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STABILIZING MIXTURE OF 4,4'-DIAMINODIPHENYL ETHER AND 2,4'-DIAMINODIPHENYL ETHER

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This invention relates to the stabilization of lubricants by incorporating therein a novel synergistic inhibitor composition of exceptionally high potency.

As will be shown by the examples appended to the present specification, the novel inhibitor composition of the present invention possesses a high degree of effectiveness far beyond that obtained through the use of each of the components singly. The synergistic effect obtained through the use of both of the components of the composition is truly unexpected because, as will be subsequently described in detail, the individual components are of similar chemical constitution and would not be expected to exert a synergistic effect. Furthermore, this synergistic effect appears to be peculiarly adapted to specific substrates. The data in the examples of the present application will show exceptionally high synergistic effects in lubricants. On the other hand, no such synergistic effect was observed in other substrates such as gasoline, rubber and fatty material.

The lubricants which are stabilized in accordance with the present invention are either solid or liquid and are obtained from any suitable source, generally being either of synthetic or petroleum origin. In recent years stringent requirements have been established for lubricants in certain applications including, for example, winter grade crank case oils, turbine engine oils, aviation instruments, automatic weapons, etc. For example, aircraft gas turbines require oils capable of providing satisfactory lubrication at temperatures ranging as low as —65° F. and as high as 275° F. during use. Temperatures up to 500° F. are encountered for intervals of from one to two hours during shutdown. It is apparent that these lubricants must possess high stability. Other lubricants also are considerably improved through the use of the novel synergistic mixture of the present invention.

As hereinbefore set forth, the lubricants may be solid and thus are included under the classification of grease. These may be either of synthetic or petroleum origin. The synthetic greases generally are referred to as lithium base grease, sodium base grease, calcium base grease, barium base grease, strontium base grease, aluminum base grease, mixed base greases, as barium-calcium, barium-lithium, etc. These greases are solid or semi-solid gels and, in general, are prepared by the addition to mineral or synthetic oils of hydrocarbon-soluble metal soaps or salts of higher fatty acids as, for example, lithium stearate, calcium stearate, aluminum naphthenate, etc. The grease may contain thickening agents such as silica, carbon black, talc, organic modified bentonite, etc., polyacrylates, amides, polyamides, aryl ureas, methyl N-n-octadecyl terephthalomate, etc. Another type of grease is prepared from oxidized petroleum wax, to which the saponifiable base is combined with the proper amount of the desired saponifying agent, and the resultant mixture processed to produce a grease. Other types of greases in which the features of the present invention are usable include petroleum grease, whale grease, wool grease, etc., and those made from inedible fats, tallow, butcher's waste, etc.

The lubricating oils may be of synthetic or petroleum origin. The synthetic lubricating oils are of various types including aliphatic esters, polyalkylene oxides, silicones, esters of phosphoric and silicic acids, highly fluorine-substituted hydrocarbons, etc. Of the aliphatic esters, di-(2-ethylhexyl) sebacate is being used on a comparatively large commercial scale. Other aliphatic esters include dialkyl azelates, dialkyl suberates, dialkyl pimelates, dialkyl adipates, dialkyl glutarates, etc. Specific examples of these esters include dihexyl azelate, di-(2-ethylhexyl) azelate, di-3,5,5-trimethylhexyl glutarate, di-3,5,5-trimethylpentyl glutarate, di-(2-ethylhexyl)pimelate, di-(2-ethylhexyl) adipate, triamyl tricarballylate, pentaerythritol tetracaproate, dipropylene glycol dipelargonate, 1,5-pentanediol-di-(2-ethylhexanonate), etc. The polyalkaline oxides and derivatives include polyisopropylene oxide, polyisopropylene oxide diether, polyisopropylene oxide diester, etc. The silicones include methyl silicone, methylphenyl silicone, etc., and the silicates include, for example, tetraisooctyl silicate, etc. The highly fluorinated hydrocarbons include fluorinated oil, perfluorohydrocarbons, etc.

The lubricating oils of petroleum origin include those referred to as motor lubricating oil, railroad type lubricating oil, marine oil, transformer oil, turbine oil, transmission oil, differential oil, diesel lubricating oil, gear oil, cutting oil, rolling oil, cylinder oil, hydraulic oil, slushing oil, specialty products oil, etc.

