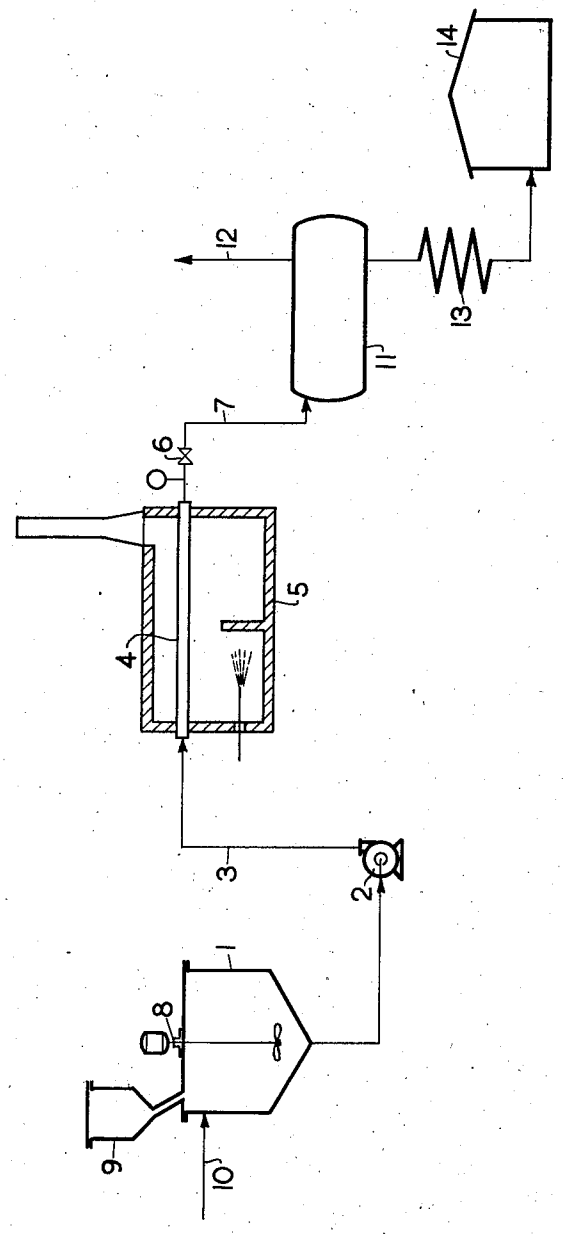
Jan. 14, 1941. R. F. BERGSTROM 2,228,500

PROCESS FOR MAKING LUBRICATING OILS

Filed April 27, 1940



Inventor: Roland F. Bergstrom

By his Attorney: S. S. Lubbers

Patented Jan. 14, 1941

2,228,500

UNITED STATES PATENT OFFICE

2,228,500

PROCESS FOR MAKING LUBRICATING OILS

Roland F. Bergstrom, Oakland, Calif., assignor to Shell Development Company, San Francisco, Calif., a corporation of Delaware

Application April 27, 1940, Serial No. 331,988

9 Claims. (Cl. 252—39)

This invention deals with a method for incorporating metal bases into lubricating oils containing organic acids.

It is known that the presence of certain metal salts of organic acids in lubricating oils have certain beneficial effects, in that these metal salts improve various properties of the oil such as corrosion protection, ringsticking prevention, extreme pressure properties, oiliness properties, wear prevention, resistance to oxidation, etc.

It is a purpose of this invention to provide a simple method whereby metal bases can be introduced into lubricating oils containing organic acid components. It is a further purpose to produce lubricating oils containing metal salts of organic acids, which salts are difficult to produce or difficult to dissolve or both; and it is a further purpose to utilize organic acids already contained in mineral oils to serve as the acid component of the salts which the finished oil shall contain.

