



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<p><b>(21) International Application Number:</b> PCT/GB91/00755</p> <p><b>(22) International Filing Date:</b> 13 May 1991 (13.05.91)</p> <p><b>(30) Priority data:</b>  9010841.6                      15 May 1990 (15.05.90)                      GB</p> <p><b>(71) Applicant (for all designated States except US):</b> EXXON RESEARCH &amp; ENGINEERING COMPANY [US/US];  P.O. Box 390, Florham Park, NJ 07932 (US).</p> <p><b>(72) Inventors; and</b>  <b>(75) Inventors/Applicants (for US only) :</b> HUTCHINGS, Timothy [GB/GB]; 8 Mallard Way, Grove, Wantage, Oxon OX12 0QG (GB). PILGREM, Kathryn, May [GB/GB]; 24 Stonesfield, Didcot, Oxon OX11 9RF (GB).</p>	<p><b>(74) Agents:</b> FLETCHER WATTS, Susan, Jane et al.; Esso Engineering (Europe) Ltd., Patents and Licences, Mailpoint 72, Esso House, Ermyn Way, Leatherhead, Surrey KT22 8XE (GB).</p> <p><b>(81) Designated States:</b> AT (European patent), BE (European patent), CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FI, FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), NL (European patent), SE (European patent), US.</p> <p><b>Published</b>  <i>With international search report.</i>  <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p><b>(54) Title:</b> GREASE COMPOSITION</p> <p><b>(57) Abstract</b></p> <p>A flame retardant grease composition comprising a lubricating base oil, a thickener selected from a simple or complex soap of lithium, calcium or sodium, and a flame retardant additive selected from an oxide, hydroxide, carbonate, bicarbonate, hydrogen carbonate or sulphate of an alkali metal or alkaline earth metal, for example calcium oxide, potassium carbonate, potassium hydrogen carbonate or potassium sulphate, or a mixture of two or more thereof.</p>		

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GREASE COMPOSITION

This invention relates to grease compositions, more especially to grease compositions exhibiting flame retardant properties.

Greases containing metal soap thickeners, for example lithium, calcium or sodium soaps, are well known for industrial and other applications. With increasing health and safety standards, there is a need to improve the flame retardant properties of these greases.

Accordingly, the present invention provides a grease composition comprising:

- (a) a lubricating base oil;
- (b) a thickener selected from a lithium, calcium or sodium soap, or a mixture of two or more thereof; and
- (c) 2 to 15% by weight based on the total weight of the composition of an additive comprising an oxide, carbonate, bicarbonate, hydrogen carbonate or sulphate of an alkali metal or alkaline earth metal, or a mixture of two or more thereof.

It has been found that by including the inorganic additive (c), the flame retardant properties of the grease can be significantly enhanced.

The alkali or alkaline earth metal employed in the additive (c) (hereafter the "flame retardant additive") may be for example, lithium, sodium, potassium, magnesium or calcium. Calcium or potassium is preferred.

The salt of the flame retardant additive is preferably an oxide, carbonate, hydrogen carbonate, or sulphate. Examples of preferred compounds include calcium oxide, potassium carbonate, potassium hydrogen carbonate and potassium sulphate.

Whilst the flame retardant properties of the grease can be improved by the addition of a single flame retardant additive, it has been found surprisingly that when a mixture of two or more of the additives are employed, the additives act synergistically and the flame retardant properties achieved are greater than those using the same amount of each additive individually. Thus in a preferred embodiment of the invention the grease composition contains a mixture of two or more, preferably two, of the above flame retardant additives.

This bicomponent flame retardant additive mixture is preferably a combination of calcium and potassium compounds, for example calcium oxide and potassium carbonate, calcium oxide and potassium hydrogen carbonate, or calcium oxide and potassium sulphate.