The synergistic composition of the present invention serves a number of important functions depending upon the particular lubricant being stabilized and the conditions under which the lubricant is stored, transported and used. These functions include oxidation inhibitor (peroxide decomposer), color stabilizer, retarding sediment or sludge formation, dispersion of sediment when formed, bearing corrosion inhibitor, ring antiplugging additive, extreme pressure additive, acidity neutralizer, antibleeding additive, etc.

In one embodiment the present invention relates to the method of stabilizing a lubricant which comprises incorporating in said lubricant a stabilizing concentration of a synergistic inhibitor composition of from about 5% to about 95% by weight of a 4,4'-diaminodiphenyl ether and from about 95% to about 5% by weight of a 2,4'-diaminodiphenyl ether.

From the above embodiment, it will be noted that the inhibitor composition of the present invention contains both a 4,4'-diaminodiphenyl ether and a 2,4'-diaminodiphenyl ether in specified proportions. In one embodiment the diaminodiphenyl ethers are free of substitutions on the nitrogen atoms. In another embodiment of the diaminodiphenyl ethers have 1, 2, 3 or 4 hydrocarbon substituents attached to the nitrogen atom or nitrogen atoms. In still another embodiment mixtures of the unsubstituted and of the substituted or mixtures of the differently substituted diaminodiphenyl ethers are employed. In any case, however, it is required that the 4,4'- and 2,4'-diaminodiphenyl ethers be utilized and that these be used in the specific proportions herein set forth. It is understood that these substituted and unsubstituted diaminodiphenyl ethers are included in the present specification and claims in the reference to a 4,4'-diaminodiphenyl ether and a 2,4'-diaminodiphenyl ether.

The synergistic effect is obtained when utilizing from about 5% to about 95% by weight of a 4,4'-diaminodiphenyl ether and from about 95% to about 5% by weight of a 2,4'-diaminodiphenyl ether. In a more specific embodiment the inhibitor composition contains from about 20% to about 80% by weight of a 4,4'-diaminodiphenyl ether and from about 80% to about 20% by weight of a 2,4'-diaminodiphenyl ether.

It generally is preferred that the substituted diamino-

diphenyl ethers are N,N'-dialkyl diaminodiphenyl ethers and that the alkyl groups are of secondary or tertiary configuration and contain from 3 to about 12 carbon atoms. Illustrative compositions include mixtures within the proportions hereinbefore set forth of the following: N,N'-diisopropyl 4,4'-diaminodiphenyl ether and N,N'-diisopropyl 2,4'-diaminodiphenyl ether, N,N'-di-sec-butyl 4,4'-diaminodiphenyl ether and N,N'-di-sec-butyl 2,4'-diaminodiphenyl ether, N,N'-tert-butyl 4,4'-diaminodiphenyl ether and N,N'-tert-butyl 2,4'-diaminodiphenyl ether, N,N'-sec-amyl 4,4'-diaminodiphenyl ether and N,N'-sec-amyl 2,4'-diaminodiphenyl ether, N,N'-tert-amyl 4,4'-diamino-diphenyl ether and N,N'-tert-amyl 2,4'-diaminodiphenyl ether, N,N'-sec-hexyl 4,4'-diaminodiphenyl ether and N,N'-sec-hexyl 2,4'-diaminodiphenyl ether, N,N'-tert-hexyl 4,4'-diaminodiphenyl ether and N,N'-tert-hexyl 2,4'-diaminodiphenyl ether, N,N'-sec-heptyl 4,4'-diaminodiphenyl ether and N,N'-sec-heptyl 2,4'-diaminodiphenyl ether, N,N'-tert-heptyl 4,4'-diaminodiphenyl ether and N,N'-tert-heptyl 2,4'-diaminodiphenyl ether, N,N'-sec-octyl 4,4'-diaminodiphenyl ether and N,N'-sec-octyl 2,4'-diaminodiphenyl ether, N,N'-tert-octyl 4,4'-diaminodiphenyl ether and N,N'-tert-octyl 2,4'-diaminodiphenyl ether, etc.

