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LUBRICATING GREASES

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This invention relates to novel lubricating greases and more particularly to block greases of spongy texture which are especially useful for lubricating bearings that operate at high temperatures since they are not rapidly consumed at high bearing temperatures.

It is an object of this invention to produce block greases that are not rapidly consumed when they are used to lubricate bearings that are operating at high temperatures. A further object is to manufacture block greases which are not rapidly consumed at high operating temperatures and which will also provide excellent lubrication for bearings that are operating at lower temperatures. Other objects will appear hereinafter.

These objects are accomplished in accordance with the present invention by making block greases which comprise a mineral oil thickened with a mixture of sodium and calcium soaps which contain a sufficient amount of the saponification products of carnauba wax or ouricury wax and of a sodium carbonate to decrease its rate of consumption at high bearing temperatures. The amount of carnauba wax or ouricury wax used in preparing these block greases may vary from 0.2 to 20 per cent by weight of the final product. Preferably the block greases of this invention comprise from about 70 per cent to about 85 per cent by weight of a mineral oil which has been thickened to a grease with from about 10 per cent to about 25 per cent by weight of a mixture of sodium and calcium soaps and which also contains from about 0.2 per cent to about 2.5 per cent by weight of carnauba wax in the form of its saponification products and from about 0.2 per cent to about 0.4 per cent by weight of excess alkali in the form of a sodium carbonate.

The following examples illustrate but do not limit the invention. All parts are given by weight except where otherwise noted.

Example 1.—To a pressure type grease kettle equipped with a mechanical agitator and means of heating with steam were charged 17 parts of tallow and 4.1 parts of stearic acid which were melted and heated to 180° F. Then 8.51 parts of a 35.9 per cent caustic soda solution were added. Heat was applied and the temperature gradually raised to 200° F. with agitation; then 0.51 part of lime mixed in a slurry with 1 part of water was charged to the kettle. Heating and stirring were continued until a temperature of approximately 250° F. was attained and the soap stock was substantially dehydrated and appreciably thickened. At this point a solution of 2 parts carnauba wax

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dissolved in 2 parts of mineral oil at 220° F. was added to the soap stock. Superheated steam at a temperature of 600° F. was admitted to the jacket of the kettle, and the temperature of the kettle raised to 450° F. During this heating period 71.2 parts of solvent refined mineral oil having a viscosity of 100 seconds SUV at 100° F. were added to the kettle at such a rate as to keep the soap stock sufficiently soft to prevent overloading of the stirring mechanism. When 450° F. temperature was reached, 0.4 part of powdered sodium bicarbonate was slowly added to the kettle and stirred into the grease for 15 minutes. Then 0.03 part of a 35.9 per cent caustic soda solution was slowly poured into the kettle. The temperature of the kettle was maintained at 450–460° F. for approximately 2 hours and then the grease was drawn from the kettle into trays for cooling. The composition and inspection data on the block grease thus prepared is set forth in the following table.

Table I

Block-type grease

Composition: per cent by weight:

100 seconds SUV at 100° F.:	
Solvent refined coastal oil	73.2
Calcium and sodium soaps of stearic acid and tallow	22.0
Excess compounding alkali or alkalis	1.0
Glycerine (theoretical)	1.8
Carnauba wax	2.0

Inspection:

Melting point: (Hawxhurst) °F.	334
Dropping point ¹ : °F.	358
Penetration, ASTM D217-44T—77° F., 150 g., 5 sec.:	
Unworked	57
Worked	198
Grease consumption test ² : g./hr.:	
Temperature of journal:	
Atmospheric temp.	6
227° F.	5
240° F.	4
260° F.	4
281° F.	7
298° F.	14
320° F.	40

¹ The dropping point test is described on page 14 of ASTM Standards on Petroleum Products and Lubricants dated September, 1937.

² The grease consumption test was performed in an apparatus described in U. S. Patent No. 2,385,656, issued September 25, 1945, to H. G. Smith. The test sample was a block measuring 3½ inches by 3½ inches by 4 inches and weighing about 700 grams. The journal was a faced cylinder 8½ inches in diameter which was revolved 267 times per minute which gave a peripheral speed of 594 feet per minute to the journal.

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Example II.—A series of block greases having a spongy texture which contained 1.5 per cent by weight of carnauba wax and from 12 per cent to 22 per cent by weight of calcium and sodium soaps in their make-up were prepared in accordance with the general procedure which is detailed above in Example I. The composition and inspection data on these block greases is set forth in the following table.

