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PROCESS OF TESTING PETROLEUM HYDROCARBONS

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The object of the invention is to provide a process of treating and inspecting petroleum hydrocarbons, and particularly gasoline and lubricating oil, whereby either substitution or adulteration can be detected.

Unscrupulous independent dealers operating filling stations dispensing one or a number of brands of gasoline not infrequently substitute, for expensive high grade gasoline, cheap low grade gasoline, in containers and pumps designed only for the storage and dispensing of such high grade gasoline. Thereby the customer is defrauded and the reputation of the refiner of the high grade gasoline is seriously impaired. In other cases a substantial percentage of low grade gasoline is added to high grade gasoline, thereby adulterating, and impairing the quality of, the high grade gasoline and defrauding the customer and damaging the reputation of the refiner as in the case of a substituted gasoline.

The object of the invention is to enable the refiner to determine, by testing the product sold at the filling station, whether there has been any such substitution or adulteration.

In our invention this object is effected by the following procedure: The standard commercial gasoline is mixed with a coloring material which, in the predetermined proportion added, is soluble in the gasoline but will not impart color thereto, but which is also soluble in, and is adapted to impart color to, another liquid which is substantially immiscible in gasoline. If, then, a quantity of the so treated gasoline is thoroughly mixed with a small quantity of said liquid, the latter, when it separates by gravity, will be colored by the coloring matter. Such procedure will demonstrate that the tested gasoline comprises, in whole or in part, the standardized gasoline so treated. In other words, such test will determine that there has been no substitution of one gasoline for another, but it will not necessarily determine that there has been no adulteration.

To test for adulteration, the coloring material must be of such character that said liquid, so colored by the coloring matter, when subjected to spectroscopic observation, will exhibit a spectrum whose characteristic will vary with the contained percentage of coloring matter. Since the percentage of coloring matter added to the treated gasoline is known, and since, in the test, a predetermined proportion of said liquid is added to a predetermined proportion of gasoline, the percentage of coloring matter in said liquid, and consequently, the character of the spectrum, is standardized, and any variation therefrom will indicate adulteration.

The process may be practiced by adding to gasoline various organic colors or dyes and by utilizing various liquids colorable by such organic colors or dyes. Examples of specific ways of carrying out the process will be given.

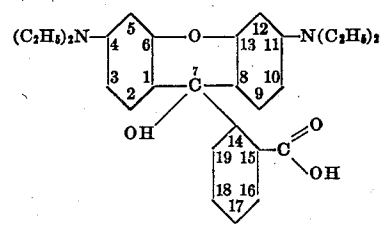
To 3,360,000 gallons (80,000 barrels) of gasoline is added one pound of 4–11 tetra ethyl diamino-7-phenyl xanthenol-15-carboxylic acid. This is the equivalent of one part by weight of the organic color to about 20,160,000 parts by weight of 62° Bé. gasoline. The proportion added may vary from one pound of color in about 1,260,000 gallons of gasoline to one pound of color in about 5,040,000 gallons of gasoline. It is preferable to first add the color to a small quantity of benzol, since in benzol it is more readily soluble in concentrated solution, and then add the treated benzol to the gasoline. The treated gasoline will not be colored by the coloring material. The coloring material is not more soluble in water than in oil and is resistant to acids and alkalis and the quality of the gasoline is therefor not affected by the treatment.

To test the treated gasoline, 5 cc. of 28% acetic acid is added to any suitable volume of gasoline up to about 90 cc., preferably about 15 cc. The mixture is thoroughly agitated. The mixture is then allowed to settle. The acetic acid separates at the bottom of the column and exhibits a pink color with yellow or orange or yellow-orange fluorescence.

The acetic acid is then subjected to spectroscopic examination, preferably in a comparison spectroscope, side by side with a standardized sample of colored acetic acid. The standardized acetic acid exhibits an absorption band of definite width and position. If the acetic acid being tested exhibits a narrower absorption band, definite proof of adulteration is afforded.

Other organic materials may be used as the coloring matter, but we prefer to use the phenyl xanthone above specified, or in place thereof 4–11 tetra methyl diamino-7-phenyl xanthenol-15-carboxylic acid, 4–11 tetra butyl diamino-7-

phenyl xanthenol-15-carboxylic acid. The preferred coloring matter has the following formula:



In claiming specifically the coloring matter having the above formula we mean to include as equivalents all operative substituted phenyl xanthones.

In place of acetic acid we may use other organic acids, such as formic acid, proprionic acid, butyric acid and oxalic acid, of proper concentration. It is posisble to use the inorganic acids, such as hydrochloric acid, sulphuric acid and nitric acid, but special care must be taken to use definite degrees of concentration.

If the process be applied to the testing of lubricating oil, the same procedure may be followed. We prefer, however, to use one pound of color to about 650,000 gallons of oil, and before testing a sample of the oil, mix the sample with an equal quantity of gasoline. It is difficult to so thoroughly mix lubricating oil with acetic acid as to effect a definite quantitative transfer of coloring matter to the acetic acid, but this can be accomplished by first mixing a definite volume of lubricating oil with a sufficient, e. g. equal, volume of gasoline, benzol, or other hydrocarbon with which the acetic acid will more readily intimately mix.

