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HYDROGENATED NAPHTHA SOAP

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This invention relates to soap and to the process of making same and relates especially to soap made with the aid of petroleum-derived products.

The invention is concerned in a specific way with detergents containing hydrogenated or hydrogen-treated petroleum solvents particularly hydroformed products ranging from the hydronaphthas through the hydrokerosenes and to oils of even higher boiling point.

These hydroformed solvents or hydrogenolates are derived from petroleum or its various liquid distillation or extraction products. They may be produced from petroleum distillates, such as burning oil or gas oil, and in general from hydrocarbon oils of a boiling range including that of gasoline and also of ranges extending up to about 650° to 700° F. or somewhat higher. In producing such solvents a petroleum distillate oil may be passed in the vapor phase with free hydrogen over a suitable catalyst at a pressure in excess of 20 atmospheres (preferably 50 to 200 atmospheres or higher) desirably at a temperature above 900° F. and preferably within the range of about 930° to 1050° F. with a suitable partial pressure of hydrogen and time of contact to secure reconstituting of the hydrocarbon oil molecules without necessarily forming appreciable amounts of polymerized or coky material. The hydrogen supplied is preferably within the range of about 1000 to 4000 cubic feet per barrel of feed oil, the amount generally depending upon the gravity and boiling range of the charging stock. A greater proportion of hydrogen may be used with suitable variation in feed rate, temperature and pressure. The feed rate depends upon the reaction temperature, and other operating conditions such as the partial pressure of hydrogen. This rate may be suitably about 1.5 to 4 volumes of oil per volume of catalyst-filled reaction-space per hour. The catalysts preferably comprise the oxides or sulfides of the elements of the 6th group together with suitable promoters of the alkaline earth or earth oxides. Such catalysts are substantially insensitive to sulfur poisoning. In the presence of hydrogen they possess the power of hydrunsulfing the petroleum feed stock, thus removing any stench due to the presence of objectionable sulfur compounds.

Although the hydroformed petroleum products obtained thereby are substantially odorless, I may remove the last traces of odor by treating them with concentrated or fuming sulfuric acid, for example in three successive steps, using about 5% by volume each time, and finally washing with water, caustic and water.

The hydroformed petroleum products obtained thereby are mostly characterized by highly desirable solvent powers throughout their boiling ranges. Ordinarily, however, I prefer to use distillate fractions boiling in a range which lies within the points included between the temperatures of 300° and 550° F., though fractions having an initial boiling point of less than 300° F., or a final boiling point higher than 550° F. are not excluded. Such fractions generally possess rather high flash points and therefore eliminate to a large degree any fire-hazard which may be encountered either during the manufacture of such hydronaphtha soaps or during such periods of time they may remain in storage. An example of such a soap-solvent hydroformed naphtha, which is admirably suited for my purpose is that of the type designated "safety fuel", and which may be prepared by subjecting the residues obtained in the distillation of heavy naphtha to a reconstituting process as just described, redistilling the product over clay, and retaining the fraction boiling between 313° and 453° F. In one case a product made in this manner was found to exhibit a flash point of 121° F.

In one form of the present invention a hydronaphtha soap in cake or bar form or other shaped form of a character substantially free from hazard from the standpoint of inflammability, possessing an adequate degree of translucency and preferably having the property of forming a substantially denser outer layer in the cake or bar of soap is sought; it being the intention to use such case-hardening, as it were, to aid in the retardation of evaporation of the naphtha material and the like.

In soaps which have been made heretofore and sold under the name "naphtha soap" the odor of the unsaponifiable oily ingredient is usually so objectionable that it is necessary to reduce the proportion to an extremely small amount, for example, to less than one percent. So small a proportion as this results in losing the cleansing values which a more substantial percentage of the solvent would afford. According to the present invention it becomes possible to use a very substantial proportion of hydrofined petroleum oil since the odor of such hydrogen-treated material is relatively mild and sweet and the presence of substantial proportions thereof in the cake of detergent does not create any unwarranted degree of inflammability. Furthermore, it becomes possible to use perfuming agents

in very moderate quantity to yield a perfumed soap, whereas with soaps containing a reasonable proportion of cruder forms of petroleum it is extremely difficult to create satisfactory perfuming conditions without the employment of too great an amount of perfume with its resulting increase in cost.

