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MINERAL OIL COMPOSITIONS AND METHODS OF SUPPRESSING FOAMING IN OILS

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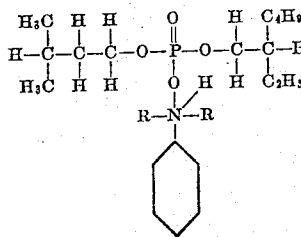
This invention relates to mineral oil compositions and methods of suppressing foaming in oils.

In lubricating machinery with oils, and in handling oils as by pumping, conditions are often such that the oil is subjected to violent agitation in the presence of air; conditions such as to produce foam or froth. In some cases foaming or frothing is quite objectionable. For example, in lubricating automobile gearing (in the gear box, and rear axle) foaming produced by the churning of the gears gives rise to excessive leakage and loss of lubricant past retainer rings, etc. Moreover, foaming seriously interferes with the proper functioning of the oil. Automobile gears such as those used in transmission and differential gear assemblies often operate at very high speeds, as well as under high tooth pressures; therefore they require a very good lubricant to prevent or retard excessive wear due to metal-to-metal contact, and foaming is undesirable. The lubricants employed are viscous oils, often containing a so-called extreme pressure agent to assist in maintaining an oil film between the teeth; sulfurized sperm oil being especially good. If the lubricant contains air dispersed through it, it lacks proper film forming properties. Moreover foaming oil is much less effective to conduct heat away from the working zone. These difficulties are often aggravated by the fact that some extreme pressure agents present in the oil actually increase the foaming characteristics of the oil composition.

Foaming is also objectionable in other situations; for example, during the pumping of oils, and in compounding oils with extreme pressure agents and other additives—in operation involving thorough agitation. Nearly all petroleum oils foam to some extent under violent agitation. The more viscous the oil the greater the amount of foam and the longer it persists after agitation is stopped.

Among the objects of our invention are the provision of a method whereby foaming or frothing of oils as described is suppressed; and the provision of gear lubricants and other lubricating compositions which are characterized by freedom from tendency to foam or froth even under severe conditions.

We have discovered that foaming of petroleum oils, even under the most violent conditions described, can be suppressed or prevented by incorporating in the mineral oil a small proportion of certain amine salts of isoamyl octyl phosphate having the following formula:



wherein R represents a substituent selected from the class consisting of methyl and ethyl groups and hydrogen.

For example, a small proportion of dimethylaniline isoamyl octyl phosphate having an adjusted acidity equivalent to a pH value within the range pH 2 to 4 is most effective. Likewise, the diethylaniline and aniline salts of isoamyl octyl phosphate, respectively, are also excellent foam inhibitors.

In general, dissolving almost any additive in oil has a tendency to increase foaming more or less. Our new agents or compounds are remarkable in that they suppress foaming, even when present in small amounts, say 0.01 to 1.0 per cent by weight of the composition. They have no deleterious effect on the lubricant properties or other properties of the oil.

Our agents can be incorporated in all sorts of oils, to achieve the stated results. One particularly advantageous field of use is in gear lubricants for automobiles and the like. As stated, these lubricants are viscous and they contain extreme pressure agents or other additives; both of which facts make for heavy foaming. By dissolving a fraction of a per cent of our agent foaming is prevented. For example, such a gear lubricant, within the purview of our invention, has the following compositions (per cent by weight):

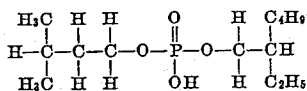
Refined high-viscosity, lubricating oil	89 to 95.0
Refined sulfurized sperm oil	5 to 10.0
Dimethylaniline isoamyl octyl phosphate	0.01 to 1.0

Another field of use is in light (low viscosity) oils used in so-called fluid drives or in hydraulic drives. With these light oils (which are often of viscosity as low as SAE 10 grade) agitation may produce considerable foam, even though the foam subsides very quickly on ceasing agitation. Our new agents are also useful in cutting oils and indeed in any oil or oily composition, whether used as a lubricant or not, in which it is desired to prevent foaming.