The process is applicable to a petroleum hydrocarbon to which is added a coloring matter adapted to impart a color to the commercial gasoline, since the special color added to, but not adapted to color, the gasoline will nevertheless impart a characteristic identifying color to the acid, which in turn will develop a characteristic identifying spectrum.

What we claim and desire to protect by Letters Patent is:

1. The process of standardizing a petroleum hydrocarbon and of subsequently determining the identity of a sample of petroleum hydrocarbon with the standardized product which comprises mixing with the petroleum hydrocarbon to be standardized a coloring material which is soluble therein and which in the proportion added will impart no color thereto, and mixing a sample of the hydrocarbon to be tested with an acid which is not permanently miscible with the hydrocarbon and in which the coloring material is soluble and which is adapted to have imparted to it by said coloring material a characteristic and identifying color.

2. The process of standardizing a petroleum hydrocarbon and of subsequently determining the identity of a sample of petroleum hydrocarbon with the standardized product, which comprises mixing with the petroleum hydrocarbon to be standardized a predetermined percentage of a coloring material which is soluble therein and which in the proportion added will impart no color thereto, mixing a sample of the hydrocarbon to be tested with an acid which is not permanently miscible with the hydrocarbon and in which the coloring material is soluble and which is adapted to have imparted to it by said coloring material a characteristic and identifying color, spectroscopically analyzing said acid so colored and comparing the spectrum thereof with the spectrum of a standardized sample of said acid colored by said coloring material.

3. The process of standardizing a petroleum hydrocarbon and of subsequently determining the identity of a sample of petroleum hydrocarbon with the standardized product, which comprises mixing with the petroleum hydrocarbon to be standardized a small quantity of 4–11 tetra ethyl diamino-7-phenyl xanthenol-15-carboxylic acid, said quantity being less than that required to change the color of the hydrocarbon, and mixing a sample of the hydrocarbon to be tested with an acid of predetermined concentration which is not permanently miscible with the hydrocarbon and in which said coloring material is soluble and which is adapted to have imparted to it by said coloring material a characteristic and identifying color.

4. The process of standardizing a petroleum hydrocarbon and of subsequently determining the identity of a sample of petroleum hydrocarbon with the standardized product, which comprises mixing with the petroleum hydrocarbon to be standardized a predetermined proportion of 4–11 tetra ethyl diamino-7-phenyl xanthenol-15-carboxylic acid, said quantity being less than that required to change the color of the hydrocarbon, mixing a sample of the hydrocarbon to be tested with an acid of predetermined concentration which is not permanently miscible with the hydrocarbon and in which said coloring material is soluble and which is adapted to have imparted to it by said coloring material a characteristic and identifying color, spectroscopically analyzing the acid so colored and comparing the spectrum thereof with the spectrum of a standardized sample of said acid in which a predetermined proportion of said coloring material has been dissolved.

5. The process of standardizing a petroleum hydrocarbon and of subsequently determining the identity of a sample of petroleum hydrocarbon with the standardized product, which comprises mixing with the petroleum hydrocarbon to be standardized a small quantity of 4–11 tetra ethyl diamino-7-phenyl xanthenol-15-carboxylic acid, said quantity being less than that required to change the color of the hydrocarbon, mixing a sample of the hydrocarbon to be tested with acetic acid, and separating the acetic acid and observing the color imparted thereto.

6. The process of standardizing a petroleum hydrocarbon and of subsequently determining the identity of a sample of petroleum hydrocarbon with the standardized product, which comprises mixing with the petroleum hydrocarbon to be standardized a predetermined portion of 4–11 tetra ethyl diamino-7-phenyl xanthenol-15-carboxylic acid, said quantity being less than that required to change the color of the hydrocarbon, mixing a sample of the hydrocarbon to be tested with a definite proportion of acetic acid, separating the acetic acid and comparing the spectrum thereof with the spectrum of a standardized sample of acetic acid colored with said coloring material.

7. The process of treating a hydrocarbon mixture to detect adulteration thereof comprising adding thereto a predetermined quantity of a substituted phenyl xanthone, said quantity being less than that required to change the color of the hydrocarbon and which when extracted with an acid solution of known pH value will impart

a pink color and substantially yellow fluorescence to the acid solution and render such acid solution capable of a quantitative spectroscopic analysis to determine the degree of adulteration, and then so extracting the substituted phenol xanthone.

8. The process of standardizing a hydrocarbon mixture and of subsequently determining the identity of a sample of a hydrocarbon mixture with the hydrocarbon mixture to be standardized which comprises mixing with said hydrocarbon mixture to be standardized a predetermined proportion of an indicating dye substance which in the quantity added will not change the color of said hydrocarbon mixture and which when extracted with another particular substance will react with and form with it a material having a particular identifying color and adding such particular substance to a sample of said hydrocarbon mixture and so extracting the indicating dye substance.

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