

(No Model.)

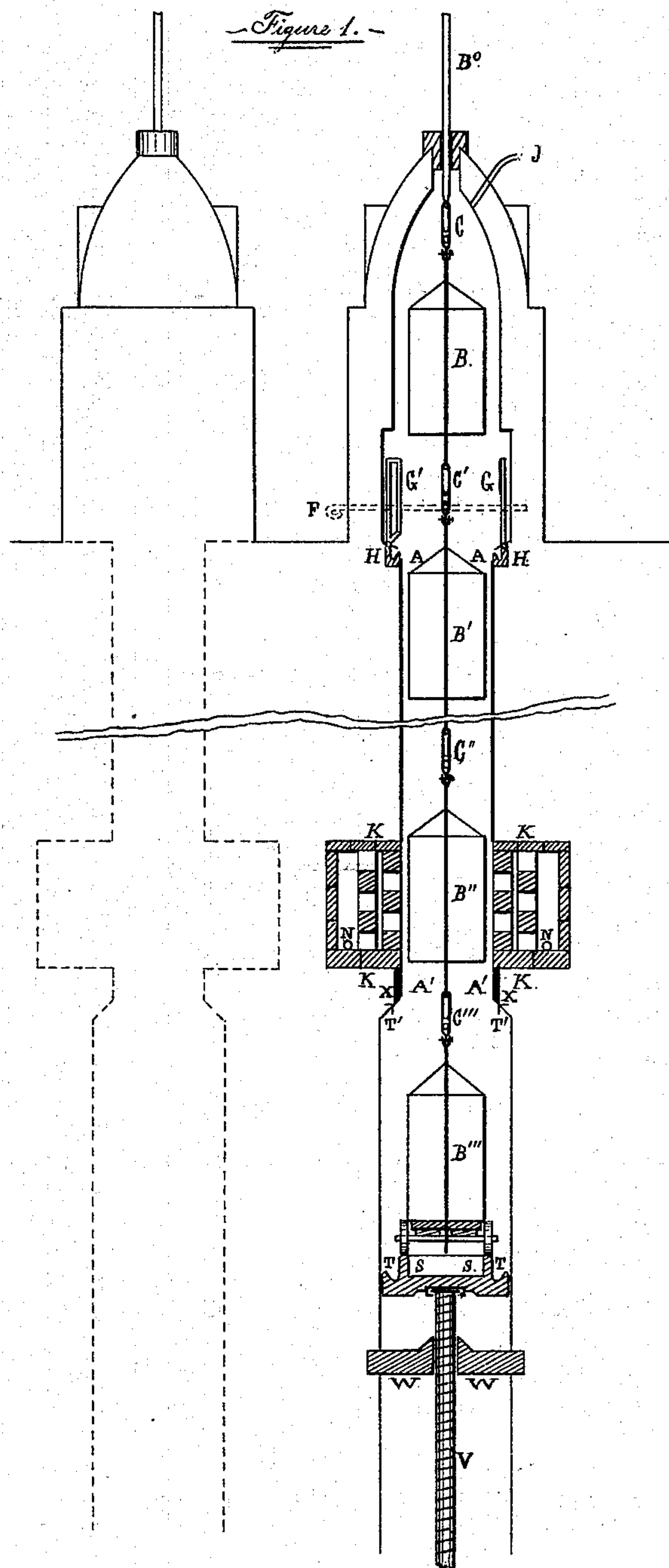
2 Sheets—Sheet 1.

H. WURTZ.

APPARATUS FOR DRY DESTRUCTIVE DISTILLATION.

No. 261,893.

Patented Aug. 1, 1882.



WITNESSES:

Walter S. Pierce
Samuel Woodman

INVENTOR

Henry Wurtz

(No Model.)