The amount of the flame retardant additive incorporated in the grease depends upon, for example, the intended purpose of the grease as well as the composition of the grease. In general, the higher the proportion of the additive the greater the flame retardant properties. However, an excessive amount of additive can cause wear, and so generally from 2 to 15%, preferably from 4 to 11%, is used by weight based on the total weight of the composition. Where the flame retardant additive is a bicomponent mixture, it usually comprises 1 to 7% by weight of each component.

The thickener (b) employed in the grease composition according to the invention may be a simple or a complex soap of lithium, calcium or sodium, preferably lithium or calcium. Most preferably the thickener is either a simple lithium soap, a complex lithium soap or a simple or complex calcium soap. The amount of thickener is generally from 2 to 30%, more preferably 5 to 20%, by weight based on the total weight of the composition.

Where the soap is a simple lithium soap, it is preferred that the soap comprises a lithium soap of C<sub>12</sub> to C<sub>24</sub> hydroxy fatty acid more preferably a C<sub>16</sub> to C<sub>20</sub> hydroxy fatty acid. A particularly preferred hydroxy fatty acid is hydroxystearic acid, for example 9-hydroxy-, 10-hydroxy-, or 12-hydroxystearic acid, more preferably the latter. Ricinoleic acid, which is an unsaturated form of 12-hydroxystearic acid having a double bond in the 9-10 position, can also be used. Other suitable hydroxy fatty acids include 12-hydroxybehenic acid and 10-hydroxypalmitic acid.

Where the soap is a complex lithium soap, it preferably comprises a lithium soap as defined above for the simple lithium soap and at least one other lithium compound. This other lithium compound is advantageously either a lithium soap of a C<sub>2</sub> to C<sub>12</sub> aliphatic dicarboxylic acid or a lithium salt of boric acid, or a mixture of both.

Where the other lithium-containing compound is an aliphatic dicarboxylic acid, the proportion of dicarboxylic acid to hydroxy fatty acid employed is generally a weight ratio of between 0.05 and 1, usually between 0.1 and 0.8, parts dicarboxylic acid per part hydroxy fatty acid. The dicarboxylic acid is preferably a C<sub>4</sub> to C<sub>12</sub>, more preferably C<sub>6</sub> to C<sub>10</sub>, aliphatic dicarboxylic acid.

Examples of suitable acids include oxalic, malonic, succinic, glutaric, adipic, suberic, pimelic, azelaic, dodecanedioic and sebacic acids. Azelaic and sebacic acids are especially preferred.

Where the other lithium-containing compound is the lithium salt of boric acid, the proportion of hydroxy fatty acid to boric acid employed is generally a weight ratio of between 3 and 100 parts, usually between 5 and 80 parts, hydroxy fatty acid per part boric acid.

The lithium soap complex may also include a second hydroxycarboxylic acid. This generally has 3 to 14 carbon atoms and an -OH group not more than 6 carbon atoms away from the carboxyl group. This acid can be either an aliphatic acid such as lactic acid, 6-hydroxydecanoic acid, 3-hydroxybutanoic acid, 1-hydroxycaproic acid, 4-hydroxybutanoic acid, 6-hydroxy-alpha-hydroxystearic acid, etc. or an aromatic acid such as parahydroxy-benzoic acid, salicylic acid, 2-hydroxy-4-hexylbenzoic acid, metahydroxybenzoic acid, 2,5-dihydroxybenzoic acid (gentisic acid); 2,6-dihydroxybenzoic acid (gamma resorcylic acid); 4-hydroxy-4-methoxybenzoic acid, etc. or a hydroxyaromatic aliphatic acid such as orthohydroxyphenyl, metahydroxyphenyl, or parahydroxyphenyl acetic acid. A cycloaliphatic hydroxy acid such as hydroxycyclopentyl carboxylic acid or hydroxynaphthenic acid could also be used. Particularly useful hydroxy acids are lactic acid, salicylic acid, and parahydroxybenzoic acid.

