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54 **High dropping-point lithium-complex grease composition having improved extreme pressure properties.**

57 High dropping-point lithium-complex grease composition having improved extreme pressure properties, comprising a lubricating oil and the following components:

- (a) at least one lithium soap a C<sub>12</sub> to C<sub>24</sub> hydroxy fatty acid,  
(b) at least one lithium salt of a boric acid, and  
(c) at least one metal salt selected from the group consisting of

- 1) a potassium salt of a boric acid,
- 2) a sodium salt of a boric acid,
- 3) an alkaline earth metal salt of a boric acid, and
- 4) a zinc salt of a boric acid.

HIGH DROPPING-POINT LITHIUM-COMPLEX GREASE  
COMPOSITION HAVING IMPROVED EXTREME  
PRESSURE PROPERTIES

The present invention relates to a high dropping-point lithium-complex grease composition having improved extreme pressure properties.

5 In the co-pending European patent application 83200077 high dropping-point lithium-complex grease compositions are described, which contain (a) as gelling component e.g. a lithium soap of a  $C_{12}$  to  $C_{24}$  hydroxy fatty acid and (b) as a complexing metal salt e.g. a lithium salt of a boric acid.

10 Furthermore a high molecular weight viscosity-index improver, and/or succinimide type dispersant and/or metal salt detergent is added in order to provide the grease compositions with improved anti-noise properties.

15 Although the present grease compositions may also contain such anti-noise additives, the purpose of the present invention is to render extreme pressure properties to high dropping-point lithium-complex grease compositions of the above type.

20 It has been found that this purpose can surprisingly be met by combining the above-mentioned components (a) and (b) with a component (c) which is a class of certain metal salts of a boric acid.

This invention therefore relates to a high dropping-point lithium-complex grease composition having improved extreme pressure properties, comprising a lubricating oil and the following components:

- 25 (a) at least one lithium soap of a  $C_{12}$  to  $C_{24}$  hydroxy fatty acid,  
(b) at least one lithium salt of a boric acid, and

- (c) at least one metal salt selected from the group consisting of
- 1) a potassium salt of a boric acid,
  - 2) a sodium salt of a boric acid,
  - 3) an alkaline earth metal salt of a boric acid, and
  - 5 4) a zinc salt of a boric acid.

#### Lubricating oil

Any conventional lubricating oil can be used. While a typical one is a mineral oil, use can also be made, singly or in combination with the mineral oil, of a synthetic oil including ester

10 oils, such as a diester oil, e.g. dioctyl azelate, and a tetra-  
ester oil, e.g. the pentaerythritol ester of an aliphatic mono-  
carboxylic acid, or poly-alpha-olefin oligomers, such as octene-  
1/decene-1 copolymers, having a viscosity of 41.0 cSt at 37.8°C, a  
viscosity index (V.I.) of 130 and a flash point of 223°C.

15 The lubricating oil may have a viscosity ranging from about  
2 to 500 cSt, preferably from 20 to 200 cSt, at 40°C.

#### Component (a)

The  $C_{12}$  to  $C_{24}$  hydroxy fatty acids include saturated or  
unsaturated monocarboxylic fatty acids containing a hydroxyl

10 radical, especially those acids containing 18 carbon atoms and a  
hydroxyl radical in the 9-, 10- or 12-position, such as 12-hydroxy  
stearic acid and ricinolic acid.

The lithium salts of the above fatty acids and hydroxy fatty  
acids can be singly used or combinedly with one or more of others.

15 In addition, when said lithium salts are prepared, said carboxylic  
acids can be reacted with lithium hydroxide not only in the form  
of free acids but also as glycerides.

#### Component (b)

Suitable salts are lithium metaborate, lithium diborate,

20 lithium tetraborate, lithium pentaborate, lithium perborate and  
lithium ortho borate, such as the monolithium salt of ortho boric  
acid ( $H_3BO_3$ ).

#### Component (c)

Suitable salts are potassium, sodium, alkaline earth metal

25 (e.g. Ca, Ba or Mg) and zinc metaborate, diborate, tetraborate,

pentaborate, perborate and ortho borate. Partial as well as full salts are suitable.

The above components (a), (b) and (c) can be present as separate compounds or as a mixed compound as explained hereinafter.

Preparation of these grease compositions

The grease composition according to the present invention can be prepared by uniformly admixing and finely dispersing the three components (a), (b) and (c) in the base oil mentioned above.

