United States Patent [19]

Sias

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[54] **PROCESS FOR PRODUCING OIL-SOLUBLE METAL SULFONATES**

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Related U.S. Application Data

[63] Continuation of Ser. No. 148,264, May 5, 1971, abandoned.

[52] **U.S. Cl.**..... **260/429 K**; 252/33; 260/429 R; 260/429.5; 260/438.5 R; 260/439 R; 260/448 R; 260/505 N; 260/513 R

[51] **Int. Cl.** **C07f 11/00**; C07f 1/00; C07f 7/28

[58] **Field of Search**..... 260/513 R, 429 R, 429 K, 260/439 R, 448 R, 505 N, 438.5, 429.5; 252/33

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[57] **ABSTRACT**

A process for producing oil-soluble metal sulfonates is disclosed wherein a metal halide is reacted with an oil-soluble sulfonic acid to produce the desired metal sulfonate. The metal constituent of the metal halide is selected from the group consisting of aluminum, indium, chromium, iron, molybdenum, vanadium, titanium, niobium, tantalum, rubidium, and osmium.

12 Claims, No Drawings

PROCESS FOR PRODUCING OIL-SOLUBLE METAL SULFONATES

This is a continuation of application Ser. No. 148,264, filed May 5, 1971 and now abandoned.

BACKGROUND OF THE INVENTION

This invention relates to oil-soluble metal sulfonates. In one aspect the invention relates to oil-soluble metal sulfonates wherein the metal constituent is selected from aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, and osmium. In another aspect the present invention relates to a process for producing oil-soluble metal sulfonate from halides of aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, and osmium and oil-soluble sulfonic acids.

BRIEF DESCRIPTION OF THE PRIOR ART

In recent years it has been found that superior standards for spectrographic equipment can be prepared from oil-soluble metal sulfonates and metal dispersions in such sulfonates by dissolving such materials in predetermined quantities in a suitable solvent. Such standards have exhibited indefinite shelf life and any combination of metals can be combined without precipitation of the metal constituents.

Further, dispersions containing certain oil-soluble metal sulfonates have acquired considerable importance as additives in fuels and lubricating oil. Such dispersions have been highly useful as additives to other materials where the problem of suspending insoluble waste materials formed in the utilization of the material and also the problem of corrosion inhibition is met. When the oil-soluble metal sulfonates are employed as additives for use in internal combustion engine lubricating compositions, such agents function to effectively disperse or peptize the insolubles formed by the fuel combustion, oil oxidation, or similar conditions obtained during the operation of the engine.

Thus, while the use of oil-soluble metal sulfonates have been established and recognized, problems have been encountered in the production of oil-soluble metal sulfonates of certain metals, such as molybdenum, aluminum and iron. Therefore, a need has long been recognized for an improved process for the production of oil-soluble metal sulfonates from readily available chemical compounds, and it is to such a process that the present invention is directed.

OBJECTS OF THE INVENTION

An object of the present invention is to provide an improved process for the production of oil-soluble metal sulfonates. Another object of the present invention is to provide an economical, dependable, and efficient method for preparing oil-soluble metal sulfonates from readily available chemical compounds.

Another object of the present invention is to provide an improved method for the preparation of oil-soluble metal sulfonate of aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, and osmium which are suitable as analytical standards while at the same time providing an oil-soluble source of such metals.

These and other objects, advantages, and features of the present invention would be apparent to those skilled in the art from a reading of the following detailed description.

SUMMARY OF THE INVENTION

According to the present invention I have found a process for producing oil-soluble metal sulfonates wherein the metal constituent is selected from aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, and osmium which comprises admixing a halide compound of such metals with an oil-soluble sulfonic acid, heating the resulting mixture to its reflux temperature for a period of time effective to allow formation of the oil-soluble metal sulfonate.

Further according to the invention I have found that it is desirable for said metal halide to be present in a stoichiometric excess of from 5 to about 200% with said oil-soluble sulfonic acid. A volatile inert solvent can be incorporated with the oil-soluble sulfonic acid to reduce the viscosity of same and to facilitate the admixing of the oil-soluble sulfonate with said metal halide.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Oil-soluble metal sulfonates have been recognized as desirable analytical standards as well as oil-soluble additives for fuels and lubricants. However, problems have been encountered in producing oil-soluble metal sulfonates such as molybdenum sulfonate, iron sulfonate and aluminum sulfonate.

