

Oct. 31, 1950

V. N. BORSOFF ET AL

2,528,466

FOAM INHIBITED OILS

Filed Sept. 18, 1947

2 Sheets-Sheet 1

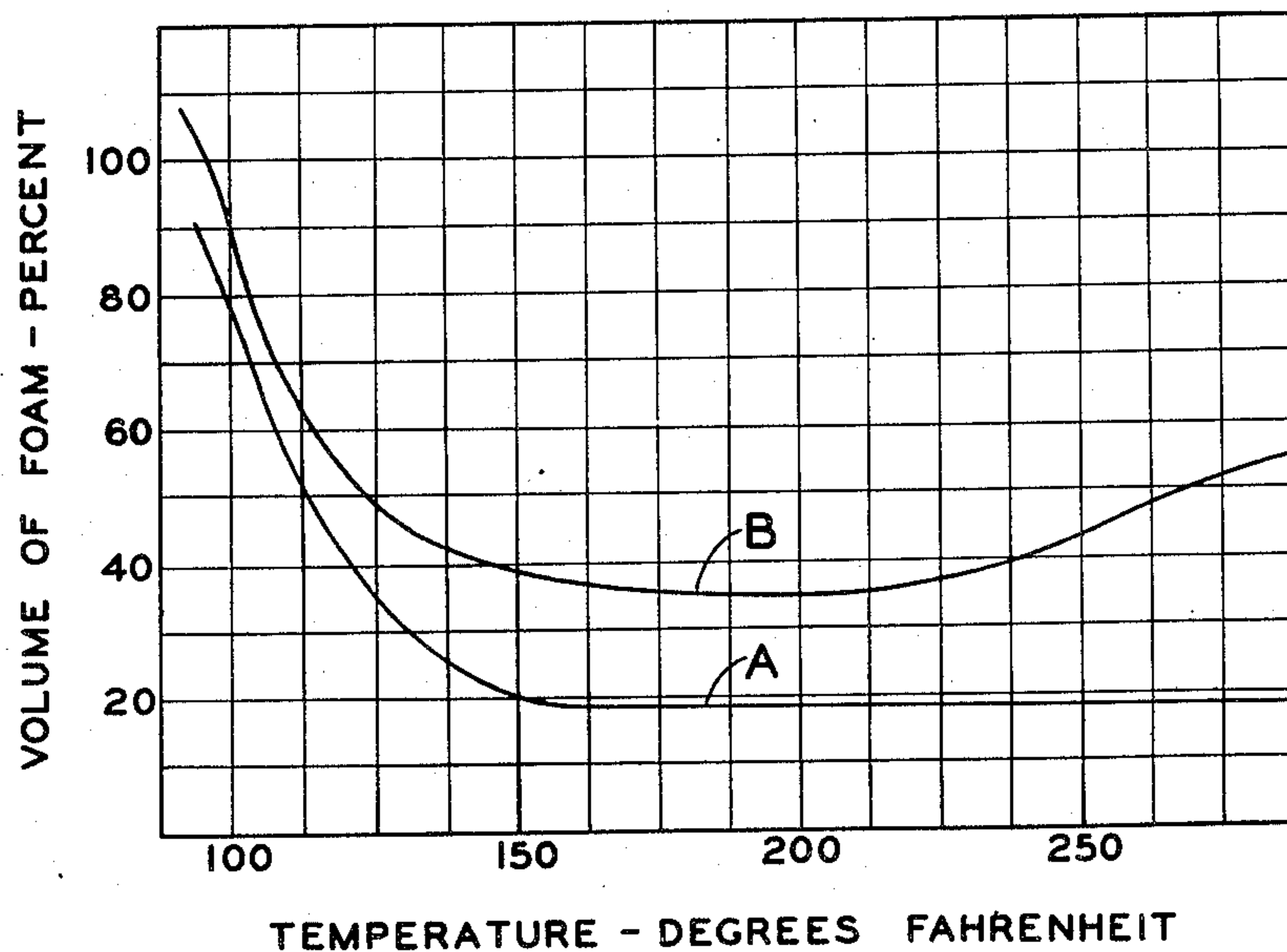


Fig. 1

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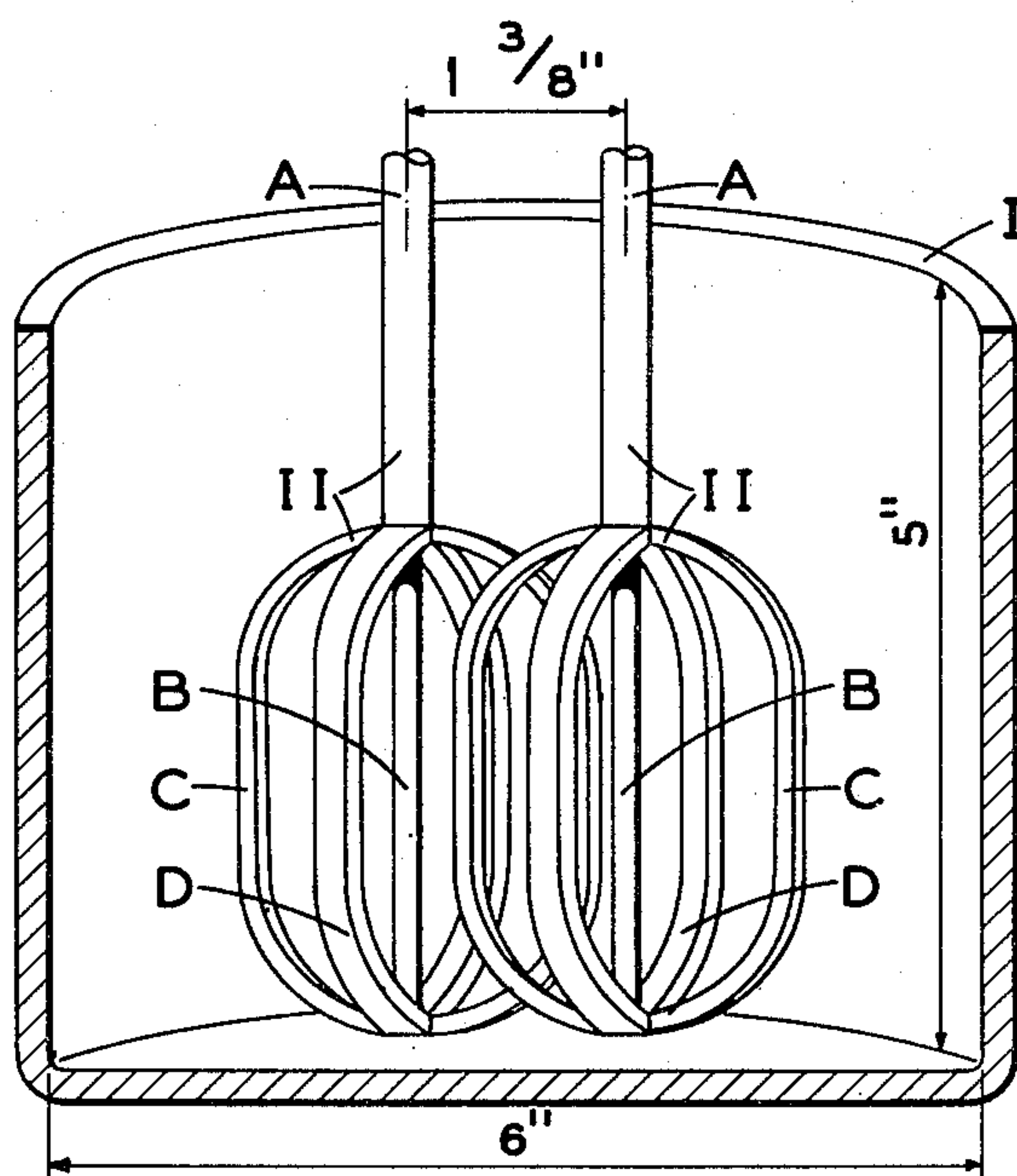


