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GREASE

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The invention described herein may be manufactured and used by or for the Government for governmental purposes without the payment to us of any royalty thereon.

The present invention relates to lubricants and more particularly to an improved grease, stabilized against deterioration in the presence of oxygen.

Prior art greases heretofore employed in the recoil mechanisms of modern artillery pieces, rapidly break down when subjected to an oxygen-containing atmosphere, particularly in the presence of copper or alloys thereof. With such prior art greases, copper or an alloy thereof appears to act as a decomposition accelerator or catalyst, greatly increasing the rate of breakdown of the grease in the presence of oxygen. Thus it has been found, using the Norma-Hoffmann accelerated stability test for lubricating greases (A. S. T. M. Preprint No. 94, 1938), that the induction period of prior aluminum base recoil greases drops from some 960+ hours, when the test is carried out in glass dishes without a catalyst, to perhaps 48 hours or lower when the test is performed in the presence of a catalyst comprising naval brass. In view of the fact that recoil mechanisms usually contain copper or alloys thereof such as brass, it is apparent that an improved stabilizer is required for recoil greases so that such greases may be made proof against oxidative deterioration in the presence of copper-containing metals.

The object of the present invention is to provide a grease characterized by increased stability to oxygen in the presence of copper or copper alloys such as brass.

A further object is to provide a means for increasing the oxygen induction period for aluminum base recoil greases when tested by the Norma-Hoffmann accelerated stability test in the presence of copper containing catalysts.

Another object is to provide a method of stabilizing recoil greases of all types, against oxidative deterioration in the presence of copper or copper containing metals.

Still another object is the provision of a new and improved grease stabilizer which greatly increases the stability of greases to the destructive effect of oxygen in the presence of a copper-containing catalyst, which stabilizer does not impair the other desirable properties of the grease.

Other objects and advantages of the present invention will appear as the invention is hereinafter more particularly described.

We have found that the foregoing objects may be accomplished in accordance with the present invention, one aspect of which involves the incorporation in the recoil grease, of a small percentage of a stabilizer selected from the group consisting of:

(*a*) Aryl-substituted naphthyl amines, such as phenyl beta naphthyl amine;

(*b*) Aryl-substituted arylene diamines, such as diphenyl para phenylene diamine; or

(*c*) Alkoxy diaryl amines, such as diparamethoxydiphenylamine.

The foregoing stabilizers may be used singly, or in combination with each other, or in combination with other compounds, as desired.

In carrying out a preferred embodiment of the present invention, the herein disclosed stabilizers, either singly or in combination, are incorporated at any suitable step in the compounding of the grease. Thus, for example, the improved stabilized grease of the present invention may be compounded by first incorporating a conventional grease-base soap (for example, aluminum stearate, oleate and/or palmitate) in a suitable grease-base oil. After the soap and oil have been suitably mixed, a small percentage, for example 1% (based on the weight of the soap and oil) of the inhibitor is added to the grease-base. In some instances it may also be desirable to incorporate a small percentage of a free fatty acid, such as stearic acid, in order to overcome any tendency of the ultimate grease, to bleed. After the components have been thoroughly mixed, the grease is maintained at an elevated temperature in order to permit the escape of any occluded air. When the air has been completely removed, the grease is allowed to chill very slowly, thereby insuring a grease having a proper jell and consistency.

In order more clearly to disclose the nature of the present invention, a preferred embodiment thereof will hereinafter be described. In this example, phenyl beta naphthyl amine will be employed as the specific stabilizer. It should be clearly understood, however, that this is done solely by way of example and is not to be construed as a limitation upon the spirit and scope of the appended claims, inasmuch as any of the foregoing stabilizers may be substituted for the

rticular compound given in the illustrative ample:

EXAMPLE

A suitable grease-base oil such as heavy recoil l is weighed out in an amount sufficient to pro- de 83.8% by weight of the ultimate grease. hereafter 15% of a metal soap such as mmercial "aluminum stearate" (usually a ixture of the stearate, oleate and palmitate) is len added to the oil and the mixture thoroughly gitated in the cold in order to wet the soap ith the oil. Thereafter the oil and soap may e permitted to stand. However, if preferred, the ixture may be heated immediately to approxi- ately 325-359° F., with constant agitation, in or- er to melt the soap. When the soap has been mpletely melted and thoroughly incorporated the oil, 1% of phenyl beta-naphthylamine is irred into the grease. Preferably there is also corporated at this time approximately 0.2% of ee fatty acid, such as stearic acid, the partic- lar amount selected being sufficient to insure at the ultimate grease will not be alkaline in eaction.

