

H. J. FENNER & F. VERSMANN.

Improvement in the Manufacture of Anthracene.

No. 130,909.

Patented Aug. 27, 1872.

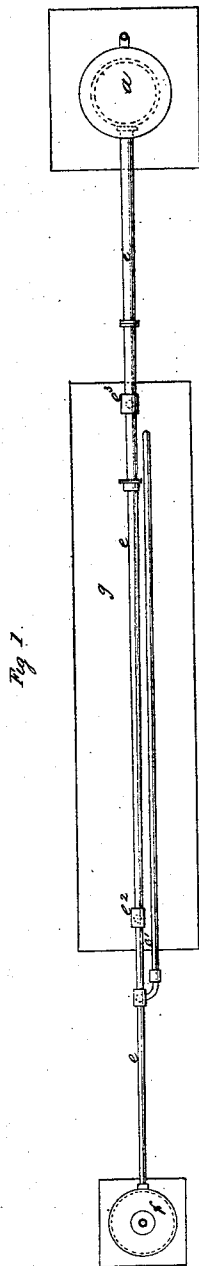


Fig. 1.

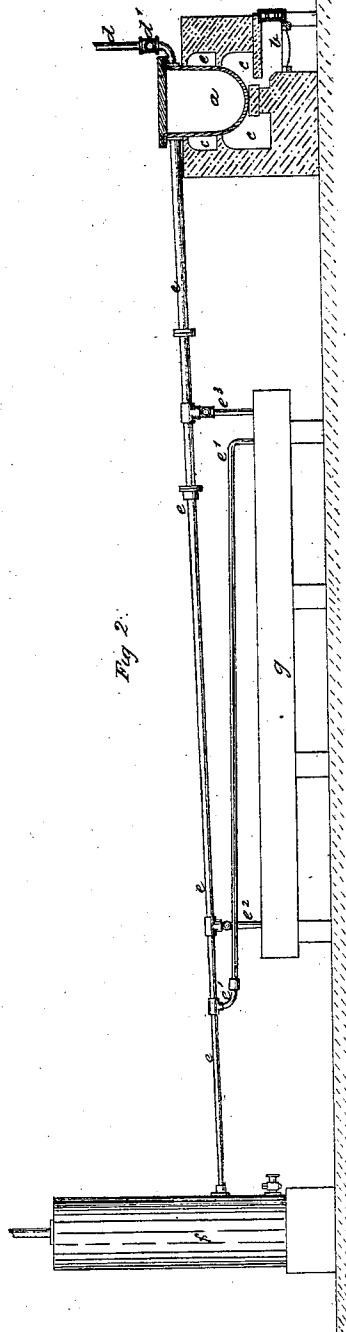


Fig. 2.

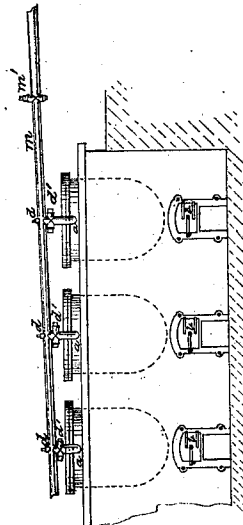


Fig. 3.

William Brooks.
Alfred George Bartlett.

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Frederick Versmann

UNITED STATES PATENT OFFICE.

HENRY JAMES FENNER, OF GREENWICH, AND FREDERICK VERSMANN, OF LONDON, ENGLAND.

IMPROVEMENT IN THE MANUFACTURE OF ANTHRACENE.

Specification forming part of Letters Patent No. 130,909, dated August 27, 1872.

To all to whom it may concern:

Be it known that we, HENRY JAMES FENNER, of Greenwich, in the county of Kent, England, and FREDERICK VERSMANN, of the city of London, England, have invented Improvements in Obtaining Anthracene; and we, HENRY JAMES FENNER and FREDERICK VERSMANN, do hereby declare that the following is a full and exact description thereof, reference being had to the accompanying drawing and to the letters of reference marked thereon—that is to say—

The object of the invention is to obtain anthracene from coal-tar pitch by the process of manufacture hereinafter described.

