(Specimens.)

2 Sheets—Sheet 1.

F. M. F. CAZIN.

METHOD OF REFINING AND DEODORIZING COAL OIL OR PETROLEUM.

No. 400.633. Patented Apr. 2, 1889.

Fig. 1.

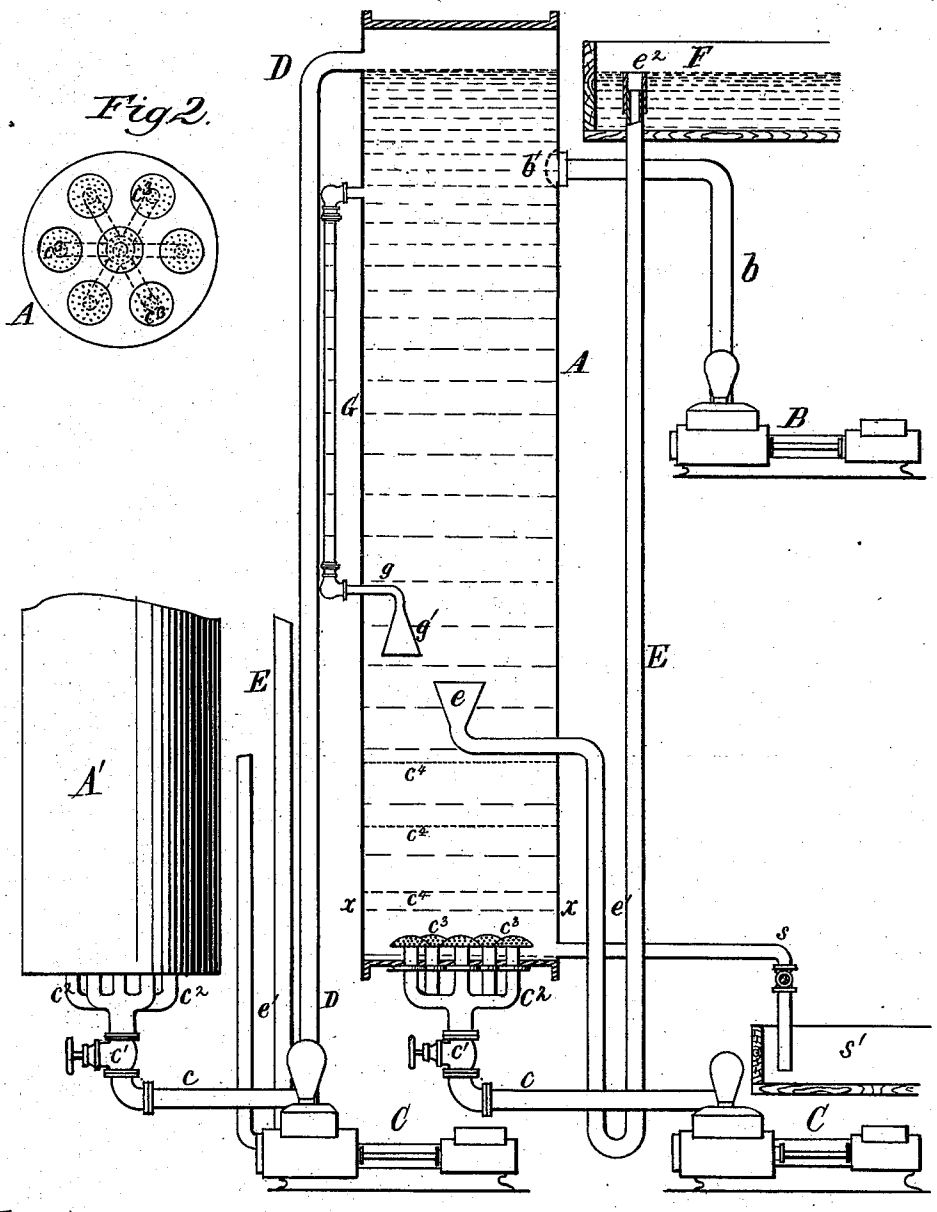


Fig. 2.

