Patented June 6, 1939 2,161,174

UNITED STATES PATENT OFFICE

2,161,174

ALKYL-SUBSTITUTED AROMATIC SULPHONIC ACIDS

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No Drawing. Application July 2, 1936, Serial No. 88,685

14 Claims. (Cl. 260—505)

The present invention relates to the use of new agents for scouring, washing, detergent and like purposes.

Broadly, the invention relates to the use of sulphonated alkylated aromatic compounds and their metallic salts, preferably the inorganic water-soluble salts, wherein said aliphatic group is obtainable from a halogenated gasoline or kerosene fraction and preferably a gasoline or kerosene fraction having a distilling range of substantially 180° C. to 280° C. More particularly the present invention relates to detergents, scouring and washing agents and the like comprising sulphonated alkylated aromatic compounds and preferably aromatic hydrocarbons, as for example benzene, toluene, naphthalene, diphenyl, chlordiphenyl, mono chlorbenzene and the like wherein said aliphatic group contains more than ten and less than sixteen carbon atoms, and more particularly wherein said aliphatic group contains fourteen carbon atoms.

The compounds of the present invention may be used alone or in combination with other suitable detergents for cleansing and scouring vegetable and animal fibers, when removing fatty or oily materials. They may be used in the washing of fruits and vegetables for spray residue removal.

As embodiments of the broader aspects of the invention kerosene was distilled and fractions collected having the distilling ranges of 140° C. to 162° C., 162° C. to 182° C., 182° C. to 204.5° C., 204.5° C. to 224.5° C., 224.5° C. to 244° C., 244° C. to 260° C. and 260° C. to 280° C., said fractions corresponding to the boiling points of nonane, decane, undecane, dodecane, tridecane, tetradecane and pentadecane respectively. The distillates so obtained were halogenated by means of a suitable halogen, for example chlorine, the halide so obtained condensed with an aromatic compound, for example benzene, toluene, naphthalene, diphenyl, chlordiphenyl, mono chlorbenzene and the like and the product so obtained sulphonated by means of a suitable sulphonating agent, and the sulphonic acid isolated preferably as the alkali metal salt.

As illustrative of the above process three molecular proportions of hydrocarbon distilling between substantially 244° C. and 260° C. are substantially one-third chlorinated on a molar basis. The product so obtained is treated with substantially 6.4 molecular proportions of benzene in the presence of aluminum chloride and a Friedel-Crafts synthesis carried out, after which the unreacted benzene and unchlorinated hydrocarbon are removed preferably by fractionation from the alkyl benzene. Substantially 50 parts by weight of the alkyl benzene so obtained and substantially 10 parts by weight of 95% sulphuric acid are placed in a suitable reactor and substantially 65 parts by weight of 25% oleum added slowly thereto, preferably with cooling. After the reaction is completed, water is added to effect separation of the major portion of the spent acid, which is drawn off, and the upper layer comprising the alkyl benzene sulphonic acid is preferably converted to the desired metal salt.

Other methods of preparing the preferred class of compounds may, of course, be employed as is obvious to those skilled in the art. Thus, it is obvious that other sulphonating agents, as for example chlor-sulphonic acid may be employed.

The metallic salts of the alkylated and sulphonated benzenes employing the kerosene fractions given above have all been prepared in a manner analogous to that hereinbefore described and tested for detergent and washing properties employing the following procedure.

Portions of woolen cloth weighing 5 grams and measuring 7½" x 7½" and containing not in excess of 0.2% of ether extractable material are impregnated with 5.0% by weight of paraffin oil by means of an ether solution thereof. After removal of the ethereal solvent by volatilization, the test strips are placed in glass jars of 500 c. c. capacity containing 200 c. c. of an aqueous solution of 1 gram of detergent and a sufficient number of Monel metal balls added to effect agitation of the liquid within the jars. The jars are immersed in water maintained at 50° C. and agitated by a suitable shaking machine. After scouring for one hour, the test strips are removed from the jars and thoroughly washed with water, dried and extracted with ether in a Soxhlet extractor. On evaporating the ether from the ethereal extract, the percentage of residual oil is a measure of the efficiency of the detergent action on the cloth.