We are aware of experimental attempts having already been made to obtain anthracene from pitch, but, so far as we have been able to ascertain, the attempts to do so have not been practically successful, and have not proved of commercial value. The adoption of the process of manufacture invented by us and employed as hereinafter described will enable the products of pitch to be obtained in regulated order, and anthracene will be produced with the requisite certainty at ascertained temperatures and known stages in the process, and when so produced will be found of a very superior character compared with any that may have been produced in the experimental attempts above referred to. The anthracene can be obtained by our process in a comparatively pure state with only a small proportion, if any, of other hydrocarbons, such as naphthaline or chrysene mixed therewith. In carrying out our improvements we either operate upon the pitch after it has been obtained by the ordinary process, and as it is met with in the market, or we combine, as hereinafter described, the operation of separating anthracene with the operation of distilling the oils, and the consequent production of pitch from the coal-tar. In any case, in order to effect our object, we employ progressively-increasing temperatures, and eventually a very high temperature—say about 600° or 800° Fahrenheit by heat externally applied. We find that in conducting our process of distilling pitch we obtain, at a temperature of about 400° Fahrenheit, an oil comparatively free from solid hydrocarbons. We continue

to raise the temperature to from 500° to 600°, when the oil becomes richer in solid matters, and is found to contain naphthaline in excess, with a small proportion of anthracene. The latter increases as higher temperatures are employed, the production of anthracene becoming abundantly in excess at from 600° to 700°. Above these temperatures we find that the production of anthracene decreases and chrysene will be chiefly obtained. In treating the pitch which has been obtained separately we usually reduce it to small convenient sizes, and then mix it with oil or residuum obtained by a previous distillation of pitch or with other dry absorbent carbonaceous matter. We do not, however, confine ourselves to the use of such mixture. The effect of using the mixture is, that the pitch, when heated in the retort or other vessel, absorbs the heat gradually from the commencement without rising or frothing, whereas pitch alone rises, and, if not carefully watched, has a tendency to froth and choke the delivery-passage as long as moisture is left in the pitch. This we have found to be a necessary and important part of the process, when treating pitch that has stood for some time exposed to the air or moisture. The pitch thus mixed with the oil or absorbent carbonaceous matter is put into a still, retort, or other suitable vessel, which we make of cast-iron, in order to enable it properly to stand the high heats which we use. In our process of manufacture, the heat is applied externally, and is progressively raised to and then continued at a very high temperature—say from 600° to 700° Fahrenheit—as long as any liquid or gaseous products pass over. The oily liquid that passes over at these high temperatures is found to be very rich in anthracene, Naphthaline and chrysene are present in comparatively small proportions. The vapors passing over are then condensed by the cooling influence of the atmosphere acting on the conducting-pipes, and the products obtained by the condensation are run into a suitable tank where they are allowed to repose or precipitate in order to complete the settling of the products. The condensation is thus effected by the action of the atmosphere on the conducting-pipes, and we do not employ the ordinary worm or coil in tanks of cold water

for that purpose, as the liquid becomes thick and the anthracene solidifies, or tends to solidify, before it reaches the end of the worm. We find it advantageous to assist the distillation by keeping up a partial vacuum in the still or retort by which the products of distillation will pass away more quickly and at a lower temperature than under ordinary pressure. This partial vacuum may be kept up by means of an exhauster or blower, or other suitable means.

Figure 1 represents a plan view, and Fig. 2 a side view, partly in section, of apparatus, such as we have found to answer well in carrying into effect our method of working.

a represents a cast-iron vessel, in which the coal-tar pitch is placed for distillation by the heat of a fire in the furnace *b*. *cc* are fluespaces by which the heat from the fire-place *b* may be retained as long as possible in contact with the surface of the vessel *a*. *d* is a pipe with a stop-cock, *d'*, by which pitch may be supplied to this vessel *a*, and *e* is a pipe by which the anthracene and matters combined therewith obtained in the distillation, are conducted in the form of vapor from the vessel *a* to a condensing-chamber, *f*; but in the passage of the vapors along the pipe *e* they become cooled by the action on that pipe *e* of the surrounding atmosphere and the products of condensation thence flow by one or other of the branch pipes *e¹ e² e³* to the receiving-tank *g*. The oil collected in the vessels or tanks, *f* and *g*, will be drawn off therefrom through suitable taps or outlets. When the product distilled at about 400° begins to issue from *a*, it passes along *e* and at first reaches to and—part being principally non-condensed vapor or gas—enters *f* as the temperature is raised, and anthracene begins to pass over. The richer oily product containing anthracene passes along *e¹* into the chamber *g*. The temperature being still further raised, or continued at a high standard, such as from 600° to 700°, the oily product distilled becomes more charged with anthracene. The tap in the branch-pipe *e²* is then opened, and, if necessary, owing to the increasing density of the oil, the tap in the tube *e³* is opened, so as to provide, as required, a short and ready passage for the distilled product into the receiving-vessel *g*.

