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ALKYL AROMATIC SULFONIC ACID

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This invention relates to an improved intermediate for the preparation of anionic detergents. More specifically this invention relates to improved alkyl aryl compounds for the preparation of alkyl aryl sulfonate detergents having superior color properties.

The detergent industry has long made use of alkylated aryl sulfonates as the active ingredient in many detergent compositions of matter. Since such detergents go into the home, the color properties of the final product are of paramount importance to the manufacturer. While the color of the detergent has no apparent effect on its properties or ability to clean, the sales potential of the product is substantially diminished when the detergent contains a yellowish or brownish tint or is otherwise off-color. The ideal color for commercial detergents is essentially snow-white in the case of a solid composition of matter and colorless or water-white in the case of liquid detergents.

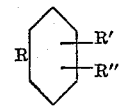
In the manufacture of detergents by the sulfonation of alkylated aryl compounds, the rather severe conditions of sulfonation effect the formation of color bodies in the product even when employing reasonably pure alkyl aromatic feed stocks for the sulfonation reaction. The exact reason for the color formation in the sulfonated product is not known; however, it is known that detergents prepared by sulfonating alkylated aromatic compounds regardless of source are all subject to this peculiar degradation which produces the aforementioned color degradation effect. Alkyl aromatic intermediates which are employed in the preparation of detergent sulfonates are generally produced by reacting an olefin of the desired chain length, e.g. C_{10}–C_{14}, with an aromatic compound and particularly a low molecular weight compound of the benzene series in the presence of an alkylating agent such as sulfuric acid, aluminum chloride, stannic chloride, zinc chloride, boron trifluoride and the like. The olefins employed for this alkylation may in turn be derived from a steam cracking process which produces relatively straight chain normal alpha olefins or they may be derived via the polymerization of C_3–C_4 olefins in a UOP type polymerization process. Alternatively, these oefins employed to alkylate benzene may be derived by the dimerization of a C_6 olefin containing steam or the trimerization of a C_4 olefin.

Other techniques for the preparation of alkylated aromatic compounds useful as detergent alkylates are well known in the art and are not particularly pertinent to the present invention. In any event the subject of the present invention relates to the detergent alkylate and in particular to detergent alkylates which are predominantly of the dodecyl benzene type. It is to be understood of course that the term "dodecyl" as employed herein is intended to include a normal C_{12} alkyl group as well as isomeric alkyl radicals having various degrees of branchiness.

As noted previously, sulfonation of alkyl aromatic compounds requires severe conditions. For example, there is employed 0.8 to 1.2 volumes and preferably about equal volumes of concentrated or fuming sulfuric acid at temperatures generally in the range of 100–250° F. for a time sufficient to effect a desired sulfonation. In lieu of sulfuric acid and/or fuming sulfuric acid, the prior art has employed chlorosulfonic acid and sulfur trioxide.

In general the alkylated aromatic compounds of this

invention fall within the scope of the following formula:



where R represents an alkyl group containing from 5 to 20 carbon atoms and averaging between 10 and 14 carbon atoms. R' may be the same as R but is preferably a lower molecular weight alkyl radical, i.e. C_1–C_4, and more preferably hydrogen. R'' is a low molecular weight alkyl radical, i.e. C_1–C_4, and more preferably hydrogen.

As noted from the above formula, the preferred alkyl aromatic intermediates of this invention are monoalkyl benzenes, although the use of dialkylated benzene, mono- or di-alkylated toluene, xylene or the like have been suggested and in some cases have been put into commercial use. While the alkylated aromatic detergents in commerce today generally contain a mixture of various isomers and homologs within the scope of the above formula, the products are generally predominantly C_{10}–C_{14} monoalkylated benzenes. The alkyl groups may be straight chained such as those derived by the reaction of a straight chain alkyl halide and benzene or they may be highly branched as in the case of a reaction product involving tetrapropylene and benzene. Intermediate degrees of branchiness such as obtained when reacting a straight chain or lightly branched internally double bonded olefin with benzene are also useful as detergent intermediates. By name, specific examples of detergent alkylates are n-dodecyl benzene, n-undecyl benzene, isododecyl benzene, n-tridecyl benzene, isotridecyl benzene, n-dodecyl toluene, didodecyl benzene or the like and mixtures thereof. Obviously a large number of compounds fall under the scope of the above formula and all would be satisfactory to some degree in the preparation of surface active alkylated aromatic sulfonates. The recitation of additional specific compounds would, however, serve no useful purpose.

