

UNITED STATES PATENT OFFICE

2,409,671

SUBSTANTIALLY OIL SOLUBLE POLYALKYL
AROMATIC SULFONATESJacob Faust, Belleville, N. J., assignor to L. Son-
neborn Sons, Inc., a corporation of Delaware.No Drawing. Application December 8, 1943,
Serial No. 513,469

3 Claims. (Cl. 260—505)

1

This invention relates to new and useful im-
provements in substantially oil soluble polyalkyl
aromatic sulfonates.

One object of the invention comprises, inter
alia, novel substantially oil soluble polyalkyl aro-
matic sulfonates possessing valuable properties
and characteristics. Another object of the inven-
tion comprises new and useful oleophilic hydro-
carbon compositions containing these novel sul-
fonates. These and still further objects of the
invention will be apparent from the following de-
scription.

The polyalkyl aromatic sulfonates in accordance
with the invention are essentially substantially
oil soluble metal—preferably alkali metal—poly-
amyl naphthalene sulfonates having at least three
amyl radicals, preferably branch chained, and one
MeSO₃ in unalkylated position on the aromatic
ring.

The oleophilic hydrocarbon composition in ac-
cording with the invention essentially comprises,
from 25 to 50% and preferably from 30 to 40% by
weight of a hydrocarbon material comprising at
least one member selected from the group con-
sisting of petroleum hydrocarbons and polyamyl
naphthalenes with at least three amyl radicals,
and from 50 to 75% and preferably from 60 to
70% by weight of a substantially oil soluble
metal—and preferably alkali metal—polyamyl
naphthalene sulfonate having one MeSO₃ radi-
cal carried by an unalkylated position of its aro-
matic ring.

Within the preferred embodiment of my in-
vention by way of the novel oleophilic hydrocar-
bon composition I find it advisable to use a poly-
amyl naphthylene sulfonate carrying branched
chain amyl groups. Although individual poly-
amyl naphthalene sulfonates as for instance tri-
tetra- or penta-amyl naphthalene sulfonates may
be used, their mixture is ordinarily more easily
available since it can be conveniently produced
from commercial polyamyl naphthalene which
normally occurs as a mixture of the tri-, tetra-
and penta- products.

The petroleum hydrocarbon in accordance with
the invention may be any suitable petroleum hy-
drocarbon or petroleum hydrocarbon cut. As is
conventionally understood in the art, when ref-
erence is made to the term "petroleum hydrocar-
bon," such term includes not only individual hy-
drocarbons but also mixtures thereof.

Where the metal polyamyl naphthalene sul-
fonate in accordance with the invention is desig-
nated as being substantially oil soluble, I mean
thereby that such product, as is well understood

2

in the art, is substantially soluble in petroleum
spirits and oils, coal tar solvents, chlorinated hy-
drocarbons and terpene hydrocarbons. Where
use is made of the term oleophilic or such similar
expression in designation of a hydrocarbon com-
position in accordance with the invention, I mean
to convey thereby the compatibility of my novel
hydrocarbon composition as such with the sol-
vents or group of solvents just recited in sub-
stantially all proportions.

A suitable polyamyl naphthalene that may be
used for the preparation of the products in ac-
cording with the invention, whether as hydro-
carbon material or as source for my novel poly-
amyl naphthalene sulfonates, is, for instance, the
commercial product constituting a light straw-
colored viscous liquid essentially of the following
physical characteristics

Specific gravity (24° C.)	0.922
Weight per gallon	7.717 lbs.
Viscosity Saybolt at 100° F.	473 secs.
Viscosity Saybolt at 210° F.	50 secs.
Viscosity index	Minus 30
Flash point	360° F.
Fire point	410° F.
Pour point	Below minus 10° F.
Color (Lovibond 1/2" cell)	5Y1.5R
Iodine value	28.2
Refractive index at 20° C.	1.545

This commercial polyamyl naphthalene com-
pound is ordinarily a mixture of tri-tetra- and
penta-amyl naphthalene in substantially the pro-
portion of 90-5-5. The amyl groups are in
branched chain, that is they are attached to
methyl, propyl, and carbonyl configurations.