2 Sheets—Sheet 2.

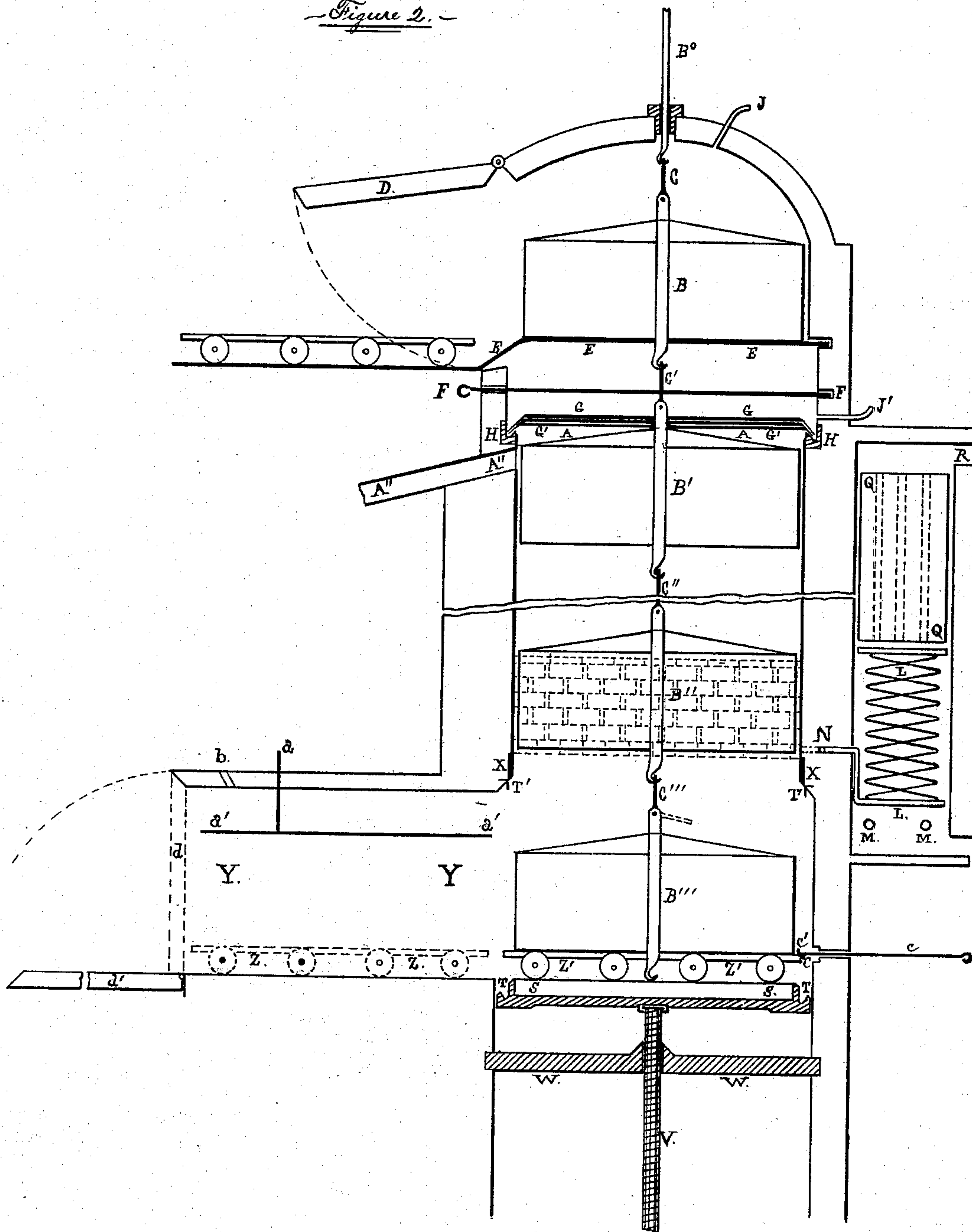
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Figure 2.



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HENRY WURTZ, OF NEW YORK, N. Y., ASSIGNOR, BY MESNE ASSIGNMENTS,
TO THE UNION CHEMICAL MANUFACTURING COMPANY, OF SAME PLACE.

APPARATUS FOR DRY DESTRUCTIVE DISTILLATION.

SPECIFICATION forming part of Letters Patent No. 261,893, dated August 1, 1882.

Application filed August 26, 1881. (No model.)

To all whom it may concern:

Be it known that I, HENRY WURTZ, a citizen of the United States, residing in New York, in the county of New York and State of New York, have invented certain new and useful improvements in apparatus for obtaining products of dry destructive distillation from solid matters; and I do hereby declare that the following is a full, clear, and exact description and specification thereof.

This invention is especially applicable to procuring from such matters the maximum proportion they are susceptible of yielding of primary distilled products of liquid nature—such as paraffine-oils, (with solid paraffine,) benzoles, phenols, and ammoniacal solutions—with the minimum proportion of naphthaline and other products of secondary decomposition or dissociation.