Fig. 2

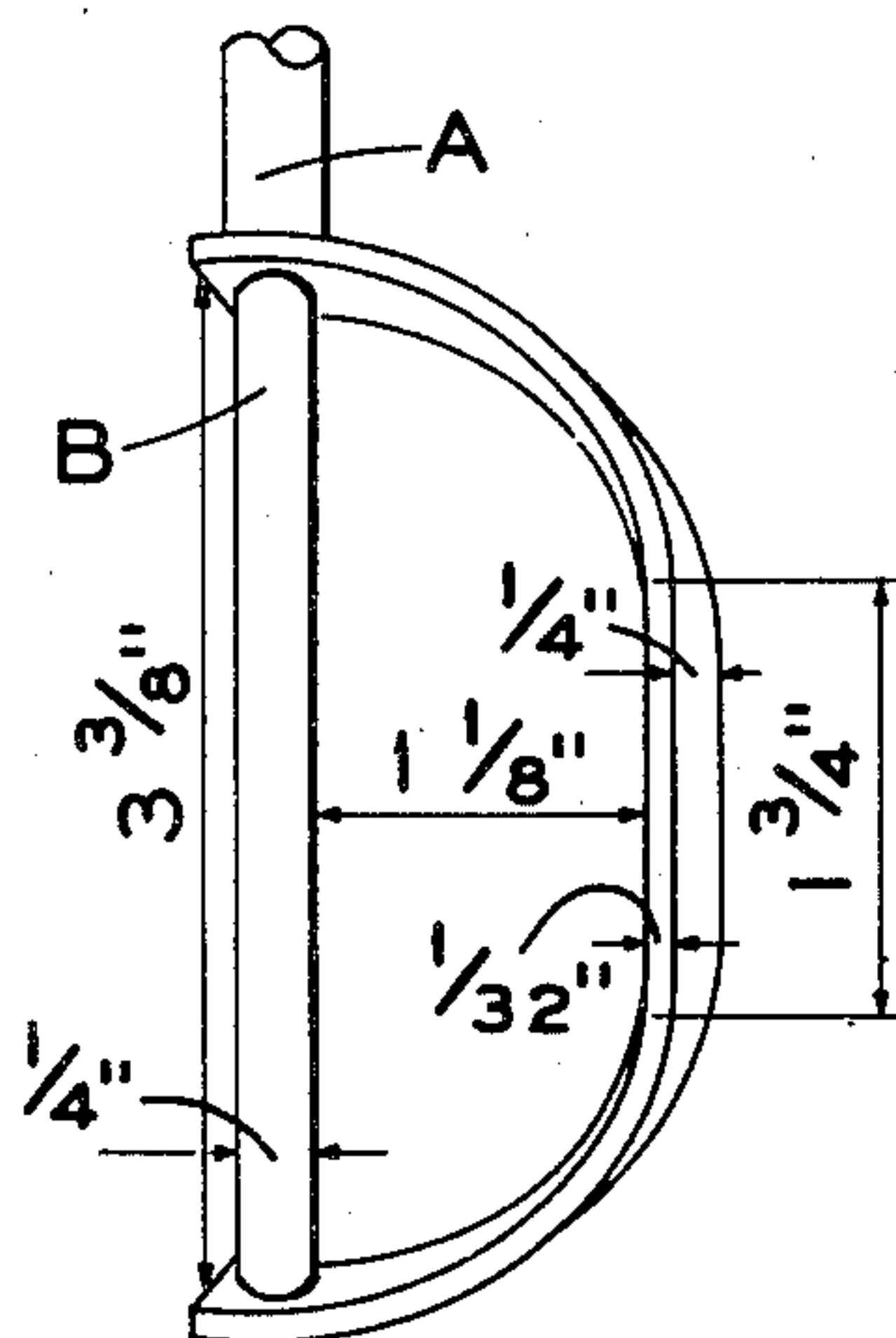


Fig. 3

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2,528,466

FOAM INHIBITED OILS

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Application September 18, 1947, Serial No. 774,744

9 Claims. (Cl. 252—32.5)

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This invention relates to oils inhibited against the formation of a stable bubble foam and to a method of inhibiting the foam formation tendency of oils.

This application is a continuation-in-part of our copending application Serial No. 450,887, filed July 14, 1942, now Patent No. 2,430,858 issued November 18, 1947.

On mixing oil with air, a certain amount of foam may be created. The amount of foam will depend, among other things, upon the character of the oil and the type of service in which it is employed. Thus most base mineral oils, that is, mineral oils uncompounded with a chemical additive, do not readily form foam, at least under ordinary service conditions. Many compounded oils tend to produce a more stable foam than the base oils from which they are compounded. Thus petroleum lubricating oils compounded with metal naphthenates, higher metal alcoholates, higher metal alkyl phenates and metal salts of higher fatty acids foam more than the corresponding base oils. Many other additives cause more foam to be produced by oils to which they are added, than is formed by the base oils.

Oil foaming, even with compounded oils which display the greatest tendency to foam, is not always troublesome. Under conditions of little or no agitation, for example, trouble due to foaming is encountered, if at all, only in very exceptional cases. On the other hand, certain drastic types of service, involving extremely violent intermixture of oil and air or combustion gases, may produce an objectionable amount of foam with many oils. Between the extremes, however, of service causing little foaming of any oil and service causing much foaming of many oils, there are types of service and certain oils which, when used in combination, cause considerable difficulty. As stated, certain compounded oils exhibit a greater tendency to foam than the base oils, and when these compounded oils are used in certain types of lubrication systems, involving greater agitation of the oil and greater intermixture with gases than the ordinary crankcase lubrication of automobiles, the greatest practical difficulty is encountered.