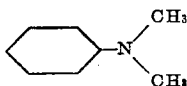
In preparing our new agents, the selected amine is reacted with isoamyl octyl acid ortho-phosphate to obtain the desired amine salt thereof; the pH of the reaction mixture being adjusted to within the range 2.0 to 5.5 in so preparing and recovering the amine salt. For example, dimethylaniline is brought into reaction with isoamyl octyl acid ortho-phosphate, and the pH of the reaction mixture is adjusted to within the range 2 to 4. With the theoretical molecular ratio of the two reactants, 1:1, the product is apt to be slightly too acid, with a pH below 2, and this is taken care of by using a slight excess of the dimethylaniline so that the molecular ratio is between 1:1 and 1:1.1. The reaction product is an oil-soluble, oily liquid and dissolving it in oils or oil compositions is very simple. Likewise, the amine salts obtained by reacting isoamyl octyl acid ortho-phosphate with aniline and diethylaniline, respectively, are oily liquids. They too readily dissolve in oils and oil compositions and can be easily incorporated and blended with mineral lubricating oils and lubricant compositions.

The proportion of the agent to be dissolved in the oil or oil base depends on the viscosity of the oil, its ingredients, and the severity of the conditions of use. Ordinarily 0.01 to 1.0 per cent of the agent is employed; these amine isoamyl octyl phosphates being very effective agents for the present purposes.

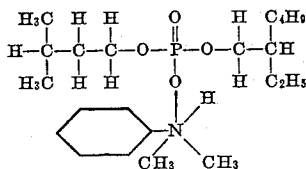
Isoamyl octyl orthophosphate, is a brown, oily liquid, its specific gravity is 1.009 at 24°/4° C., and its viscosity 210, 75 and 29 centipoises at 25, 50 and 75° C. respectively. It is insoluble in water and soluble in mineral oils to the extent of 11.70 parts in 100 parts of oil at 20° C. The orthophosphate decomposes at 302 to 311° F. It has the structural formula:



Dimethylaniline is a yellow or brown liquid, of specific gravity 0.962 at 15° C., having a melting point of 1.67° C. (29° F.) and a boiling point 192.5–193.5° C. (379–380° F.) and is soluble in alcohol and ether. It has a molecular weight of 121.09 and has the following formula:



Our preparation, the reaction product of the above, has the probable formula:



The following example illustrates one advantageous way of preparing our foam suppressor:

Example I.—One hundred and twenty-one pounds of dimethylaniline were added to an iron vessel equipped with an air lance and 281 pounds of isoamyl octyl ortho-phosphate were introduced over a period of one hour; the slow rate of addition being desirable to maintain the temperature of the reaction mixture below 185° F. At the conclusion of this reaction, the temperature was 180° F., and the pH of the mixture was 2.0. A pH of 3.0 was desired; 12.0 pounds of dimethylaniline were added, which brought the pH

to 3.0. The reaction product was an oily substance, readily soluble in mineral oils and having the following properties:

Gravity, ° API	7.4
Specific gravity, 60°/60° F.	1.019
Viscosity, SUV 100° F.	468
Pour, ° F.	—50

As the dimethylaniline isoamyl octyl phosphate is substantially insoluble in water, the pH equivalent is measured by the following expedient. Normal butanol (which contains a small amount of water) is adjusted to exact neutrality, pH 7.0, and the sample is dissolved therein. The pH of the solution is then measured in the ordinary way as for aqueous solutions, by electrometric or colorimetric methods. The butanol appears to serve as a blending agent, for dissolving the sample in the water, so to speak.

It will be noted that the measured pH of the aminophosphate is low despite the fact that a slight excess of amine is present. This is because the reaction involves neutralizing a rather strong acid with a very weak base.

Moreover, traces of mono-, di- and tribasic acids may be formed under the conditions of the pH determination. At any rate, the results are as stated.

The following example illustrates the preparation of a gear lubricant in accordance with the invention.

Example II.—A highly viscous, highly refined Pennsylvania oil was selected as the base, and in it was dissolved 0.04 per cent of the oily reaction product of Example I. As shown in the following table the change in viscosity and in color by incorporation of the agent was negligible.