The polyamyl naphthalene sulfonates in ac-
cording with the invention are obtained by sub-
jecting a suitable polyamyl naphthalene of the
requisite type and characteristics or a mixture of
such polyamyl naphthalenes to the action of a
sulfonating agent under sulfonating conditions.

The sulfonation reaction may be carried out
in any suitable conventional manner such as, for
instance, by treatment with gaseous SO₃ or by the
addition of fuming sulfuric acid such as, for in-
stance, from 50 to 100% by weight of a normal
20% fuming sulfuric acid. In the latter case the
acid may be added as desired, either in a single
treatment or in a multiple number of shots. In
most cases and particularly with acid amounts
approaching or equaling 100% the sulfonation
approaches quantitative yields. It may be some-
times advisable, dependent upon special condi-
tions and treatments, to carry out the sulfona-

tion reaction in the presence of a substantially non-sulfonatable organic solvent as, for instance, a suitable halogenated hydrocarbon, such as carbon tetrachloride, ethylene dichloride, amyl chloride, polyamyl chloride and chloroform.

Upon completion of the sulfonation reaction, the reaction mass is allowed to settle and such acid sludge as may have formed is withdrawn. The remaining layer is then suitably neutralized, preferably with the aid of an aqueous caustic solution such as aqueous caustic potash or soda solution, to, for instance, phenol-phthaleine end point neutrality. The crude polyalkyl naphthalene sulfonate obtained may then be purified after the removal of the aqueous layer, by for instance extraction with 50% aqueous alcohol. Alternatively, the sulfonation mix may be dissolved in benzol and the benzol solution may be thereupon washed with water to free the same from salts, thereafter distilling off the benzol and recovering the purified product.

The polyamyl naphthalene sulfonates obtained in accordance with the invention are substantially soluble in petroleum hydrocarbons even in the cold and they are colloiddally soluble in water. These novel sulfonate products, when incorporated into petroleum hydrocarbons, render the same substantially spontaneously emulsifiable in aqueous media without the use of any auxiliary linking agents or solvents and the oleophilic hydrocarbon products containing the novel sulfonates in accordance with the invention possess excellent foaming, wetting and penetrating power.

Still further the polyamyl naphthalene sulfonates in accordance with the invention are valuable for textile processing and treating agents. They also constitute valuable agents as plasticizers in the preparation of synthetic rubber, conditioning agents for asphalts, dispersing aids in paint and paint pigment compositions, dispersing agents for insecticides and fungicides in agricultural sprays, assistants in leather processing and finishing, solubilizing agents for resins, dyes and other materials normally insoluble in oil and as ingredients in corrosion inhibiting and rust proofing compositions.

The following examples are furnished by way of illustration and not of limitation:

Example I

50 parts by weight of oleum having a 20% SO_3 content were slowly stirred into 100 parts by weight of a commercial polyamyl naphthalene of the description and properties hereinabove recited. A sulfonate mass formed which was allowed to settle at a temperature of 140 to 150° F. A small amount of acid sludge was produced and was drawn off, whereupon the upper layer was diluted with an equal volume of benzol and exactly neutralized with 20% caustic soda solution using phenol-phthaleine as indicator. The resulting aqueous layer was allowed to settle and was then drawn off. The remaining upper layer was distilled to remove the benzol, thus obtaining a purified product. The percent conversion to polyamyl naphthalene sulfonate was 55.5%. The resulting product contained about 44% unreacted polyamyl naphthalene.

Example II

100 parts by weight of oleum having a 20% SO_3 content were slowly stirred into 100 parts by weight of a commercial polyamyl naphthalene of the description and properties hereinabove re-

cited; a sulfonate mass formed which was allowed to settle at a temperature of 140 to 150° F. A small amount of acid sludge was produced and was drawn off, whereupon the upper layer was diluted with an equal volume of benzol and exactly neutralized with 20% caustic soda solution using phenol-phthaleine indicator. The aqueous layer was allowed to settle and drawn off. The upper layer was distilled to remove the benzol, obtaining a purified product. The percent conversion to polyamyl naphthalene sulfonate was 90.4%. The resulting product was almost free from unreacted polyamyl naphthalene.

Example III

To 100 parts by weight of the polyamyl naphthalene sulfonate product obtained in Example II were added 40 parts by weight of 100 viscosity (Saybolt at 210° F.) Gulf Coast pale oil and the mixture stirred cold to obtain a homogeneous composition containing approximately a polyamyl naphthalene sulfonate content of 65% and an inert hydrocarbon carrier content of approximately 35%.