The sodium salts of the alkylated and sulphonated benzenes above described were found to be efficient cleansing agents.

As further specific embodiments of the present invention toluene, xylene, naphthalene, diphenyl, chlordiphenyl and mono chlor benzene have been reacted with the halogenated kerosene distillates exemplified above, for example the alkyl halide prepared from the kerosene distillate having a distilling range of 162° to 182° C., and the alkylated hydrocarbon after sulphonating, found, on testing, to function as desirable and

valuable washing, cleansing and emulsifying agents. Further, diphenyl and chlordiphenyl have been reacted with the alkyl halide prepared from a kerosene distillate having a distilling range of 182° to 204.5° C. and the alkylated compound, after sulphonating, found to possess desirable detergent action.

As a preferred embodiment of the present invention aliphatic hydrocarbons have been prepared by careful refractionation of cuts obtained from commercial kerosene. The octane, nonane, decane, undecane, dodecane, tridecane, tetradecane, pentadecane and hexadecane fractions so prepared were halogenated by means of chlorine, and the halide so formed condensed with an aromatic hydrocarbon for example benzene in the presence of aluminum chloride as a catalyst and the alkyl benzene sulphonated in a manner analogous to that employed above in the preparation of an alkylated sulphonated aromatic compound employing halogenated kerosene cuts as the alkylating agent.

Heretofore, certain sulphonated alkylated aromatic hydrocarbons have been employed as detergents, cleansing and emulsifying agents. According to the present invention it has been discovered, however, that the most efficient detergent, cleansing and like action are present in those sulphonated alkylated aromatic compounds which contain more than ten and less than sixteen carbon atoms in the side chain. This is demonstrated and will be evident from the following data wherein the cleansing action of the sodium salts of the sulphonated alkylated benzenes containing eight, nine, ten, eleven, twelve, thirteen, fourteen, fifteen, sixteen and twenty-three carbon atoms in the side chain are compared by testing according to the method of detergency hereinbefore defined.

Table I

Detergent	Percent residual oil
Octyl benzene sodium sulphonate	1.65
Nonyl benzene sodium sulphonate	1.09
Decyl benzene sodium sulphonate	0.57
Undecyl benzene sodium sulphonate	0.20
Dodecyl benzene sodium sulphonate	0.16
Tridecyl benzene sodium sulphonate	0.18
Tetradecyl benzene sodium sulphonate	0.21
Pentadecyl benzene sodium sulphonate	0.20
Hexadecyl benzene sodium sulphonate	0.71
Tricosyl benzene sodium sulphonate	2.87

It is thus clearly shown that the use of salts of sulphonated alkylated aromatic compounds containing more than ten and less than sixteen carbon atoms in the side chain as washing, detergent and emulsifying agents comprises an important advance in the art.

What is claimed is:

1. A detergent comprising essentially a mixture of mono-sulphonated alkylated benzene hydrocarbons obtained by substantially one-third chlorinating on a molar basis a kerosene fraction selected from a kerosene having a distilling range of substantially 182° C. to 280° C. and consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of undecane, dodecane, tridecane, tetradecane and pentadecane, employing said chlorinated hydrocarbons as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith by a Friedel-Crafts synthesis in the presence of aluminum chloride, separating the alkylated benzene hydrocarbons therefrom, and sulphonating the alkylated benzene hydrocarbons so obtained.

2. A method of preparing a detergent which comprises chlorinating a kerosene fraction consisting substantially of paraffinic hydrocarbons having more than ten and less than sixteen carbon atoms and having a distilling range of substantially 182° to 280° C. by the introduction of chlorine into an excess of said kerosene so as to produce a preponderant quantity of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith by means of a Friedel-Craft synthesis and sulphonating the condensation product to form the monosulphonic acids thereof.

3. A detergent comprising essentially a mixture of water-soluble salts of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of undecane, dodecane, tridecane, tetradecane and pentadecane and having a distilling range of substantially 182° C. to 280° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith, sulphonating the alkylated benzene compounds and converting the sulphonic acids to the alkali metal salts.