	Straight oil	Improved oil
Gravity, ° API	26.6	26.5
Viscosity, SUV:		
100° F.	1.918	1.905
210° F.	141.7	142.2
Color, NPA.	4.75	4.75

To evaluate the foaming properties of the straight oil and the improved oil, samples (500 cc.) of the two oils were subjected to foam test No. 1 (described below). The results were as follows:

Composition	Straight oil	Improved oil
End of stirring (15 minutes):		
Temperature, ° F.	84	83
Oil+foam, cc.	1,740	540
Ratio of volume of oil+foam to the volume of the original oil	3.48	1.08
After 1 hr. standing:		
Temperature, ° F.	77	77
Oil+foam, cc.	660	500
Ratio of volume of oil+foam to the volume of the original oil	1.32	1.0
Nature of foam	Fine	No foam

It will be observed that the straight oil developed twice its volume of foam under the conditions of the test and that an appreciable volume of foam remained even after an hour's standing; while the improved oil developed only a negligible amount of foam, which was all gone at the end of an hour.

Decreasing the percentage to 0.01 still leaves the oil much less susceptible to foaming than the straight oil.

The foam suppressor as stated can be added to compounded lubricants to prevent foaming thereof. It is compatible with most of the other

types of improvement agents now employed and the amount required to prevent foaming in such compounded lubricants is relatively small and does not deleteriously affect the other properties of compounded lubricants. This is an important part of the invention, as certain of the agents employed to impart particular properties to lubricating oils have been found also to promote the foaming of the oil composition when agitated. By adding even traces of the present foam suppressor, such compounded lubricants become very resistant to foaming, even under drastic service conditions.

In making up compounded lubricants of this type it is advantageous to dissolve the aminophosphate in the oil first, as thereby foaming is prevented during the step of mixing in the extreme pressure agent or other additive. But so far as performance of the lubricant in service is concerned, the order of addition makes no difference.

The following example illustrates the properties of an extreme pressure gear lubricant prepared in accordance with the invention.

Example III.—A conventional gear lubricant contains 92.5 per cent by volume of a highly refined, viscous Pennsylvania oil and 7.5 per cent of refined sulfurized sperm oil (Smith & Cantrell Patent 2,179,064). A similar lubricant prepared according to the invention contains these oils in proportions 92.46 and 7.5 per cent respectively and 0.04 per cent of the product of Example I.

The viscosity of the conventional lubricant was 1918 SUV at 100° F., 767 at 130° F., and 141.7 at 210° F. The viscosity of the improved lubricant was only negligibly different from this, and the same was true of the other characteristics—gravity, viscosity, index, flash and fire tests, pour test, color, sulfur, copper strip tests, centrifuge test (for gravity-separable matter), and carbon residue. The neutralization of the new lubricant was 0.56 against 0.50 for the old. The surface tensions were nearly identical. The Almen and Timken tests, indicative of the lubricating value of the oil, as secured on the compounded oil before the addition of the foam suppressing agent, were the same as the corresponding tests made after the addition of the indicated amount of this foam suppressor.

In other words, the characteristics of the two lubricants were practically the same, except for the foaming properties. The following are comparable tests (the nature of which is described in detail below) on these properties. The samples were 500 cc. each, the speed of the agitators 550 R. P. M. and the initial temperature 77° F.

Foam tests

	Old lubricant	New lubricant	
After stirring for.....	15 min.	15 min.	44 hr.
Temperature, °F.....	84.....	83.....	83.....
Oil+foam, cc.....	1.740.....	540.....	590.....
Vol. of oil+foam.....	3.48.....	1.08.....	1.18.....
Vol. of oil.....			
After standing 1 hr. after stirring stopped:			
Temperature, °F.....	77.....	77.....	77.....
Oil+foam, cc.....	660.....	500.....	540.....
Vol. of oil+foam.....	1.32.....	1.00.....	1.08.....
Vol. of oil.....			
Nature of foam.....	Coarse.....	None.....	

To evaluate the foaming tendency of petroleum oils and compositions a test was devised which affords an exceptionally accurate indica-

tion of the comparative foaming tendencies. A sample of oil is subjected to very drastic foaming conditions under a standardized procedure which makes it possible to determine quantitatively the tendency of the oil to foam and the stability or permanency of the foam produced. In general it resembles a somewhat similar testing method employed by the General Motors Corporation for determining the foaming tendencies of gearing lubricants, but has been modified in the direction of greater accuracy and in order to make it possible to record more comprehensible test data.