When combining the production of anthracene according to our invention with the distillation of heavy oils from coal-tar, thus making one continuous process of the two operations—a method of manufacture which we have found to give beneficial results that have not hitherto been obtained—we proceed as follows: We distil the heavy oils from coal-tar, in the usual way, in a wrought-iron still or boiler, *n*, such as has been hitherto used for such distilling process, and we connect such vessel with a set of cast-iron stills or retorts, or other suitable vessels, *a*, such as have been already referred to, taking care, however, that they may be capable of withstanding the high heat

which we use in our process. The connection thereto is made by means of a pipe or pipes provided with stop-cocks, or other simple regulating means. As soon as the heavy oils have been worked off in the wrought-iron vessel, then a residuum of pitch is left, and without allowing the pitch to cool, it is at once run off into a set of vessels, *a*, which have been previously heated, and in which the distillation is at once proceeded with until the liquid or gaseous products desired have passed over to the condenser, aided in some cases by the use of a partial vacuum, such as above described.

This continuous treatment of the pitch constitutes a novel feature in the process of manufacture invented by us, and will be found economical in regard to cost and beneficial in result.

Fig. 3 represents, by a sectional view, apparatus arranged to operate according to this method of working. *m* is a pipe by which liquid pitch may be conducted from the boiler, which is not shown in the drawing, to branches *d* with taps *d'*, by which to supply vessels *a*, such as already referred to. *m'* is a tap in the pipe *m* to regulate or to stop the supply as required.

The vapor generated in these vessels *a* will be conducted to a condensing-chamber or condensing-chambers *f* and *g*, or to other suitable condensing apparatus for the condensation, as stated. It would be possible after distilling the oils to continue to treat the pitch residuum in the same vessel by applying higher temperatures; but we have found that the cost of carrying on such a mode of manufacture would be so great, owing to the destruction of the wrought-iron apparatus, which is found suitable for the distillation of the oils from the tar, above referred to, that we do not recommend the continued operation to be carried on in the same vessel; but we recommend it to be carried on in separate vessels, as above described. The employment of heat internally applied, either by means of steam or hot air, is not advantageous and forms no part of our invention. We find, as has been already mentioned, that the anthracene obtained from coal-tar pitch by our process of distillation is at the comparatively lower temperatures, mixed more or less with naphthaline, and at the higher temperatures is more or less mixed with chrysene. When it is desired to obtain the anthracene as pure as possible, this is effected by employing a mean of the temperatures between that which gives excess of naphthaline and that which gives excess of chrysene. By testing the nature of the distilled product from time to time, it may soon be found, by experience, in what way and to what extent the temperatures may be varied and regulated. The anthracene distilled from pitch by our process, as above described, will be found to be mixed with a viscid heavy oil, from which it may be readily separated by filtration and washing with oils of less specific gravity, or

petroleum or other spirit, or by other convenient modes of separation.

Having thus described our invention, and the means which we adopt in carrying the same into effect, we would have it understood that we do not confine ourselves to the apparatus described, as such may be varied without departing from the peculiar character of our invention; but we would have it understood that what we claim is—

1. The process, above described, of obtaining anthracene from coal-tar pitch, by the employment of regulated high temperatures externally applied, and substantially as described.

2. Also, we claim the use of a partial vacuum to assist the passage of the products of distillation, as above described.

3. Also, we claim the combination of the manufacture of anthracene from coal-tar pitch with the distillation of oils from coal-tar by a continuous process, substantially as described.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

HENRY JAMES FENNER.
FREDERICK VERSMANN.

Witnesses to HENRY JAMES FENNER:

N. F. FENNER,
E. K. MITTING.

Witnesses to FREDERICK VERSMANN:

WM. SCOTT,
LOUIS LUTZ.