I have now found that oil-soluble metal sulfonates of aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, and osmium can readily be prepared by reacting a halide compound, or a mixture of a halide compound and oxide compound, of such metal with an oil-soluble sulfonic acid at elevated temperatures for a period of time effective to allow said halide compound or a mixture of a metal halide and a metal oxide compound to react with said oil-soluble sulfonic acid to produce the desired oil-soluble metal sulfonate.

The present invention can be carried out as either a batch process or a continuous process. However, for the sake of simplicity the process of the present invention will be described as a batch process.

The metal halide and the oil-soluble sulfonic acid are charged to a reaction vessel equipped with heating means, a stirring means and a reflux means. Generally, it is desirable to introduce an effective amount of an inert volatile solvent to the reaction mixture to reduce the viscosity of the oil-soluble sulfonic acid thereby facilitating the mixing and contact between the reactants. The amount of inert volatile solvent employed can vary widely depending upon the viscosity of the particular oil-soluble sulfonic acid employed as well as the viscosity desired in the reaction mixture but will generally be in an amount ranging from about 25 to 150 weight percent, based on the weight of the reaction mixture. The amount of the reactants can vary widely. However, the metal halide should be present in a stoichiometric excess. Generally, the excess will range from about 5 to 200 percent with the most desirable amount ranging from 5 to about 15 percent.

Once the reactants have been introduced into the reaction vessel the reactants are thoroughly agitated and the reaction mixture is heated to its reflux temperature

which will generally be within the range of about 60° to 105° C. When desirable an additional amount of the oil-soluble sulfonic acid can be introduced into the reaction mixture during the heating period before the mixture reaches its reflux temperature. However, care must be exercised to insure that the introduction of the additional oil-soluble sulfonic acid does not dilute the reaction mixture to such an extent that the metal halide is no longer present in a stoichiometric excess. Generally, when additional oil-soluble sulfonic acid is introduced the amount will range from about 50 to 200 weight percent based on sulfonic acid present and at a temperature in the range of about 60° to 105° C.

When the reaction mixture reaches its reflux temperature it is maintained at such temperature under reflux conditions for an effective period of time to allow the metalhalide and oil-soluble sulfonic acid to react and form the desired oil-soluble meal sulfonate. The reflux time of the reaction mixture can vary widely but will generally range from about 1 to about 6 hours. It is often desirable to introduce to the mixture after same has refluxed for about 1 to 6 hours from about 1 to 25 weight percent water based on sulfonic acid. The reaction mixture containing the water is then maintained at reflux conditions for an additional period of time ranging from 0.1 to 2 hours.

After the above-described reflux steps have been carried out the mixture is stripped of the volatile components. Any suitable method for removing the volatile components can be employed such as heating the mixture to a temperature from about 125° to 175° C. From about 20 to 300 weight percent of a nonvolatile organic carrier component (based on sulfonic acid) is introduced at any convenient point, such as during the reflux period. Residual volatile material is removed by any suitable means such as vacuum stripping or stripping said mixture with a gas such as nitrogen, carbon dioxide, air and the like for a period of time ranging from 0.2 to 6 hours. The stripped product normally is clarified by filtration of the stripped product through a desirable inert absorbent such as alumina, diatomaceous earth, pumice and the like.

The metal halide which can be employed in the production of the oil-soluble metal sulfonates can be any suitable halide of aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, and osmium. Examples of such halides are aluminum chloride, aluminum bromide, aluminum fluoride, chromium chloride, chromium bromide, chromium fluoride, ferric chloride, ferric bromide, ferric fluoride, molybdenum fluoride, vanadium chloride, vanadium bromide, vanadium fluoride, titanium chloride, titanium bromide, titanium fluoride, indium chloride, indium bromide, indium fluoride, niobium chloride, niobium bromide, niobium fluoride, tantalum chloride, tantalum bromide, tantalum fluoride, rubidium chloride, rubidium bromide, rubidium fluoride, osmium chloride, osmium bromide, and osmium fluoride. Especially desirable results have been obtained wherein the metal halide is the metal chloride. In addition, mixtures of the metal halide and a metal oxide can be employed. When such a mixture is employed the metal halide will be present in such mixture in an amount ranging from about 0.25 to 8 moles per mole metal oxide. Examples of suitable mixtures of the halide and oxide components are: $AlCl_3$-Al_2O_3 (and hydrates); $FeCl_3$-Fe_2O_3; $CrCl_3 \cdot 6H_2OCr_2O_3$; $TiCl_4$-TiO_2, and the like.