I have discovered that oils containing varying amounts of organic acids can be made to react with metal oxides or hydroxides if the acid-containing oil is heated with a suitable amount of the metal base to an elevated temperature and under a superatmospheric pressure in the presence of a small amount of aqueous ammonia. The reaction proceeds readily if the salt produced thereby is oil-soluble. Hence, a metal base should be selected which produces an oil-soluble salt with the acid contained in the oil. It is in general preferred to carry out the reaction with a stoichiometric amount or a slight excess thereover, of the metal base required to neutralize the acid contained in the oil, or, if desired, to produce the basic salt if a polyvalent metal base is employed.

Organic acids which may be conveniently converted to their salt solutions by my process are, for example, phenols, alkyl phenols, thiophenols, alkyl naphthols, naphthenic acids, fatty acids, aromatic fatty acids, aromatic carboxylic acids in which the carboxyl radical is directly attached to the aromatic nucleus, rosin acids, paraffin acids produced by oxidation of paraffins, wool fat acids, chlorinated or hydroxy derivatives of the above acids, oil-soluble sulfonic acids, such as mahogany sulfonic acids, etc.

Practically all metal bases which form oil-soluble salts with the above acids may be used in the process. Thus the alkali metal hydroxides, alkali earth oxides or hydroxides, particularly magnesium or calcium oxides or hydroxides; the oxides or hydroxides of Cu, Zn, Cd, Al, As, Sb, Bi, Cr, Mo, W, Mn, Fe, Co, and Ni may be introduced successfully.

Many of the salts of the above acids and bases are very difficult to produce by other methods, and some salts, even if obtainable, tend to decompose when it is attempted to dissolve them in lubricating oils, or they refuse to go into solution. For example, calcium or aluminum cresylates are almost impossible to dissolve in lubricating oil by means other than those herein described, whereas by my method, no difficulty is experienced in obtaining fairly concentrated solutions of these cresylates.

A particular advantage of my method resides in the fact that basic salts can easily be produced, which salts are often difficult to obtain, particularly when weak acids or weak bases or both are involved.

The amount of aqueous ammonia required may vary over a considerable range. In general, from about .1% to 5% by volume of concentrated (28% to 30%) aqueous ammonia may be added to the oil, or an equivalent amount of more dilute ammonia. In general, however, it is proposed to use ammonia of at least about 10% concentration.

The temperature of reaction may vary from about 300° F. up to the incipient cracking temperature of the oil and is preferably between about 500° to 700° F. The time of reaction generally required may vary from about .01 to 10 seconds, depending upon the temperature used, although a longer time may be allowed at temperatures low enough to prevent undesirable side reactions. The superatmospheric pressures maintained may range advantageously from about 15 to 500 pounds per square inch (gauge), but preferably 50 to 200 pounds per square inch (gauge).

After having contacted the mixture of oil containing the organic acid, metal oxide base and aqueous ammonia at the desired elevated temperature and pressure, the pressure is released, whereupon water and ammonia escape, and the reacted oil is then cooled at the reduced pressure, which is most conveniently atmospheric pressure. Usually the resulting oil is somewhat cloudy, containing unreacted metal base, which can be removed by simple filtration or centrifuging.

Depending upon the solubility of the salt produced by the reaction, various amounts of salts can be incorporated by my method. Saturated solutions, and occasionally even supersaturated solutions, may be produced.

My process will be more fully understood from the accompanying drawing which represents one form of an apparatus which may be used con-

veniently in producing my compounded oils. Lubricating oil containing an amount of an organic acid metal base and aqueous ammonia is pumped from storage tank 1 by pump 2 in line 3 into reaction tube 4 situated in furnace 5. Pressure on the reaction tube is maintained by valve 6 in rundown line 7.

Tank 1 is equipped with a stirrer 8 and hopper 9, the latter being used to introduce the metal base. Aqueous ammonia is introduced into tank 1 through line 10. During operation, stirrer 8 keeps a uniform suspension of the metal oxide and ammonia in the oil.

Reacted oil flows through rundown line 7 to separator 11 equipped with vent line 12. Hot oil from the separator passes through cooling coil 13 into storage tank 14.