Alternatively a lower alkyl ester, usually, a C<sub>1</sub> to C<sub>4</sub> alkyl ester of the above second hydroxycarboxylic acid can be used instead of the free acid. This is especially beneficial when boric acid is employed as the ester aids dispersion of the insoluble salt. As example of a preferred ester is methyl salicylate. When a second hydroxy-

carboxylic acid is employed in the grease composition, the proportion is generally a weight ratio of 0.1 to 10 parts, usually 0.5 to 5 parts, second hydroxycarboxylic acid per part of the acid component of the said other lithium-containing compound, for example boric acid.

Where the soap is a calcium complex soap, it is preferably a soap as defined above for the lithium complex soap, substituting calcium for lithium, except that the other calcium compound may be a mono or dicarboxylic acid, or a calcium salt of boric acid, or a mixture thereof.

The lubricating base oil employed in the grease composition can be any of the conventionally used lubricating oils and is preferably a mineral oil, a synthetic oil or a blend of mineral and synthetic oils. In general these lubricating oils have a viscosity in the range of 15 to 220, preferably 30 to 150 cSt at 40°C, and a viscosity index in the range of 30 to 170, preferably 30 to 140. Mineral lubricating oil base stocks used in preparing the greases can be any conventionally refined base stocks derived from paraffinic, naphthenic and mixed base crudes. Synthetic lubricating oils that can be used include esters or dibasic acids, such as a di-2-ethylhexyl sebacate, esters of glycols such as C<sub>13</sub> oxo acid diester or tetraethylene glycol, or complex esters such as one formed from 1 mole of sebacic acid and 2 moles of tetraethylene glycol and 2 moles of 2-ethylhexanoic acid. Other synthetic oils that can be used include synthetic hydrocarbons such as alkyl benzenes, e.g. alkylate bottoms from the alkylation of benzene with tetrapropylene, or the polymers and copolymers of alpha olefins; silicone oils, e.g. ethyl phenyl polysiloxanes, methyl polysiloxanes, etc.; polyglycol oils, e.g. those obtained by condensing butyl alcohol with propylene oxide; carbonate esters, e.g. the product of reacting C<sub>8</sub> oxo alcohol with ethylcarbonate

to form a half ester followed by reaction of the latter with tetraethylene glycol, etc. Other suitable synthetic oils include the polyphenyl ethers, e.g. those having from about 3 to 7 ether linkages and about 4 to 8 phenyl groups (see US Patent 3,424,678, column 3).

In addition to the components already mentioned, the grease may also contain other additives conventionally incorporated in greases, which include, but are not limited to, corrosion inhibitors, antiwear agents, pour point depressants, tackiness agents, extreme pressure agents, viscosity improvers, oxidation inhibitors, rust inhibitors, dyes and the like.

The grease composition according to the invention can be used in a variety of applications where flame retardants and fire resistance are important, for example in the lubrication of industrial and domestic machinery and appliances, in vessels and aircraft, and in public places where a fire would be a serious hazard, for example in enclosed spaces or areas catering for high volumes of people.

The invention will now be illustrated by the following examples.

#### Example 1 (Comparative)

For comparative purposes, the flame retardant properties of a conventional simple lithium soap grease were tested. The grease comprised, by weight based on the total weight of the grease, 84.9% base oil and 10.6% of the lithium soap of 12-hydroxystearic acid, the balance consisting of an additive package of a sulphur-phosphorus extreme pressure agent, an amine-type antioxidant, a zinc naphthenate corrosion inhibitor and zinc dialkyl dithiophosphate.



The flame retardant properties of the grease were measured using the following test: 25 grams of the test grease were mixed with 0.5 gram cotton wool. 10 grams of this mixture was formed into a cone and the top of the cone pulled out to form a wick. The wick was ignited with a match, and the cumulative weight loss in grams and rate of weight loss in grams per minute of the grease sample measured at minute intervals. If the grease sample self-extinguished, the sample was reignited where possible by pulling out a further wick of the cotton wool/grease mixture and igniting this, and the test continued.