Illustrative of the type of lubrication system which, when used with compounded lubricating oils having a strong tendency to foam, causes practical difficulty, is the dry sump lubrication system employed in many aircraft engines. In this system, oil from the moving parts of the

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motor is collected in the crankcase and pumped from there to an external tank or sump by a scavenging pump. Oil from this sump is pumped back to the motor in the usual manner by an oil pump. The foaming difficulty arises from the fact that the scavenging pump operates at excess capacity; it pumps faster than required to pump all of the available oil in the crankcase. It therefore pumps a great deal of air along with the oil, and this at a rapid rate, and the result is entrainment of air in the oil and the formation of foam. As a consequence, excessive foaming occurs when oils such as the aforementioned compounded oils are used and oil is lost through the breather pipes, insufficient lubrication results because of the presence of air in the oil supplied to the motor, and other disadvantages result.

Oil also forms a more persistent foam if it contains water (as in solution) and is then heated and suddenly subjected to a very low pressure, as, for instance, in an airplane engine climbing from a low altitude to 40,000 feet at a high speed.

In other instances, as in the lubrication of gears with heavy oils, foaming difficulties may also be encountered.

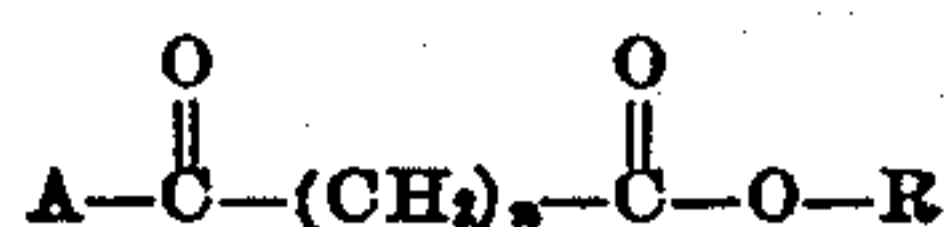
It is an object achieved by the present invention to inhibit the foaming of oils.

It is a further object achieved by the present invention to inhibit the foaming of compounded mineral lubricating oils.

It is a still further and a particular object achieved by the present invention to obviate the problem of excessive foaming of compounded mineral lubricating oils when used in types of lubrication service involving severe conditions of intermixture of oil and gases.

Other objects achieved by the invention will be apparent from the following description and from the claims.

We have discovered that foaming of oils can be inhibited by incorporating therein a small amount of an aliphatic compound containing at least one ester group and at least one aldehyde or acid carbonyl group. The active hydrogen atom on the aldehyde or carboxyl group in these compounds is believed responsible for imparting anti-rusting properties in addition to foam-suppressing properties. Illustrative of this group of compounds are those represented by the formula:



wherein A may be hydrogen or hydroxyl, R is a lower alkyl group such as methyl, ethyl, propyl, butyl, isobutyl, tertiary butyl, pentyl, hexyl, etc., and n is a small integer such as 0, 1, 2 or 3.

Preferably we use as foam inhibitors oxygen-containing aliphatic compounds containing not less than about 25 percent by weight of oxygen, and having a molecular weight not higher than about 225. We also prefer to use as inhibitors those compounds which are soluble in mineral oil to the extent used. Less soluble inhibitors are more likely to be removed by filtration or to settle out on standing. However, oil solubility is not essential to effectiveness in reducing foam and a solubility on the order of 0.001 percent by weight based on the oil is adequate.

We have found that such oxygen-containing aliphatic compounds are effective in reducing foaming even when used in very small amounts, and that they are most effective in inhibiting the foaming of compounded lubricating oils. They are, however, not limited in their effectiveness to compounded oils but may be used to advantage in dealing with a variety of foaming problems. Certain of the foam inhibitors falling within the designated class are more effective than others for a given set of conditions, whereas for another set of conditions a different group of inhibitors will be found to be superior.

Specific examples typifying the class of inhibitors which we have found to be effective are the following:

Inhibitor	Molecular Structure	Molecular Weight	Per Cent by Weight of Oxygen
Methyl glyoxalate	CH ₃ .OOC.CHO	88	59.5
Monoethyl adipate	C ₂ H ₅ .OOC(CH ₂) ₄ .COOH	174	36.8
Ethyl butanalate	C ₂ H ₅ .OOCCH ₂ CH ₂ CHO	130	36.9

Still further examples of foam inhibitors are monomethyl oxalate, monoethyl oxalate, monoethyl malonate, etc.

oil may be prepared. Such concentrates may also contain other compounding agents. By diluting the concentrate with more oil, a finished oil can be produced containing foam inhibitor, or foam inhibitor and other compounding agent or agents in the desired amount. Conveniently, concentrates containing from about 1.0 to 20 per cent by weight of foam inhibitor based on the total concentrate are thus prepared, in cases where the foam inhibitor is sufficiently oil-soluble.