It has now been found that the incorporation of a minor amount of C_1–C_4 lower alkanoyl amide such as formamide in the alkyl aromatic compound or as it is termed herein the detergent alkylate provides a product which upon sulfonation will have improved color properties over a product sulfonated in the absence of such an amide. The amounts of additive employable are 0.5 to 2.5 wt. percent based on alkyl aromatic compound. Preferably, and a range in which outstanding results have been obtained, there may be used 1.0 to 1.5 wt. percent based on detergent alkylate. The lower alkanoyl amides falling within the scope of this invention are soluble on contact with the detergent alkylate and include formamide, acetamide, propionamide, n-butyramide, isobutyramide or mixtures thereof.

The amide may be employed in one of several ways. In a preferred embodiment the detergent alkylate is contacted with the prescribed amounts of amide prior to sulfonation. In another embodiment of this invention the amide may be added during the sulfonation reaction if desired. Insofar as it is understood, the amide does not react with the alkylated aromatic compound or its contaminants so that an improved detergent alkylate will comprise the alkylated aromatic compound such as described above containing from 0.5 to 2.5% of the lower alkanoyl amide. The alkyl aromatic compound may be contacted with the amide at ambient or elevated temperatures if desired, e.g., 50 to 150° F. Contact time should be sufficient to permit solubilization of the requisite wt. percent of amide in the detergent alkylate.

To demonstrate the additive of this invention, reference is now had to the following examples.

EXAMPLE 1

50 milliliters (about 44 grams) of an alkyl benzene having an average of about 12 carbon atoms per alkyl group, wherein said alkyl group was derived by polymerizing propylene to form a tetrapropylene fraction, was placed in a one-liter flask. The alkylate was then cooled from ambient temperatures to 0° C. with a Dry Ice-isopropyl alcohol cooling bath. To the flask there was added 20% fuming sulfuric acid equal to 1.4 times the weight of the alkylate, i.e. about 60 grams. The sulfuric acid was added over a period of 10 minutes with continuous stirring. The temperature in the flask was allowed to rise to about 15° C. and was then maintained at between 15 and 20° C. for the remainder of the sulfuric acid addition. After all the acid was added, the cooling bath was removed and the temperature of the reaction mixture was raised to 55° C. over the next 10 minutes by heating with a hot water bath. After a total reaction time of 20 minutes the sulfonated mixture was then poured onto about 300 grams of clean ice in a two-liter flask with the addition of approximately 400 milliliters of cold water. The diluted sulfonated mixture was the neutralized with 30 wt. percent aqueous sodium hydroxide to a pH of 6.8 to 7.0 maintaining the temperature below 60° C. To determine the color of the product, the resultant aqueous sodium dodecyl benzene sulfonate was diluted to 1768 grams with water to a concentration of 3.54 wt. percent sodium dodecyl benzene sulfonate and the diluted aqueous solution was tested for light transmission in a Bausch and Lomb colorimeter in a 425 mu one centimeter cell. The product showed a light transmittance of 61.0% and a Gardner color of 2.5.

EXAMPLE 2

Example 1 was repeated exactly except that the dodecyl benzene detergent alkylate was first contacted with 0.44 gram of formamide which dissolved rapidly in a detergent alkylate product. Sulfonation and work-up of the product was identical to Example 1. The detergent of this example showed a light transmittance of 70.0% under the same test conditions as employed in Example 1 with a Gardner color of 1.5. To demonstrate the results more graphically, reference may be made to the following table.

Table

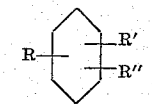
COLOR IMPROVEMENT IN SULFONATION DUE TO FORMAMIDE ADDITION

Reactants Used	Percent Transmittance, 425 mu. 1 cm. Cell		Gardner Color	
	Treated	Untreated	Treated	Untreated
20–23% Oleum + Dodecyl Benzene	[a]70.0	[a]61.0	[a]1.5	[a]2.5

[a]On a 16:1 (wt./wt.) aqueous dilution of the acid reaction mixture.

What is claimed is:

1. A process for producing an alkyl aromatic sulfonic acid which comprises reacting an alkyl benzene having the formula:



wherein R is a C_5 to C_{20} alkyl radical, R' is selected from the group consisting of hydrogen and C_5 to C_{20} alkyl radicals, and R'' is selected from the group consisting of hydrogen and C_1 to C_4 alkyl radicals with a sulfonating agent in the presence of color-inhibiting amounts of a lower alkanoyl amide.

2. A process in accordance with claim 1 wherein said lower alkanoyl amide is formamide.

3. A process for producing a monoalkylbenzene sulfonic acid which comprises sulfonating a monoalkylbenzene having an average of 10 to 14 carbon atoms in the alkyl group, said monoalkylbenzene containing a color-inhibiting amount of a lower alkanoyl amide.

4. A process in accordance with claim 3 wherein said alkanoyl amide is present in an amount of from 0.5 to 2.5 wt. percent based on said alkylated aromatic compound.

5. A process in accordance with claim 4 wherein said alkanoyl amide is formamide.

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