The results are given in Table 1. The grease ignited easily and continued to burn at an increasing rate. After 7 minutes over half the sample in weight had been lost, and the flame was deliberately extinguished.

### Example 2

A grease composition containing 10 wt % calcium oxide was formulated by mixing calcium oxide powder into the simple lithium soap grease having the composition as described in Example 1. The calcium oxide was incorporated into the grease using conventional laboratory scale mixing techniques. On a larger scale the calcium oxide can be incorporated using conventional grease mixing or milling techniques.

The flame retardant properties of the resulting grease composition were measured using the test procedure as set out in Example 1. The results are given in Table 1.

The results show that the grease containing calcium oxide has significantly improved flame retardant properties compared to the same grease containing no calcium

TABLE 1

Example No. 1 (Comparative)

2

3

4

Additive	None		10% CaO		7% K <sub>2</sub> CO <sub>3</sub>		7.5% KHCO <sub>3</sub>	
	Wt Loss (g)	Rate (g/min)	Wt Loss (g)	Rate (g/min)	Wt Loss (g)	Rate (g/min)	Wt Loss (g)	Rate (g/min)
1	.12	.12	.05*	.05	.15	.15	.08	.08
2	.41	.29	.15	.1	.32	.17	.11	.03
3	.94	.43	.22	.07	.49	.17	.15	.04
4	1.94	1.0	.45	.23	.64	.15	.28	.13
5	3.3	1.4	.66	.21	.74	.1	.34*	.06
6	4.4	1.1	.78	.12	.81*	.07	.37	.03
7	5.4	1.0	.89*	.11	.83	.02	.46	.06
8	PUT OUT	-	.91	.02	.95	.12	.48*	.05
9			.95	.04	.96	.01	.53	.05
10			1.1	.2	.97*	.01	.62	.09
11			1.5	.4	1.0*	.03		

\* Indicates sample self-extinguished and had to be reignited.

oxide (Example 1). The calcium oxide-containing grease was difficult to ignite and after 7 minutes it self-extinguished and had to be reignited to continue the test. After 11 minutes only 1.5g of the sample had been lost.

### Example 3

A grease composition was formulated as described in Example 2 except that the calcium oxide was replaced with 7 wt % potassium carbonate.

The flame retardant properties of the resulting composition were measured using the test procedure as set out in Example 1. The results are given in Table 1.

The results show that the grease containing potassium carbonate has significantly improved flame retardant properties compared to the same grease containing no flame retardant additive (Example 1). The grease containing potassium carbonate was difficult to ignite and self-extinguished 3 times during the test. After 11 minutes only 1.0g of the sample had been lost.

### Example 4

A grease composition was formulated as described in Example 2 except that the calcium oxide was replaced with 7.5 wt.% potassium hydrogen carbonate.

The flame retardant properties of the resulting composition were measured using the test procedure as set out in Example 1. The results are given in Table 1.

The results show that the grease containing potassium hydrogen carbonate has significantly improved flame retardant properties compared to the same grease contain-

ing no flame retardant additive (Example 1). The grease containing  $\text{KHCO}_3$  was difficult to ignite and self-extinguished twice during the 10 minute test. At the end of the test only 0.62 g of the sample had been lost.

#### Example 5

A grease composition containing 5 wt % calcium oxide and 5 wt % potassium carbonate was formulated by mixing these inorganic compounds, in powder form, into the simple lithium soap grease having the composition as described in Example 1. The inorganic compounds were mixed in using conventional laboratory scale techniques. On a larger scale, conventional grease mixing or milling techniques can be used. Preferably the potassium compound is milled with the grease at room temperature and then the calcium oxide is mixed in as a subsequent step.

The flame retardant properties of the resulting grease composition were measured using the test procedure as set out in Example 1. The results are given in Table 2.