The following specific examples will serve to illustrate the advantages and effectiveness of the inhibitors of the invention in reducing foam formation:

Example I.—The effectiveness of the present anti-foaming agents may be shown by means of the A. S. T. M. Foaming Test, which is also known as the U. S. Army Specification 2-104B-Test AXS 1562, a detailed description of which is obtainable from the Office, Chief of Ordnance, War Department, Washington, D. C. In this test, a column of oil is aerated under controlled conditions, and the volume of foam is recorded after 5 minutes; then the introduction of air is stopped and the amount of foam recorded for regular intervals of time. This test was applied to a compounded oil consisting of an S. A. E. blend of 70% Western acid treated naphthenic base oil and 30% Western paraffinic base oil compounded with 0.55% by weight of a calcium alkyl phenate, 0.5% by weight of a calcium alkylphenyl thiophosphate, and 0.17% by weight of an ali-

Table I

Oil Description	Temp.	cc. Foam after bubbling 5 minutes	cc. Foam uncollapsed after				
			5 min.	10 min.	15 min.	20 min.	30 min.
Compounded Oil	200° F.	650	610	580	490	390	300
	Room	460	400	390	370	360	350
Compounded Oil+0.25% Monoethyl adipate	200° F.	30	No foam after 45 seconds				
	Room	440	280	250	220	190	100

The inhibitors of our invention may be advantageously used in a wide variety of oils, including naphthenic base, paraffin and mixed base mineral oils, in synthetic oils, and in oils of viscosity ranging from 40 SSU or lower at 210° F. to 150 SSU or higher at 210° F.

Preferably the foam inhibitors of the present invention are used in the finished oil in an amount equal to about 0.001 to 1 per cent by weight based on the oil. If the oil is a compounded oil containing 0.1 to 5 per cent by weight of an additive which promotes foaming, the foam inhibitor is preferably used in an amount equal to about 10 to 200 per cent by weight based on the additive.

Concentrated solutions of foam inhibitor in

Example II.—The foaming qualities of various oils were also observed in another manner. The base stock was a solvent treated, paraffinic base oil having a viscosity of 120 SSU at 210° F. and a viscosity index of 86. The first oil tested consisted of this base stock alone and the second oil consisted of the base stock compounded with 0.25 per cent by weight of a 2:1 admixture of calcium alkyl phenate and calcium cetyl phosphate. The calcium alkyl phenate so used was the salt of an alkyl substituted phenol prepared by condensing butene polymers, having an average molecular weight of about 196, with phenol. The calcium cetyl phosphate so used was the salt prepared from a mixture of monocetyl- and di-

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cetyl-phosphoric acids. The test conditions were as follows:

500 cc. of the oil were placed in a cylindrical metal container 6 inches in diameter by 5 inches high and a "Mix-Master" stirrer of the type used in household kitchens was lowered into the oil. The oil was slowly heated by means of an electric hot plate and the amount of foam produced was measured at various temperatures up to 300° F. The speed of the stirrer was 1100 revolutions per minute. The test results at 275° F., under identical operating conditions except for variation of the oil composition, are set forth in Table II below:

Table II

	Per cent increase in volume
Uncompounded oil	11
Compounded oil	57

The per cent increase in volume referred to in the above table, and again in Table III below, is arrived at by observing the increase in volume of the mixture of foam and oil over the original volume of oil, dividing the increase by the original volume of oil, and multiplying by 100. It is, therefore, the volume of foam produced reckoned as per cent of the original volume of oil.

Example III.—Oils were tested in an apparatus comprising a reservoir adapted to be heated by a hot plate, a sump situated below the reservoir, a drain provided with a regulating valve and leading from the bottom of the reservoir to the sump, and a return conduit connected with the bottom of the sump, provided with a pump and extending into the reservoir under the surface of the oil therein. Oil, maintained at 200° F. in the reservoir, dropped from the reservoir through the drain to the sump at a rate controlled by the regulating valve. The pump in the return conduit was operated at a capacity three times the rate of flow of oil into the sump; therefore, both oil and air were pumped from the sump to the reservoir and foaming was caused in the reservoir. At the end of each 30 minutes of operation, the volume of foam and oil in the reservoir was observed. The amount of foam was calculated as the per cent increase in volume over the original volume of oil. Under identical operating conditions, various oils produced increase in volume as given in Table III below:

Table III

	Per cent increase in volume
Uncompounded oil	30
Compounded oil	200+

As stated above, the inhibitors of the invention are not all equally effective in inhibiting foam formation. Some of the inhibitors are less effective than others under all or most conditions. Some inhibitors are the most effective under certain conditions, whereas others are the most effective under other conditions.