The hydroformed naphthas and hydroformed kerosenes have the advantage of being perfume-sensitive, that is to say, their odor blends with a considerable number of the usual soap perfumes in consequence of which there is rather the tendency for the hydroformed material to act as a fixative for the perfume, which is of course advantageous. Hydroformed naphthas and oils of this character I term perfume-sensitive, by which term it is understood that a relatively small proportion of perfume such as would normally be used in a perfumed toilet soap suffices to blend with the odor of the hydroformed material whereby the resultant soap cake or bar can be given an agreeable odor.

Hydronaphthas such as those of the type designated "safety fuel" have a desirable plasticizing soap-solvent action on soap that has a moderate or minimum proportion of water. For example, a commercial grade of white chip soap so low in water that it may be ground readily to a powder will when triturated or masticated with the hydronaphtha readily agglomerate on pressure or extrusion to a semi-gelatinized, transparent to translucent mass quite free from the opacity of the initial powder. This plasticizing effect is useful in the production of cakes or bars of soap containing a maximum soap content with minimum of water and with a substantial or influential proportion of the soap-solvent hydronaphtha. This plasticizing property has particular advantage when hydronaphthas are used for the preparation of extruded soap. Such a product may be made by forcing a hydronaphtha soap composition through a die to form a thin ribbon, strip, curl or crepe sheet, and the like. The extruded soap possesses an adequate degree of translucency and also a substantial percentage of hydrosolvent which serves to increase the cleansing value of the soap. Furthermore, when prepared by using a distillate of the safety fuel type, the product is substantially free from hazard from the standpoint of inflammability. Owing to the objectionable odor of the unsaponifiable oily ingredient in the product heretofore sold under the name of "naphtha soap" it does not usually contain more than about 1% of the "naphtha" ingredient and this amount of oil is not sufficient to plasticize a relatively dry soap to the extent required for extrusion. In other words, if such a soap is to be extruded it must be additionally plasticized by water. However, according to the present invention, relatively large percentages of hydronaphtha can be used while the water content is kept very low.

Soaps may also be prepared in powder form as will be described hereinafter.

Soaps which are made by any of the well-known processes and which contain a low content of water are suitable for my purposes. Such soaps are usually produced by the saponification of tallow or other vegetable or animal fats or oils by means of alkalies, as for example sodium or potassium hydroxide, or by the direct action of such alkalies on the higher fatty acids, as for example stearic acid or distilled cottonseed fatty acids, or on wax acids, or on mixtures of these acids.

By wax acids is meant the acids derived by the oxidation of paraffin wax, or other mineral waxes such as those from petrolatum, ceresin, ozokerite, Palembang wax, slop wax, wax-tailings, and in some instances Montan wax, by means of strong nitric acid, air, oxygen, or oxidizing gases. Such oxidation is generally carried out at temperatures corresponding to the melting point of the waxes or higher (e. g. 300–400° F.), and preferably in the presence of catalysts such as previously oxidized wax, manganese or cobalt resinates, barium cinnamate and the like, mixtures of these acids, or other appropriate catalysts. Also the acids, obtained by the oxidation of heavy petroleum oils, and similar substances are not excluded.

In making hydronaphtha soaps I prefer to grind soap of low water content to a fine powder and then thoroughly incorporate the hydronaphtha with the finely ground soap. The thoroughly masticated mixture can thereupon be molded into any desired shape by placing in a mold and subjecting to pressure. The pressure required will vary with the quantity of hydronaphtha incorporated with the powdered soap but generally pressures from about 50 to 2000 lbs. per square inch will be sufficient. I have found that as high as 50 percent by weight of the soap-solvent hydronaphtha may be incorporated with the soap. This quantity of hydronaphtha is much larger than is generally required, and I prefer to use a smaller quantity in most instances, say 10 to 12.5 percent, though with some soaps as little as 2 to 5 percent may be all that is required. The particular use which is to be made of the soap will be decisive in determining the proportion of hydronaphtha incorporated with the soap.