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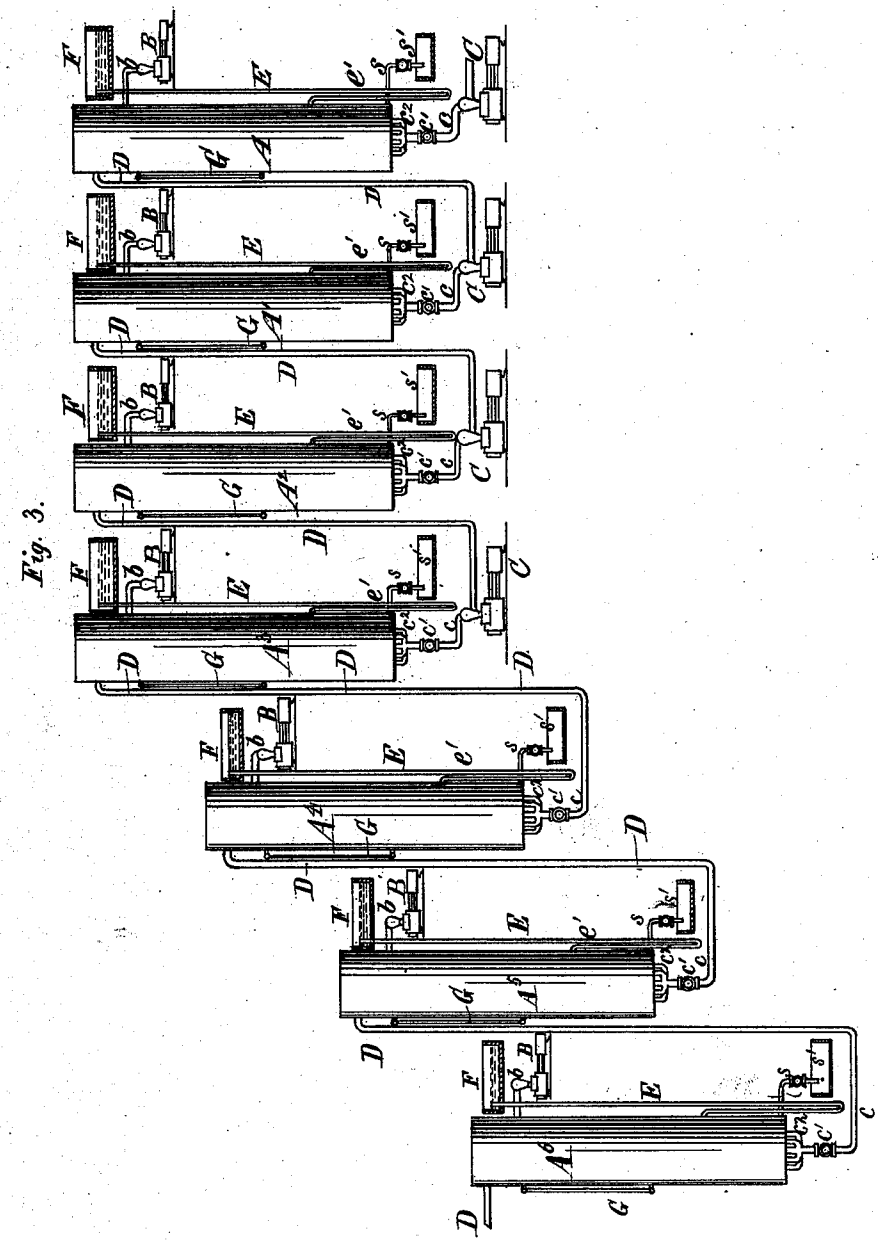
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Fig. 3.



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UNITED STATES PATENT OFFICE.

FRANCIS M. F. CAZIN, OF NEW YORK, N. Y.

METHOD OF REFINING AND DEODORIZING COAL-OIL OR PETROLEUM.

SPECIFICATION forming part of Letters Patent No. 400,633, dated April 2, 1889.

Application filed September 3, 1887. Serial No. 248,756. (Specimens.)

To all whom it may concern:

Be it known that I, FRANCIS M. F. CAZIN, of the city and county of New York, in the State of New York, have invented a new and useful Improvement in the Method of Deodorizing Coal-Oil or Petroleum, of which the following is a specification.