In the specific compounds hereinbefore set forth, the alkyl groups are of the same chain length and configuration. It is understood that, in another embodiment, the alkyl groups may be different and the synergistic composition then will include mixtures such as the following: N,N'-diisopropyl 4,4'-diaminodiphenyl ether and N,N'-di-sec-butyl 2,4'-diaminodiphenyl ether, N,N'-diisopropyl 4,4'-diaminodiphenyl ether and N,N'-di-tert-butyl 2,4'-diaminodiphenyl ether, N,N'-di-sec-butyl 4,4'-diaminodiphenyl ether and N,N'-di-tert-butyl 2,4'-diaminodiphenyl ether, etc., N-isopropyl-N'-tert-butyl 4,4'-diaminodiphenyl ether and N-isopropyl-N'-tert-butyl 2,4'-diaminodiphenyl ether, N-sec-amyl 4,4'-diaminodiphenyl ether, N-sec-hexyl-N'-tert-octyl 2,4'-diaminodiphenyl ether, etc.

While it is preferred that the alkyl groups attached to the nitrogen atoms are of secondary or tertiary configuration, in some cases the alkyl groups may be of primary configuration and are selected from methyl, ethyl, propyl, butyl, amyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, etc. In still another embodiment the substitution may include a mixture of the primary and secondary or tertiary alkyl groups as, for example, compositions comprising a mixture of N-methyl-N'-isopropyl 4,4'-diaminodiphenyl ether and N-methyl-N'-isopropyl 2,4'-diaminodiphenyl ether, N-methyl-N,N'-diisopropyl 4,4'-diaminodiphenyl ether and N-methyl-N,N'-diisopropyl 2,4'-diaminodiphenyl ether, N,N'-dimethyl-N,N'-diisopropyl 4,4'-diamniodiphenyl ether and N,N'-dimethyl-N,N'-diisopropyl 2,4'-diaminodiphenyl ether, etc.

From the above description, it will be noted that a number of different 4,4'-diaminodiphenyl ethers and 2,4'-diaminodiphenyl ethers may be used in accordance with the present invention. These different mixtures may be manufactured by separately preparing the 4,4'-diaminodiphenyl ether and the 2,4'-diaminodiphenyl ether and subsequently mixing them in the desired proportions, or the mixture of the desired proportions may be manufactured by utilizing selected reactants in the preparation of these compounds. For example, when the composition is prepared by starting with nitrophenol and nitrochlorobenzene, both para-nitrochlorobenzene and ortho-nitrochlorobenzene are used in the proportions desired in the final composition. Regardless of the method of preparing or mixing the different components of the composition, it is necessary, as hereinbefore set forth, that both components are present in the final composition and that they are present in the specific proportions hereinbefore set forth.

It is understood that the different compositions which are employed in the present invention are not necessarily equivalent, but all of them will exhibit a synergistic effect. The specific composition to be used will depend upon the availability of the reactants and the cost of preparation, as well as upon the particular lubricant in which the composition is to be used. As another advantage to the composition of the present invention, it may be prepared as a mixture of compounds and will not require separation of the individual components of the mixture, but the mixture as produced is used and thus avoids the time and expense which otherwise would be required in separating individual components.

In general, the inhibitor composition of the present invention is utilized in a concentration of from about 0.001% to about 5% and preferably of from about 0.01% to about 3% by weight of the lubricant, although in some cases higher or lower concentrations may be employed.

It is understood that the inhibitor composition of the present invention may be used along with other additives incorporated in lubricants including, for example, higher alcohols, esters, organic amines, polybutene, sulfurized fatty materials, sulfur chlorine compounds, dyes, fillers, etc. In some cases it may be of advantage to also include a metal deactivator as, for example, disalicylal diaminopropane, etc., or to include other additives such as tricresyl phosphate, dialkylphenols, trialkyl phenols including 2,6-di-tert-butyl-4-methylphenol, 2,4-di-methyl-6-tert-butylphenol, alkylated diphenyl amines, phenyl naphthyl amines, dialkyl phenylene diamines, diarylphenylene diamines, diamino diphenyl methanes, tetraalkyl diaminodiphenyl methanes, diphenylamine, aminophenols, alkylamino phenols, phenothiazine, organic selenium compounds, zinc dialkyldithiocarbamates, cadmium, dialkyldithiocarbamates, naphthols, hydroxydiphenylamines, dinaphthyl-p-phenylene diamine-p-phenylene diamine, N-phenyl-N'-cyclohexyl-p-phenylene diamine, reaction products of sulfur dichloride or sulfur monochloride with alkylphenols, aniline, alkylanilines, reaction products of formaldehyde and alkylphenols, guaiacol, etc.