The invention consists, first, in the use of a vertical fire-brick-lined structure or distilling-tower of oblong horizontal section, through which is made to descend successively, by continuous steps, a series of iron boxes or cases, of similar oblong form, charged with the materials to be distilled, which cases are suspended constantly, or during the whole time, from the upper part of the tower, being linked together in the manner of a chain, with certain equal interspaces between them, by link-work of special construction, in such manner as to bring each case at each such downward step under the influence of a higher heat, until the focus of highest temperature is reached just above the sole of the working-section or distilling-chamber of the tower; second, in so constructing the sole of the distilling-chamber of this tower as to be movable, by suitable means, downward from its seat to the level of the floor of a lower lateral chamber, called a "cooling-chamber," into which the case containing the incandescent residuum of the final stage of the distillation may be removed after being also lowered, the movable sole being then elevated again to its seat, with which it is made to form a gas-tight junction by means of a liquid seal or otherwise; third, in so constructing, as hereinafter described in detail, the hook and link connections between the iron boxes or cases of the chain that the lowest member of the chain

will unlink itself on slightly elevating again the movable sole, together with the case of coke resting thereon, after the two shall have been lowered to the level of the cooling-chamber; fourth, in the attachment to or superposition upon the upper end of the distilling-chamber of another chamber to serve as a vestibule or antechamber, through which the newly-charged boxes or cases of material are introduced into the distilling-tower, this antechamber being provided also, as hereinafter explained in detail, with hinged doors or valves which close gas-tight between it and the distilling-chamber below, both before opening the door of the antechamber to introduce a new charge and after lowering the said charge into the distilling-chamber; fifth, in combining with the lower part of the vertical distilling-chamber lateral recesses in its two broader sides, having gas-tight linings or casings of iron plate, in which bricks of refractory material are arranged cellularly next to the material to be distilled, highly-heated gas, which serves as the source or vehicle of heat for the distillation, being introduced behind this cellular brick-work, between the same and the gas-tight casing, this combination serving, among other purposes, that of distributing the heated gas uniformly, the bricks themselves becoming heated at the same time and aiding in imparting heat by radiation to the materials under distillation, while the current of gas introduced serves also to remove the primary products of distillation at once away from the focus of the greatest heat, thus preventing secondary dissociation with gasfaction of these products and formation of naphthaline, &c.; sixth, in the prevention of explosions in inclosed spaces containing air which are to be put into communication with spaces containing hot gas—as in the case of the antechamber over the distilling-chamber and that of the lateral cooling-chamber—the attachment to such space of pipes at the top and bottom for displacing such air with a current of a gas lighter than air forced in at the top pipe, the air being thus forced out through the bottom pipe before opening communication with the hot-gas chamber.

In the drawings, Figure 1 presents a front

view of a tier of these distilling-towers, one tower being represented in section through a central plane. Fig. 2 presents a sectional view of one of the distilling-towers with its attachments, from front to rear, through a central plane.

A A A' A', Figs. 1 and 2, represent the interior of the working portion or distilling-chamber of the tower, containing, as here represented, but two, B' B'', of the iron cases holding the material to be distilled, although in practice three, or even more, such receptacles may be thus included within the working portion of the tower, the latter being vertically prolonged in proportion. The internal horizontal section of the tower is a rectangular parallelogram greatly elongated from front to rear, and the cases or boxes have a corresponding shape, leaving a space of an inch or more between their sides and ends and the internal walls of the tower. These internal walls may be lined for some distance above the place of introduction of the heated gases with removable sheets of rolled plate-iron to prevent carbonaceous deposits from attaching themselves to the fire-brick walls, and to facilitate the ready removal of such incrustations when thick enough to constitute obstructions, the incrustated plates being replaced very easily by clean ones and the incrustations got rid of by bending the plates. The fire-brick lining of the distilling-chamber should be accurately set to prevent leakage of gas; but defects of this kind, if not too great, mend themselves through the carbonization of the heavier hydrocarbons, which soon closes up pores and fissures. This does not apply, however, to the cellular chambers attached to the lower part of the distilling-chamber, as explained below, for distributing the gaseous-heat vehicle. As this latter gas should contain no hydrocarbons or vapors which deposit carbon by dissociation, and as it would therefore percolate readily through porous brick walls, this part of the apparatus must be made imperforate by a casing of iron plates joined gas tight around these cellular chambers.

The cases or receptacles for containing the materials to be distilled (marked B B' B'' B''' in Figs. 1 and 2,) may vary in construction and character, to some extent, with variations of the materials. When cannels, shales, bones, or other matters are operated on which do not soften, flow, or swell to any extent during the early stages of their distillation, they may with advantage be a sort of iron basket, of either cast or rolled iron, having extensively-perforated walls, the perforations of which may, indeed, be greater in aggregate area than the continuous surface; but when the material is caking in its character these walls must be wholly continuous, so as to confine the soft dilating mass. In this latter case these receptacles are boxes, rather than baskets, the material of their walls being either cast or rolled plates. A frame-work of iron rods may be so constructed as to hold together removable sections of

such plates, so that if a section, under prolonged action of high heats, becomes damaged or defective, it may be readily removed and replaced. In operating on non-caking materials it may be found economical, in the long run, to employ for the walls of the receptacles sections of some refractory non-metallic material suitably perforated, such as soapstone, pyrophyllite, or a fire-clay, porcelain, asbestos, or black-lead composition. Such can scarcely be employed, however, with caking coals, for these, iron plates from which adherent carbon may be detached without injury being advisable.

