000+ hours expected of a mineral oil based formulation.

The failure of vegetable oils under these conditions is largely due to the inclusion of water. Vegetable oils undergo relatively rapid hydrolysis under these conditions to yield free fatty acids and hence their acid number quickly rises to greater than 2.0mgKOH/g*

An alternative, water-free, evaluation, ie. the Baader oxidation test (DIN 51554) has been developed for hydrolytically sensitive base oils, and for vegetable oils is usually carried out at 95°C.

Unfortunately the Baader test was outwith the scope of this present evaluation, however an alternative method, ie. ASTM D2893 (also a water-free technique), was used to evaluate the oxidative stabilities of the Biolube and Envirolube base oils.

A second attractive feature of this test was that relevant vegetable oil data were available in the literature (20).

In its simplest form, the selected oxidation procedure (ASTM D2893) consists of the following:

The oil sample, 300ml, is subjected to a temperature of 95°C for 312 hours whilst being 'blown' with 10 litres per hour of dry air. Viscosity increase and precipitation number are reported.

The test duration can, however, be decreased to 50 hours by increasing the temperature to 121°C and replacing the air with oxygen. This modified procedure gives the same degree of oxidation severity as the basic test (1). The modified procedure was used during this study.

The data obtained after 50 hours oxidation for the neat base oils, ie. no added antioxidants, are shown in Table 12. For comparison, the results obtained for a mineral oil, a semi-synthetic ester, a synthetic ester and several other vegetable oils, including a high oleic sunflower oil (HOS), are also shown.

* This level of acidity would be reached after ca 1.0% of the available glyceride bonds were hydrolysed.

Table 12 Base Oil Oxidation Stabilities (No added antioxidants)

Base Oil	*Kinematic Viscosity (40°C)			Final Soluble Acidity* (mgKOH/g)
	Initial (cSt)	Final (cSt)	Final Initial	
Biolube 32	35	406	11.6	6.7
Biolube 46	43	284	6.6	4.2
Biolube 68	64	169	2.7	7.1
Biolube 100	92	397	4.3	5.3
Envirolube 22	24	117	4.8	15.6
Envirolube 32	35	243	6.9	1.6
Envirolube 46	46	359	7.8	8.1
Synthetic Ester	7.8	7.6	0.97	0.24
Semi-Synthetic Ester	4.5	37	8.2	Δ
Mineral Oil	85	138	1.6	5.3
High Oleic Sunflower Oil	42	284	6.7	16.6
Olive Oil	37	219	5.9	16.2
Standard Sunflower Oil	27	1144	42.3	8.5

* Average of three repeats

Δ Not quantified due to severe discoloration of the oil

The results show that the synthetic ester is particularly stable to oxidation showing only a minimal change in viscosity and acidity. The mineral oil was the second most stable in terms of viscosity change, but, interestingly, gave a level of acidity higher than the Biolube 46 and the Envirolube 32 base oils.

Of the vegetable base oils studies, Biolube 68 underwent the lowest relative viscosity change followed by Biolube 100, Envirolube 22 and olive oil. Biolube 46, the high oleic sunflower oil and Envirolube 32 gave similar results, but, interestingly the HOS gave a very high level of acidity, as did the olive oil. Both the HOS and the olive oil have very low levels of polyunsaturation (4-5%) and this high acidity level may reflect this, ie. oils with very low levels of polyunsaturation might possibly oxidise to give acidic residues rather than high viscosity polymeric products.

The standard sunflower oil underwent the greatest change in viscosity, with this being due to its highly polyunsaturated nature.

For the vegetable oils therefore, the order of decreasing oxidation stability (based solely on viscosity results) is (most stable first):

Biolube 68 > Biolube 100 > Envirolube 22 > olive oil > Biolube 46 ≈ high oleic sunflower ≈ Envirolube 32 > Envirolube 46 > Biolube 32 >> standard sunflower.

5.8.1 Addition of Antioxidant

The compatibility of the base oils with an antioxidant was assessed, and the data obtained are shown in Table 13.

For comparison, the results obtained for eight commercially available lubricants (including two mineral oil based samples) are also shown.

