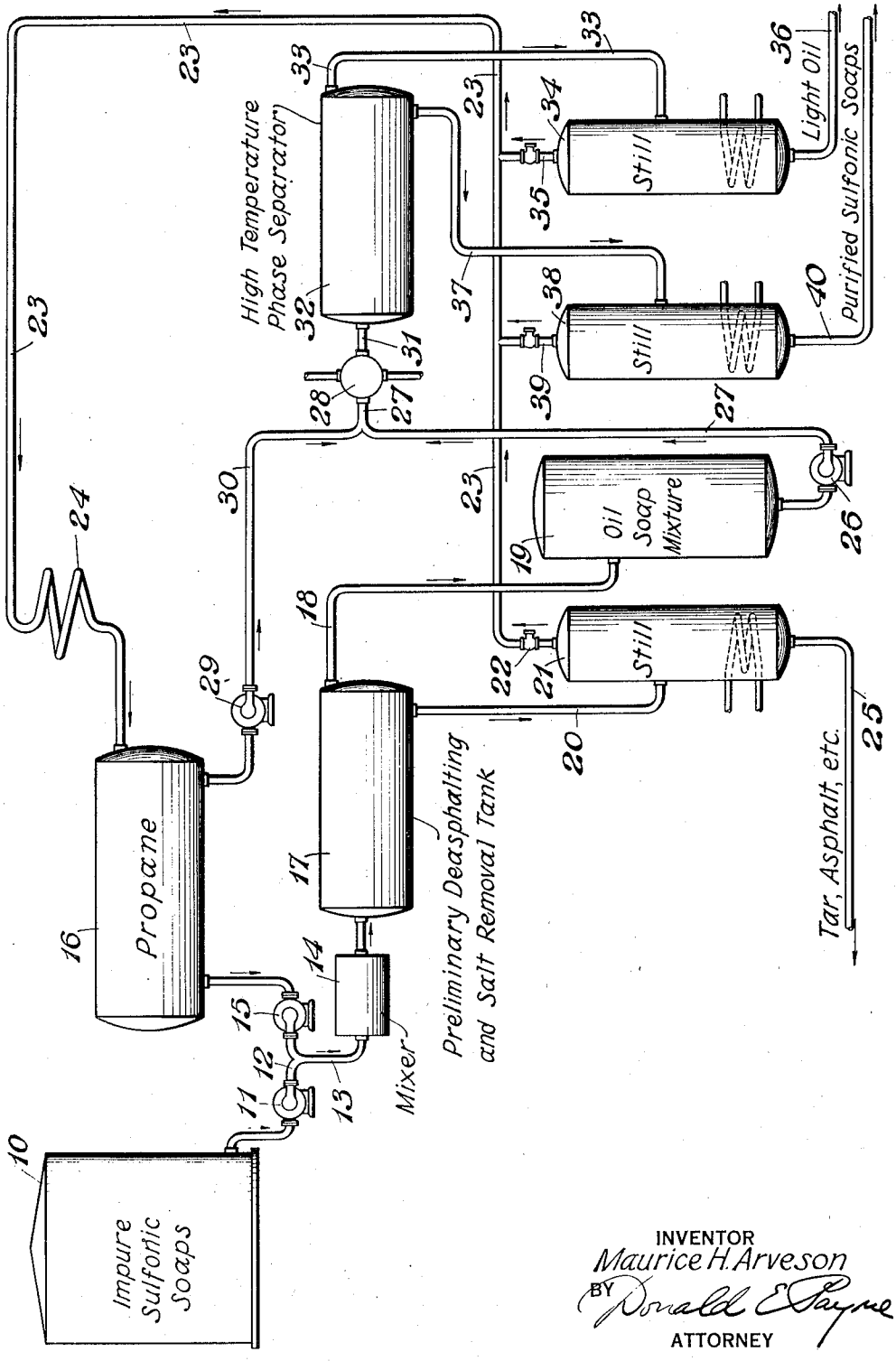
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PURIFYING MAHOGANY SOAP

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PURIFYING MAHOGANY SOAP

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This invention relates to the purification of sulfonic compounds and it pertains more particularly to the purification of mahogany soap by means of normally gaseous solvents such as propane at elevated temperatures.

In the refining of mineral oils with concentrated or fuming sulfuric acid, particularly in the refining of viscous mineral oils for the production of medicinal oil or white oil, the sulfuric acid reacts with certain of the oil components to form sulfonic compounds. Some of these compounds are preferentially water soluble and are recovered from the acid sludge; these are usually of a greenish color and lower molecular weight and they are commonly referred to in the art as "green acids" or "green acid soaps," depending on whether or not the acid sludge material has been neutralized. Certain high molecular weight reaction compounds are oil soluble, but may be recovered from the oil by extraction with dilute alcohol; these compounds are of characteristic brownish or mahogany color and they are consequently called "mahogany acids" or "mahogany soaps." In other refinery processes a pressure tar may be treated with concentrated or fuming acid to yield sulfonic compounds and an oil which imparts a desired bloom to other oils with which it is blended. Other materials which may be treated with concentrated or fuming sulfuric acid to produce sulfonic compounds include waxes, gas oils, pressure tar distillates, coking still distillates, and liquids obtained from polymerization or hydrogenation of hydrocarbon materials.