In other cases, especially in lubricating oils, the synergistic mixture may be used in combination with the following bearing corrosion inhibitors, peroxide decomposers or antioxidants: zinc, dialkyldithiophosphates, phosphorus pentasulfide-various olefin reaction products, sulfur-olefin reaction products, sulfurized terpenes, aromatic hydroxysulfides and disulfides and their neutralization products with calcium, barium and magnesium oxides or hydroxides, various calcium, barium, magnesium sulfonates, dialkyldithiophosphates, barium or calcium alkylphenates, etc., as well as additional corrosion inhibitors, extreme pressure additives, viscosity index improvers, detergents, etc. When desired, the inhibitor composition of the present invention may be prepared as a mixture with one or more of these other additives and incorporated in the lubricant in this manner.

The inhibitor composition of the present invention is incorporated in the lubricant in any suitable manner and at any suitable stage of preparation. For example, in the preparation of grease, the inhibitor composition may be added to one or more components of the grease prior to compositing or processing thereof or it may be added to the mixture at any time, preferably before final processing in order to obtain intimate mixing and dissolving of the inhibitor composition in the grease.

The following examples are introduced to illustrate further the novelty and utility of the present invention but not with the intention of unduly limiting the same.

EXAMPLE I

The grease used in this example is a lithium base grease and was prepared by the following general method. Approximately 92% of a commercial Mid-Continent lubricating oil having a S.A.E. viscosity of 20 was mixed with approximately 8% by weight of lithium stearate. The mixture was heated to about 450° F. with constant agitation. Subsequently the grease was cooled while

agitating to 320° F. and at this temperature the inhibitor in the proper concentration was added. Agitation was continued and the mixture was allowed to cool to about 250° F. and the grease then further cooled slowly to room temperature.

The stability of the grease was tested according to ASTM D–942 method, in which method a sample of the grease is placed in a bomb and maintained at a temperature of 210° F. Oxygen is charged to the bomb, and the time required for a drop of 5 pounds' pressure is taken as the induction period.

In this set of runs, the inhibitors, when employed, were incorporated in the grease in a concentration of 0.3% by weight. The first sample of grease did not contain an inhibitor and was used as a control. The second sample of grease contained 0.3% by weight of N,N'-diisopropyl-2,4'-diaminodiphenyl ether. The third sample of grease contained 0.3% by weight of N,N'-diisopropyl-4,4'-diaminodiphenyl ether. The last sample of grease contained 0.3% by weight of a synergistic mixture of about 70% by weight of N,N'-diisopropyl-4,4'-diaminodiphenyl ether and about 30% by weight of N,N'-diisopropyl-2,4'-diaminodiphenyl ether. The results of these runs are reported in the following table:

Table I

Number	Inhibitor	Induction Period (Hours to 5 pounds pressure drop)
1	None	9.5
2	N,N'-diisopropyl-2,4'-diaminodiphenyl ether.	830
3	N,N'-diisopropyl-4,4'-diaminodiphenyl ether.	513
4	Synergistic mixture of about 70% of compound No. 3 and about 30% of compound No. 2.	1,873

From the data in the above table, it will be noted that the mixture of compounds exhibited an exceedingly high degree of synergism. Actually the run was terminated after 1873 hours and before a pressure drop of 5 pounds was reached. It was apparent that this mixture was so exceptionally synergistic that the stability imparted to the grease was far beyond that required for general use. Therefore, it was unnecessary to continue the run. The pressure drop after 1873 hours was only 3.5 pounds.

It will be noted that the 1873 hours far exceeded the expected stability. Normally it would be expected that the stability would be somewhere between the 830 and 513 hours obtained through the use of these compounds individually, more specifically about 600 hours. In contrast to this expected stability, the stability obtained through the use of the mixture reached the surprising figure of greater than 1873 hours.