Table 13 Biolube/Envirolube Antioxidant Compatibility

Base Oil	*Kinematic Viscosity (40°C)			Final Soluble Acidity* (mgKOH/g)
	Initial (cSt)	Final (cSt)	Final Initial	
Biolube 32	35	406	11.6	6.7
Biolube 32 plus antioxidant	35	35	1.0	0.47
Biolube 46	48	284	6.6	4.2
Biolube 46 plus antioxidant	48	58	1.2	1.96
Biolube 68	64	169	2.7	7.1
Biolube 68 plus antioxidant	64	68	1.1	1.1
Biolube 100	92	397	4.3	5.3
Biolube 100 plus antioxidant	92	92	1.0	1.1
Envirolube 22	24	117	4.8	15.6
Envirolube 22 plus antioxidant	24	25	1.0	1.5
Envirolube 32	35	243	6.9	1.6
Envirolube 32 plus antioxidant	35	35	1.0	1.8
Envirolube 46	4.6	359	7.8	8.1
Envirolube 46 plus antioxidant	4.6	46	1.0	1.76
Commercial Product A	41	71	1.7	*
Commercial Product B	84	1095	13	9.2
Commercial Product C	55	254	4.6	*
Commercial Product D	62	180	2.9	*
Commercial Product E	37	43	1.1	0.9
Commercial Product F	46	46	1.0	1.7
Commercial Product G ^Δ	103	77	0.74	*
Commercial Product H ^Δ	32	32	1.0	0.05

Δ Mineral oil based

* Too discoloured to quantify

The data shown in Table 13 clearly indicate that both the Biolube and Envirolube oils respond well to antioxidants. By comparison with the uninhibited oils, a very marked reduction was found for both the viscosity increase and the level of acidity after oxidation.

For the commercial oils, only samples E, F, G and H indicated similar levels of stability to the inhibited Biolube/Envirolube oils. The other oils, ie. A-D showed a much greater relative increase in their viscosities and, with the exception of sample B, became very black.

In general, an indication of good oxidation stability under these conditions would be:

- Viscosity increase <10.0%
- Acidity 2.0mgKOH/g (max)

The results obtained for both the Biolube and Envirolube oils show that these performance specifications can be attained with the use of suitable antioxidants.

5.8.2 Extended Oxidation Test

The oxidation data previously reported were obtained after a 50 hour test duration. In this study the 'longer-term' stability of the oils was assessed by extending the oxidation period to 150h.

Results (viscosity and acidity) were obtained for the Biolube and Envirolube oils, and the high oleic sunflower oil. No antioxidants were added to these samples. The viscosity and acidity data are shown in Tables 14 and 15 respectively. For comparison, the 50 hour data are again reported.

Table 14 Extended Oxidation - Viscometry

Sample	Test Duration (hours)/Viscosity (cSt, 40° C)			
	0h	50h	150h	150 0h
Biolube 32	35	406	792	22.6
Biolube 46	43	285	415	9.6
Biolube 68	64	169	477	7.6
Biolube 100	92	397	512	5.5
Envirolube 22	24	117	519	21.6
Envirolube 32	35	243	1231	35.1
Envirolube 46	43	359	455	10.5
High Oleic Sunflower Oil	42	284	1086	25.8

Table 15 Extended Oxidation - Acidity

Sample	Test Duration (hours)/Acidity (mgKOH/g)		
	0h	50h	150h
Biolube 32	0.11	6.7	13.1
Biolube 46	0.11	4.2	13.8
Biolube 68	0.11	7.1	20.0
Biolube 100	0.11	5.3	19.2
Envirolube 22	0.25	15.6	27.4
Envirolube 32	0.11	1.6	8.4
Envirolube 46	0.11	8.1	12.8
High Oleic Sunflower Oil	0.11	16.6	29.1

After 150 hours oxidation all of the samples had undergone a very marked degree of change. The order of decreasing oxidation stability (based on relative viscosity change) was:

Biolube 100 > Biolube 68 > Biolube 46 > Envirolube 46 > Envirolube 22 > Biolube 32 > high oleic sunflower > Envirolube 32.