Foaming test (No. 1)

An agitating means is provided which is an adaptation of an ordinary commercial motor-driven household mixer—a "Sunbeam Mixmaster," Model 1, manufactured by the Flexible Shaft Company, Chicago, Ill. The device employed in the test is the usual household model with two slight changes: the turntable of the usual household mixer is replaced by a rigid platform, and a cylindrical brass container having an inside diameter of 7 inches and an inside height of 4 inches is substituted for the usual household glass mixing bowl. The container is fitted with a gage for measuring the depth of oil or oil and foam therein before and after agitation.

The agitator device itself comprises a pair of motor driven beaters which are of the convex outside surface type as described in U. S. Patent 2,161,881, each beater having a pair of blades of the type indicated and being so positioned with respect to each other that the two pairs of beater elements are at right angles and rotate in opposite directions in closely spaced, overlapping paths. In operating position, the beaters are perpendicular to the base of the mixer, as shown in Patent 2,161,881. In the present test they are centered in the container and the bottom of the beaters is spaced approximately $\frac{1}{8}$ inch from the bottom of the pan when the latter is positioned on the rigid platform. With 500 cc. of oil in the container, the beaters are submerged in the initial oil sample only to a depth of $\frac{3}{4}$ of one inch. The beaters are rotated at a speed of 550 R. P. M. controlled within ± 10 R. P. M.

A measured sample of 500 cc. (± 5 cc.) of oil is introduced into the container, the temperature of the sample is brought to 77° F. and the container is then placed in position and the beater elements lowered into operating position.

The motor is started and adjusted to the speed indicated above. The beaters agitate the oil and beat air into the sample. Agitation is continued for exactly 15 minutes.

The motor is now stopped, the beaters are removed from the oil, and any oil or foam adhering to the beaters is permitted to drain into the container, which takes one or two minutes. The foam level is then immediately determined, and the temperature of the sample is measured. It is then possible to calculate the ratio of the volume of oil and foam to the volume of the original oil, with correction for any temperature changes.

The container is removed and allowed to stand quiet for one hour (measured from the time the stirring is stopped). The volume and temperature measurements are taken again, and serve to indicate stability or permanence of the foam produced.

The test procedure may of course be varied, as for example by changing the size of the sample, the speed of agitation or the time of agitation, or by taking the final measurements at an earlier or later period. However, in the test referred to in the specific example above the procedure was precisely as indicated.

Test No. 2 is the same as test No. 1 except that agitation is continued for 44 hours instead of 15 minutes. (Longer periods of agitation may be employed if a still more drastic test is required.)

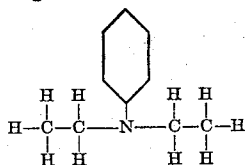
In some very low viscosity oils considerable foam may develop during agitation, which disappears very quickly when agitation is stopped. These conditions are found, for example, in marine turbine lubrication systems (which use light oils) at the point where returned oil is discharged violently into a reservoir. To evaluate foaming in such cases there is employed a third test No. 3, similar in all respect to test No. 1 except that the measurements of oil and foam are made while the motor is still running. The following example shows how a turbine oil treated according to the invention behaves under such conditions.

Example IV.—A highly refined Pennsylvania turbine oil was subjected to test No. 3. After 15 minutes agitation the oil plus foam measured 800 cc. as compared with 500 cc. for the original oil.

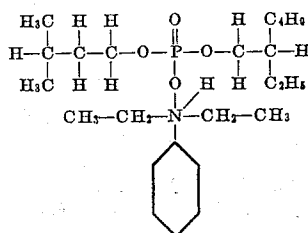
The same oil after treating with 0.01 per cent of dimethylaniline isoamyl octyl phosphate tested only 540 cc. for the oil plus foam.

In the foregoing examples, we have illustrated certain embodiments of our invention employing dimethylaniline isoamyl octyl phosphate which is an excellent and advantageous agent for the purposes of this invention. However, as described ante, we may also employ with advantage other amine salts of isoamyl octyl phosphate prepared by reacting the acid phosphate ester with aniline and diethylaniline respectively. Both of these amines are well known compounds and are commercially available and can be readily converted into agents suitable for the present purposes.