EXAMPLE II

Another set of runs was made similar to that described in Example I except that 0.1% by weight of the inhibitor, when employed, was incorporated in the grease. The results of these runs are shown in the following table:

Table II

Number	Inhibitor	Induction Period (Hours to 5 pounds pressure drop)
5	None	9.5
6	N,N'-diisopropyl-2,4'-diaminodiphenyl ether.	160
7	N,N'-diisopropyl-4,4'-diaminodiphenyl ether.	160
8	Synergistic mixture of about 70% of compound No. 7 and about 30% of compound No. 6.	378

Here again, is seen that the synergistic mixture imparted stability to the grease far beyond that which would be expected.

EXAMPLE III

As hereinbefore set forth, special uses of grease require a high degree of stability at higher temperatures. In order to evaluate the stability of the grease at a higher temperature, the following runs were made with different samples of the grease described in Example I and in the same manner described therein, except that the temperature was 250° F. instead of 210° F. The inhibitors, when employed, were utilized in a concentration of 0.3% by weight of the grease. The results of these runs are shown in the following table:

Table III

Number	Inhibitor	Induction Period (Hours to 5 pounds pressure drop)
9	None	2
10	N,N'-diisopropyl-2,4'-diaminodiphenyl ether.	116
11	N,N'-diisopropyl-4,4'-diaminodiphenyl ether.	39
12	A synergistic mixture of about 60% of compound No. 10 and about 40% of compound No. 11.	362

It is seen that even at this higher temperature, the synergistic mixture served to impart a stability to the grease far beyond that expected through the use of each of the compounds singly. As before, it would be expected that the stability of the grease would be somewhere between the 116 hours and 39 hours obtained through the use of each of these compounds separately, more specifically about 85 hours. In contrast, the actual stability obtained through the use of the synergistic mixture is the surprisingly high period of 362 hours.

EXAMPLE IV

This example reports results using different mixtures of the inhibitor compounds described in Example III and shows that the synergistic effect prevails in all these mixtures. These runs were made using different samples of the grease and in the same manner as described in Example III, and 0.3% by weight of the different inhibitor mixtures. The results of these runs are shown in the following table:

Table IV

Number	Percent by weight of N,N'-diisopropyl-2,4'-diaminodiphenyl ether	Percent by weight of N,N'-diisopropyl-4,4'-diaminodiphenyl ether	Induction Period (Hours to 5 pounds pressure drop)
13	10	90	274
14	30	70	309
15	40	60	362
16	50	50	250
17	60	40	249
18	70	30	2 4

Here again, it will be noted that the mixture is synergistic throughout the ranges shown in the table. These results are to be compared with those of compounds 10 and 11 shown in Table III. This particular set of runs did not include compositions containing 5% of one of the compounds and 95% of the other, but other runs have shown that the synergistic effect also prevails through these ranges. It will be noted that synergistic mixture 15 is the same as synergistic mixture 12 reported in Table III and also that in this particular set of runs this mixture appears to be somewhat better than the other mixtures. It is understood that the various mixtures will not necessarily be equivalent in the same or

different lubricants but that all of the mixtures within the proportions specified herein will exhibit a synergistic effect.

EXAMPLE V

This example reports runs made in a lithium base grease prepared with a different commercial Mid-Continent lubricating oil of S.A.E. 20. The lithium base grease was prepared in substantially the same manner described in Example I except for the different oil, and was evaluated at 250° F. This different grease had a control (no inhibitor) stability of 4 hours, whereas the grease used in Examples I and II had a control stability of 2 hours.

In these runs the unsubstituted diaminodiphenyl ethers were utilized. The results of these evaluations are reported in the following table:

Table V

Number	Inhibitor	Induction Period (Hours to 5 pounds pressure drop)
19	None	4
20	2,4'-diaminodiphenyl ether	121
21	4,4'-diaminodiphenyl ether	142
22	Synergistic mixture of about 70% of compound No. 21 and about 30% of compound No. 20.	224

Here again, it will be noted that the mixture exerted a synergistic effect. As before, it would be expected that the stability imparted to the grease would be between 121 hours and 142 hours which were obtained with the compounds singly, more specifically about 136 hours. In contrast, the synergistic mixture imparted a stability to the grease of 224 hours.