We have observed that our inhibitors are more effective at elevated temperatures than at lower temperatures. At about the ordinary temperature, the reduction in foaming produced by the inhibitors of the invention is small, although substantial. At about 120° to 150° F. the reduction of foaming begins to be large, and above about 150° F. the reduction in foaming is very large.

The effect of temperature upon foaming of uninhibited and inhibited oils is shown by Fig. 1 of the accompanying drawings.

In Fig. 1, abscissae represent temperature in degrees Fahrenheit and ordinates represent the

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volume of foam calculated as percentage on the original volume of oil. Curve A represents the uncompounded oil of Example II, and curve B represents the same oil compounded with 0.25 per cent by weight of a 2:1 mixture of calcium alkyl phenate and calcium cetyl phosphate (the same as used in Example II). The test conditions were the same as in Example II, except that observations were made at a number of different temperatures.

It will be noted that foaming was great in the case of both oils at about 100° F., though less with oil A (uncompounded oil) and oil C (compounded oil). As the temperature increased, the foaming of both oils decreased, but the foaming of oil A and C decreased more rapidly than that of oil B. At about 190° F., however, oil B began to foam more with increase in temperature, whereas oil A continued to foam to about the same degree.

Crankcase temperatures are generally above about 200° F., although the crankcases of some engines operate at temperatures as low as about 150° F. It is at temperatures above about 150° F., especially above about 200° F., that excessive foaming is likely to occur. At these temperatures the inhibitors of our invention are highly effective in reducing foam formation.

The phenate-phosphate mixture referred to above is effective in inhibiting oxidation of lubricating oils and in reducing ring sticking. It is given by way of example of the oil-soluble metal organic salts which are widely used in lubricating oils for such purposes. The tendency of such compounding agents to promote foaming detracts somewhat from their utility, and the obviation of this defect, by the use of our foam inhibitors, is one of the principal objects accomplished by the invention.

It has been found that certain compounded oils which foam excessively also develop surfaces having the characteristics of plastic solids; that is, the surface is highly viscous, yields to and is deformed by a pressure in excess of a minimum yield value, but does not flow freely like a liquid under the force of gravity. This fact has been brought to light through the use of the torsion pendulum. This apparatus and its use to determine surface plasticity and viscosity of aqueous solutions is described by R. E. Wilson and E. D. Ries in "Surface Films as Plastic Solids," Colloid Symposium Monograph, volume I, pages 145-173, published by the Department of Chemistry, University of Wisconsin, 1923. The same apparatus and method were used to determine surface plasticity and viscosity of mineral oils.

Thus an SAE 30 blend of 70% Western acid treated naphthenic base oil and 30% Western paraffinic base oil compounded with 0.25% by weight of a 2:1 mixture of a calcium alkyl phenate and a calcium cetyl phosphate (the same as Example II) was investigated by means of the torsion pendulum. The base oil is a non-foaming oil and the compounded oil exhibits a marked tendency to foam under the conditions of Example II. It was found that at room temperature the surface of the compounded oil, on ageing, became very viscous and similar to a plastic solid. However, when 0.1% of a foam inhibitor was dissolved in the compounded oil, a similar viscosity increase occurred on ageing following which a slow decrease in surface viscosity set in and upon continued standing the surface became thixotropic; could even be made liquid by rotation of the torsion pendulum. A similar

phenomenon occurs at more elevated temperatures, such as 200° F.

Thus there is a correlation between surface viscosity and foaming tendency; the excessively foaming oil formed a surface, on ageing, similar to a plastic solid, but the foam inhibited oil formed a liquid surface or a plastic solid surface of very low yield point.

Therefore, those oils whose surfaces on ageing (a matter of minutes) exhibit the properties of plastic solids with high yield points are excessively foaming oils, while oils whose surfaces on ageing remain liquid or exhibit the properties of plastic solids with very low yield points are not excessively foaming oils. The most probable explanation of these observed phenomena seems to be as follows: All oils, when agitated with air produce foam, which consists of small portions of air each surrounded by a film of oil. In many cases this foam is unstable, probably due to the instability of the film of oil. This film is so fluid that continued agitation and collisions of the foam bubbles with each other and the sides of the vessel and draining of the liquid from the film cause rupture of the film and destruction of the bubbles. In those cases, however, where the oil surface (hence the film of oil enclosing the air in the foam) is a plastic solid, the film is not as easily ruptured because the liquid cannot drain from the film due to capillary forces set up by the plastic solid surfaces, and the foam is stable. The plasticity and stability of the oil film clearly depend, in part at least, upon the nature and amount of solutes in the oil. Thus metal salts such as metal naphthenates, metal soaps of higher fatty acids, higher metal alkyl phenates, metal salts of phosphoric acid partly esterified by long chain alcohols, and higher metal alcoholates are apparently adsorbed by the film of oil and render it more viscous, plastic and tough. We do not wish to infer that this is the only factor influencing foaming or that all foaming is due to the formation of a plastic solid film, but this is one of the principal contributing factors. The inhibitors of this invention apparently function by reducing the amount of adsorption of the foam-producing salts in the film, or are themselves adsorbed and counteract the adsorbed foam-producing salts.