Suitable oil-soluble hydrocarbon sulfonic acids include alkane sulfonic acid, aromatic sulfonic acid, alkaryl sulfonic acid, aralkyl sulfonic acid, and the natural petroleum mahogany sulfonic acids. The mahogany sulfonic acids include any of those materials which may be obtained by concentrated or fuming sulfuric acid treatment of petroleum fractions, particularly the higher boiling lubricating oil distillates and white oil distillates. The higher molecular weight petroleum oil-soluble mahogany sulfonic acids are condensedring compounds, which condensed-rings may be aromatic or hydroaromatic in nature. Alkyl and/or cycloalkyl substituents may be present in the mahogany sulfonic acids.

The terms "oil-soluble sulfonic acids," as used herein, refers to those materials wherein the hydrocarbon portion of the molecule has a molecular weight in the range of about 300 to about 1,000. Preferably, this molecular weight is in the range of about 370 to about 700. These oilsoluble sulfonic acids can be either synthetic sulfonic acids or the so-called mahogany or natural sulfonic acids. The term "mahogany sulfonic acid" is believed to be well understood, since it is amply described in the literature. The term "synthetic sulfonic acids" refers to those materials which are prepared by sulfonation of hydrocarbon feedstocks which are prepared synthetically. The synthetic sulfonic acids can be derived from either alkyl or alkaryl hydrocarbons. In addition, they can be derived from hydrocarbons having cycloalkyl (i.e., naphthenic) groups in the side chains attached to the benzene ring. The alkyl groups in the alkaryl hydrocarbons can be straight or branched chain. The alkaryl radical can be derived from benzene, toluene, ethyl benzene, xylene isomers, or naphthalene.

An example of a hydrocarbon feedstock which has been particularly useful in preparing synthetic sulfonic acids is a material known as postdodecylbenzene. Postdodecylbenzene is a bottoms product of the manufacture of dodecylbenzene. The alkyl groups of postdodecylbenzene are branched chain. Postdodecylbenzene consists of monoalkylbenzenes and dialkylbenzenes in the approximate mole ratio of 2:3 and has typical properties as follows:

Specific gravity at 38 degrees C	0.8649
Average molecular weight	385
Percent sulfonatable	88
ASTM D-158 Engler:	
I.B.P., degrees F	647
5 degrees F	682
50 degrees F	715
90 degrees F	760
95 degrees F	775
F.B.P. degrees F	779
Refractive index at 23 degrees C	11.4900
Viscosity at:	
−10 degrees C, centistokes	2800
20 degrees C, centistokes	280
40 degrees C, centistokes	78
80 degrees C, centistokes	18
Aniline point, degrees C	69
Pour Point, degrees F	−25

An example of another hydrocarbon feedstock which is particularly useful in preparing synthetic sulfonic acids is a material referred to as "dimer alkylate". "Dimer alkylate" has a long branched-chain alkyl group. Briefly described, dimer alkylate is prepared by the following steps:

1. dimerization of a suitable feedstock, such as cat poly gasoline; and

2. alkylation of an aromatic hydrocarbon with the dimer formed in step (1).

Preferably, the dimerization step uses a Friedel-Crafts alkylation sludge as the catalyst. This process and the resulting product are described in U.S. Pat. No. 3,410,925.

An example of another hydrocarbon feedstock which is particularly useful for preparing synthetic sulfonic acids which can be used in my invention is a material which I refer to as "NAB Bottoms." NAB Bottoms are predominantly di-n-alkyl aromatic hydrocarbon wherein the alkyl groups contain from eight to 18 carbon atoms. They are distinguished primarily from the preceding sulfonation feedstocks in that they are straight chain and contain a large amount of disubstituted material. A process of preparing these materials and the resulting product are described in application Ser. No. 62,211, filed Aug. 7, 1970, and being a continuation-in-part of application Ser. No. 529,284, filed Feb. 23, 1966, and now abandoned. Application Ser. Nos. 62,211 and 529,284 have the same assignee as the present application. The product is also described in U.S. Pat. No. 3,288,716, which is concerned with an additional use for the product, other than sulfonation feedstock. Another process of preparing these materials is described in application Ser. No. 53,352, filed Aug. 6, 1970, and having the same assignee as the present application. Application Ser. No. 53,352 is a continuation-in-part of application Ser. No. 529,284. Still another process of preparing a di-n-alkaryl product is described in application Ser. No. 104,476, filed Jan. 7, 1971, which is a continuation-in-part of application Ser. No. 521,794, filed Jan. 20, 1966, and now abandoned.