Table II.—Stabilized block greases

	22% Soap Content Grease	17% Soap Content Grease	12% Soap Content Grease
Composition, per cent by Wt.:			
100 Seconds SUV at 100° F.— Solvent refined Mid-Continent oil	73.7	79.3	84.95
Calcium and sodium soaps of tallow and stearic acid	22.0	17.0	12.0
Excess compounding alkali or alkalis	1.0	0.8	0.55
Glycerine (Theoretical)	1.8	1.4	1.0
Carnauba wax	1.5	1.5	1.5
Inspection:			
Melting Point (Hawxhurst), °F.	340	332	314
Dropping Point, °F.	358	359	345
Penetration, ASTM D-217- 44T 77° F., 150 G., 5 Sec.:			
Unworked	59	65	82
Worked	180	206	229
Grease Consumption Test, G./Hr.—			
Temperature of Journal:			
Atmospheric Temp.	7	1	3
227° F.	5	3	14
240° F.	4	3	7
260° F.	4	5	5
281° F.	7	10	22
298° F.	27	10	44
320° F.	102	19	46

Example III.—The following table illustrates the fact that a grease which is prepared without the inclusion of sodium bicarbonate in its make-up is consumed far more rapidly than a block grease to which sodium bicarbonate has been added during its preparation. The greases described in the following table were both prepared in accordance with the general procedure described above in Example I except that in the preparation of the grease "without NaHCO₃" the step of adding sodium bicarbonate was omitted.

Table III.—Block greases

	Without NaHCO ₃	With NaHCO ₃
Composition, Per cent by Wt.:		
150 Sec. SUV at 100° F.—Solvent re- fined coastal oil	74.2	77.4
Calcium and sodium coaps of Hydro- genated Fatty Acids	25.0	22.0
Excess compounding alkali or alkalis ..	0.5	0.4
Carnauba wax	0.3	0.2
Inspection:		
Melting Point (Hawxhurst), °F.	298	302
Dropping Point, °F.	330	320
Penetration, ASTM D217-44T-77° F., 150 G., 5 Sec.:		
Unworked	52	59
Worked	168	175
Grease Consumption Test, G./Hr.—		
Temperature of Journal:		
227° F.	49	8
240° F.	51	13
260° F.	3,541	99
281° F.	-----	338

The dropping points and grease consumption data given in Tables II and III are determined in accordance with the procedures indicated in the footnotes to Table I.

Example IV.—A block grease having a spongy texture which contained 1.5 per cent by weight of ouricury wax and 22 per cent by weight of calcium and sodium soaps in its make-up was prepared in accordance with the general proce-

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dure which is detailed above in Example I. The composition and inspection data on this block grease are set forth in the following table.

Table IV

Composition: per cent by wt.:	
100 seconds SUV at 100° F. solvent re- fined Mid-Continent oil	74.1
Calcium and sodium soaps of stearic acid and tallow	22.0
Excess compounding alkali or alkalis ..	1.0
Glycerine (theoretical)	1.4
Ouricury wax	1.5
Inspection:	
Melting point: (Hawxhurst) °F.	332
Dropping point: °F.	360
Penetration, ASTM D217-44 T-77 °F., 150 g., 5 sec.:	
Unworked	58
Worked	185
Grease consumption test: g./hr.:	
Temperature of journal:	
260° F.	9
281° F.	15
298° F.	24

The dropping point and grease consumption data given in the above table are determined in accordance with the procedures outlined in the footnotes to Table I.

In the preparation of these block greases any suitable lubricating oil may be used such as solvent refined coastal and midcontinent oils having viscosities ranging from 100 to 150 seconds SUV at 100° F.

The sodium and calcium soaps used in thickening these oils to greases may be prepared from any suitable fats or higher fatty acids such as tallow, stearic acid or hydrogenated fish oil fatty acids. Any suitable mixture of these fats and fatty acids may also be employed.

In the presence of excess alkali carnauba wax is saponified to form a soap of the carnauba wax acids and the higher alcohols which are found in carnauba wax. Neither the soap nor the higher alcohols after being formed are removed during the later stages of grease manufacture.

In order that the greases of the present invention will not be consumed rapidly at high bearing temperatures it is also necessary to include in their composition a small amount of excess alkali in the form of a sodium carbonate. As shown above in Example I this excess alkali in the form of a sodium carbonate may be introduced into the grease by adding sodium bicarbonate to the batch during the manufacture of the grease. Alternatively, gaseous carbon dioxide may be used in place of sodium bicarbonate. In this alternative procedure the gaseous carbon dioxide is contacted with the melted grease for the length of time necessary to give the desired results during the finishing process of the grease. Whether the carbonate in the grease exists in the form of sodium carbonate or sodium bicarbonate is not precisely known and, therefore, we refer to it as excess alkali in the form of a sodium carbonate.