In making extruded hydronaphtha soaps I prefer first to grind the soap to a fine powder and then thoroughly incorporate the hydronaphtha with the finely ground powder, as by stirring or triturating. The thoroughly incorporated mixture of powdered soap and hydronaphtha may then be pressed to force it through a die in the form of a thin film, ribbon or sheet. One method of accomplishing this is to place the mixture of powdered soap and hydronaphtha in a pressure chamber in which is situated an extrusion nozzle of the requisite size and shape for the purpose specified. Upon the application of sufficient pressure to a plunger in the pressure chamber the soap composition is extruded through a ribbon die in the form of curled fragments. Usually a pressure of 500 to 2500 lbs. per inch will be sufficient, though greater or less pressures may be required. The pressure actually needed will depend upon such factors as the proportion of hydronaphtha incorporated with the powdered soap, the aperture of the die and the diameter of the plunger. In general, the shape taken by the extruded soap is that already indicated, namely, films, shavings or curled strips. Plain flat strips also may be produced or even sheets of a smooth surface that is free from undulations. On the other hand, the sheet or ribbon may be extruded in such a manner as to cause it to take the form of crepe, that is, to have a crepe or undulating surface. Large sheets may be broken up into small fragments or chips, if desired. A die which gives a tubular form can, for example, be used producing a ribbon bent into the form of a tube. Such tubes can be cut into short lengths as desired.

Care should be taken not to exceed a certain maximum of soap-solvent hydronaphtha plasticizer, as otherwise stickiness of the soap may result causing the ribbons or other extruded shapes to stick together when standing, for example, in package form for a considerable time. It is desired to produce a soap in accordance with the present invention which will be made up of flakes, sheets, shavings, and the like, which do not stick together but can be introduced into water intended for washing purposes in the form substantially as originally produced from the extrusion apparatus, thereby retaining a maximum degree of solubility or readiness to dissolve due to the large surface exposed to the action of water.

An optimum proportion of hydronaphtha for general cleansing purposes ranges from somewhat less than 10 percent up to approximately 20 percent, a suitable working proportion being between 12 and 15 percent. Plasticized extruded hydronaphtha soap preferably is packaged in metal cans having tight fitting covers whenever the naphtha used is of a somewhat volatile type. As the grade of naphtha increases in its boiling point, becoming definitely a non-volatile kerosene type, precautions of this kind to prevent evaporation are not necessary.

One method of making hydronaphtha soap powders is to reduce a soap of low water content to a fine powder and then thoroughly incorporate the hydronaphtha with the soap powder by grinding. While large proportions of hydronaphtha, as high as 50 percent, may be incorporated in this manner, I prefer to use about 10 to 12 percent. This proportion of hydronaphtha yields a dry, pulverulent hydronaphtha soap powder, while with larger proportions the powder tends to become somewhat sticky and more difficult to grind. In some instances lesser proportions of hydronaphtha, say 2 to 5 percent, may be all that is required. The particular use which is to be made of the soap will be decisive in determining the proportion of hydronaphtha incorporated with the soap powder.

Another procedure which may be used in the preparation of such soap powders is to mix thoroughly a relatively large proportion of solid alkali, such as sodium carbonate, with, for example, the solid or semi-solid acids obtained by the oxidation of waxy materials. During heating the mixture is kept well agitated. The product thus obtained is a dry, fluffy powder with which hydronaphtha may be incorporated to yield a hydronaphtha soap powder.

The soap powders or soap materials made from the lower fractions will be of a softer nature than those products made from the higher fractions. Those fractions which contain acids in the stearic range, that is, 18 carbon atoms, generally give the harder particles of soap product. By the use of one fraction, or mixtures of these fractions as desired, I may obtain soap products of a harder or softer nature.

In the preparation of soap powders within the confines of the present invention I prefer to prepare these in a form which permits a free-flowing product to be secured. Owing to the plasticizing or gel-forming qualities of hydronaphtha the proportion of the latter therefore should be held below that which would cause agglomeration of the soap powder in the package and prevent ease of distribution of such soap powder at the time of use. For example, it is convenient to package such a product in metal boxes with perforations in the cover and the powder should retain such a condition of pulverulency or free-flowing quality that it may be discharged from such packages in the manner desired. Ordinarily the introduction of more than 10 percent of soap-solvent hydronaphtha having a powerful plasticizing action on ordinary water-soluble soaps is undesirable if agglomeration is to be avoided. However, the addition of fillers and abrasives greatly promotes flowability and permits the employment of higher proportion of hydronaphtha when desired. The addition of 2 or 3 parts of powdered pumice to 1 part of soap, or other powdered abrasive in somewhat similar proportion, is useful for the purpose. Likewise sodium carbonate in the commercial form of soda ash used to the extent of several times the amount of the powdered soap is of great assistance in preventing agglomeration and giving free-flowing properties to the product.