4. A detergent comprising essentially a mixture of water soluble salts of mono-sulphonated, mono-alkylated benzene compounds obtained by introducing chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of undecane, dodecane, tridecane, tetradecane and pentadecane and having a distilling range of substantially 182° C. to 280° C., so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing a substantial molecular excess, based on the alkyl monochlorides present, of an aromatic hydrocarbon of the benzene series therewith to obtain substantially monoalkylated benzene compounds, sulphonating said monoalkylated benzene compounds and converting the sulphonic acids to the alkali metal salts.

5. A detergent comprising essentially a mixture of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons and having a distilling range of substantially 224.5° C. to 244° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl chlorides as an alkylation medium by condensing therewith an aromatic hydrocarbon of the benzene series and sulphonating the alkylated benzene compounds.

6. A detergent comprising essentially a mixture of mono-sulphonated alkylated benzene compounds obtained by introducing chloride into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons and having a distilling range of substantially 244° C. to 260° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith and sulphonating the alkylated benzene compounds.

7. A detergent comprising essentially a mixture of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons and having a distilling range of substantially 260° C. to 280° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith and sulphonating the alkylated benzene compounds.

8. A detergent comprising essentially a mixture of water soluble salts of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons and having a distilling range of substantially 224.5° C. to 244° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing therewith an aromatic hydrocarbon of the benzene series, sulphonating the alkylated benzene hydrocarbons and converting the sulphonic acids to the alkali metal salts.

9. A detergent comprising essentially a mixture of water soluble salts of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons and having a distilling range of substantially 244° C. to 260° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing therewith an aromatic hydrocarbon of the benzene series, sulphonating the alkylated benzene hydrocarbons and converting the sulphonic acids to the alkali metal salts.

10. A detergent comprising essentially a mixture of water soluble salts of mono-sulphonated alkylated benzene compounds obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons and having a distilling range of substantially 260° C. to 280° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith, sulphonating the alkylated benzene hydrocarbons and converting the sulphonic acids to the akali metal salts.

11. A detergent comprising essentially a mixture of alkali metal salts of mono-sulphonated alkylated benzene hydrocarbons obtained by introducing chlorine into an excess of a kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is selected from the group consisting of undecane, dodecane, tridecane, tetradecane and pentadecane, and having a distilling range of substantially 182° C. to 280° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing an aromatic hydrocarbon of the benzene series therewith, separating the alkylated benzene hydrocarbons therefrom, sulphonating the alkylated benzene hydrocarbons, adding water to the sulphonated reaction products to form an upper layer of the alkylated benzene sulphonic acids and a lower aqueous layer containing the major portion of the spent sulphonating agent, withdrawing the lower layer and neutralizing the upper layer of reaction products to form the alkali metal salts.

12. A detergent comprising essentially a mixture of water-soluble salts of benzene mono-sulphonic acids having a nuclear hydrogen substituted by an alkyl radical obtained by introducing chlorine into an excess of a paraffinic base kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is a tridecane, and having a distilling range of substantially 224.5° C. to 244° C., so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing benzene therewith, sulphonating the alkylated benzenes and converting the sulphonic acids to the alkali metal salts.

13. A detergent comprising essentially a mixture of water-soluble salts of benzene mono-sulphonic acids having a nuclear hydrogen substituted by an alkyl radical obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is a tetradecane, and having a distilling range of substantially 244° C. to 260° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing benzene therewith, sulphonating the alkylated benzenes and converting the sulphonic acids to the alkali metal salts.

14. A detergent comprising essentially a mixture of water-soluble salts of benzene mono-sulphonic acids having a nuclear hydrogen substituted by an alkyl radical obtained by introducing chlorine into an excess of a paraffin base kerosene fraction consisting substantially of paraffinic hydrocarbons, the major proportion of which is a pentadecane, and having a distilling range of substantially 260° C. to 280° C. so as to produce a preponderant proportion of alkyl monochlorides, employing said alkyl monochlorides as an alkylation medium by condensing benzene therewith, sulphonating the alkylated benzenes and converting the sulphonic acids to the alkali metal salts.

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