In order to make my disclosure even more complete, U.S. Pat. No. 3,410,925 and application Ser. Nos. 53,352; 62,211 and 104,7476, are made a part of this disclosure.

In addition to the sulfonic acids derived from the foregoing described hydrocarbon feedstock, examples of other suitable sulfonic acids include the following: mono- and poly-substituted naphthalene sulfonic acid, dinonyl naphthalene sulfonic acid, diphenyl ether sulfonic acid, naphthalene disulfide sulfonic acid, dicetyl thianthrene sulfonic acid, dialauryl betanaphthol sulfonic acid, dicapryl nitronaphthalene sulfonic acid, unsaturated paraffin wax sulfonic acid, hydroxy substituted paraffin wax sulfonic acid, tetraamylene sulfonic acid, mono- and poly-chlorosubstituted paraffin wax sulfonic acid, nitrosoparaffin wax sulfonic acid, cycloaliphatic sulfonic acid such as lauryl-cyclohexyl sulfonic acid, mono- and poly-wax-substituted cyclohexyl sulfonic acid, and the like.

The corresponding hydrocarbon sulfonic acid is usually prepared by treating the hydrocarbon with concentrated sulfuric acid, fuming sulfur acid or sulfur trioxide. The sulfonation of hydrocarbons is well known and details need not be given. The sulfonic acid may also be purified by any suitable means: i.e., treatment with inorganic base, ion exchange, water washing and the like.

As previously stated the oil-soluble sulfonic acid is often diluted with a volatile solvent. The volatile solvent can be any suitable hydrocarbon, preferably a low boiling hydrocarbon such as hexane or naphtha which may readily be removed from the metal sulfonate product when desired.

With respect to the types of nonvolatile carriers which may be utilized in the process, a wide variety of materials have been found suitable for such usage. The principal requisites desired in the nonvolatile carrier are that it will dissolve the dispersing agents utilized in the process, and that such solutions will be relatively stable when the basic metallic compounds are peptized in the dispersion by the dispersing agent. Examples of such nonvolatile carriers which may be employed include mineral lubricating oil obtained by any of the conventional refining procedures; vegetable oils, such as corn oil, cotton-seed oil, castor oil, etc; animal oil, such as lard oil, sperm oil, etc; and synthetic oils, such as polymers of propylene, polyoxyalkylenes, polyoxypropylene, dicarboxylic acid esters, such as esters of adipic and azelaic acids with alcohols such as butyl, 2-ethyl hexyl and dodecyl alcohols, and esters of acids of phosphorus, such as diethyl ester of decanephosphonic acid and tricresyl phosphate. The preferred nonvolatile carriers are liquid lubricating oils, either mineral or synthetic. In addition, sulfonic acid stock such as previously described hereinabove can be employed as the nonvolatile carrier. If desired, the nonvolatile carriers may be diluted with a solvent to reduce the viscosity. Suitable solvents include petroleum naphtha or hydrocarbons, such as hexane, heptane, octane, benzene, toluene, or xylene.

In order to more fully illustrate the nature of the present invention the following examples are given. However, it is to be understood that the examples are for illustrative purposes only and are not intended to unduly limit or restrict the scope of the present invention. In each example the sulfonic acid was derived frm an alkylaromatic which was predominantly di-n-alkylbenzenes having a combined molecular weight of about 420, unless otherwise specified.

EXAMPLE 1

To a creased 1-liter flask was charged 212.0 grams of sulfonic acid and 27.4 grams of anhydrous $MoCl_5$ during mechanical agitation. Heat was applied and the reaction was taken to 70° C, whereupon an additional 212.0 grams of sulfonic acid was charged and the reaction taken to reflux temperature and refluxed for 2 hours, 5 ml. water was charged followed by additional refluxing, then the volatiles were taken overhead to a pot temperature of 150° C; 170 grams of 80 pale oil was charged at about 110° C. The product was then stripped with N_2 gas for 15 minutes and filtered through Hyflo. The product was analyzed and found to contain 2.6 weight percent molybdenum and 0.04 weight percent chlorine.