In addition to perfumes I may also add disinfectants to the hydronaphtha soap. This may be done, for example, by incorporating first the disinfectant with the finely ground soap and then the hydronaphtha, or when solubility permits the disinfectant may be dissolved in the hydronaphtha and the hydronaphtha solution incorporated with the ground soap. After incorporation of both the hydronaphtha and the disinfectant the powdered soap is molded into any desired shape by subjection to pressure. The degree of disinfecting power possessed by various disinfectants will largely determine the proportion in which they are incorporated with the soap.

When desired, wetting agents may be added to the hydronaphtha soaps in order to increase their efficiency as detergents. This addition of wetting agents may be made in several ways. When the wetting agent can be ground to a fine powder it may be mixed in this form with the powdered soap, and the hydronaphtha then incorporated with the soap mixture. In other instances the wetting agent may be mixed first with the hydronaphtha to secure either a solution of the agent in the hydronaphtha or a homogeneous paste of hydronaphtha and wetting agent, and this solution or paste incorporated with the powdered soap. Still another procedure, particularly applicable to wetting agents which are soluble in water, is to grind the wetting agent with a small quantity of water to secure a concentrated solution of the wetting agent in water, and after incorporating the water solution with the powdered soap finally to incorporate the hydronaphtha with the soap mixture. In this last procedure care must be exercised not to employ an excessive quantity of water otherwise the soap mixture will become pasty and cannot be readily incorporated with the hydronaphtha. While the quantity of wetting agent employed will be determined largely by its degree of efficiency, in most instances the amount required will vary from about 1 percent to 10 percent of the weight of the soap.

Rosin soaps may also be incorporated with these hydronaphtha soaps. One method of effecting such an incorporation is to mix thoroughly, for example, wax acids and rosin, add an equivalent quantity of potassium hydroxide, or other alkali, dissolved in water, and evaporating the water. Hydronaphtha may then be incor-

porated with the mixed soaps. Another procedure is to make the separate soaps of wax and of rosin, for example, and then mixing the dried soaps and incorporating the hydronaphtha with the soap mixture.

The following examples serve to illustrate various phases of the present invention. The proportions given are parts by weight.

Example 1.—To a mixture consisting of equal parts of wax acids, stearic acid, and distilled cottonseed acids was added an equivalent quantity of sodium hydroxide dissolved in water. This mixture was evaporated to dryness and a solid soap obtained. This soap could be readily incorporated with 5 percent of hydronaphtha (boiling from 94° to 398° F.) to yield a solid homogeneous hydronaphtha soap.

This soap also could be incorporated readily with 10 or 20 per cent, respectively, of the hydronaphtha to yield hydronaphtha soaps.

Example 2.—A sample of commercial white chip soap, having a low water content, was ground to a powder and then 5 percent of safety fuel (hydronaphtha) having a boiling range of 312° to 406° F. was thoroughly incorporated with the powdered soap stock. This hydronaphtha soap stock was then formed into the shape of a cake by subjecting it in a mold to a pressure of about 500 pounds per square inch.

In a similar manner hydronaphtha soaps containing 0.5%, 1%, 2%, 10%, 33⅓% and 50% of hydronaphtha were prepared. These were likewise molded into the form of cakes, the pressures required varying from about 25 to 2000 pounds per square inch; the higher pressures being used with those compositions containing the lesser proportions of soap-solvent hydronaphtha. The plasticizing action of hydronaphtha on dry soap is useful in the shaping of the soap product.

Example 3.—4.5 parts of lavender perfume were added to 100 parts of safety fuel. 5 parts of this solution were incorporated with 100 parts of ground soap chips, and the hydronaphtha soap likewise was shaped into the form of a cake or bar by subjecting it, in a mold, to pressure.

Example 4.—100 parts of white soap flakes were pulverized, and to this there were added 3 parts of powdered para tertiary butyl phenol. The two substances were thoroughly mixed by grinding and then 10 parts of the safety fuel were incorporated. The hydronaphtha soap so obtained could be molded in a similar manner as above.