EXAMPLE VI

This example illustrates the use of the synergistic mixture of the present invention in a calcium base grease, which is prepared by the following general method. The calcium soap is prepared by mixing the fat, hydrated lime equivalent to about one-seventh of the fat, mineral oil sufficient to make about one-fourth of the total charge, and about 0.5% of water. The ingredients are mixed and heated to about 140° F. and then dry lime is added, agitation and heat continued until saponification is completed. Additional oil then is added and the mixture heated to 220–230° F. Additional oil is added to bring the mixture to the desired consistency, and then is heated at 170°–180° F. At this point, 0.02% by weight of a synergistic mixture of about 80% by weight of N,N'-di-sec-butyl 4,4'-diaminodiphenyl ether and about 20% by weight of N,N'-di-sec-butyl 2,4'-diaminodiphenyl ether, along with other additives, is added, and the mixing is continued until formation of grease of the desired structure.

EXAMPLE VII

This example illustrates the use of the synergistic mixture of the present invention in a motor lubricating oil of petroleum origin. These runs were conducted in a Lauson engine, using a jacket temperature of 210° F. and an oil temperature of 280° F. A commercial lubricating oil of S.A.E. 20 viscosity was used and the runs were continued for 115 hours. Pertinent results of these runs, using a sample of the lubricating oil which did not contain an inhibitor, and a different sample of the same oil containing 0.5% by weight of a synergistic mixture of about 70% by weight of N,N'-diisopropyl 2,4'-diaminodiphenyl ether and about 30% by weight of N,N'-diisopropyl 4,4'-diaminodiphenyl ether are reported in the following table:

Table VI

Additive	None	Synergistic Mixture
Bearing weight loss, grams	2.9021	0.01
Oil consumption, ml./hr.	6.03	5.70
Used oil neutralization number	10.78	0.18
Used oil saponification number	46	0.66
Used oil pentane insolubles, percent	5.16	0.11
Used oil, viscosity, SSU at 100° F	742	368
Used oil, viscosity, SSU at 210° F	74.7	56.3

Referring to the data in the above table, it will be noted that the synergistic mixture of the present invention served to considerably reduce bearing weight loss, which illustrates the corrosion inhibiting and acidity neutralizing properties of the synergistic mixture. It also will be noted that the used oil neutralization number is considerably lower, thus illustrating the antioxidant or peroxide decomposing properties of the synergistic mixture. The lower viscosities of the used oil containing the synergistic mixture illustrate the effectiveness of the mixture to inhibit formation of heavier products.

EXAMPLE VIII

This example illustrates the use of the synergistic mixture of the present invention in a synthetic lubricant. The effectiveness of the synergistic mixture was determined in an accelerated test in which a sample of the synthetic lubricant is placed in a glass test cell fitted with a condenser and a gas delivery tube extending through the top to near the bottom of the test cell. The test cell is immersed in an oil bath to maintain a temperature of 204° C. Air is passed through the cell at a rate of 5 liters of air per hour. Samples of the lubricant are withdrawn periodically for analysis. The induction period is taken as the number of hours required to reach an acid number of 5.

The synthetic lubricant used in this example is di-(2-ethylhexyl) sebacate. The synergistic mixture used in this example contained about 70% by weight of N,N'-diisopropyl 2,4'-diaminodiphenyl ether and about 30% by weight of N,N'-diisopropyl 4,4'-diaminodiphenyl ether.

When evaluated in the above manner, the control sample (not containing inhibitor) reached the induction period in 12 hours. On the other hand, the sample of synthetic lubricant containing the synergistic mixture did not reach the induction period until 50 hours. Thus, it will be seen that the synergistic mixture effectively stabilized the synthetic lubricant.

I claim as my invention:

1. A synergistic inhibitor mixture of from about 5% to about 95% by weight of a 4,4'-diaminodiphenyl ether and from about 95% to about 5% by weight of a 2,4'-diaminodiphenyl ether.

2. A synergistic inhibitor mixture of from about 5% to about 95% of N,N'-diisopropyl 4,4'-diaminodiphenyl ether and from about 95% to about 5% by weight of N,N'-diisopropyl 2,4'-diaminodiphenyl ether.

3. A synergistic inhibitor mixture of from about 5% to about 95% of N,N'-di-sec-butyl 4,4'-diaminodiphenyl ether and from about 95% to about 5% by weight of N,N'-di-sec-butyl 2,4'-diaminodiphenyl ether.

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