E. A. STARKE
PRODUCING BENZENE OR ITS HOMOLOGUES FROM PETROLEUM.
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913,780.

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Witnesses:

Inventor:

Eric A. Starke
by H. F. Booth
his Attorney.
To all whom it may concern:

Be it known that I, ERIC A. STARKE, a citizen of the United States, residing at Berkeley, in the county of Alameda and State of California, have invented certain new and useful Improvements in Producing Benzene or Its Homologues from Petroleum, of which the following is a specification.

My invention relates to the treatment of petroleum, and it consists in a novel process of producing benzene or its homologues.

My process is only applicable to those petroleums the distillates of which contain the benzene nucleus, by which I mean petroleum which contains hydro-carbons having benzene or its homologues, either free or in some form of combination, such as for example "California" or "Japanese" petroleum. Though these combinations have, as yet, not been accurately determined, certain hydrogenized homologues of benzene as for example di-hydro-toluol, di-hydro-xylene, and others have been recognized; and besides these forms, benzene or its homologues exist in the free state in some oils. I have found that if the distillates of these petroleums which contain the benzene nucleus, either free or in some form of combination, are acted upon by sulfuric acid, with the aid of heat, oxidation takes place, resulting in the formation of sulfuric acid and sulfuric dioxid. This is taken advantage of in my process, which I carry out as follows:

The figure in the accompanying drawing represents an apparatus in which the hydrolytic distillation may be carried out. I take of the distillates coming from the said petroleum, 10 parts by weight, and mix therewith 3 parts by weight of concentrated sulfuric acid of, approximately, 96% H$_2$SO$_4$, and I then heat the mixture in a suitable stirring apparatus, keeping up the agitation during the heating. The distillate fractions best available for my process, are those which pass over, say from 260 degrees to 500 degrees F., and which constitute the illuminating oils; for the fractions which contain the benzene nucleus are those boiling below 500 degrees F. The temperature to which the mixture is heated depends upon the boiling point of the particular distillate being treated. In any case, it is below the boiling point of said distillate. For example, if the boiling point of the distillate is less than 300 F., then the mixture is heated to a temperature of about 150 F.; and if the boiling point is above 300 F., then a good working temperature for the mixture is approximately, 260 F. I may state, incidentally, that if 260 F. is exceeded, excessive frothing takes place, which hinders the operation, and causes loss by decomposition of sulfuric acids already formed. The length of time during which the mixture is thus heated under agitation is determined by the time when the evolution of sulfur dioxide ceases or nearly ceases. In practice this is about one hour. When the reaction is over, the mass is allowed to settle for about 24 hours, when it will be found that it has separated into two layers, the lower being a tarry liquid containing the sulfuric acids and the upper being petroleum. The tarry liquid is then drawn off and is subjected to distillation to recover the benzene. This distillation may be carried out in any suitable form, as, for example, in the apparatus here shown. The tarry liquid is pumped into a retort 1, and is here treated with superheated steam, at a temperature of about 500 F. The steam enters the retort through a pipe 2 and is discharged into the tarry liquid through a heavy leaden pipe 3 at the bottom of the retort. The action of the steam begins at once, and temperature of the mass gradually rises until it exceeds 300 F., during which time the benzene will have been distilled over and condensed in a lead pipe condenser 4. It may then be washed with alkali and subjected to fractional distillation in a column still. After the benzene has been distilled, the contents of the retort is run into a settler 5 for proper treatment to recover the sulfuric acid for use again, and the acid tar, which may be used as a fuel. During the distillation of the benzene, a certain amount of sulfuric acid is reduced to SO$_3$. This may be caught by a gas-trap 6 and conducted to condensing towers or chambers.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

The process of producing benzene or its homologues which consists in mixing a distillate of petroleum which contains a benzene nucleus, with concentrated sulfuric acid; then heating the mixture during agita-
tion until the evolution of sulfur dioxid has
about ceased; then allowing the mass to set-
tle until the sulfonic acids have separated
from the petroleum; and then recovering
the benzene from said sulfonic acids by dis-
tillation.
In testimony whereof I have signed my
name to this specification in the presence of
two subscribing witnesses.

ERIC A. STARKE.

Witnesses:
N. A. ACKER,
L. E. WILKINS.