The invention is primarily concerned with mahogany soaps and particularly with preferentially oil soluble sulfonic compounds which contain so much oil, tarry matter, and other deleterious substances that they can not readily be used as emulsifiers, detergents, wetting agents, etc. It is common practice to segregate mahogany soaps into the grade A class which is exceptionally useful for emulsifying oil in hard water, and the grade B class which does not possess the desired emulsifying properties and which contains such a high percentage of impurities that it is practically worthless. For years vast amounts of this grade B mahogany soap have been accumulating in oil refineries and attempts to purify it and use it in industry have proved futile. The object of my invention is to purify sulfonic compounds and particularly to purify grade B mahogany soap.

Ordinarily one skilled in the art would expect mahogany soap to have solubilities in normally gaseous hydrocarbons such as propane similar to the solubilities of oxidized hydrocarbon compounds and tarry matters. Since oxidation products, resins, tars, and asphalts are readily precipitated from an oil-propane solution, it would reasonably be expected that mahogany soap would likewise be precipitated from a propane solution at ordinary room temperatures. I have discovered, however, that sulfonic compounds, and particularly grade B mahogany soap, are soluble in propane throughout a relatively wide temperature range,—from 0 to over 100° F. I have also discovered that at extremely high temperatures, temperatures of from about 120° to 200° F., a large amount of relatively pure mahogany soap is thrown out of propane solution and I have found that as the temperature increases more and more of the mahogany soap is thus precipitated. In practicing my invention, therefore, I may first remove asphaltic and tarry matters from the mahogany soap and I may then treat it with a light hydrocarbon such as propane at a temperature which, in the case of propane, may be about 120° to 200° F.

The physical phenomena on which my invention is based may be demonstrated by subjecting a propane-mahogany soap mixture to increased temperatures and pressures in a glass tube and observing the appearance of the mixture. At 75° F. this mixture will have a milky appearance, but there will be no phase separation. Increase in pressure up to 750 pounds per square inch has no effect on this appearance. As the temperature is increased, the white material forms a curd which settles out slowly. Increase in pressure reduces the size of the particles. At 104° to 106° F. this curd appears to coalesce or melt to form a yellow, muddy-colored liquid layer. Increase in pressure causes this liquid to partially dissolve in the upper liquid layer and the white curd to reappear. As the temperature is increased, the volume of the lower liquid layer increases. At about 130° to 135° F. and 350 pounds per square inch pressure, the upper liquid layer is nearly colorless and the lower liquid layer is translucent and of a muddy yellow color and it occupies about 15% of the total volume of the mixture. Increasing the pressure to 850 pounds per square inch reduces the volume of the lower layer to about 5% of the total volume and changes its appearance to a muddy yellow color which looks somewhat like a flocculent precipitate. As the temperature is gradually increased, the volume of the lower layer increases unless the pressure is likewise markedly increased.

A method of making practical use of these

phenomena is illustrated in the accompanying drawing which forms a part of this specification and which illustrates a flow diagram of apparatus for practicing the invention.

Grade B mahogany soaps from storage tank 10 may be forced by pump 11 through lines 12 and 13 to mixer 14 wherein they may be admixed with about 2 to 15 volumes, preferably about 6 volumes, of propane which is introduced into line 13 by pump 15 from storage tank 16. The mixture of propane and impure sulfonic soaps is introduced from mixer 14 into preliminary deasphalting chamber 17, preferably at a temperature of about 80° to 120° F. Hydrocarbon oils and sulfonic soaps are present in the upper propane solution and are withdrawn through line 18 to oil soap storage tank 19. Resinous, tarry or asphaltic matter is removed as a lower layer from tank 17 through line 20 to a suitable still 21, the propane being withdrawn through valved line 22 and returned to storage thru header 23 and condenser 24 to tank 16, asphaltic material, etc. being withdrawn through line 25.

The salt content (sodium sulfate, etc.) of the mahogany soap may be somewhat lowered by the preliminary deasphalting step, but these salts appear to be closely bound to the sulfonic soaps and they will therefore probably go with the propane-oil-soap solution. This solution may therefore be treated at this stage in any conventional manner for the removal of these salts. If the neutralization of the sulfonic acids has been effected by slowly adding the sour oil to a dilute caustic solution, the salt content may be sufficiently low to eliminate the necessity of any further salt removing treatment.

