United States Patent Office.

GEORGE M. SAYBOLT, OF JERSEY CITY, NEW JERSEY, ASSIGNOR TO THE STANDARD OIL COMPANY OF NEW YORK, OF NEW YORK, N. Y.

PROCESS OF REFINING PETROLEUM DISTILLATES.

SPECIFICATION forming part of Letters Patent No. 565,040, dated August 4, 1896.

Application filed February 5, 1896. Serial No. 578,138. (No specimens.)

To all whom it may concern:

Be it known that I, GEORGE M. SAYBOLT, a citizen of the United States, residing at Jersey City, in the county of Hudson and State of New Jersey, have invented certain new and useful Improvements in the Processes of Refining Petroleum Distillates; and I do declare the following to be full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

My improved process is designed for use in the refining of the burning distillates of crude petroleum of less than .850 specific gravity, but it is applicable to the refining of the like distillates of similar hydrocarbon oils, such as shale-oil, coal-oil, and the distillates of bitumen, asphalt, &c. I shall describe it as I have practiced it in refining petroleum distillate.

As stated in another patent application, Serial No. 572,082, filed December 13, 1895, I have discovered that by treating petroleum distillate (after the usual sulfuric-acid treatment) with powdered material such as fullers' earth I am enabled to secure a perfectly refined decolorized product free from all objectionable sulfonic compounds, such as will develop on the application of heat, without the need of applying to the distillate the ordinary treatment with water and alkaline solution, and that I thus secure a much better and cheaper product than possible to be obtained from like materials by methods herestofore used.

The discovery on which the present specification is based is that if after the sulfuricacid treatment I wash the light petroleum distillate (of .850 specific gravity or less) with water, with or without treatment with an alkaline solution, and agitate the distillate with powdered fullers' earth or its equivalent, as hereinafter explained, I accomplish a result similar to that above indicated, and while the refining is not so thorough I can use less of the fullers' earth, and the fullers' earth can by washing be more easily restored, so as to be capable of repeated use in the practice of the process.

In carrying my present invention into effect I take the unrefined petroleum distillate

and subject it to the ordinary sulfuric-acid treatment, that is, I place it in a suitable vessel and add thereto the non-fuming sulfuric acid of commerce. Fuming sulfuric acid will 55 attack the distillate itself as well as the impurities, and is to be avoided as deleterious in the practice of my invention. The amount of sulfuric acid to be employed will be understood by those skilled in the art. The dis- 60 tillate and acid are then thoroughly mixed by agitation. The impurities of the oil attacked by such acid treatment are allowed to settle in the form of sludge acid and are removed. Such acid treatment is well known (5 and does not require further description. I then agitate the distillate thoroughly with water, allow the water with the impurities to settle and draw it off, and then I may agitate the distillate with a weak alkaline solution, 70 such as a solution of caustic soda, preferably of about 5° Baumé, though this is not necessary, and in some respects I deem it undesirable. Then after settling and removing the alkaline solution I add to the distillate 75 some powdered fullers' earth, and thoroughly agitate the oil therewith for about an hour, more or less, depending upon the character of the oil and the quantity of distillate under treatment. The effect of this agitation So is to remove thoroughly the objectionable sulfonic salts, which have been produced by reaction of the sulfuric acid with the oil. After such treatment, and the settling out from the oil of the pulverized fullers' earth, 85 the oil is decanted and drawn of, when it will be found to be deodorized, clear in color, completely refined, and free from objectionable compounds.

The process here employed is not a process 90 of filtering, but it is a process of agitation, only a relatively small amount of the fullers' earth being employed. The proportion of fullers' earth proper to be used depends upon the proportion of sulfuric acid originally employed. When .6 per cent. of sulfuric acid of 66° Baumé has been used, I prefer to add to the distillate 1.5 per cent. of fullers' earth, and as the percentage of acid is increased to proportionately increase the percentage of fullers' earth, though the proportions may be varied, and my invention is not limited in

scope to the employment of particular pro-

portions.

Other comminuted or crushed solid substantially non-alkaline substances, (which 5 will not dissolve in the acid-treated oil and produce permanent objectionable sulfur compounds,) such, for example, as the silicates of alumina, bauxite, alumina, clay, charcoal, either vegetable or animal, or a mixture of 10 them or any of them, may be used, though I deem fullers' earth and clay to be most suit- $\mathrm{able}.$ The second second

m I~claim1. The method hereinbefore described of 15 purifying petroleum distillate, which consists in treating the burning oil distillate of petroleum of less than .850 specific gravity, after its final distillation, with non-fuming sulfuric acid, and removing the impurities remaining 20 after the acid treatment by agitating such distillate with water and then agitating it

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with finely divided non-alkaline solid material, and thereby removing the sulfonic salts produced by reaction of the sulfuric acid with the oil.

2. The method hereinbefore described of purifying petroleum distillate, which consists in treating the burning oil distillate of petroleum of less than .850 specific gravity, after its final distillation, with non-fuming sulfuric 30 acid, and removing the impurities remaining after the acid treatment by agitating such distillates with water and then agitating it with finely-divided clay or earth, and thereby removing the sulfonic salts produced by re- 35 action of the sulfuric acid with the oil.

In testimony whereof I now affix my signature in presence of two witnesses.

Witnesses: GEORGE M. SAYBOLT.

THOMAS W. BAKEWELL,
BENJ. MARTIN, Jr.