The stages of processing at which the carnauba wax and sodium bicarbonate may be added to the grease batch are variable. We have found it desirable to add the sodium bicarbonate at a maximum temperature. After the grease containing all the other ingredients has been melted, the water of reaction liberated from the addition of the sodium bicarbonate and subsequent minor portion of caustic soda solution rapidly evaporates without causing an excessive or per-

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sistent emulsion. For this reason, it is likewise desirable to add the carnauba wax at an early stage of the process, as described above in Example I; however, it also may be charged with the fats and/or fatty acids used for the soap formation.

We prefer in preparing our new block greases to use from 14 per cent to 22 per cent by weight of a mixture of sodium and calcium soaps made from a mixture of tallow and stearic acid and from 1.5 per cent to 2.5 per cent by weight of carnauba wax and to employ sodium bicarbonate to introduce excess alkali in the form of a sodium carbonate. The block greases of the present invention which have the most desirable physical properties have compositions falling within the following range:

Component	Per Cent by Wt.
Lubricating Oil, 100 sec. SUV at 100° F.....	69.7-85.2
Sodium soap of tallow.....	7.0-14.4
Calcium soap of tallow.....	1.8-4.0
Sodium soap of stearic acid.....	0.0-4.4
Calcium soap of stearic acid.....	0.0-0.8
Glycerine (Theoretical).....	0.6-1.8
Carnauba Wax.....	0.2-2.5
Excess sodium hydroxide.....	0.3-0.7
Excess sodium carbonate.....	0.2-0.4

Greases prepared according to the present invention have improved consumption characteristics and give excellent lubrication. Most of these greases lubricate bearings at 320° F. with moderate grease consumption; however, they also give excellent lubrication at lower temperatures with very low consumption. These greases have a spongy texture. We do not limit the carnauba wax content of these greases to 0.2-2.5 per cent since we are able to make improved greases containing from 0.2 per cent to 20 per cent of carnauba wax.

The block greases of the present invention are particularly useful in the lubrication of journals and other bearings operating at high temperatures such as the hot roll bearings of machinery used in the manufacture of paper and cement. These block greases afford excellent lubrication to bearings operating at high temperatures and are not consumed at an excessive rate while they are lubricating such hot bearings.

By the addition of from 1 per cent to 2 per cent of carnauba wax we have been able to prepare a block grease containing but 12 per cent of a mixture of sodium and calcium soaps which has superior physical properties to a block grease that has been prepared in a similar manner and which contains 22 per cent of a mixture of sodium and calcium soaps but no carnauba wax. The block grease containing 12 per cent of soap and from 1 per cent to 2 per cent of carnauba wax will lubricate bearings operating at high temperatures better than a grease similarly prepared which has a sodium and calcium soap content of 22 per cent but which contains no carnauba wax. The grease which contains 22 per cent of sodium and calcium soaps but no carnauba

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ba wax will not lubricate bearings that operate at temperatures above 240° F. without being excessively consumed. On the other hand, a block grease prepared in accordance with our invention which contains 12 per cent of sodium and calcium soaps and 1.5 per cent of carnauba wax will lubricate bearings operating at 320° F. without being excessively consumed.

Resort may be had to such modifications and variations as fall within the spirit of the invention and the scope of the appended claims.

What we claim is:

1. A block grease of spongy texture which is not rapidly consumed at high bearing temperatures which comprises a major amount of a soda lime base block grease, and minor amounts, sufficient to decrease the rate of consumption of said block grease at high bearing temperatures, of the alkali saponification products of a wax selected from the group consisting of carnauba wax and ouricury wax, and of excess alkali in the form of a sodium carbonate, the amount of said wax in the form of its alkali saponification products being from 0.2 to 20 per cent by weight, and the amount of said excess alkali being from about 0.2 to about 0.4 per cent by weight.

2. The grease of claim 1, wherein the wax is carnauba wax.

3. A block grease of spongy texture which is not rapidly consumed at high bearing temperatures which comprises from about 70 per cent to about 85 per cent by weight of a mineral oil which has been thickened to a grease with from about 10 per cent to about 25 per cent by weight of a mixture of sodium and calcium soaps and which also contains from about 0.2 per cent to about 2.5 per cent by weight of carnauba wax in the form of its alkali saponification products and from about 0.2 per cent to about 0.4 per cent by weight of excess alkali in the form of a sodium carbonate.

4. The grease of claim 3, wherein the alkali saponification products of the carnauba wax include the sodium soaps of the carnauba wax acids and the carnauba wax alcohols.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
26,693	McMunn	Jan. 3, 1860
1,599,854	Van Gundy	Sept. 14, 1926
2,148,292	Francis	Feb. 21, 1939
2,197,263	Carmichael	Apr. 16, 1940
2,321,384	Hemker	June 8, 1943

OTHER REFERENCES

"Lubricating Greases," Klemgard, page 587, published by the Reinhold Publishing Corp., 1937, New York, N. Y.