It is of interest to note that the process should be operated by the continuous method described in the drawing, rather than by the batch method. It has been found that batch operation is accompanied by difficulties, possibly because of the impossibility of properly controlling the various factors of time element, quick pressure release to remove water and ammonia just at the proper moment, etc. Thus, while the presence of water may be a cause for difficulty under some circumstances, it has been observed that, on the other hand, its presence, together with that of ammonia, is essential for the promotion of the reaction.

The following examples further illustrate my invention:

Example I

A lubricating oil containing 20% of an alkyl phenol mixture was mixed with 7% by weight calcium hydroxide. 5% by weight of 28% aqueous ammonia was added. The resulting mixture was strongly agitated to maintain a uniform suspension, which was fed at a rate of about 3 to 4 gallons per hour through an externally heated reaction tube 12 inches in length and ¼ inch I. D. A pressure was maintained of 100 pounds per square inch. The oil leaving the tube had a temperature of 650° F. and was collected in an open vessel from which water and ammonia escaped. After cooling, the oil was filtered. Upon diluting the compounded oil with lubricating oil free from salts to produce a blend containing approximately 1% of calcium alkyl phenolate, an oil was produced having good resistance to oxidation and fair anti-ringsticking properties.

Example II

A mineral oil containing 20% naphthenic acid was treated with 7% by weight magnesium hydroxide under the conditions of Example I. An oil was produced containing 22% basic magnesium naphthenate. This oil was filtered and blended with straight lubricating oil to produce a blend having a content of basic magnesium naphthenates of 1.2%. This blend proved to be a lubricating oil having excellent anti-ringsticking properties.

I claim as my invention:

1. Process for incorporating a metal base in a lubricating oil containing an amount of an organic acid, said metal base and organic acid being capable of forming an oil-soluble salt, comprising mixing said oil with said metal base and a small amount of strong aqueous ammonia, heating the mixture to a temperature between 300° F. and incipient cracking temperature of said oil at a superatmospheric pressure for a period of about .01 to 10 seconds, releasing the pressure whereby water and ammonia are allowed to escape, and cooling the resulting oil.

2. Process of claim 1 wherein the aqueous ammonia has a concentration of about 10% to 30%.

3. Process of claim 1 wherein the amount of aqueous ammonia is equivalent to between .1% to 5% of concentrated ammonia by volume of said oil.

4. Process of claim 1 wherein the temperature is 300° to 700° F.

5. Process of claim 1 wherein the pressure is 15 to 500 pounds per square inch gauge.

6. Process for producing solutions of salts of organic acids in lubricating oils comprising mixing a lubricating oil containing an organic acid with an amount of a base of a metal capable of producing oil-soluble salts with said acid, said amount being at least equivalent to said acid, and a quantity of a strong aqueous ammonia equivalent to .1% to 5% concentrated ammonia by volume of said oil, heating said mixture to a temperature from 300° F. to incipient cracking temperature of the oil for .01 to 10 seconds at a pressure of 15 to 500 pounds, releasing the pressure, whereby water and ammonia are allowed to escape, cooling and filtering the resulting oil.

7. Process of claim 6 wherein the organic acid is a mixture of alkyl phenols, and the metal is an alkaline earth metal.

8. Process of claim 6 wherein the organic acid is a mixture of acids naturally associated with said oil.

9. Process for producing solutions of basic salts of organic acids in lubricating oils, comprising mixing a lubricating oil containing an organic acid with an amount of a base of a metal capable of producing oil-soluble salts with said acid, said amount being substantially in excess of the equivalent of said acid, and a quantity of a strong aqueous ammonia equivalent to .1% to 5% concentrated ammonia by volume of said oil, heating said mixture to a temperature from 300° F. to incipient cracking temperature of the oil for .1 to 10 seconds at a pressure of 50 to 500 pounds, releasing the pressure, whereby water and ammonia are allowed to escape, cooling and filtering the resulting oil.

ROLAND F. BERGSTROM.