If the oil-soap mixture in tank 19 is the product of an initial deasphalting step, it will be combined with a substantial volume of propane; this partially purified oil-soap mixture may be prepared in any other convenient manner preliminary to the final purification step to be hereinafter described.

Oil-soap mixture from storage tank 19 is forced by pump 26 and line 27 to heater 28. Similarly, propane from tank 16 is forced by pump 29 and line 30 to line 27 and heater 28 wherein it is intimately admixed with the oil-soap mixture and heated to a temperature above 120° F.,—preferably to about 135° to 160° F. The volume of propane will depend somewhat upon the nature of the soap stock undergoing treatment, desired results to be obtained, temperatures to be employed, etc., but I prefer to use about 4 to 12, preferably about 8, volumes. This mixture at a temperature of about 140° F. may be introduced from heater 28 through line 31 into separator 32 which may consist of a simple, horizontal, cylindrical vessel. The pressure in tank 32 is maintained sufficient to keep the propane in liquid phase and to prevent undue agitation,—preferably it is between 200 and 1,000 pounds per square inch.

The propane-oil solution is withdrawn from the upper part of tank 32 through line 33 and introduced into still 34, the propane being withdrawn thru valved line 35 and returned to storage thru header 23 and condenser 24 to tank 16, the light oil being withdrawn through line 36 into suitable storage tanks. The soap fraction is withdrawn from the base of chamber 32 through line 37 and introduced into still 38, the propane being returned by lines 39 and 23 and condenser 24 to storage tank 16 and purified sulfonic soaps being withdrawn through line 40 to suitable storage tanks.

While I have described a simple, high temperature propane treating process for purifying grade B mahogany soaps, it should be understood that the invention has many other applications. Instead of a simple, one-stage process, I may successively treat a mahogany soap at higher and higher temperatures or with increasing amounts of propane to obtain different fractions of sulfonic soaps. Starting with an impure soap containing about 39% of oil, I have found that the soap precipitated at 130° F. contains only about 16% of oil while the material precipitated at 145° F. contains less than 15% of oil.

While I have described the use of propane, it should be understood that ethane, isobutane, butane, and similar normally gaseous treating agents may be used in the place of, or in admixture with, propane, the treating temperatures being lowered with lighter diluents and being increased with heavier diluents.

I have found that unneutralized sulfonic acids do not respond quite like the soaps to the high temperature propane fractionation purification. However, when the sulfonic acids are reaction products obtained from residual stocks, they may be separated from lighter oils by means of propane at the high temperatures hereinabove indicated. The sulfonic acids appear to be more responsive to ethane than to the 3 and 4 carbon atom diluents, but heavy oils are likewise insoluble in ethane. It is sometimes possible, however, to separate sulfonic acids from the oil by precipitation from ethane at temperatures of the order of —100° F.

Primarily, however, my invention relates to the purification of sulfonic soaps and to the use of the high temperature propane fractionation on materials which have preferably undergone a preliminary purification as hereinabove described. This fractionation may be in a single batch treatment, it may be in a multiple batch treatment at different temperatures or propane concentrations, it may be in counter-current towers, or it may be in accordance with any other known methods of procedure. This invention should not be limited to the specific details hereinabove set forth, but the appended claims should be construed as broadly as the prior art will permit.

The term "propane" as used in the following claims is intended to cover not only the pure hydrocarbon C_3H_8, but mixtures of ethane, propane and butane.

I claim:

1. The method of purifying a sulfonic soap produced by the reaction of concentrated or fuming acid on a mineral oil, which method comprises intimately admixing an impure sulfonic soap with propane heating said mixture to a high temperature and under a pressure sufficient to maintain the propane in liquid phase, removing one phase from the other, and removing the diluent from each separated phase.

2. The method of claim 1 wherein the temperature is between 120° and 200° F.

3. The method of purifying impure oil soluble sulfonic compounds resulting from the reaction of sulfuric acid on mineral oil compositions, which method comprises heating said impure compounds with about 4 to 12 volumes of propane to a temperature of about 120° to 200° F. under a pressure sufficient to maintain the propane in liquid state whereby there is a separation of a propane-oil phase, removing said propane-oil phase from the sulphonic compounds, and removing propane from the propane-oil phase and

from the resulting sulfonic compounds, respectively.

4. The process of claim 3 wherein the sulfonic compounds undergoing treatment are components of grade B mahogany soap.

5. The method of purifying grade B mahogany soap which comprises removing therefrom those asphaltic, tarry and resinous materials which are insoluble in propane at temperatures of about 100° F., and subsequently heating it with about 4 to 12 volumes of propane at a temperature high enough to effect separation of a propane-oil solution from the mahogany soap and at a pressure high enough to maintain the propane in the liquid state, separating the propane solution from the soap layer, and removing propane from said solution and from said layer, respectively.

6. The method of claim 5 which also includes the step of removing the salt from the impure grade B mahogany soap.

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