For instance, diethylaniline is a colorless to yellow liquid. It has a melting point of -34.5°C . and a boiling point of 213.5°C . It is soluble in alcohol, ether, chloroform and insoluble in water. It has a molecular weight of 149.13 and has the following formula:



The reaction product of this amine with isoamyl octyl acid ortho-phosphate has the following probable formula:

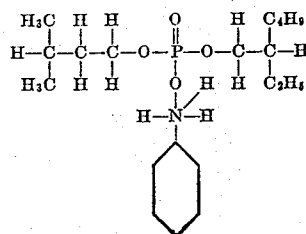


The above compound is an excellent agent for the present purposes.

Another effective agent is the amine salt prepared from aniline. This amine, also known as phenylamine, is a colorless to yellow liquid having a boiling point of 184.4°C . It is soluble in alcohol and ether and slightly soluble in water. It has a molecular weight of 93.12 and has the following formula:



The reaction product of this amine with isoamyl octyl acid ortho-phosphate has the probable formula:



The above amine salt is readily soluble in oils and is another excellent agent for the present purposes.

The two amine salts described above may be used in lieu of the dimethylaniline isoamyl octyl phosphate in the examples given ante. Additional embodiments of our invention are further illustrated in the examples given post which illustrate other advantageous ways of practicing our invention.

Example V.—One hundred and forty-nine pounds of diethylaniline were added to an iron vessel equipped with an air lance, and 281 pounds of isoamyl octyl ortho-phosphate were introduced over a period of one hour; the slow rate of addition being desirable to maintain the temperature of the reaction mixture below 185°F . At the conclusion of this reaction, the temperature was 180°F . and the pH of the mixture was 2.0. A pH of 3.5 was desired; a further 15 pounds of diethylaniline were added, which brought the pH to 3.5. The reaction product was an oily liquid. It was readily soluble in mineral oils.

As the diethylaniline isoamyl octyl phosphate is substantially insoluble in water, the pH equivalent is measured by the following expedient described ante in Example I. Here again, the measured pH of the aminophosphate is low despite the fact that a slight excess of amine is present.

The following example illustrates the preparation of a gear lubricant in accordance with our invention using the aminophosphate obtained in Example V.

Example VI.—A highly viscous, highly refined Pennsylvania oil was selected as the base, and in it was dissolved 0.04 per cent of the oily reaction product of Example V. As shown in the following table the change in viscosity and in color by incorporation of the agent was negligible.

Composition	Straight oil	Improved oil
Gravity, $^{\circ}\text{API}$	26.6	26.5
Viscosity, SUV:		
100 $^{\circ}\text{F}$	1,918	1,905
210 $^{\circ}\text{F}$	141.7	142.3
Color, NPA.....	4.75	4.75

To evaluate the foaming properties of the straight oil and the improved oil, samples (500 cc.) of the two oils were subjected to foam test No. 1 (described ante). The results were as follows:

Composition	Straight oil	Improved oil
At end of stirring:		
Temperature, °F.....	84	84
Volume of oil and foam, cc.....	1,740	540
Ratio ¹	3.48	1.08
After 1 hr. standing:		
Temperature, °F.....	77	77
Volume of oil and foam, cc.....	660	500
Ratio ¹	1.32	1.0
Nature of foam.....	Fine	No foam

¹ Ratio of $\frac{\text{volume of oil and foam}}{\text{volume of original oil}}$

It will be observed that the straight oil developed twice its volume of foam under the conditions of the test and that an appreciable volume of foam remained even after an hour's standing; while the improved oil developed only a negligible amount of foam, which was all gone at the end of an hour.

Decreasing the percentage to 0.01 still leaves the oil much less susceptible to foaming than the straight oil.

Further, this foam suppressor can also be added to compounded lubricants to prevent foaming thereof. It was compatible with most of the other types of improvement agents now employed and the amount required to prevent foaming in such compounded lubricants is relatively small and does not deleteriously affect the other properties of compounded lubricant. By adding even traces of diethylaniline isoamyl octyl phosphate, such compounded lubricants become very resistant to foaming even under drastic service conditions.

The following example illustrates the properties of one such extreme pressure gear lubricant prepared in accordance with our invention.