When only partially sulfonating a suitable polyamyl naphthalene, as is for instance the case when using amounts of acid materially less than 100%, the resulting product may be recovered free from unreacted polyamyl naphthalene, or such unreacted polyamyl naphthalene may be left in the resulting sulfonate product, the latter to be used as such with the unreacted polyamyl naphthalene as a diluent therein. In such case, when incorporating the polyamyl naphthalene sulfonate product in a suitable hydrocarbon oil within the scope of the invention, the unreacted polyamyl naphthalene, depending upon the amount present, may be treated as the sole hydrocarbon material or as part of the hydrocarbon material to which the sulfonate is to be added in preparing the oleophilic hydrocarbon composition in accordance with the invention.

The exceptionally high surface activity exercised by our novel oleophilic polyamyl naphthalene sulfonate hydrocarbon composition is, for example, shown by the product resulting from Example III, which in 0.25% aqueous solution shows a wetting time (standard Draves test) of about two seconds and a surface tension of about 36.4 dynes per cm.

Another exceptional and valuable property of our novel sulfonates resides in the fact that they are capable of emulsifying chlorinated hydrocarbons, such as chloroform, carbon tetrachloride, dichlor ethyl ether and the like, to yield emulsions of an exceptionally high degree of stability as is required, for example, in the use of such chlorinated hydrocarbon emulsion for dormant agricultural sprays. None of these chlorinated hydrocarbons has hitherto satisfactorily responded to such emulsification by other wetting agents.

The properties and characteristics inherent in and exercised by the novel polyamyl naphthalene sulfonates are substantially unaffected by the particular hydrocarbon material with which they are compounded in accordance with one of the embodiments of the invention and it is therefore in most cases immaterial whether the same is a petroleum hydrocarbon or unreacted polyamyl naphthalene. To this extent the product resulting, for instance, from Example I shows wetting and surface tension characteristics of substantially the same order as those hereinabove described as exhibited by the product obtained in

5

accordance with Example II. One of the prime advantages inherent in the presence of unreacted polyamyl naphthalene, however, whether present alone or present in conjunction with petroleum hydrocarbons, essentially resides in the ease of blendability with other components in the preparation of compositions to be compounded, for instance, for textile use, cutting oil use and compositions for such other uses to which the polyamyl naphthalene sulfonates, as an ingredient in accordance with the invention, may lend themselves. For this reason I find it often of advantage, within the scope of one preferred embodiment of my invention, to only partially sulfonate a polyamyl naphthalene in a manner yielding from 25 to 50% and preferably from 30 to 40% by weight of unreacted polyamyl naphthalene, and from 50 to 75% and preferably from 60 to 70% by weight of polyamyl naphthalene sulfonate of the herein described character and nature. Such a product results, for instance, when proceeding in accordance with Example I. When desiring to obtain a product in accordance with the invention containing in the requisite amounts only unreacted polyamyl naphthalene as the hydrocarbon material, it is possible to achieve this result by using from 50 to 75% of fuming sulfuric acid in the sulfonation treatment herein referred to.

6

The foregoing description is for purposes of illustration and not of limitation and it is therefore our intention that the invention be limited only by the appended claims or their equivalents wherein I have endeavored to claim broadly all inherent novelty.

I claim:

1. A new substantially oil-soluble polyalkyl aromatic sulfonate which consists of a substantially oil soluble polyamyl naphthalene sulfonate having one metal sulfonate radical carried by an unalkylated position of the aromatic ring and having at least three amyl radicals.

2. A new substantially oil-soluble polyalkyl aromatic sulfonate in accordance with claim 1 in which said polyamyl naphthalene sulfonate carries branched chain amyl groups.

3. An oleophilic petroleum hydrocarbon composition which comprises from 25 to 50% of a hydrocarbon material comprising at least one member selected from the group consisting of petroleum hydrocarbons and polyamyl naphthalenes with at least three amyl radicals and from 50 to 75% of a polyamyl naphthalene sulfonate having one metal sulfonate radical carried by an unalkylated position of its aromatic ring and having at least three amyl radicals.

JACOB FAUST.