5.9 Hydrolytic Stability

It should be assumed that a lubricant will become exposed to water sometime during its use. Instability in a wet environment can lead to corrosion, pumping and filterability problems and a shortened fluid and equipment lifetime. It is therefore essential to have knowledge of the hydrolytic stability of any lubricant.

The hydrolytic stability of an oil can be assessed using the technique described in ASTM D2619.

Briefly, the test is conducted by sealing the test oil (75g), distilled water (25g) and a copper strip in a beverage bottle (6oz) and rotating it, end-over-end, for 48 hours in an oven at 200°F (93.3°C).

The acid content for both the oil and water layers and the appearance of the copper strip are recorded.

The test was carried out on the full range of Biolube and Envirolube base oils. For comparison, data were also recorded for a high oleic sunflower oil and a mineral oil. The results obtained are shown in Table 16.

Table 16 Hydrolytic Stability

Sample	Final Acidity (mg/KOH/g)		Copper Strip	
	Oil Layer	Aqueous Layer	Weight loss	Appearance
Biolube 32	0.13	0.22	0.3	1b
Biolube 46	0.44	0.15	0.3	1b
Biolube 68	0.44	0.03	0.1	1b
Biolube 100	0.44	0.03	0.0	1b
Envirolube 22	1.1	0.02	0.2	1a
Envirolube 32	0.25	0.34	0.3	1b
Envirolube 46	0.22	0.27	0.1	1b
High Oleic Sunflower	0.38	0.20	0.2	2a
Mineral	0.01	0.01	0.0	2a

1a, b - Slight tarnish
2a - Moderate tarnish

By comparison with the mineral oil, all of the vegetable oil based materials underwent significantly greater hydrolysis. It has been reported (8) that acid scavengers can be utilised to reduce the hydrolytic instability of vegetable oils.

5.10 Greases

Conventional greases consist of a non-biodegradable mineral oil base, into which thickeners are blended, eg. lithium, calcium or aluminium soaps, and additives, eg. corrosion and anti-wear compounds.

Most greases are identified by their thickeners, eg. lithium based, and are classified according to a system developed by the National Lubricating Grease Institute (NLGI). This system was first introduced in 1991, and, although specifically relating to automotive applications (chassis and wheel bearings), is widely recognised throughout industry.

Greases bearing the NLGI mark have been tested in accordance with ASTM D4950 and have been found to comply with performance specifications for wheel bearings (GC), chassis (LB), or dual-service (GC-LM) applications. The full grease testing regime is shown in Table 17.

Table 17 Tests for Characterising Greases

Property	Test Method	Description
Shear Stability	ASTM D 217 ASTM D 1831 ASTM D 4290	Multistroke penetration Roll stability Wheel bearing leakage
Oxidation Resistance	ASTM D 942 ASTM D 3527 ASTM D 3336	Bomb oxidation Wheel bearing life High-temperature performance
Water Resistance	ASTM D 1264 ASTM D 4049	Water washout Water spray-off
Bleed Resistance	FTM 321.3 ASTM D 1742	Oil separation (static) Pressure Oil separation
Extreme-Pressure/Antiwear	ASTM D 2596 ASTM D 2509 ASTM D 2266	Four-ball Timken method Four-ball wear
Corrosion	ASTM D 1743 - ASTM D 4048	Rust test Emcor Copper corrosion
Pumpability	ASTM D 4693 US Steel LT37	Low-temperature torque Mobility
Identification & Quality Control	ASTM D 2265	Dropping Point

Completion of this entire suite of tests was outwith the scope of this initial study, sufficient data were, however, obtained to classify the rapeseed oil based products (Biogrease and Envirogrease) in terms of their consistency, upper temperature limit, wear prevention properties and water repellency. The tests were run on the basic grease, ie. without additives. Since the greases were based on the same rapeseed oil as had been used during the 'oil' study, it is therefore reasonable to assume that they

will also be compatible with lubricant additives, and hence their performance could be markedly improved if required.

The test data and relevant specification are shown in Table 18. A brief summary of each test method follows.

