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

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**US-A-3 907 691**  
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## Description

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 (EP—A—84910) high dropping-point lithium-complex grease compositions are described, which contain (a) as gelling component e.g. a lithium soap of a  $C_{12}$ — $C_{24}$  hydroxy fatty acid and (b) as a complexing metal salt e.g. a lithium salt of a boric acid.

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  
10 properties.

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.

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

From US—A—3,907,691 a grease is known which comprises a lubricating oil, lithium hydroxy stearate, an alkali metal salt of a boric acid, and an alkaline earth metal salt of a boric acid. The lithium salt of a boric acid is less preferred. The combination of the metal salts of boric acid gives the grease improved extreme-pressure properties.

20 It has now been found that very high-dropping point grease having improved extreme-pressure properties can be obtained by using salts other than alkaline earth metal salts.

This invention therefore provides a 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 of a  $C_{12}$ — $C_{24}$  hydroxy fatty acid,  
25 (b) at least one lithium salt of a boric acid, and  
(c) at least one other metal salt of a boric acid, characterized in that the metal salt of component (c) is selected from

- (1) a potassium salt,
- (2) a sodium salt, and
- 30 (3) a zinc salt.
- (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
  - 35 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 oils, such as a diester oil, e.g.  
40 dioctyl azelate, and a tetraester oil, e.g. the pentaerythritol ester of an aliphatic monocarboxylic 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.

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

### 45 Component (a)

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

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

55 Suitable salts are lithium metaborate, lithium diborate, 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 (e.g. Ca, Ba or Mg) and zinc metaborate, diborate, tetraborate, pentaborate, perborate and ortho borate. Partial as well as full salts are suitable.

60 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

65 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;

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

Component (b): 0.05 to 20 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;

10 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

15 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

20 preparation.

It is also possible to add an aqueous slurry of LiOH · H<sub>2</sub>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.

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

25 basic metallic detergents, such as basic Ca salicylate, or ashless dispersants, rust inhibitors (e.g., paraffin oxide, amino imidazoline or barium 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 oils and fats, or tricresyl phosphate), in the proportions

30 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.

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

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#### Example 1

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

40 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.

#### 45 Example 3 (Comparative Example)

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

#### Example 4

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

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

	TABLE			
	1	2	3	4
55 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

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#### Claims

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

65 (a) at least one lithium soap of 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 other metal salt of a boric acid, characterized in that the metal salt of component (c) is selected from

- (1) a potassium salt,
  - 5 (2) a sodium salt, and
  - (3) a zinc salt.
2. Composition as claimed in claim 1, characterized in that the boric acid is ortho boric acid.
3. Composition as claimed in claim 1 or 2, characterized in the proportions per 100 pbw of base oil are:
- Component (a): 2—40 pbw
- 10 Component (b): 0.05—20 pbw
- Component (c): 0.05—20 pbw
4. Process for the preparation of a composition according to any one of claims 1—3 comprising mixing and heating together in a lubricating oil a C<sub>12</sub> to C<sub>24</sub> hydroxy fatty acid, a boric acid, lithium hydroxide and an oxide or a hydroxide from a metal selected from the group consisting of potassium, sodium and zinc.

#### 15 Patentansprüche

1. Schmierfettzusammensetzung auf der Basis eines Lithium-Komplex mit hohem Tropfpunkt und mit verbesserten Eigenschaften bei extremen Druckbedingungen, enthaltend ein Schmieröl und die folgenden
- 20 Komponenten:
  - (a) mindestens eine Lithiumseife einer C<sub>12</sub>—C<sub>24</sub>-Hydroxyfettsäure,
  - (b) mindestens ein Lithiumsalz einer Borsäure, und
  - (c) mindestens ein anderes Metallsalz einer Borsäure, dadurch gekennzeichnet, daß das Metallsalz der Komponente (c) ausgewählt ist aus
- 25
  - (1) einem Kaliumsalz,
  - (2) einem Natriumsalz, und
  - (e) einem Zinksalz.
2. Zusammensetzung wie in Anspruch 1 beansprucht, dadurch gekennzeichnet, daß die Borsäure Orthoborsäure ist.
- 30 3. Zusammensetzung wie in Anspruch 1 oder 2 beansprucht, dadurch gekennzeichnet, daß die Anteilsmengen je 100 Gewichtsteile Basisöl wie folgt sind:

Komponent (a): 2 bis 40 Gewichtsteile

Komponente (b): 0,05 bis 20 Gewichtsteile

Komponente (c): 0,05 bis 20 Gewichtsteile
- 35 4. Verfahren zur Herstellung einer Zusammensetzung gemäß einem der Ansprüche 1 bis 3 umfassend das Vermischen und Erhitzen in einem Schmieröl einer C<sub>12</sub>—C<sub>24</sub>-Hydroxyfettsäure, einer Borsäure, Lithiumhydroxid und eines Oxids oder eines Hydroxids eines Metalls ausgewählt aus der Gruppe bestehend aus Kalium, Natrium und Zink.

#### 40 Revendications

1. Composition de graisse à base d'un complexe de lithium avec un haut point de goutte ayant des propriétés d'extrême pression améliorées, comprenant une huile lubrifiante et les composants suivants:
- 45
  - (a) au moins un savon de lithium d'un hydroxyacide gras en C<sub>12</sub> à C<sub>24</sub>,
  - (b) au moins un sel de lithium d'un acide borique, et
  - (c) au moins un autre sel métallique d'un acide borique, caractérisée en ce que le sel métallique du composant (c) est choisi parmi
- 50
  - (1) un sel de potassium,
  - (2) un sel de sodium, et
  - (3) un sel de zinc.
2. Composition comme revendiqué dans la revendication 1 caractérisée en ce que l'acide borique est l'acide orthoborique.
3. Composition comme revendiqué dans la revendication 1 ou 2 caractérisée en ce que les proportions pour 100 parties en poids d'huile de base sont:
- 55
  - composant (a): 2—40 parties en poids
  - composant (b): 0,05—20 parties en poids
  - composant (c): 0,05—20 parties en poids.
4. Procédé pour la préparation d'une composition selon l'une quelconque des revendications 1 à 3 comprenant le mélange et le chauffage ensemble dans une huile lubrifiante d'un hydroxyacide gras en C<sub>12</sub> à C<sub>24</sub>, d'un acide borique, d'hydroxyde de lithium et d'un oxyde ou d'un hydroxyde d'un métal choisi dans
- 60 le groupe constitué par le potassium, le sodium et le zinc.