The results show that the grease containing a mixture of calcium oxide and potassium carbonate as a flame retardant additive exhibits extremely good flame retardant properties. The grease was difficult to ignite and self-extinguished many times during the test. At all stages the weight loss was considerably better than that of the base grease containing no flame retardant additive (Example 1), and also better than that containing solely calcium oxide or potassium carbonate as the flame retardant additive (Examples 2 and 3 respectively).

**TABLE 2**

Example No.	5	6	7	8
	5% CaO + 5% K <sub>2</sub> CO <sub>3</sub>	5% CaO + 5% KHCO <sub>3</sub>	5% CaO + 5% K <sub>2</sub> SO <sub>4</sub>	2% CaO + 4% K <sub>2</sub> SO <sub>4</sub>
Additive	Wt Loss (g)	Wt Loss (g)	Wt Loss (g)	Wt Loss (g)
Time(min)	Rate (g/min)	Rate (g/min)	Rate (g/min)	Rate (g/min)
1	.01*	.04*	.03	.02
2	.03*	.12	.07	.04
3	.08*	.13	.22	.05
4	.11*	.23	.39	.07
5	.1	.27*	.55	.08*
6	.15*	.32	.69	.09
7	.17*	.37*	.74*	.15
8	.25*	.44	.95	.23
9	.28*	.45*	1.04	.33
10	.31	.97*	1.17	.44
11	.34*	.48*	1.25	.57*
12			1.28*	.65

\* Indicates sample self-extinguished and had to be reignited.

### Example 6

A grease composition was formulated as described in Example 4 except that the calcium oxide and potassium carbonate were replaced with a flame retardant additive mixture consisting of 5 wt % calcium oxide and 5 wt % potassium hydrogen carbonate.

The flame retardant properties of the resulting grease composition were measured using the test procedure as set out in Example 1. The results are given in Table 2.

The results show that the grease containing a mixture calcium oxide and potassium hydrogen carbonate as the flame retardant additive also exhibits very good flame retardant properties.

### Example 7

A grease composition was formulated as described in Example 4 except that the calcium oxide and potassium carbonate were replaced with 5 wt % calcium oxide and 5 wt % potassium sulphate.

The flame retardant properties of the resulting grease composition were measured using the test procedure as set out in Example 1. The results are given in Table 2.

The results show that this grease composition exhibits good flame retardant properties, although the weight loss is more than that for the other grease compositions containing 10 wt % of a bicomponent additive (Examples 4 and 5).

### Example 8

A grease composition was formulated as described in Example 4 except that the calcium oxide and potassium carbonate were replaced with 2 wt % calcium oxide and 4 wt % potassium sulphate.

The flame retardant properties of the resulting grease composition were measured using the test procedure as set out in Example 1. The results are given in Table 2.

The results show that the grease composition exhibits very good flame retardant properties, and that for the calcium oxide/potassium sulphate additive an amount of additive of 2 wt % CaO/4 wt % KHCO<sub>3</sub> is preferred to 5/5 wt % (Example 6).

### Example 9

A bicomponent flame retardant additive consisting of 4 wt % calcium oxide and 4 wt % potassium carbonate was mixed, using conventional laboratory scale mixing techniques, into a conventional simple lithium soap grease having the following formulation: 90.2 wt % base oil, 5.3 wt % lithium soap of 12-hydroxystearic acid, the balance consisting of an additive package of a sulphur-phosphorus extreme pressure agent, an amine-type antioxidant, a zinc naphthenate corrosion inhibitor and zinc dialkyl dithiophosphate.

The flame retardant properties of this resulting grease composition were measured using the test procedure as set out in Example 1. The results are given in Table 3.

TABLE 3Additive: 4% CaO + 4% K<sub>2</sub>CO<sub>3</sub>

Time (min)	wt loss (g)	Rate (g/min)
1	.07	.07
2	.16	.09
3	.24	.08
4	.29	.05
5	.33*	.04
6	.34	.01
7	.40	.06
8	.45*	.05
9	.47	.02
10	.59	.12
11	.7*	.11
12	.72	.02

The results show that the grease composition exhibits very good flame retardant properties.