(i) Penetration - IP50/ASTM D217

Data from this method gives information on the consistency of the product. A standard cone is allowed to drop freely into the grease for 5 seconds. The maximum penetration of the cone, measured in tenths of millimetres, is recorded. The test is done at 25°C and is preferably carried out on a sample of grease which has been 'worked' on a 'Grease Worker'.

(ii) Dropping Point- IP32/ASTM 2265

This procedure gives the temperature at which the grease passes from a semi-solid to a liquid state.

(iii) Wear Prevention - ASTM D2266

Results from this procedure give an evaluation of the anti-wear properties of the grease. The method is similar to the four-ball method described earlier.

(iv) Water Washout - ASTM D1264

This method estimates the resistance of greases to water washout, and hence is a measure of the suitability of a grease to a wet environment. The grease is packed into a bearing, rotated at 600rpm and sprayed with water (5ml/sec) at a specified temperature (80°C) for one hour. The weight loss of grease from the bearing is recorded.

Table 18 Biogrease/Envirogrease Test Data

Procedure	Biogrease	Envirogrease	NLGI Specification
Worked Penetration (mm/10)	230	308	220-340
Dropping Point (°C)	>300	246	220
Wear Prevention (mm)	0.664	0.630	0.9
Water Washout % (80°C)	*	2.01	15 max.

* Biogrease is intended only for dry environments

Within the limits of the tests carried out, both the Biogrease and Envirogrease met the necessary specifications.

6 FIELD TRIAL/COMMERCIAL POTENTIAL

6.1 Biolube 100 Chainsaw Oil

After completion of the initial product development phase of the project, a trial was carried out by Forest Enterprise Ltd, Edinburgh, which compared the chainsaw lubrication performance of Biolube 100 with the performance characteristics of a commercially available vegetable oil based chainsaw oil (Scandinavian origin).

The test was carried out in the field, by allowing an experienced Forester to use the test oil during a normal days work. During the test the amount of wear on the cutting blades was assessed along with how much "sticking" of the blade occurred as it cut through the tree trunks. An estimation of the rate of consumption of each of the products was also made.

In general, the Biolube 100 performed well during the test and gave wear and sticking prevention results similar to those observed for the commercial product. The rates of consumption of the two oils were also similar.

A more extensive, laboratory based, evaluation is currently underway which should hopefully give more quantifiable data regarding the relative lubricity and anti-wear characteristics of Biolube 100.

6.2 Biogrease

This product is being evaluated by Revolv Lubricants Ltd, Newcastle. There are two potential areas of interest:

- i) as a general purpose grease for the food industry; and
- ii) as a speciality product able to withstand organic solvents, eg hexane.

6.3 Biolube

Revolv Lubricants Ltd have also shown an interest in the Biolube range as hammer oils, ie the lubricants used in the quarrying industry.

7 SUMMARY

- (i) Two series of lubricant base oils were produced, ie. the Biolube and the Envirolube ranges, exhibiting viscosity characteristics which meet the internationally accepted viscosity profiles for a range of lubricant types.
- (ii) The Biolube range is formulated using materials of the highest quality, ie. in terms of health and safety, and hence could find application in, for example, the pharmaceutical, cosmetic or food industries.
- (iii) The Envirolube range exhibit high viscosity indices (greater than 200), and hence are able to maintain their rheological properties over a wide temperature range.
- (iv) Both product ranges comfortably met the pour point, flash point, anti-foaming and demulsification specifications for lubricants, and, after the addition of an additive, also passed the corrosion prevention tests.
- (v) The prepared oils were prone to oxidation, and during testing exhibited viscosity and acidity increases. In terms of viscosity increase, many of the oils gave figures similar to those found when using a high oleic sunflower oil. Both the Biolube and the Envirolube oils were, however, found to be compatible with an antioxidant which greatly improved their oxidation stability. The data obtained for these inhibited oils were, in some cases, superior to the findings obtained whilst testing commercially available products.
- (vi) A field trial, which compared the chainsaw lubricating power of Biolube 100 with a commercially available chainsaw oil, concluded that Biolube 100 performed as effectively as the commercial product.
- (vii) During testing, Biogrease has exhibited the ability to withstand the solubilising effects of organic solvents. This unexpected property could potentially open up new areas of opportunity for this product.

8 REFERENCES

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