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PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

A Lubricant

We, SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ N.V., a company organised under the laws of The Netherlands, of 30 Carel van Bylandtlaan, The Hague, The Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a lubricant which is particularly but not exclusively of interest as an engine oil.

According to the present invention, a lubricant comprises a major proportion of a lubricating oil and a minor proportion of halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride, the minor proportion of the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride being from 0.05 to 2.5% by weight based on the weight of the lubricating oil.

The presence of the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride enables the lubricant to have a variety of useful properties, for example extreme pressure properties and anti-corrosion properties.

Preferably, the minor proportion of the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride lies in the range 0.25 to 1.5% by weight. Preferably, tetrachlorophthalic acid or anhydride or a mixture thereof is used. An example of a halogenated naphthalic acid or anhydride is 3,4,5,6-tetrachloronaphthalic acid or anhydride in which the four chlorine atoms are bound to one of the two fused phenyl residues. The halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride can be in the form of a mixture of any of the aforesaid acids and/or anhydrides, for example a technical mixture of chlorophthalic anhydrides, which will usually contain a proportion of the corresponding acids.

The lubricating oil can be a mineral, synthetic or vegetable oil or a mixture thereof. When the lubricating oil is a mineral oil, it can have a viscosity in the range 3—22 centistokes at 210° F, preferably 4—18 centistokes at 210° F and a viscosity index of at least 35 and preferably at least 80. When the lubricating oil is a synthetic oil, it can be a synthetic hydrocarban oil, for example an oil made by the Fischer Tropsch process or the Zeigler process, in which case oils having the viscosity and viscosity index characteristics recited above are preferred, or it can be an ester, by which is meant a simple ester, a complex ester, a polyester or a mixture thereof. Simple, complex and poly-esters suitable for the purposes of the present invention are defined and exemplified in our Patent Specification No. 1,036,695.

When the lubricating oil is a mineral oil, dissolution of the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride can be facilitated by adding said acid or anhydride to the heated oil; for this purpose an oil temperature of about 150° C is usually adequate. Alternatively, the acid or anhydride can be dissolved in a solvent that is soluble in or miscible with the oil. Such a solvent must be one that will be compatible with the use to which the lubricant will be put. When the lubricant is to be used as an engine oil, a convenient solvent is an alkyl pyrrolidone, preferably N-methyl pyrrolidone. Examples of lubricants in accordance with the present invention wherein the lubricating oil forming the major proportion of the lubricant was either a mineral oil (Examples I—IV) or an ester (Example V) were made as follows:

[*Price 4s. 6d.*]

		% by wt.
Example I	SAE 20 mineral oil (1)	99.49
	Tetrachlorophthalic anhydride	0.51
Example II	HVI 65 mineral oil (2)	77.5
	HVI 160 mineral oil (2)	21.5
	Tetrachlorophthalic anhydride	1.0
Example III	HVI 65 mineral oil	99.5
	Tetrachlorophthalic anhydride	0.5
Example IV	HVI 65 mineral oil	99.5
	Tetrabromophthalic anhydride	0.5
Example V	Di-isooctyl axelate	99.0
	Tetrachlorophthalic anhydride	1.0

(1) A mineral oil having an SAE viscosity number of 20 according to the SAE Classification of Lubricants.

(2) The expressions "HVI—65" and "HVI—160" mean a high viscosity index oil having viscosities of 65 seconds and 160 seconds Redwood I at 140° F. respectively; in the Examples, these oils had viscosity indices above 80.

The tetrachlorophthalic anhydride was incorporated in the mineral oils comprising Examples I—III and the tetrabromophthalic anhydride in the mineral oil comprising Example IV by heating the oils to a temperature of approximately 150° C; in the preparation of Example V, the tetrachlorophthalic anhydride was dissolved in the ester at room temperature.