Many of the coal-oils or petroleums obtained from recently-worked wells, particularly those in the State of Ohio, and which are called, when refined, "Lima oils," contain, in addition to the simple hydrocarbon compounds, other hydrocarbon compounds which also contain sulphur. These sulphur-containing compounds have, almost without exception, a pungent alliaceous odor, and as their boiling temperature, with only very few exceptions, lies within the same limits of temperature under which distillation of coal-oil is at present practiced, they are not eliminated, but pass over into the refined oil intended for market. Not only do these sulphur compounds in the refined oil impart a very disagreeable odor thereto, but when burned in lamps sulphurous acids (and under favorable circumstances sulphuric acid) are produced, increasing the intolerable odor, and under given circumstances, especially when the flame is as is termed "reducing" for want of sufficient oxygen, they form an incrustation upon the wick and a deposit on the glass chimney. Hence the Ohio refined oil cannot in quality compete with Pennsylvania oil, and therefore does not find so ready a market.

The object of my invention is to remove the sulphur compounds from the refined oil, and thereby increase its market value.

The invention consists in the improvement in the method of refining and deodorizing oil, consisting in subjecting the oil in a state of fine division or diffusion to intimate contact with water or a watery solution.

In carrying out my method it becomes necessary to employ a series of bodies of watery solutions containing different chemicals through which the oil, always in a state of fine division or diffusion, is passed, and by each of which certain impurities contained in the oil are eliminated.

In carrying out my method I prefer to supply the fresh water or solution near the top of the body through which the oil is passed, and I permit the overflow of oil from the top of the body and also the discharge of water from a point near the bottom of the body and its overflow at about the level of the oil overflow. Simultaneously with the passage of the oil in a state of fine diffusion upward through a body of water or watery solution I provide for the discharge from the bottom of the body of the precipitant deposited from the oil.

In carrying out my invention I may advantageously employ an apparatus which forms the subject of my application for Letters Patent, Serial No. 248,100, filed August 29, 1887, and in the accompanying drawings I have illustrated such an apparatus.

In the drawings, Figure 1 is a sectional elevation of one vessel with its appurtenances and a partial elevation of a second vessel in the series. Fig. 2 is a horizontal section on about the plane indicated by the dotted line $x\,x$, Fig. 1; and Fig. 3 is an elevation, upon a smaller scale, of the entire series of vessels for carrying out my invention.

Similar letters of reference designate corresponding parts in all the figures.

My apparatus comprises a series of substantially-similar vessels, which may be made of boiler-iron and which are of a very considerable height proportionate to their diameter. I have here represented seven vessels, respectively designated by the letters A A′ A^2 A^3 A^4 A^5 A^6. Each vessel is provided with a water-inlet pipe, *b*, leading from a pump, B, or an overhead tank, and each vessel is supplied with oil through a pipe, *c*, in which is a cock or valve, *c′*, and which terminates in a number of branches, c^2, which communicate with the bottom of the vessel, and are prolonged upward slightly within the vessel, as shown in Fig. 1. The oil-supply pipe *c* from the first vessel, A, leads from a pump, C, and the pumps C are also employed between several of the succeeding vessels, although each pump after the first receives its oil from an overflow-pipe, D, leading from near the top of the preceding vessel.

Where pumps C are employed for maintaining uniform supplies of oil to the several vessels, they may be arranged upon substantially the same level; but I may arrange all

of the vessels in the series one below another, as represented at the left hand of Fig. 3, in which case no pumps C are necessary, and the oil-supply pipe of each vessel forms simply a continuation of the overflow-pipe D, leading from the preceding vessel.

It is necessary to produce a fine division or diffusion of the entering oil before it passes upward through the water, and to this end I provide each of the upwardly-prolonged branches c^2 with a rose-head or distributer, c^3, and I also provide at a slight distance above the oil-inlet screens or sieves c^4, which extend transversely across the vessel, and which may be of perforated or reticulated material. These combined means effect with certainty the most minute division or diffusion of the entering oil, and therefore bring it into most intimate contact with the water. The water entering through the pipe *b* may be finally distributed by means of a rose-head or distributer, *b'*.

In connection with each vessel I employ an overflow-pipe, E, which communicates with the vessel between its ends, and which from its point of connection with the vessel is carried first downward and then upward, so as to form a downwardly-extending loop or leg, *e'*, and at its top it communicates with an overflow trough or tank, F.