Compounding agents, including oil-soluble polyvalent metal salts of organic and of organic-substituted inorganic acids, such as salts of organic substituted acids of phosphorus, which tend to cause foaming, are illustrated in Farrington et al. 2,228,659 (polyvalent metal salts of organic substituted acids of phosphorus), Rutherford et al. Reissue 22,829 (polyvalent metal salts of sulfur-containing acids of phosphorus having at least 24 carbon atoms), and Gardiner et al. 2,228,661 (alkaline earth metal alkyl phenates). Types of metal salts of organic acids are metal salts of higher fatty or aliphatic acids, metal salts of naphthenic salts, metal salts of oil-soluble substituted phenols, and metal salts of carboxylic acids containing an aryl substituent. A number of examples of these metal salts of organic acids are given in Denison et al. 2,346,155 (pp. 17-18); examples of various mixed and complex additives are given in Neely et al. 2,322,307.

Our invention, however, is not limited in its application to those oils which, because of certain salt-like compounding agents, form viscous, plastic surfaces. Our invention is applicable broadly to the inhibition of oil foaming wherever it occurs and comprises the addition of our foam in-

hibiting agents to any oil which foams excessively.

For the purpose of clarity, the terms "excessive foaming," "excessively foaming," and the like, as applied to oils herein and in the claims, refer to performance under the conditions of the following reproducible test, described with reference to Figs. 2 and 3 of the accompanying drawings. An oil which, when agitated under the conditions of this test at 275° F., increases in volume due to foaming by more than 20 per cent of its original volume, is an "excessively foaming" oil, and oils which foam less than this amount are not "excessively foaming" oils.

500 cc. of oil are placed in a cylindrical flat bottomed container 6" in diameter and 5" high (internal dimensions), the oil in the container is brought to and maintained at 275° F. by any suitable means such as a hot plate or a jacket, and the stirring elements of a "Mix-Master" stirrer of the type used in household kitchens is lowered into the container. The "Mix-Master" stirrer is the product of and obtainable from the Chicago Flexible Shaft Co., 1124 South Central Avenue, Chicago, Illinois. The stirring elements are similar in construction and action to the familiar household egg beater, consisting of two shafts oppositely rotated by a motor, and two elliptical loops attached to each shaft, one loop being at right angles to the other and the two loops having a common long axis. The dimensions and form of the vessel and stirring elements will be understood, and can be duplicated by, reference to Figs. 2 and 3.

Fig. 2 shows, partly in cross-section and partly in front elevation, the container and the stirring elements, while Fig. 3 shows in front elevation a detailed view of one-half of one loop of one stirring element.

Referring to Fig. 2, the apparatus comprises a cylindrical flat bottomed container I and two stirring elements II constructed exactly alike. The internal dimensions of the container, as shown, are a diameter of 6" and a height of 5". The stirring elements consist each of an upper shaft A geared at the top to a motor (not shown) and rigidly secured at the bottom to a lower shaft B and to two elliptical loops C and D which are disposed at right angles to each other and have each the shaft B as their long axis and as a supporting member. Shaft B is rigidly secured to the tops and bottoms of loops C and D. In the preferred construction, as shown, shaft A is sturdier than shaft B, but this is not necessary; for example, a single shaft of uniform diameter may take the place of shafts A and B, passing through the tops of loops C and D and being secured thereto at the top and bottom of the loops. The stirring elements II are so placed in vessel I that shafts A are parallel and 1 3/8" apart (center to center) and they are placed in vessel I so that a line drawn half way between and in the plane of the shafts coincides with the axis of vessel I, and the distance from the bottom of loops C to the bottom of vessel I is 1/4 inch. The shafts A are so geared to the motor that they rotate at equal speeds and in opposite directions.

Referring to Fig. 3, shafts A and B and one-half of loop C are shown. The other half of loop C, and both halves of loop D are identically constructed.

Shaft B is cylindrical and has a diameter of 1/4" and a length of 3 3/8". Loop C consists of a flat strip of metal or equivalent material 3/2" thick and 1/4" wide. It is bent approximately in the shape of an ellipse with a short radius of 1 1/8".