Example VII.—A modern gear lubricant contains 92.5 per cent by volume of a highly refined, viscous Pennsylvania oil and 7.5 per cent of refined monoester of a sulfurized fatty acid. A similar lubricant prepared according to the invention contains these oils in proportions 92.46 and 7.5 per cent respectively and 0.04 per cent of the product of Example V. The viscosity of the modern gear lubricant was 1920 SUV at 100° F., 768 at 130° F. and 141.8 at 210° F. The viscosity of the improved lubricant was only negligibly different from this, and the same was true of the other characteristic—gravity, viscosity index, flash and fire tests, pour test, color, sulfur, copper strip tests, centrifuge test (for gravity-separable matter), and carbon residue. The neutralization number of the new lubricant was 0.56 against 0.50 for the old. The surface tensions were nearly identical. The Almen and Timken tests, indicative of the lubricating value of the oil, as secured on the compounded oil before the addition of the foam suppressing agent, were the same as the corresponding tests made after the addition of the indicated amount of this foam suppressor.

In other words, the characteristics of the two lubricants were practically the same, except for the foaming properties. The following are comparable tests (the nature of which is described in detail ante; see foaming test No. 1) on these properties. The samples were 500 cc. each, the speed of the agitators 550 R. P. M. and the initial temperature 77° F.

Foam tests

	Old lubricant	New lubricant	
After stirring for.....	15 min.	15 min.	44 hr.
Temperature, °F.....	84	83	83
Oil+foam, cc.....	1,740	540	590
Vol. of oil+foam.....	3.48	1.08	1.18
Vol. of oil.....			
After standing 1 hr. after stirring stopped			
Temperature, °F.....	77	77	77
Oil+foam, cc.....	660	500	540
Vol. of oil+foam.....	1.32	1.00	1.08
Vol. of oil.....			
Nature of foam.....	Coarse	None	

The above tests and data clearly show the superiority of our new lubricant containing diethylaniline isoamyl octyl phosphate.

The following example shows how a turbine oil can be improved according to our invention:

Example VIII.—A highly refined Pennsylvania turbine oil was subjected to test No. 3. After 15 minutes' agitation the oil plus foam measured 800 cc. as compared with 500 cc. for the original oil.

The same oil after treating with 0.01 per cent of diethylaniline isoamyl octyl phosphate tested only 540 cc. for the oil plus foam.

The following example illustrates one advantageous way of preparing another of our foam suppressors, namely, aniline isoamyl octyl phosphate:

Example IX.—Ninety-three pounds of aniline were added to an iron vessel equipped with an air lance and 281 pounds of isoamyl octyl ortho phosphate were introduced over a period of one hour; the slow rate of addition being desirable to maintain the temperature of the reaction mixture below 212° F. At the conclusion of this reaction, the temperature was 180° F. and the pH of the mixture was 2.0. A pH of 3.5 was desired; a further 9 pounds of aniline were added, which brought the pH to 3.5. The reaction product was an oily liquid. It was readily soluble in mineral oils.

As the aniline isoamyl octyl phosphate is substantially insoluble in water, the pH equivalent is measured by the following expedient. Normal butanol (which contains a small amount of water) is adjusted to exact neutrality, pH=7.0, and the sample is dissolved therein. The pH of the solution is then measured in the ordinary way as for aqueous solutions, by electrometric or colorimetric methods. The butanol appears to serve as a blending agent for dissolving the sample in water, so to speak.

It will be noted that the measured pH of this aminophosphate is low despite the fact that a slight excess of amine is present. This is because the reaction involves neutralizing a rather strong acid with a very weak base.

Moreover, traces of mono-, di and tribasic acids may be formed under the conditions of the pH determination. At any rate, the results are as stated.

The following example illustrates the preparation of a gear lubricant in accordance with the invention:

Example X.—A highly viscous, highly refined Pennsylvania oil was selected as the base, and in it was dissolved 0.04 per cent of the oily reaction product of Example IX. As shown in the following table the change in viscosity and in color by incorporation of the agent was negligible.