The overflow-pipe E may have a funnel-shaped mouth, *e*, at its end which is within the vessel, and at its outer end it may have an adjustable sleeve or section, e^2, for controlling the overflow-level. The overflow-pipe E should be so proportioned or adjusted as to be about on a level with the oil-overflow pipe D, so that the water may be carried off through the pipe E, while the oil will pass from each vessel through the pipe D, and the sleeve or adjustable section e^2 provides for such ready adjustment.

It is important to have the downwardly-extending leg or loop *e'* in the overflow-pipe E, because then globules of oil which may be carried down with the water have an opportunity of escaping from the water back into the vessel by its lesser specific gravity, and are not necessarily carried with the water to the overflow tank or trough F.

I also employ a glass gage, G, on the exterior of each vessel, and which has its lower limb, *g*, provided within the vessel with a funnel-shaped mouth, *g'*, inverted and serving to catch some of the globules or drops of oil rising in the vessel, so that they will be deflected through the gage-glass G and brought to the view of the attendant. I likewise employ at the bottom of each vessel an escape-pipe, *s*, which serves for the removal of any precipitant in the vessel to a tank or receptacle, *s'*.

To the several vessels of the series there may be supplied clear water or watery solutions of such chemicals as will be effective in eliminating the objectionable compounds from the oil. For example, the first vessel, A, may contain clear water; the second, A', may contain an alkaline solution; the third, A^2, may contain a solution of a salt of a heavy metal—such, for instance, as acetate of lead; the fourth may contain water with chloride or oxide of copper, mercury, or other heavy metal finely divided and in a state of suspension therein, but this step in the process may be omitted; the fifth, A^4, may contain a diluted solution of sulphuric acid; the sixth, A^5, may contain an alkaline solution, and the seventh, A^6, may contain clear water.

It will be observed that in each vessel the intimate contact of the oil and water is prolonged by the upward flow of the oil being opposed to the downward movement or circulation of the water.

It will be understood that although I have above mentioned those chemicals which are useful in separating a certain line of impurities from the oil, such other chemicals may be employed as will be effective in separating other and different impurities.

The several vessels of the series being supposed to contain solutions, as above described, the operation and its effects will be briefly described, as follows: During the passage of the oil through the first vessel, A, such of the sulphur-containing compounds as are soluble in water—as, for instance, ethyl sulphate—will be dissolved in the water and carried off thereby, and such of the compounds—as, for instance, dimethylic sulphate—as are decomposed by water will have their products of decomposition carried off by water. Such of the compounds as have a higher density than water—as, for instance, methyl bisulphide, ethyl sulphites, and methyl sulphonic acid—and are unable to ascend against a descending column of water will collect on the bottom of the vessel A, and when sufficiently accumulated are drawn off through the pipes *s*. During the passage of the coal-oil through the alkaline solution in the second vessel, A', such objectionable compounds as are essentially acid in character, as methyl sulphate, and which have escaped the previous water, are retained. Such of the specified compounds as being of less density than water and pass off with the coal-oil and which also resist absorption by alkaline solution, and which, in consequence, have passed through the vessels A and A' of the series—as, for instance, such compounds as methyl mercaptan—are absorbed while passing through the solution of acetate of lead or other salt of a heavy metal in the vessel A^2 and form a new compound with the lead, if lead be used, being a yellow precipitate and a color of as high value as white lead. This precipitate collects on the bottom of the vessel A^2, and may be drawn off at intervals through the pipe *s;* and such of the objectionable compounds—as, for instance mercaptan—being of as low density as coal-oil, and resisting the action of both alkali and acetate of lead, should lead be used, are, when brought into contact with oxides or

chlorides of heavy metals, as copper or mercury, when passing through the fourth vessel, A^3, absorbed and obtained as a precipitate or otherwise in the water, and in the fifth vessel, A^4, other compounds, which are dissolved by diluted acids, are separated from the oil and carried off in the watery solution; and in the sixth vessel the remaining acid is neutralized, and in the seventh vessel the ultimate cleansing of the refined and deodorized oil by clear water takes place.

What I claim as my invention is—

The process of deodorizing oil, consisting in first passing the same in a finely-divided state through water; second, passing the same through an alkaline solution; third, passing the same through a solution of a salt of a heavy metal; fourth, passing the same through a solution of sulphuric acid; fifth, passing the same through an alkaline solution and ultimately washing the same, substantially as herein described.

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