It has flat sides $1\frac{3}{4}$ " long, and the portions between the flat sides and shaft B have approximately the curvature of a circle.

Small variations of the structure of the loop and the diameter of shaft B may be made without altering substantially the results obtained.

In operation, the oil container I is maintained at 275° F. and stirring elements A are rotated in opposite directions each at the rate of 1100 revolutions per minute. The volume increases due to foaming is determined by measuring the height of the oil before foam formation and after foam formation.

We claim:

1. A foam inhibited compounded mineral oil, comprising a major quantity of a petroleum lubricating oil containing an oil-soluble foaming-promoting metal salt selected from the group consisting of polyvalent metal phenates and polyvalent metal salts of organo substituted acids of phosphorus and in an amount sufficient substantially to increase the tendency of the lubricating oil to foam excessively at temperatures above 150° F. under conditions of severe oil agitation and intermixture with gas, and a minor quantity, sufficient to inhibit foaming, of an aliphatic compound consisting of elements selected from the group consisting of carbon, hydrogen, and oxygen having a molecular weight not greater than 225, containing not less than about 25% by weight of oxygen and which is represented by the formula



wherein A is selected from the class consisting of hydrogen atom and hydroxyl group, R is a lower alkyl group, and n is an integer.

2. A foam inhibited compounded mineral oil, comprising a major quantity of a petroleum lubricating oil containing an oil-soluble foaming-promoting metal salt selected from the group consisting of polyvalent metal phenates and polyvalent metal salts of organo substituted acids of phosphorus and in an amount sufficient substantially to increase the tendency of the lubricating oil to foam excessively at temperatures above 150° F. under conditions of severe oil agitation and intermixture with gas, and a minor quantity, sufficient to inhibit foaming, of an aliphatic compound consisting of elements selected from the group consisting of carbon, hydrogen, and oxygen, containing one ester group and one carboxyl group, having a molecular weight not greater than 225 and containing not less than about 25% by weight of oxygen.

3. A foam inhibited compounded mineral oil comprising a major quantity of a petroleum lubricating oil containing an oil-soluble foaming-promoting metal salt selected from the group consisting of polyvalent metal phenates and polyvalent metal salts of organo substituted acids of phosphorus and in an amount sufficient substantially to increase the tendency of the lubricating oil to foam excessively at temperatures above 150° F. under conditions of severe oil agitation and intermixture with gas, and a minor quantity, sufficient to inhibit foaming, of an aliphatic compound consisting of elements selected from the group consisting of carbon, hydrogen, and oxygen, containing one ester group and one aldehyde group, having a molecular weight not greater than 225 and containing not less than about 25% by weight of oxygen.

4. A foam inhibited compounded mineral oil

comprising a major quantity of a petroleum lubricating oil containing an oil-soluble foaming-promoting metal salt selected from the group consisting of polyvalent metal phenates and polyvalent metal salts of organo substituted acids of phosphorus and in an amount sufficient substantially to increase the tendency of the lubricating oil to foam excessively at temperatures above 150° F. under conditions of severe oil agitation and intermixture with gas, and a minor quantity, sufficient to inhibit foaming, of an aliphatic compound consisting of elements selected from the group consisting of carbon, hydrogen, and oxygen, containing one ester group and one carbonyl group in a radical selected from the group consisting of carboxyl and aldehyde radicals, having a molecular weight not greater than 225 and containing not less than about 25% by weight of oxygen.

5. The compounded oil of claim 4, wherein said metal salt is an oil-soluble polyvalent metal phenate.

6. The compounded oil of claim 4, wherein said metal salt is an oil-soluble polyvalent metal salt of an organo-substituted acid of phosphorus.

7. The compounded oil of claim 4, wherein said aliphatic compound is monoethyl adipate.

8. The compounded oil of claim 4, wherein said aliphatic compound is methyl glyoxalate.

9. A compounded petroleum lubricating oil comprising a major proportion of petroleum oil of lubricating viscosity, a small amount of an oil-soluble polyvalent metal salt of a phenol and a small amount of an oil-soluble polyvalent metal salt of an organo-substituted acid of phosphorus, said metal salt of a phenol and said metal salt of an acid of phosphorus being present in amount sufficient to increase substantially the tendency of the oil to foam when subjected to agitation with air, and about 0.001 to 1 per cent by weight, sufficient substantially to inhibit foaming of the oil when subjected to agitation with air, of an aliphatic compound consisting of elements selected from the group consisting of carbon, hydrogen, and oxygen, containing one ester group and one carbonyl group in a radical selected from the group consisting of carboxyl and aldehyde radicals, having a molecular weight not greater than 225 and containing not less than about 25% by weight of oxygen.

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