The agitation of the mixture is continued for bout half an hour after which the material is rawn or run into shallow containers which are hen placed in an oven heated to approximately 50°. The containers are allowed to remain in he oven for approximately 24 hours in order to llow occluded air to escape. The initial tem- erature of 350° F. may be very gradually reduced ver the twenty-four hour period, or the grease nay be held at a constant temperature for the nitial twenty-four hour period and thereafter ermitted to chill very slowly. The latter method f cooling is usually preferable inasmuch as it nsures a grease which has a proper jell and vhich also will not show any tendency to bleed.

In some instances, it may be desirable to add ther stabilizers in addition to the phenyl beta- aphthylamine employed in the foregoing ex- mple. Thus for example, the stabilizer may onsist of two parts by weight phenyl beta- aphthylamine, one part by weight dipara- nethoxydiphenylamine and one part of diphen- lparaphenylene diamine, this mixture being mployed in the grease to the extent of 1% by veight. In other instances it may be desirable o use the various stabilizers individually or in various proportions. Although we have discov- ered that the employment of a stabilizer com- prising substantially one per cent (1%) by weight of the total weight of the grease provides optimum antioxidant performance from the economic standpoint it will be apparent that other percentages of stabilizer may also be em- ployed, the antioxidant effect thereof being de- pendent to a certain extent upon the quantity or proportion used.

In order to establish the stability of greases prepared in accordance with the present inven- tion, experiments were conducted using the Nor- ma-Hoffmann accelerated stability test for lu- bricating greases. This procedure included the loading of various greases, prepared under iden- tical conditions, in a Norma-Hoffmann bomb. Each dish of the bomb contained a naval brass catalyst having a surface area of 0.756 square inch. The subsequent details of the tests were similar to those set forth in the A. S. T. M. Pre- print mentioned above. The results are indicat- ed in the table.

Table

Inhibitor and content	Induction period (I. P.)	Pressure drop during I. P./100 hrs.(P.D.)	Condition of grease after oxidation	Condition of catalyst after oxidation
A [1] (1%)	189	4.8	Semi-liquid, rancid.	Green coating.
B [2] (1%)	384	4.75	Liquid, rancid	Slight corrosion.
B (0.50%) + C [3] (0.25%) + D [4] (0.25%)	413	4.44	Black semi-solid, rancid.	Green coating.

[1] A—phenyl alpha naphthylamine.
[2] B—phenyl beta naphthylamine.
[3] C—diparamethoxydiphenylamine.
[4] D—diphenyl para phenylene diamine.

As will be apparent from the induction peri- ods noted in the foregoing table, a grease-con- taining phenyl beta naphthylamine is substan- tially more stable under the conditions of the test than a grease containing the alpha position isomer (384 compared with 189). Further im- provement was noted when the other inhibitors were incorporated along with the phenyl beta naphthylamine, as indicated by the last example in the table (I. P.=413). The grease represent- ed by sample "A" is, in turn, far more stable than the same grease without the phenyl alpha naphthylamine, as indicated by the fact that a similar grease containing no inhibitor will fre- quently show an induction period below 30, and greases containing the usual commercial "metal deactivaters" do not show any measurably greater stability under the conditions of the test. It will thus be seen that the incorporation of a very small percentage of the improved stabilizers of the present invention results in an entirely unexpected (and indeed, inexplicable) enhance- ment in stability of greases under the conditions of the Norma-Hoffmann test, when carried out in the presence of a copper-containing metal.

In the foregoing detailed description it will be apparent to those skilled in the art that many variations may be made without departing from the spirit and scope of the present invention. Thus for example, although the invention has been illustrated by particular reference to the phenyl-substituted beta-naphthylamines, the other aryl amino stabilizers hereinbefore de- scribed may be employed where desired, the par- ticular compound selected being determined in part by the availability of the compound in ques- tion. Many other variations will be apparent to those skilled in the art. We therefore intend to be restricted only in accordance with the follow- ing patent claims.

We claim:

1. A grease comprising an oil, a metal soap, and a stabilizer consisting substantially of a mix- ture of about 25% diparamethoxydiphenylamine, about 25% diphenylparaphenylene diamine and about 50% phenyl beta naphthylamine.

2. A grease comprising a metal soap, an oil, and an antioxidant stabilizer comprising sub- stantially one per cent (1%) by weight of said grease, said stabilizer comprising substantially a mixture of about 25% dipara methoxy-diphenyl- amine, about 25% diphenyl paraphenylene di- amine and about 50% phenyl beta naphthyl- amine, and wherein said soap comprises an aluminum salt of a long chain fatty acid.

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CERTIFICATE OF CORRECTION.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, first column, line 15, for "325-359° F." read --325-350° F.--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 19th day of June, A. D. 1945.

Leslie Frazer

(Seal) Acting Commissioner of Patents.