EXAMPLE 2

An experiment was conducted employing the procedure of Example 1 except that all of the sulfonic acid was charged at ambient temperature and the product was stripped at 150° C for 30 minutes under house vacuum. The charge employed in this experiment was as follows:

212.0	grams	Sulfonic Acid
12.8	grams	Anhydrous $MoCl_5$
80.1	grams	80 Pale Oil
10	ml.	Water

The product produced was filtered as in Example 1 and found to contain 1.7 weight percent molybdenum.

EXAMPLE 3

The general procedure described in Example 2 was followed. The charge employed was as follows:

250	grams	Sulfonic Acid
34.9	grams	$CrCl_3 \cdot 6H_2O$
120	grams	80 Pale Oil

The mixture of the acid and chromium compound was heated to its reflux temperature and maintained under reflux conditions for 2 hours. The pale oil was then added to the mixtufe at 100° C. After additional refluxing the product was heated to 150° C and stripped for 15 minutes with N_2 gas. The stripped product was then filtered and analyzed to contain 2.4 weight percent chromium and 0.02 weight percent chlorine.

EXAMPLE 4

The procedure of Example 1 is employed in this example. The sulfonic acid was charged in two equal increments of 125 grams. The total charge to the reaction flask is as follows:

250	grams	Sulfonic Acid
16.4	grams	Anhydrous $CrCl_2$
120	grams	80 Pale Oil

The initial reaction mixture was heated to its reflux temperature and refluxed for 2 hours. Ten ml. of water were then charged to the reaction mixture and the resulting mixture was heated to its reflux temperature and maintained under reflux conditions for ten minutes. The volatiles were then taken overhead to a pot temperature of 150° C. The 80 pale oil was charged to the mixture and the mixture was then stripped with N_2 gas for 15 minutes at 150° C. The stripped product was 2.2 weight percent chromium and less than 0.01 weight percent chlorine.

EXAMPLE 5

An experiment was conducted on the production of iron sulfonates using the general procedure of Example 2 wherein all the sulfonic acid was charged at ambient temperature. The charge employed was as follows:

235	grams	Sulfonic Acid
30.0	grams	80 Pale Oil
13.6	grams	Anhydrous $FeCl_3$
10.0	grams	Water

The sulfonic acid-$FeCl_3$ mixture was heated to its reflux temperature and refluxed for 2 hours. Ten milliliters of water was then charged followed by additional refluxing. The volatiles were then taken overhead to a pot temperature of 150° C. The pale oil was then charged to the mixture at about 110° C. The resulting product was then stripped with N_2 gas for about 15 minutes and filtered. The product was analyzed and found to contain 2.4 weight percent iron.

EXAMPLE 6

To a creased one-liter flask was charged 113.3 grams of sulfonic acid and 21.8 grams of $FeCl_3 \cdot 6H_2O$ during mechanical agitation. The sulfonic acid was diluted with 50 milliliters of n-heptane. Heat was applied and the reaction mixture was taken to 85° C whereupon an additional 113.0 grams of sulfonic acid was charged to the mixture. The resulting reaction mixture was then heated to a pot temperature of about 95° C at which point about 136 milliliters of volatile materials were removed overhead. The mixture was then refluxed for 2 hours. At the end of the reflux period the volatile components remaining were taken overhead to a pot temperature of 150° C. The product remaining was then stripped with N_2 gas at 150° C for 45 minutes. The pale oil was then charged to the stripped product. The resulting product was filtered and found to contain 2.5 weight percent iron and less than 0.01 weight percent chlorine.

EXAMPLE 7

In this experiment the sulfonic acid was charged to a reaction flask and residual water was removed by azeotropic distillation. The sulfonic acid was then employed to prepare a niobium sulfonate composition as follows:

The charge employed was:

209	grams	Sulfonic Acid
12	grams	Anhydrous $NbCl_5$
18.3	grams	80 Pale Oil

The general procedure of Example 1 was followed. The sulfonic acid was charged in equal increments and the reflux period was 2 hours. The 80 pale oil was charged to the mixture at 125° C and the product was stripped with N_2 gas for 15 minutes at 150° C. The product was filtered and found to contain 3.3 weight percent niobium and less than 0.01 weight percent chlorine.

The above examples clearly indicate the preparation of oil-soluble metal sulfonates by the process of the present invention.