Samples of the lubricants represented by Examples I—V, and, for comparative purposes, of the lubricating oils used in the preparation thereof (i.e. the corresponding lubricating oils), were evaluated by means of the following test procedures:—

Lead and copper corrosion tests

The procedure of Standard Method 5321, 5th Dec. 1956 of Federal Test Method Std. No. 791 was followed, except that both the lead and the copper panels were weighed. In brief, the method comprises exposing a test panel of specified dimensions to the lubricant under test in a sample tube of specified dimensions. The test temperature employed was 163° C.

Rust test

In this test, which is a vapour phase corrosion test which has been found to give good correlation with engine rusting tests, mild steel test panels of dimensions 2" × 1" and drilled for suspending by a cotton thread are cleaned by 0 and 00 gauge emery cloth, washed with petroleum spirit and when dry weighed to within 0.2 mg. The panels are then fully immersed in 100 mls of the oil under test for one hour, thereafter being removed from the oil, suspended by a cotton thread already attached and allowed to drain for 15 minutes. 500 mls of water and 30 mls of hydrobromic acid of specific gravity 1.46 are placed in a 5 litre beaker having a Perspex (R.T.M.) cover. The oil coated test panels are then suspended in the 5 litre beaker by means of hooks from the Perspex cover (i.e. the panels are suspended in the vapour space above the liquid in the beaker). The beaker is placed on a metal block heater, the temperature of which is controlled by a contact thermometer set at 38° C, the contact thermometer being placed in a thermometer pocket mounted in the Perspex cover of the beaker. The test is run for a period of two days after which the test panels are removed, cleaned with petroleum spirit and cotton wool or tissue paper. After all of the oil and rust has been removed the panels are reweighed. The panels which were most heavily rusted are those which show the greatest weight loss after cleaning.

Four-ball test

This test, which provides a measure of the extreme pressure properties of a lubricant, is described in the article entitled "Four-ball test for testing boundary lubricating properties under high mean pressures," G. D. Boerlage and H. Block, Engineering, (1937) Vol. 144, p.1 et seq.

Test results

In the lead and copper corrosion tests, Example I and the corresponding mineral lubricating oil gave the following results:—

	Lead weight loss in milligrams		Copper weight loss in milligrams
	After 1 hour	After 16 hours	After 16 hours
Example I	0.0006	0.0926	0.0016
Lubricating oil of Example I	0.0482	0.6825	0.0102

In the rust test, Examples II and V and the corresponding mineral lubricating oil and ester lubricating oil gave the following results:—

	Steel weight loss in milligrams
Example II	5.4
Lubricating oil of Example II	35.6
Example IV	6.0
Lubricating oil of Example V	28.0

In the four ball test, Examples III and IV and the corresponding mineral lubricating oils gave the following results:—

	Initial seizure load in kilogrammes
Example III	85
Lubricating oil of Example III	40
Example IV	60
Lubricating oil of Example IV	40

From the foregoing test results, it is clearly seen that a lubricant in accordance with the present invention gives less lead and copper corrosion and less rusting of a steel surface than does the corresponding lubricating oil forming the major proportion of the lubricant and furthermore, has extreme pressure properties superior to those of the lubricating oil.

Similar results are obtained when the tetrachlorophthalic anhydride is incorporated into the oil in the form of a 50% by wt. solution in N-methyl pyrrolidone.

If desired, the lubricant may also contain one or more supplementary additives known to be suitable for incorporation into lubricating oils, e.g. antioxidants, extreme pressure agents, antiwear agents, viscosity index improvers, thickeners, dispersants, detergents and anti-foam agents.

As further exemplification of a lubricant in accordance with the present invention, a lubricant wherein the lubricating oil contained supplementary additives was made and tested as follows:—

EXAMPLE VI.

	% by wt.
Lubricating oil	99
Tetrachlorophthalic anhydride	1

The lubricating oil used in the preparation of Example VI had the following composition:—

	% by wt.
HVI.65 mineral oil	77.5
HVI.160 mineral oil	16.25
Acryloid 917 (1)	4.5
AN—2 (2)	0.75
Triphenyl phosphate	1.0

(1) Acryloid 917 (the name "Acryloid" is a Registered Trade Mark) is a proprietary viscosity index improver comprising a copolymer of lauryl methacrylate and N-vinyl pyrrolidone.

(2) AN—2 is the trade name for an anti-oxidant comprising 4,4'-methylene-bis (2,6-ditert.butyl phenol).

A sample of the lubricant represented by Example VI and, for comparative purposes, of the lubricating oil used in the preparation thereof was subjected to the rusting test with the following results:

Example VI	1.4
Lubricating oil of Example VI	35.2

From the result of this test, it is again seen that a lubricant in accordance with the present invention gives less rusting of a steel surface than does the lubricating oil used in the preparation thereof.

To illustrate further the benefits to be obtained from the present invention, a lubricant having the composition set forth below as Example VII, wherein the lubricating oil comprising the major proportion of the lubricant contained supplementary additives was made and subjected to a Modified Oldsmobile (R.T.M.) Scuffing Test. This test is based on the Sequence I of the Engine Test Sequences for Evaluating Automotive Lubricant for API Service MS specified by the American Petroleum Institute for approval purposes, "Service MS" being considered to represent the most severe service encountered in the operation of the gasoline and other spark ignition engines. The engine operating test conditions are fully described in ASTM Special Technical Publication No. 315 under the heading "Reference Sequences I, II, III" and at the end of the test cycle prescribed therein the number of scuffed cams is ascertained; one or more scuffed cams is regarded as an unacceptable level of cam scuffing. The results given in Table V were obtained by operating the Oldsmobile (R.T.M.) engine in accordance with the procedure set out in ASTM Special Technical Publication No. 315 for the Reference Sequence I except that in order to increase the severity of the test, valve spring pressures were increased from 240 to 290 lbs.

EXAMPLE VII.

	% by wt.
Mineral lubricating oil	99.5
Tetrachlorophthalic anhydride	0.5

The mineral lubricating oil used in the preparation of Example VII had the following composition.

	% by wt.
Mineral oil — viscosity 4 centistokes at 210° F	94.75
Acryloid 917	4.5
AN—2	0.75

No cams were found to be scuffed at the end of this test. By way of contrast, the mineral lubricating oil used in the preparation of the lubricant represented by Example VII produced severe scuffing of several cams in this test.

No evidence of pitting of tappets was obtained from engine tests using lubricants in accordance with the present invention, i.e. lubricants comprising a major proportion of a lubricating oil and a minor proportion of a halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride.

WHAT WE CLAIM IS:—

1. A lubricant comprising a major proportion of a lubricating oil and a minor proportion of halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride, the minor proportion of the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride being from 0.05 to 2.5% by weight based on the weight of the lubricating oil.

2. A lubricant according to claim 1 wherein the minor proportion of the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride lies in the range 0.25 to 1.5% by weight.

3. A lubricant according to claim 1 or 2, comprising a solvent for the halogenated phthalic acid or anhydride or halogenated naphthalic acid or anhydride, the solvent being soluble in or miscible with the lubricating oil.

4. A lubricant according to claim 3 wherein the solvent is an alkyl pyrrolidone.

5. A lubricant according to claim 4 wherein the alkyl pyrrolidone is N-methyl pyrrolidone.

6. A lubricant according to any one of claims 1 to 5 wherein there is present tetrachlorophthalic anhydride.

7. A lubricant according to any one of claims 1 to 5 wherein there is present terrabromophthalic anhydride.

8. A lubricant according to any one of the preceding claims wherein the lubricating oil is a mineral oil having a viscosity in the range 3—22 centistokes at 210° F and a viscosity index of at least 35.

9. A lubricant according to claim 8 wherein the mineral oil has a viscosity in the range 4—18 centistokes at 210° F.

10. A lubricant according to claim 8 or 9 wherein the mineral oil has a viscosity index of at least 80.

11. A lubricant according to any one of claims 1—7 wherein the lubricating oil is a synthetic oil.

12. A lubricant according to claim 11 wherein the synthetic oil is an ester.

13. A lubricant according to claim 12 wherein the ester is di iso octyl azelate.

14. A lubricant substantially as herein described with reference to any one of Examples I—VII.

15. A lubricant according to claim 1, substantially as herein described.

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