While the amounts of these three components compounded in the base oil above are not necessarily critical but can be variable for individual components, it is generally advantageous to use them in the following proportions per 100 weight parts (pbw) of the base oil;

Component (a): 2 to 40 weight parts, preferably 5 to 30 weight parts

Component (b): 0.05 to weight parts, preferably 0.1 to 10 weight parts

Component (c): 0.05 to 20 weight parts, preferably 0.1 to 10 weight parts

Also, it is particularly preferable to use the components (a) and (b) in the following mol ratio within the above ranges of the proportions;

Component (a)/Component (b) mol ratio = 0.5 to 10, especially 1 to 5.

The present grease composition can also be prepared as follows. The base oil is admixed with a hydroxy fatty acid for forming a component (a), followed by an e.g., equivalent, amount of LiOH to form a soap. Then, a boric acid for a component (b) is admixed, followed successively by an, e.g equivalent, amount of LiOH, another portion of a boric acid and an e.g., equivalent, amount of a metal compound of a metal of component (c), e.g. an oxide or hydroxide of K, Na, an alkaline earth metal or Zn. Alternatively, the total of the two portions above of boric acid may be admixed simultaneously in the first mixture, followed by LiOH and said metal compound.

These procedures, which result into so-called mixed compounds, may be carried out at 70 to 90°C. Subsequently, the reaction mixture is dehydrated gradually at e.g. 140 to 225°C to complete the preparation.

5 It is also possible to add an aqueous slurry of  $\text{LiOH} \cdot \text{H}_2\text{O}$ , the metal compound of a metal of components (c) and boric acid to e.g. an autoclave containing lubricating oil and hydroxy fatty acid.

10 In the grease composition according to the present invention, use can be made, in addition to the three components (a), (b) and (c) above, of other metal borates or other lubricant-additives, such as neutral or basic metallic detergents, such as basic Ca salicylate, or ashless dispersants, rust inhibitors (e.g., paraffin oxide, amino imidazoline or barium  
15 dinonylnaphthalene-sulphonate), oxidation inhibitors (e.g. 2,6-ditertiary butyl-4-methylphenol), N-phenyl- $\alpha$ -naphthylamine or diphenylamine octylate) and conventional extreme-pressure additives (e.g. zinc naphthenate or other zinc compounds such as zinc dithiophosphates or zinc-oxide, lead naphthenate, sulphurized  
20 oils and fats, or tricresyl phosphate), in the proportions ordinarily employed.

The present grease compositions can be used, for example, for the lubrication of the bearings of electric motors as well as of the wheel bearings in automotive bearing/axle integrated structures.  
25

In the following, the present greases are further illustrated by means of examples (the boric acid used was ortho boric acid).

#### Example 1

30 Component (a) was lithium 12-hydroxy stearate, component (b) lithium borate and component (c) potassium borate.

This grease was prepared in situ by mixing and heating 9.00 %w of 12-hydroxy stearic acid, 2.96 %w boric acid, 2.33 %w lithium hydroxide, 0.45 %w potassium hydroxide and 85.26 %w mineral lubricating oil.

Example 2

As Example 1 except for (c) which was sodium borate.

Example 3

As Example 1 except for (c) which was calcium borate.

5

Example 4

As Example 1 except for (c) which was zinc borate.

Test results of these greases are shown in the following Table.

10

TABLE

	1	2	3	4
Consistency (worked penetration)	265-296	250-277	229-271	285
Dropping point, °C	247->260	242->260	239-255	261
Shell 4-ball test weld load, kg	282-355	282-355	316	316

C L A I M S

1. High dropping-point lithium-complex grease composition having improved extreme properties, comprising a lubricating oil and the following components:
  - (a) at least one lithium soap of a  $C_{12}$  to  $C_{24}$  hydroxy fatty acid,  
5 acid,
  - (b) at least one lithium salt of a boric acid, and
  - (c) at least one metal salt selected from the group consisting of
    - 1) a potassium salt of a boric acid,
    - 10 2) a sodium salt of a boric acid,
    - 3) an alkaline earth metal salt of a boric acid, and
    - 4) a zinc salt of a boric acid.
2. Composition as claimed in claim 1, wherein the boric acid is ortho boric acid.
- 15 3. Composition as claimed in claim 1 or 2, wherein the alkaline earth metal salt is a calcium, barium or magnesium salt.
4. Composition as claimed in any one of claims 1-3, wherein the proportions per 100 pbw of base oil are:
  - Component (a): 2-40 pbw
  - 20 Component (b): 0.05-20 pbw
  - Component (c): 0.05-20 pbw
5. Process for the preparation of a composition according to any one of claims 1-4 comprising mixing and heating together in a lubricating oil a  $C_{12}$  to  $C_{24}$  hydroxy fatty acid, a boric acid,  
25 lithium hydroxide and an oxide or a hydroxide from a metal selected from the group consisting of potassium, sodium, an alkaline earth metal and zinc.