Example 5.—One part of the wetting agent, the mixed sodium salts of low molecular weight sulfo-acids from acid sludge, was incorporated by grinding with 10 parts of the safety fuel. The mixture of safety fuel and wetting agent was then incorporated with 100 parts of finely ground soap chips to yield a hydronaphtha soap. This hydronaphtha soap was molded into the shape of a cake.

Example 6.—40 parts of oleic acid were added to 17.8 parts of a 44.62 percent (by weight) solution of potassium hydroxide. After mixing, the excess water was removed by evaporation. The dry soap stock so obtained was ground to a powder and 10 parts of hydronaphtha incorporated with 100 parts of the soap stock. The hydronaphtha soap was shaped into cake form by placing in a mold and subjecting to pressure.

Example 7.—50 parts of soap prepared from oleic acid, as in Example 6, were thoroughly mixed by grinding with 50 parts of rosin soap. 10 parts of hydronaphtha were incorporated with 100 parts of this soap mixture and the resulting hydronaphtha soap was molded into the shape of a cake by placing in a die and subjecting to pressure.

Example 8.—A sample of commercial white chip soap, having a low water content, was ground to a fine powder and then 5 percent of safety fuel (hydronaphtha) having a boiling range of 312° to 406° F. was thoroughly incorporated with the powdered soap. This hydronaphtha soap was then placed in a circular chamber, containing a plunger, and expressed in an extruded form by the application of pressure.

Example 9.—40 parts of wax acids, having an apparent molecular weight of 287.5, were added to 17.5 parts of a 44.62 percent (by weight) solution of potassium hydroxide. After thoroughly mixing, the water was removed by evaporation and a solid soap obtained. This soap stock was ground to a fine powder and 10 parts by weight of hydronaphtha added. The hydronaphtha soap stock was converted to the extruded finished form as described in Example 8.

Example 10.—40 parts of oleic acid were added to 17.8 parts of a 44.62 percent (by weight) solution of potassium hydroxide. After mixing, the excess water was removed by evaporation. The dry soap stock so obtained was ground to a powder and 10 parts of hydronaphtha were incorporated with 100 parts by weight of the soap stock. The hydronaphtha soap stock was converted to the extruded finished form as described in Example 8.

Example 11.—A sample of commercial white chip soap, having a low content of water, was ground to a fine powder. 5 parts of safety fuel (hydronaphtha possessing a boiling range of 312° to 406° F.) was thoroughly incorporated with 100 parts of the soap powder by grinding. The hydronaphtha soap powder so made was a white, soft powder, easily ground, and could be used in cleaning operations, particularly those involving the removal of grease or oil.

Example 12.—1 part of wax acids was thoroughly incorporated with 2 parts of soda ash and the mixture heated to a temperature of about 284° F. with constant agitation. After reaction was completed the soap composition was cooled and ground to a powder. 10 parts of this soap powder were incorporated with 1 part of hydronaphtha to yield a hydronaphtha soap powder. This hydronaphtha soap powder lathered well with water and could be used as a detergent.

Example 13.—A hydronaphtha soap powder containing wetting agents was made in the following manner. 5 parts of the sodium salts of mixed sulfonic acids, derived from the sludge obtained on the treating of petroleum oil with sulfuric acid, were reduced to a powder. The finely ground wetting agent was thoroughly incorporated with 50 parts of the soap powder made from wax acids and soda ash (sodium carbonate) as described in Example 12. This soap powder was afterwards incorporated with 1 part of hydronaphtha, thus resulting in the formation of a hydronaphtha soap powder.

While it is not necessary to employ for making the soap cakes or bars a soap stock which has been strongly dried, the preferred method is carried out advantageously with a soap of low water content. For example, a chip soap can be rather well dried to a water content of only a few percent. While, therefore, the employment of well dried soap represents a preferred embodiment of the invention, it should be under-

stood that the hydronaphtha may be incorporated in saponified material of higher water content or even in soap pastes and in various other saponified products.

Any of these products may contain various fillers, silicate of soda, soda ash, coloring matter, and the like.