Having thus described the invention, I claim:

1. A process for producing oil-soluble sulfonates containing metal constituents, which sulfonates have a long shelf life without precipitation of the metal constituents, comprising:

a. mixing at least a stoichiometric amount of a metal halide selected from the group consisting of aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, osmium, and mixtures thereof, with water and an oil-soluble sulfonic acid having a molecular weight in the range of about 300 to about 1000 to form a reaction mixture,

b. agitating and heating said reaction mixture to a temperature in the range of 60° to 105°C,

c. introducing into the reaction mixture an additional amount of the oil-soluble sulfonic acid in an amount of from 50 to 200 weight percent based on the oil-soluble sulfonic acid already in the reaction mixture,

d. continuing the agitation and heating of the reaction mixture to the reflux temperature of said mixture for a period of time effective to allow formation of a metal sulfonate substantially free of said halide, and

e. recovering from the reaction product of step (d) the metal sulfonate.

2. The process of claim 1 wherein said oil-soluble sulfonic acid is diluted with from about 25 to 150 weight percent of an inert volatile solvent and said reflux temperature is in the range of about 60° to 105° C.

3. The process of claim 2 wherein said inert volatile solvent is a low boiling hydrocarbon selected from the group consisting of hexane and naphtha.

4. The process of claim 1 wherein said reaction mixture is maintained at its reflux temperature for a period of time ranging from about 1 to 6 hours.

5. The processs of claim 4 which includes the step of admixing from about 1 to about 25 weight percent water, based on the amount of sulfonic acid employed, to said mixture after same has refluxed and then heating the mixture to its reflux temperature and maintaining same under reflux condition for a period of time ranging from 0.1 to 2 hours.

6. The process of claim 1 wherein the refluxed mixture is stripped of volatile components by heating said refluxed mixture to a temperature within the range of about 125° to 175°C and includes the step of admixing from about 20 to 300 weight percent of a nonvolatile organic carrier component to said reflux mixture during refluxing of same.

7. The process of claim 6 which includes the additional purification steps of stripping the product with an inert gas selected from the group consisting of nitrogen, carbon dioxide, air, and mixtures thereof for a period of time ranging from about 0.2 to 6 hours and filtering the gas stripped product through an inert absorbent material selected from the group consisting of alumina, diatomaceous earth and pumice.

8. The process of claim 7 wherein said metal halide is present in a mixture of said metal halide and a metal oxide, said metal halide being present in said mixture in an amount ranging from about 0.25 to 8 moles of said metal halide per mole of said metal oxide.

9. The process of claim 8 wherein said oil-soluble sulfonic acid has a molecular weight in the range of about 370 to about 700 and is produced synthetically by the sulfonation of an alkylate selected from the group consisting of dimer alkylate and NAB Bottoms alkylate, and said nonvolatile carrier component is pale oil.

10. The process of claim 9 wherein said nonvolatile carrier is diluted with a solvent selected from the group consisting of petroleum naphtha, hexane, heptane, octane, benzene, toluene, and xylene.

11. A process for producing oil-soluble sulfonates containing metal constituents, which sulfonates have a long shelf life without precipitation of the metal constituents, comprising:

a. mixing at least a stoichiometric amount of a metal halide selected from the group consisting of aluminum, chromium, iron, molybdenum, vanadium, titanium, indium, niobium, tantalum, rubidium, osmium, and mixtures thereof, with water and an oil-soluble sulfonic acid having a molecular weight in the range of about 300 to about 1,000 to form a reaction mixture,

b. agitating and heating said reaction mixture to the reflux temperature of said mixture for a period of time effective to allow formation of a metal sulfonate substantially free of said halide; and

c. admixing from about 1 to about 25 weight percent water, based on the amount of sulfonic acid employed, to said mixture after same has refluxed and then heating the mixture to its reflux temperature and maintaining same under reflux condition for a period of time ranging from 0.1 to 2 hours,

d. recovering from the reaction product of step (c) the metal sulfonate.

12. The process of claim 11 which includes the step of admixing from about 50 to 200 weight percent additional oil-soluble sulfonic acid to said reaction mixture during the heating of said mixture, and while said mixture is at a temperature within the range of about 60° to 105°C.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,897,470

DATED : July 29, 1975

INVENTOR(S) : Roy C. Sias

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 18, change "meal" to"metal"

Signed and Sealed this

seventeenth Day of *February 1976*

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks