

[54] **DETERGENT ACTIVE MATERIALS**

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[58] Field of Search .....252/161, 535, 554; 260/513, 260/459, 503, 504

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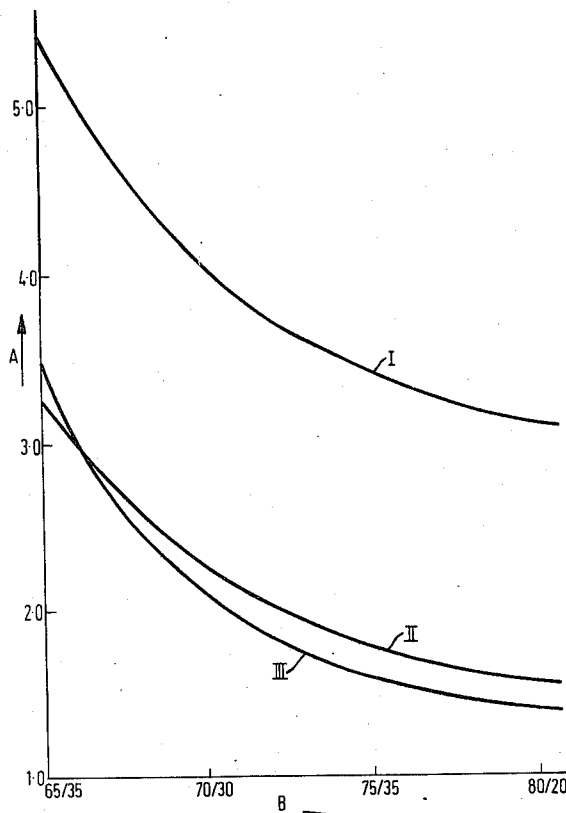
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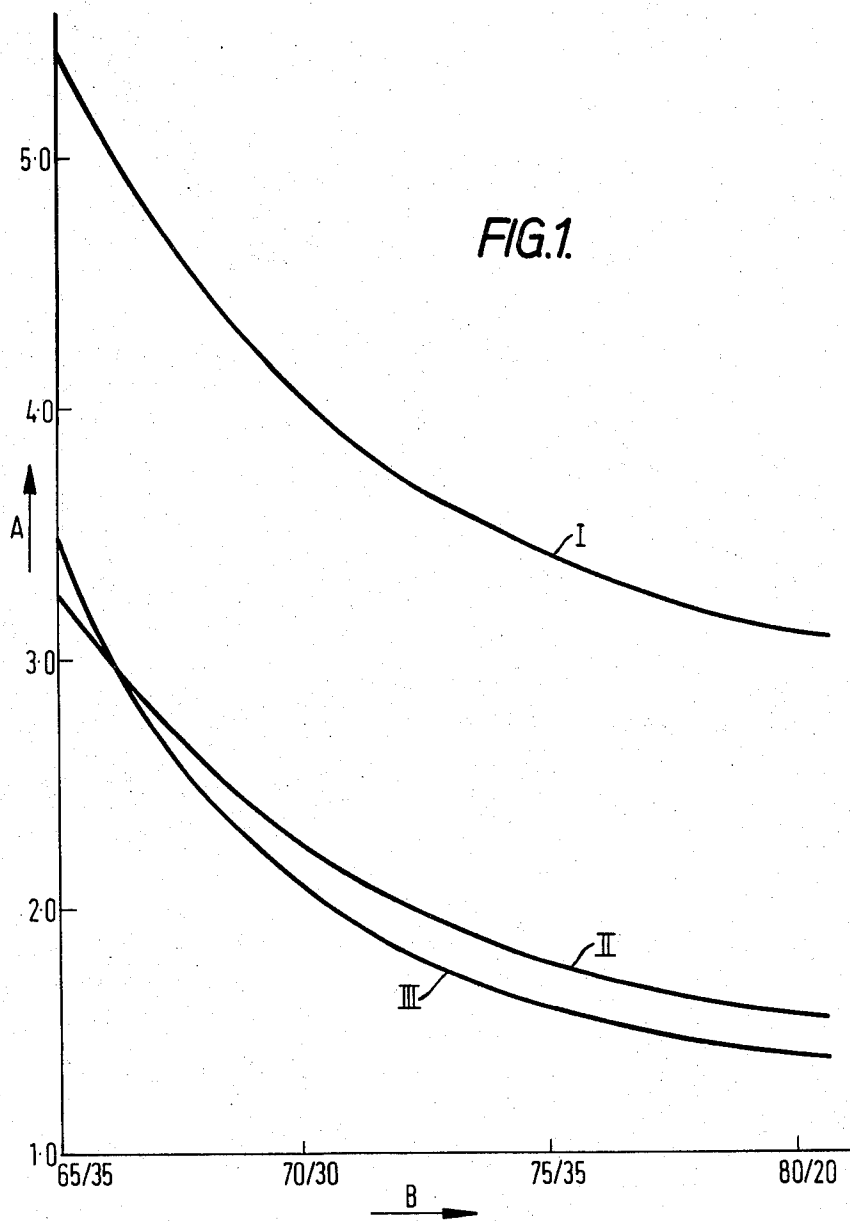
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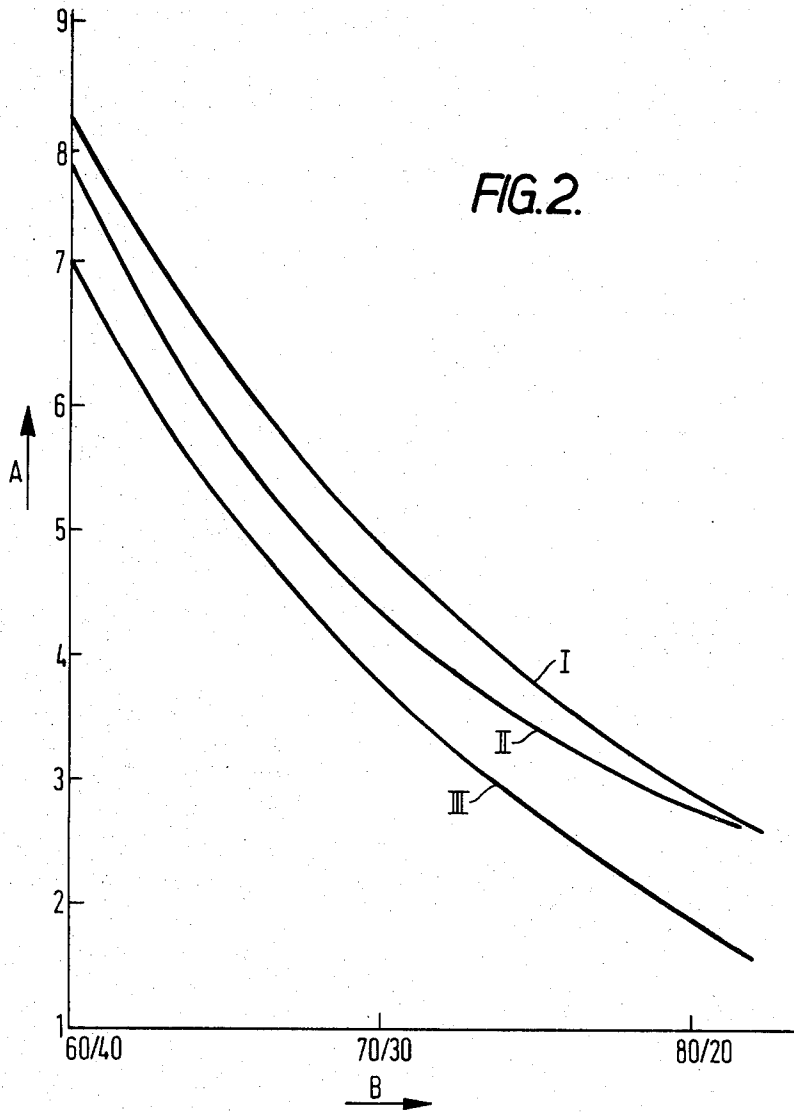
[57] **ABSTRACT**

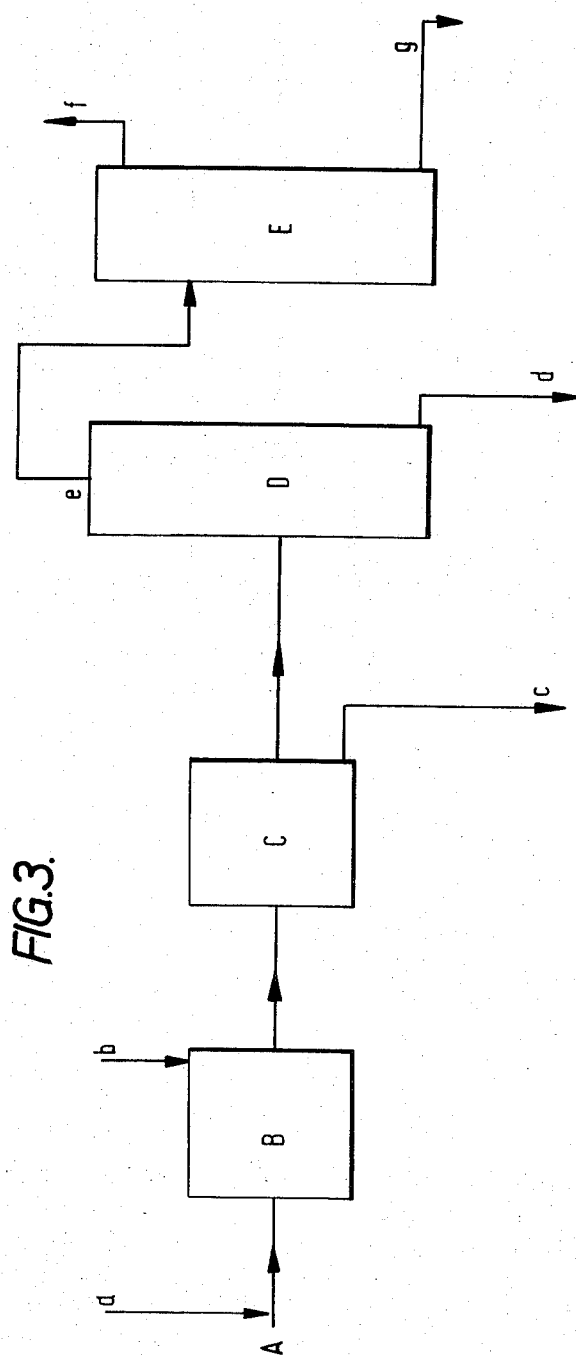
The inorganic salt content of alkane sulphonates, alcohol sulphates and olefin sulphonates can be reduced by treating the detergent active with a mixture of water and isopropanol and/or isobutanol with the conversion of bisulphite ions into sulphite ions. The treatment is performed under reflux at a temperature between boiling and 20°C below boiling. A proportion of the inorganic material precipitates and is separated from the liquor which contains the active.

**8 Claims, 6 Drawing Figures**









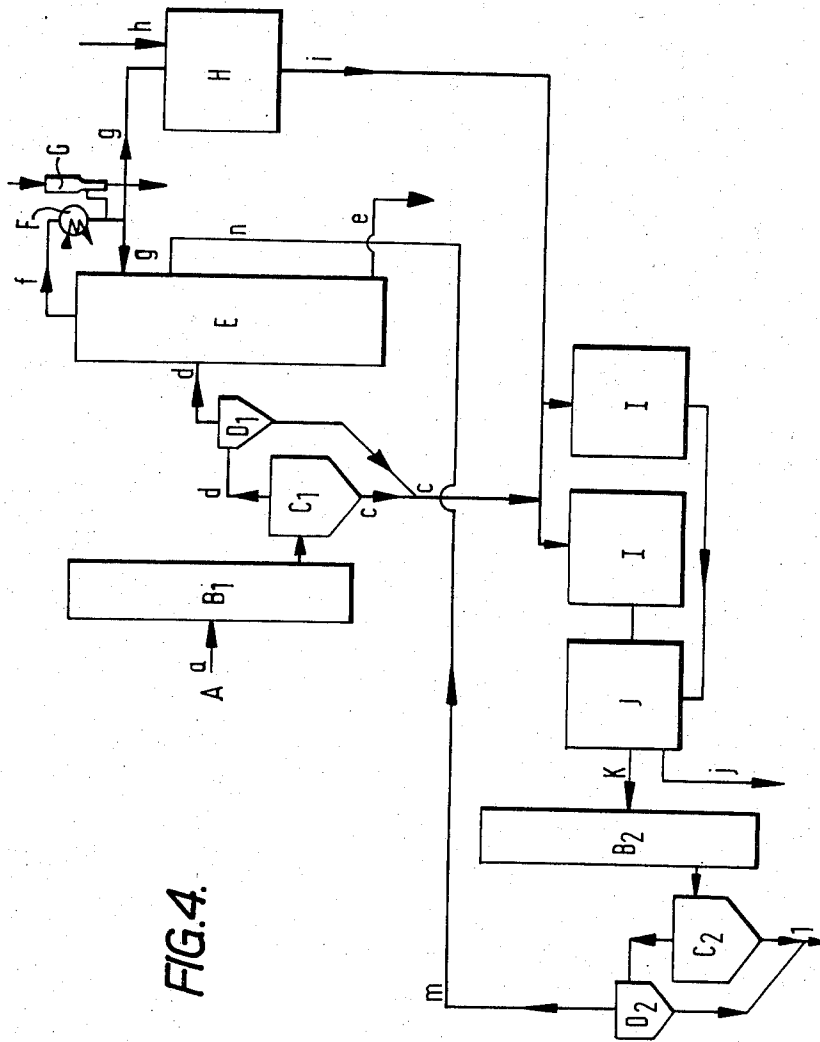


FIG. 4.

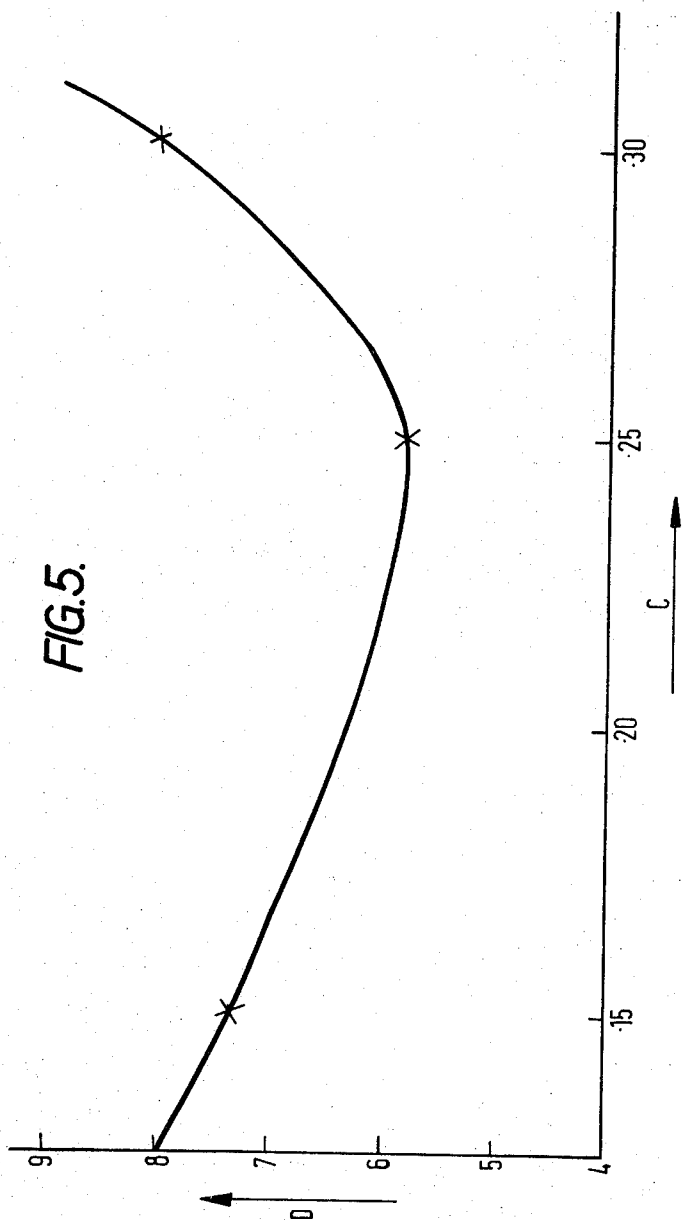
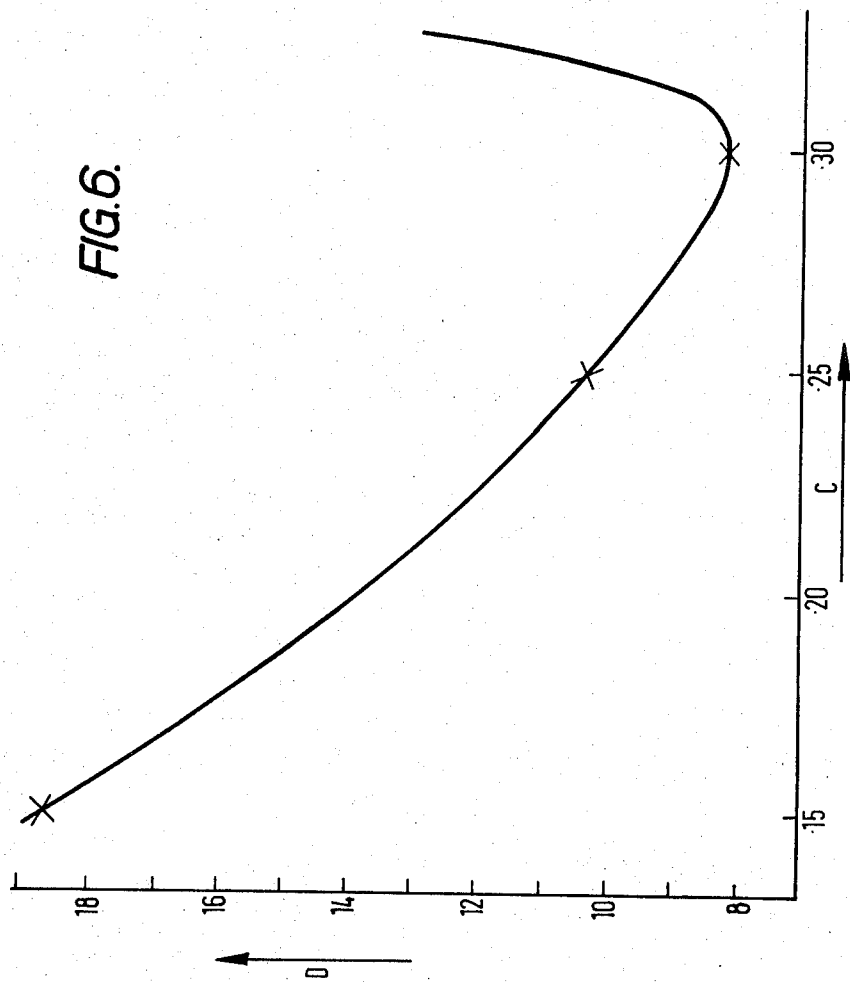


FIG. 6.



## DETERGENT ACTIVE MATERIALS

The invention relates to a process for reducing the inorganic salt content of a detergent active.

Detergent actives often contain inorganic salts. Such salts are often formed concomitantly, with the detergent active. For instance, when sodium alkane sulphonate is formed by the reaction of sodium bisulphite with an olefin the product usually contains sodium sulphite and sodium bisulphite together with traces of sodium sulphate.

The invention is particularly useful for reducing the inorganic salt content of such a product.

Many methods for separating inorganic salts have been tried but all for one or more reasons have been unsatisfactory.

The invention provides a method of reducing the inorganic material content of a detergent active material comprising the steps of;

- i. forming the active powder or paste into a monophasic solution with a solvent mixture of water and isopropyl alcohol, and/or tertiary butyl alcohol.
- ii. converting any bisulphite ions present into sulphite ions before or after (if necessary),
- iii. heating under reflux in a temperature range from boiling to 20°C below to precipitate a proportion of the inorganic material,
- iv. separating the precipitated material from the liquor and
- v. recovering the active material from the liquor.

The bisulphite conversion can be performed before the monophasic solution is formed. It has been found advisable to seed the solution to speed up the process of precipitation and thus increase the amount of inorganic material separated.

The term "monophasic" refers to the liquid phase which must exist as a single phase although it may contain some undissolved solid material when first formed.

The ratio of water to alcohol in the solvent mixture will be determined by the user of the invention having regard to the amount of inorganic material in the detergent active material. Thus a good separation of inorganic material is achieved in the region of the azeotropic ratio but because of the greatly reduced solubility of the detergent active material a relatively large amount of solvent mixture is required. As the temperature at which the solvent mix is heated is reduced the separation improves, because of the reduced solubility of the active material. The applicant has found that a useful separation of inorganic material is achieved at temperatures down to 20°C below the boiling point of the mixture.

To use sodium alkane sulphonate as an active in some special formulations, it is desirable to have the concentration of inorganic material below 2 percent. This is achieved by using a solvent mix containing more than 60 percent by weight of isopropyl alcohol or more than 78 percent by weight of tertiary butanol at boiling reflux. The upper concentration limit will be determined by the amount of alcohol the user is prepared to use per unit of detergent active.

Sodium alkane sulphonate suitable for use as a detergent active usually contains from eight to 22 carbon atoms in a straight chain with the sulphonate group at one end of the chain. Such compounds can be prepared by, for instance, free-radical reaction of sodium bisulphite

with an alpha-olefins. Alpha-olefins normally contain antioxidants to minimize their deterioration in the presence of atmospheric oxygen. It has been found that pretreatment of alpha-olefins as described in GB Pat. No. 1,159,728 removes a substantial proportion of such antioxidants.

The reaction of alpha-olefin with sodium bisulphite gives a product containing sodium sulphate together with sodium sulphite and sodium bisulphite. It has been found that better removal of salt from alkane sulphonate is obtained if sodium bisulphite present is converted to sodium sulphite by addition of alkali prior to the alkane sulphonate being heated in solution by aqueous alcohol. This obviates a tendency for a liquid phase containing much of the inorganic salts to form; to separate the inorganic salts once this liquid phase forms is especially difficult.

The invention is particularly applicable to sodium alkane sulphonate prepared by the free-radical reaction of sodium bisulphite with alpha-olefin in aqueous isopropanol. The weight ratio of isopropanol to water is usually less than 65:35 during the addition reaction, and after completion the concentration of isopropanol can be brought to the desired level for salt separation.

Experiments to illustrate the invention will now be described with reference to FIGS. 1 and 2 which show the reduction in inorganic content of sodium alkane sulphonate using respectively isopropanol and tertiary butanol in the concentrations with water shown.

In FIGS. 1 and 2, axis A shows the concentration of inorganic material in the detergent active and axis B shows the ratio by weight of the isopropanol/water and tertiary butanol/water mixtures.

In each of these figures there are three graphs which were determined under the following conditions I. Without conversion of bisulphite

II. With conversion of bisulphite

III. With seeding of the heated mix.

In order to determine graph I 250 g, 225 g, 150 g, 150 g, and 95 g of crude sodium C<sub>11-14</sub> alkane sulphonate containing respectively 12.25 percent, 7.75 percent, 6.83 percent, 8.5 percent and 7.75 percent of inorganic material were boiled under reflux in 65 percent (500 g), 70 percent (580 g), 72 percent (500 g), 75 percent (500 g) and 80 percent (500 g) by weight of isopropanol in water. After precipitation of the inorganic material the liquor was filtered and the inorganic material content measured.

This procedure was repeated, to provide Graph II, after the sodium bisulphite had been converted in the sulphite by addition of the calculated amount of 25 percent sodium hydroxide solution. Pure isopropanol was added to retain the isopropanol/water ratio at the desired level and the mixture boiled for three hours.

The procedure for Graph II was repeated with the further additional step of seeding. The mixture in each sample was seeded with 0.2 g of sodium sulphate and 0.2 g of sodium sulphite. After seeding the mixture was stirred for 3 hours before separating the precipitate and determining the inorganic material content of the filtrate.

Repeating the experiments at room temperature (20°C) using 500 g of 70 percent by weight of isopropanol in water with 33 g of crude active, it was found that a good separation of inorganic material was



achieved. However, a larger quantity of solvent is required which not only leads to greater expense in the purchase of isopropanol but also to the requirement for a larger crystallizer and increased amounts of steam for recovery of the isopropanol.

FIG. 2 shows the results of experiments using tertiary butanol and starting with a crude  $C_{11-14}$  sodium alkane sulphonate containing 12.25 percent inorganic material i.e. sodium bisulphite, sulphite and sulphate.

With both these solvents it has been found that an effective separation of inorganic material is achieved at temperatures down to 20°C below the boiling point.

The process of the invention allows the inorganic salt content to be reduced to the region of 2 percent. The use of an isopropanol/water mixture containing at least 70 percent by weight of the alcohol or a tertiary butanol/water mixture containing at least 78 percent by weight of the alcohol provides an alkane sulphonate with an inorganic material concentration of not more than 2 percent.

The invention also extends to processes for a reduction of inorganic material content in detergent active materials.

A preferred process according to the invention consists of the following stages;

a. neat isopropanol or an azeotrope of isopropanol and water is added to sodium alkane sulphonate in aqueous isopropanol to bring the weight ratio of isopropanol to water to greater than 70:30;

b. crystalline inorganic salt is separated at a temperature from 20°C below reflux to reflux;

c. the aqueous isopropanol is removed to give the sodium alkane sulphonate with reduced inorganic salt content;

d. the aqueous isopropanol is fed to a distillation column where it is separated into water and isopropanol or azeotrope; and,

e. the isopropanol or azeotrope is used in stage (a).

It should be noted that the separation in stage (d) need not be complete; azeotrope can be formed. Likewise the water can contain some isopropanol; the water will in any case contain a major proportion of any oily matter originally mixed with the sodium alkane sulphonate, for instance, paraffin and residual olefine. This is a further advantage for this process. Also the inorganic salts separated at stage (b) will be in a convenient form to extract sodium sulphite for re-use in the bisulphite reaction with olefin for making the alkane sulphonate.

Preferably in stage (b) crystallization is initiated by the addition of seed quantities of crystalline sodium sulphite and sulphate.

In a particularly preferred process according to the invention aqueous isopropanol, in which the reaction between  $\alpha$ -olefin and sodium bisulphite has occurred, is flashed off from the sodium alkane sulphonate and inorganic salts. Neat isopropanol or, preferably, an azeotrope of isopropanol and water is added to the sodium alkane sulphonate and inorganic salts and the solution so formed heated at a temperature from 20°C below reflux to reflux so that the inorganic salts start to crystallize. Crystalline inorganic salts are then separated and isopropanol and water flashed off to give sodium alkane sulphonate with reduced inorganic salt content.

Preferably the aqueous isopropanol, in which the reaction has occurred, and the water and isopropanol after separation of crystalline inorganic salts are fed to a distillation column to give an azeotrope of isopropanol and water and water which contains the bulk of any oily matter originally mixed with the sodium alkane sulphonate.

By the use of flash evaporators before the distillation column foaming in the column is reduced in comparison with a procedure within the scope of the invention where, after separation of crystalline inorganic salts, the sodium alkane sulphonate and isopropanol and water are separated in a distillation column.

Also use of a flash evaporator to flash off the isopropanol and water, in which the reaction between  $\alpha$ -olefin and sodium bisulphite has occurred, before the sodium alkane sulphonate is heated with isopropanol and water at a temperature from 20°C below reflux to reflux enables an azeotrope of isopropanol and water to be added to the sodium alkane sulphonate for the crystallization rather than neat isopropanol or azeotrope and neat isopropanol. Neat isopropanol is expensive to produce.

Use of the flash evaporator also enables the amount of azeotrope and hence the size of the crystallizers to be minimized. The amount of azeotrope added at the crystallization stage can be related to the solubility of the active at the temperature concerned.

Preferably the distillation column should be operated under slight vacuum. Thereby the operation of the flash evaporators is aided.

Examples of processes according to the invention will now be described with reference to FIGS. 3 and 4.

In FIG. 3, an isopropanol and water solution of sodium alkane sulphonate together with organic and inorganic impurities is produced, A. To this mixture is added sufficient sodium hydroxide, *a*, to convert any residual sodium bisulphite to sodium sulphite. The mixture is then fed to a crystallizer, B, where it is heated to or maintained at reflux temperature, ca 82°C, and sufficient isopropanol, *b*, is added to raise the weight ratio of isopropanol to water in the mixture to 70:30. Crystallization of inorganic salts occurs and the crystalline salts, *c*, are separated in centrifuge, C.

The isopropanol and water solution of sodium alkane sulphonate is fed to a flash-evaporator D. Sodium alkane sulphonate, *d*, with a reduced inorganic salt content is obtained. The flashed off isopropanol and water, *e*, is fed to an azeotropic or extractive distillation column, E, where the isopropanol, *f*, and water, *g*, are at least to a major extent separated.

Referring now to FIG. 4 isopropanol and water solution of sodium alkane sulphonate together with organic and inorganic impurities is produced. To this mixture is added sufficient sodium hydroxide, *a*, to convert any residual sodium bisulphite to sodium sulphite. The mixture is then fed to a tubular flash dryer, *b*<sub>1</sub>, backed by a flash chamber, C<sub>1</sub>. Sodium alkane sulphonate and inorganic impurities are fed, *c*, to Crystallizers I.

Isopropanol and water vapors *d*, from the flash chamber, C<sub>1</sub>, are fed to a cyclone separator, D<sub>1</sub>. Sodium alkane sulphonate, *c*, from the separator is fed to the Crystallizers, I. The vapors *d*, from the separator are fed to a distillation column, E, where they are fractionated into water, *e*, which contains the bulk of the

organic impurities in the sodium alkane sulphonate, and an azeotrope, *f*, of isopropanol and water.

The distillation column, E, is backed by a condenser, F, and a steam ejector system, G.

A proportion of the azeotrope, *g*, from the condenser, F, is returned to the distillation column. The remainder is fed to a storage vessel, H. Provision is made for isopropanol, *h*, to be added to the storage vessel, H, to compensate for isopropanol lost in the system. Sufficient of the azeotrope or azeotrope enhanced with isopropanol is fed, *i*, to the crystallizers, I, to dissolve the sodium alkane sulphonate at a temperature from 20°C below reflux to reflux. In a metafilter, J, crystalline inorganic salts, *j*, are separated.

Isopropanol and water *k*, from the metafilter, J, are fed to a second tubular flash dryer, B<sub>2</sub>, backed by a flash chamber, C<sub>2</sub>, and cyclone separator, D<sub>2</sub>. Sodium alkane sulphonate with a reduced inorganic salt content, *l*, is obtained from the flash chamber, C<sub>2</sub>, and the cyclone separator D<sub>2</sub>. The isopropanol and water vapors *m*, from the cyclone the separator, D<sub>2</sub>, are fed to the distillation column, E.

In a modification of the apparatus shown in FIG. 4 a second distillation column is provided immediately before dryer B<sub>2</sub>. Isopropanol and water vapors are fed back to the distillation column E and the concentrated alkane sulphonate solution supplied to dryer B<sub>2</sub>.