A hydronaphtha of the safety fuel type may be converted to a clear jelly-like substance by heating it with 2 or 3 percent of soap, especially a soap containing wax acids. The clear water-white gelatinized material thus obtained may be employed for cleansing purposes in various ways. A soap made by saponifying wax acids with potassium hydroxide, drying and dissolving in the safety fuel by heating with it to about 150° C. gives a jelly which is somewhat darker in color but is remarkably free from syneresis and which therefore is better suited for employment in cleansing compounds of this general character than products in which the gel in the course of time shrinks with exudation of naphtha in an unthickened condition.

As has been indicated, by the term "hydronaphtha" I mean any hydrogenated naphtha, kerosene, and the like, within the scope of the present invention that is acting as a soap-solvent, including hydrofined petroleum solvents of various boiling ranges extending through light naphtha, heavy naphtha, light kerosene, heavy kerosene, and even higher. I prefer, however, hydrofined products substantially or wholly free from stench forming hydrogenated naphthalenes, such as tetralin. The term "hydronaphtha" as employed herein, therefore, should be understood to embrace petroleum fractions of a boiling range higher than that ordinarily comprehended by the term naphtha, such fractions, however, having a bland sweet odor and being substantially free from naphthalene derivatives such as tetralin; the boiling range of the hydronaphtha preferably being in the zone embracing so-called heavy naphtha or light kerosene.

Hydronaphtha of the safety fuel type may be used in quite large proportions without rendering the soap inflammable. By reference to Example 2 it will be noted that I have therein set forth compositions containing various proportions of this grade of hydronaphtha. If soap cakes or bars are so made and shavings obtained from these it will be found that on attempting to ignite the tips of such shavings by holding thereunder the flame of a match ignition of the shaving does not occur with a product containing 10 per cent of the naphtha. On continued holding of the soap chip in the match flame the hydronaphtha soap does not catch fire, as might be expected, but slowly melts and becomes charred accompanied occasionally by very small localized spits or flashes. With 33⅓ percent and especially with 50 percent of hydronaphtha similar shavings are fairly inflammable when exposed to ignition conditions in this manner. A desirable range for preservation of non-inflammability is a soap containing between 10 and 20 percent of the naphtha material. Ordinarily safety fuel can be used in soap in a proportion up to 12 percent or possibly 15 percent while still remaining below the point of ready inflammability as determined by exposing a shaving of the soap to a flame.

The above examples are given for illustrative purposes only and my invention is not to be limited to any of the substances named therein or to the proportions in which these substances are used.

I claim:

1. The process of plasticizing soap, which comprises incorporating it with a non-flammable hydrogenated petroleum distillate of a boiling range substantially of 300° to 400° F. as the essential plasticizing agent.

2. Plasticized soap in solid form containing hydrofined heavy naphtha boiling substantially between 300° and 400° F. as the essential plasticizing agent.

3. The process of treating soap, which comprises incorporating it with a non-flammable hydrogenated petroleum distillate as the essential plasticizing agent, said distillate boiling substantially between 300° and 400° F. and possessing soap-plasticizing properties, whereby a plasticized hydronaphtha soap in solid form is obtained.

4. A solid detergent composition consisting essentially of a soap and hydroformed soap-plasticizing naphtha boiling substantially between 300° and 400° F., said composition being substantially non-flammable.

5. A solid detergent composition comprising a soap and a hydroformed petroleum distillate boiling substantially between 300° and 400° F. as the essential plasticizer, the proportion of the distillate being more than 1 per cent but not exceeding about 20 per cent of the weight of the soap, said composition being substantially non-flammable.

6. Bar soap containing hydroformed naphtha as the essential plasticizing agent, the latter possessing a boiling range substantially of 300° to 400° F., the proportion of said naphtha being about 1 to 20 per cent of the weight of the soap, said bar soap being substantially non-flammable.

7. An extruded plasticized soap in the form of thin pieces containing hydroformed naphtha as the essential plasticizing agent, the latter possessing a boiling range substantially of 300° to 400° F., the proportion of said naphtha being about 5 to 15 per cent of the weight of the soap, said extruded soap being substantially non-flammable.

8. A soap powder comprising a water-soluble soap and a hydroformed naphtha possessing a boiling range substantially of 300° to 400° F. as the essential plasticizing agent, the proportion of said naphtha being about 5 to 15 per cent of the weight of the soap, said soap powder being substantially non-flammable.

9. A non-flammable plasticized hydronaphtha soap in solid form containing hydrofined naphtha having an initial boiling point of about 300° F. as the essential plasticizing agent.

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