Composition	Straight oil	Improved oil
Gravity, °API.....	26.6	26.5
Viscosity, SUV:		
100° F.....	1,918	1,005
210° F.....	141.7	142.3
Color, NPA.....	4.75	4.75

To evaluate the foaming properties of the straight oil and the improved oil, samples (500 cc.) of the two oils were subjected to foam test No. 1 (described ante). The results were as follows:

Composition	Straight oil	Improved oil
At end of stirring (15 minutes):		
Temperature, °F.....	84	83
Volume of oil and foam, cc.....	1,760	635
Ratio ¹	3.52	1.07
After 1 hr. standing:		
Temperature, °F.....	77	77
Volume of oil and foam, cc.....	660	500
Ratio ¹	1.32	1.0
Nature of foam.....	Fine	No foam

¹ Ratio of volume of oil and foam
volume of original oil

It will be observed that the straight oil developed more than twice its volume of foam under the conditions of the test and that an appreciable volume of foam remained even after an hour's standing; while the improved oil developed only a negligible amount of foam, which was all gone at the end of an hour.

Decreasing the percentage of this agent to 0.01 still leaves the oil much less susceptible to foaming than the straight oil.

Likewise, this foam suppressor, namely, aniline isoamyl octyl phosphate, can also be added to compounded lubricants to prevent foaming thereof. It is compatible with most of the other types of improvement agents now employed and the amount required to prevent foaming in such compounded lubricant is relatively small and does not deleteriously affect the other properties of compounded lubricants. By adding even traces of aniline isoamyl octyl phosphate, such compounded lubricants become very resistant to foaming even under drastic service conditions.

The following example illustrates the properties of one such extreme pressure gear lubricant prepared in accordance with our invention.

Example XI.—A modern gear lubricant contains 92.5 per cent by volume of a highly refined, viscous Pennsylvania oil and 7.5 per cent of refined monoester of a sulfurized fatty acid. A similar lubricant prepared according to the invention contains these oils in proportions 92.46 and 7.5 per cent, respectively, and 0.04 per cent of the product of Example IX.

The viscosity of the modern gear lubricant was 1918 SUV at 100° F., 768 at 130° F. and 141.8 at 210° F. The viscosity of the improved lubricant was only negligibly different from this, and the same was true of the other characteristics—gravity, viscosity index, flash and fire tests, pour test, color, sulfur, copper strip tests, centrifuge test (for gravity-separable matter), and carbon residue. The neutralization number of the new lubricant was 0.56 against 0.50 for the old. The surface tensions were nearly identical. The Almen and Timken tests, indicative of the lubricating value of the oil, as secured on the compound oil before the addition of the foam suppressing agent, were the same as the corresponding tests made after the addition of the indicated amount of this foam suppressor.

In other words, the characteristics of the two lubricants were practically the same, except for the foaming properties. The following are comparable tests (the nature of which is described in detail ante; see Foaming test) on these properties. The samples were 500 cc. each, the speed of the agitators 550 R. P. M. and the initial temperature 77° F.

Foam tests

	Old lubricant	New lubricant
After stirring for.....	15 min.	15 min.
Temperature, °F.....	84.	83.
Oil+foam, cc.....	1,740	540.
Vol. of oil+foam.....	3.48	1.08
Vol. of oil.....		
After standing 1 hr. after stirring stopped:		
Temperature, °F.....	77	77
Oil+foam, cc.....	660	500
Vol. of oil+foam.....	1.32	1.00
Vol. of oil.....		
Nature of foam.....	Coarse	None

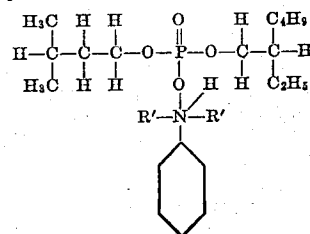
The above tests and data clearly show the superiority of our new lubricants containing aniline isoamyl octyl phosphate.

The following example shows how a turbine oil can be improved according to our invention, using aniline isoamyl octyl phosphate as the improvement agent or foam suppressor.

Example XII.—A highly refined Pennsylvania turbine oil was subjected to test No. 3. After 15 minutes agitation the oil plus foam measured 800 cc. as compared with 500 cc. for the original oil.

The same oil after treating with 0.01 per cent of the aniline isoamyl octyl phosphate tested only 540 cc. for the oil plus foam.