CLAIMS

1. A grease composition comprising:
  - (a) a lubricating base oil;
  - (b) a thickener selected from a lithium, calcium or sodium soap, or a mixture of two or more thereof; and
  - (c) 2 to 15% by weight based on the total weight of the composition of an additive comprising an oxide, carbonate, bicarbonate, hydrogen carbonate or sulphate of an alkali metal or alkaline earth metal, or a mixture of two or more thereof.
2. A grease composition according to Claim 1 wherein the alkali metal or alkaline earth metal of additive (c) is selected from calcium or potassium, or a mixture thereof.
3. A grease composition according to Claim 1 wherein the additive (c) is selected from calcium oxide, potassium carbonate, potassium hydrogen carbonate or potassium sulphate, or a mixture of two or more thereof.
4. A grease composition according to Claim 1 wherein the additive (c) is a mixture of calcium oxide and a potassium compound selected from potassium carbonate, potassium hydrogen carbonate or potassium sulphate.
5. A grease composition according to Claim 4 wherein the additive (c) comprises 1 to 7% calcium oxide and 1 to 7% potassium compound, by weight based on the weight of the total composition.
6. A grease composition according to any preceding Claim wherein the amount of additive (c) is from 4 to 11 weight per cent.

7. A grease composition according to any preceding Claim wherein the thickener (b) is a simple or complex lithium soap.

8. A grease composition according to any of Claims 1 to 6 wherein the thickener (b) is a complex calcium soap.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 91/00755

**I. CLASSIFICATION OF SUBJECT MATTER** (if several classification symbols apply, indicate all)<sup>6</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.C1.5                    C 10 M 169/06    //(C 10 M 169/06                    C 10 M 115:00  
 C 10 M 117:04            C 10 M 117:06                    C 10 M 125:10                    C 10 M 125:22 ),  
 C 10 N 30:00, 50:10

**II. FIELDS SEARCHED**

Minimum Documentation Searched<sup>7</sup>

Classification System	Classification Symbols
Int.C1.5	C 10 M

Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are Included in the Fields Searched<sup>8</sup>

**III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup>**

Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
P,X	US,A,4986923 (J.A. WAYNICK) 22 January 1991, see column 11, line 22 - column 14, line 30; column 28, examples 44-47 ---	1-3,6,8
X	FR,A,2572089 (COMPAGNIE FRANCAISE DE RAFFINAGE) 25 April 1986, see page 1, lines 20-30; page 2, lines 17-25; column 3, line 24 ---	1,2,7
X	EP,A,0296362 (AMOCO) 28 December 1988, see claim 1; page 16, example 20; page 17, example 24; page 3, line 4 ---	1-3,6,8
X	US,A,3090750 (C.R. BERGER) 21 May 1963, see column 12, example 11 ---	1,2
A	DE,A,3431585 (MAGYAR SZENHIDROGENIPARI KUTATO) 11 April 1985, see page 11, lines 1-7 --- -/-	1-3,6

<sup>10</sup> Special categories of cited documents :

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**IV. CERTIFICATION**

Date of the Actual Completion of the International Search  07-08-1991	Date of Mailing of this International Search Report  18 OCT 1991
International Searching Authority  EUROPEAN PATENT OFFICE	Signature of Authorized Officer  MISS D. S. KOWALCZYK

## III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	US,A,2748081 (W.H. PETERSON) 29 May 1956, see column 2, lines 5-10 -----	1-3

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.**

GB 9100755  
SA 47329

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.  
The members are as contained in the European Patent Office EDP file on 17/09/91  
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 4986923	22-01-91	None	
FR-A- 2572089	25-04-86	None	
EP-A- 0296362	28-12-88	US-A- 4787992 JP-A- 1026698	29-11-88 27-01-89
US-A- 3090750		None	
DE-A- 3431585	11-04-85	CH-A- 662127	15-09-87
US-A- 2748081		None	

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82