This invention relates also to an improvement in the process of reacting a bisulphite material for example the sodium salt, with an alpha-olefinic material to give an alkane sulphonate detergent active. The reaction is normally performed in a reaction medium comprising a mixture of isopropanol and water. The inventor has found that the amount of isopropanol required in the reaction mixture per ton of active per hour can be minimized to give reduced requirements for raw materials and equipment.

The invention is directed to a process wherein a bisulphite material is reacted with a C<sub>10</sub> to C<sub>20</sub> alpha olefin in a reaction medium comprising isopropanol and water characterized in that the weight of alpha olefin taken as starting material is sufficient to give an amount of alkane sulphonate after conversion; the ratio between the amount of alkane sulphonate and total amount of isopropanol, water and alkane sulphonate being in the range of 0.2 to 0.3. For a C<sub>11-14</sub> olefin the ratio is preferably about 0.25 and for a C<sub>15-18</sub> olefin the ratio is preferably about 0.3.

FIGS. 5 and 6 show respectively the tons of isopropanol required per ton of active produced per hour (axis D) plotted against the ratio of the weight of active produced to the total weight of active, isopropanol and water (axis C). It is seen that minima occur in the graphs at 0.25 in FIGS. 5 which relates to C<sub>11-14</sub> alpha olefins and at 0.30 in FIG. 6 which relates to C<sub>15-18</sub> alpha olefins.

In an example to demonstrate the invention 2,580 lbs of C<sub>11-14</sub> alpha olefin (available olefins 85 percent) were added to a reaction vessel with 4,460 lbs of isopropanol and 3,940 lbs of water. The mixture was heated to its boiling point and reacted with a sodium bisulphite solution consisting of 1,255 lbs of sodium metabisulphite (97.5 percent pure), and 125.5 lbs of sodium hydroxide (98 percent pure) dissolved in 2.761

lbs of water. The final product was 1.66 tons (3,733 lbs) of sodium alkane sulphonate. The ratio as defined by the invention was 0.25.

The example was repeated for C<sub>15-18</sub> olefin (available olefin present 89 percent) using 3,300 lb of olefin, 4,125 lb of isopropanol and 3,186 lb of water in the reaction vessel. The solution added consisted of 1,370 lb of sodium metabisulphite (97.5 percent pure), 137 lb of sodium hydroxide (98 percent pure) and 3,014 lb of water. The final product was 4,480 lb of sodium alkane sulphonate and the ratio as defined by the invention was 0.305.

What is claimed is:

1. A process for reducing the inorganic salt content of an alkane sulphonate containing about eight to about 22 carbon atoms, prepared by reacting an alkali metal bisulphite with an olefinic compound, comprising the steps of;

i. preparing a monophasic solution of the sulphonate in a solvent mixture of water and at least 70 percent by weight of a compound selected from the group consisting of isopropanol, tertiary butanol, and mixtures thereof,

ii. adding a basic material to the monophasic solution to convert bisulphite ions to sulphite ions.

iii. heating the solution under reflux at a temperature in the range from about the boiling point of the solution to about 20°C below this point,

iv. separating the precipitated inorganic salts from the solution, and

v. recovering the alkane sulphonate from the solution by evaporation of the mixed solvent.

2. A process according to claim 1 wherein the solvent mixture contains at least 78 percent by weight of tertiary butanol.

3. A process according to claim 1 wherein a crystalline material selected from the group consisting of alkali metal bisulphites and sulphates is added to the solution to assist crystallization of the inorganic salts.

4. A process according to claim 1 wherein the solvent mixture is azeotropic.

5. A process for reducing the inorganic salt content of an alkane sulphonate containing about eight to about 22 carbon atoms prepared by reacting an alkali metal bisulphite with an olefinic compound, comprising the steps of:

i. adding a basic material to the alkane sulphonate reaction product to convert bisulphite ions to sulphite ions,

ii. preparing a monophasic solution of the alkane sulphonate in a solvent mixture of water and at least 70 percent by weight of a compound selected from the group consisting of isopropanol tertiary butanol and mixtures thereof,

iii. heating the solution under reflux at a temperature in the range from about the boiling point of the solution to about 20°C below this point,

iv. separating the precipitated inorganic salts from the solution, and

v. recovering the alkane sulphonate from the solution by evaporation of the mixed solvent.

6. A process according to claim 5 wherein the solvent mixture contains at least 78 percent by weight of tertiary butanol.

7. A process according to claim 5 wherein a crystalline material selected from the group consisting of alkali metal bisulphites and sulphates is added to the solution to assist crystallization of the inorganic salts.

8. A process according to claim 5 wherein the solvent mixture is azeotropic.

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