That is, as shown in the above examples, aniline isoamyl octyl phosphate is an effective foam suppressor and is useful in many embodiments of our invention. However, in the general practice of our invention it is advantageous to employ the foam suppressors prepared from isoamyl octyl acid phosphate and the tertiary amines, namely dimethyl or diethyl aniline. This advantageous class of foam suppressors may be represented by the following generic formula:



wherein R' represents a methyl or ethyl group. This class of compounds is readily soluble in mineral oils and lubricants and are excellent foam suppressors. Their use in mineral lubricating oils and compounded lubricants has been fully illustrated ante.

While our invention has been described above with reference to various specific examples and embodiments, it will be understood that the invention is not limited to such illustrative examples and embodiments, but may be variously practiced within the scope of the claims herein made.

This application is a continuation-in-part of our prior application Serial No. 421,842, filed December 5, 1941. The aromatic amine salts of isoamyl octyl orthophosphate herein disclosed are new chemical compounds which are claimed per

se in our continuing application, Serial No. 639,-701, filed January 7, 1946.

What we claim is:

1. A mineral oil composition resistant to foaming, comprising mineral oil and a small proportion of dimethylaniline isoamyl octyl orthophosphate exhibiting a pH value between approximately 2 and 4.

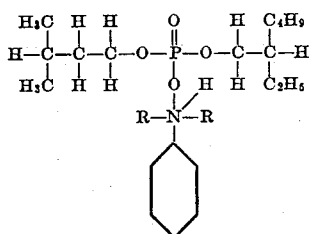
2. The composition of claim 1 wherein the proportion of said orthophosphate is between 0.01 and 1.0 per cent by weight of the composition.

3. A mineral oil resistant to foaming comprising a small proportion of the reaction product of dimethyl-aniline and isoamyl octyl orthophosphate, said reaction product being an oily oil-soluble liquid and having an acidity equivalent to a pH value between 2 and 4.

4. A method of making mineral oil compositions which are resistant to foaming, which comprises incorporating in a mineral oil composition a small proportion of dimethylaniline isoamyl octyl phosphate, of pH value in the range 2 to 4.

5. A method of suppressing foaming of mineral oils and oil compositions which comprises incorporating and maintaining therein during use a small proportion of dimethylaniline isoamyl octyl phosphate of pH value in the range 2 to 4.

6. A mineral oil composition resistant to foaming, comprising mineral oil and a small proportion of an amine salt of isoamyl octyl phosphate having the formula



wherein R represents a substituent selected from the class consisting of methyl and ethyl groups and hydrogen.

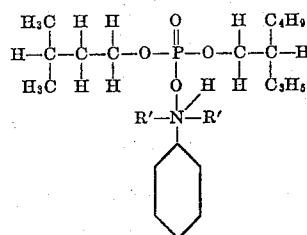
7. The composition of claim 6 wherein the proportion of said orthophosphate salt is between 0.01 and 1.0 per cent by weight of the composition.

8. The composition of claim 6 wherein said amine sale is aniline isoamyl octyl phosphate.

9. The composition of claim 6 wherein said amine salt is diethylaniline isoamyl octyl phosphate.

10. The composition of claim 6 wherein said amine salt is dimethylaniline isoamyl octyl phosphate.

11. A mineral oil composition resistant to foaming, comprising mineral oil and a small proportion of a dialkylaniline salt of isoamyl octyl phosphate having the formula:



wherein R' represents a substituent selected from the class consisting of methyl and ethyl groups.

12. A mineral oil resistant to foaming, comprising a small proportion of the reaction product of diethylaniline and isoamyl octyl phosphate, said reaction product being an oily liquid readily soluble in oil and having an acidity equivalent to a pH value between 2.0 and 5.5.

13. A mineral oil resistant to foaming, comprising a small proportion of the reaction product of aniline and isoamyl octyl phosphate, said reaction product being an oily liquid readily soluble in oil and having an acidity equivalent to a pH value between 2.0 and 5.5.

14. A mineral oil resistant to foaming, comprising a small proportion of a reaction product of isoamyl octyl orthophosphate and an amine having the following formula:



wherein R represents a substituent selected from the class consisting of methyl and ethyl groups and hydrogen, said reaction product being an oily liquid readily soluble in mineral oil.

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