In Figs. 1 and 2, C C' C'' C''' indicate the link-work connecting the boxes or cases into a constantly-suspended chain, such chain being strengthened and conjoined throughout by long flat cast-iron links passing through the center of each box or case, as shown in the drawings, and kept rigidly in position by being fastened both to the bottom plate of the box and to a cross-piece at the top. This long flat link terminates below the box in a strong hook cast in one piece with the link, while above the box it has hinged to it a shorter flat link which is capable of swinging only in the plane of the curvature of the hook interlinked with it above. This shorter link has in it two apertures or eyes, as shown in Fig. 1, the lower eye being of circular form and provided for the insertion of the rod F F, as explained farther on, while the upper eye, for the insertion of the hook of the case above, is vertically elongated to a considerable extent. It results from this construction, by reason of the lateral swing of the shorter link and the rigidity of the hook, that on a slight elevation of any one of these boxes or cases the inner slope of the shank of the hook will force the link to tilt over and fall off, breaking the chain at such point, as is shown in Fig. 2 at C'''. If greater strength be desired, the long flat link passing through and forming part of the box or case may be made broad enough to allow the hook and link connections between the cases to be doubled in number, or even tripled, if great weight is to be sustained. The hook C, Fig. 2, to which the topmost case, B, is hung on its introduction into the antechamber or vestibule, is attached to the lower end of a cylindrical steel rod, B^o, which slides vertically, like a piston-rod, through a stuffing-box at the apex of the antechamber. The door D, which is packed gas-tight with rubber, or with a porous packing saturated with tar or glycerine, closes the antechamber after the introduction of B and its connection at C and C'.

E E E indicate a movable pair of rails or a platform placed so as to support the case B while it is being hooked fast to C, after which said platform is removed out of the way.

F F, Fig. 2, is a strong steel rod used to support B, with the rest of the chain below, after B has been lowered to the position B' and inclosed in the distilling-chamber while it is being detached from C, to enable the vertically-sliding rod B^o to be elevated again and admit

of the introduction of another charged case. In Fig. 1 this horizontal rod F F is represented in dotted lines as occupying a position at right angles to that in Fig. 2—a construction which may be resorted to when materials or charges of great weight are operated on, as F F being here very much shorter can bear a greater weight in proportion. In this case it is obvious that the connecting-links C C' C'' C''' must have positions at right angles with those figured.

G G', Figs. 1 and 2, indicate the doors or valves, which close gas-tight between the antechamber and the distilling-chamber, and which are opened to allow of the descent of the charged boxes or cases into the latter. One of the several functions essential to the system, fulfilled by the intervals or interspaces between the several cases composing the vertical chain, is to afford room for the opening and shutting of these doors in these said interspaces. In Fig. 1 these doors are represented as open and in Fig. 2 as closed. G'—the door which closes first—is hollow, and may have circulating through it a current of cold water while closed to prevent heating of its upper surface. It may also be packed with non-conducting material. G, which laps over on G', is faced with a thick glycerine-soaked porous material to form a gas-tight joint across the top. The joint all around the edges of these doors may be made gas-tight by being formed of a continuous channel of fusible metal, H H, Figs. 1 and 2, into which the down-turned edges dip, this channel being formed in an iron casting which surrounds and forms the upper rim of the distilling-chamber. The alloy used in this channel may be of two parts tin and one of lead, which melts at about 360° Fahrenheit. If it be considered advisable to use metal of greater fusibility, bismuth may be added. An alloy containing forty or fifty per cent. of bismuth will melt at a little above the boiling-point of water. Each of the doors G G' may have a projecting flap of thin elastic metal lined with felt soaked in glycerine, these two flaps being so constructed and adjusted that when the doors are shut they meet with some slight pressure, so as to clasp and form a gas-tight junction around the upper end of the flat link that passes centrally through B'. In these two sections these flaps are not represented. These doors may be opened and shut by means of two crank-handles altogether outside the apparatus, so attached as to turn round axially two rods which operate as hinges. The rod which turns the lower door may be hollow and have a current of water circulating through it and into and out of the hollow interior, as before stated.

J, Fig. 2, is a tube for the introduction of gas to expel air from the antechamber, J' being an exit-pipe for the air. This expulsion must be effected after the closing in of the freshly-charged case within the antechamber and before opening the doors G G', which opening would otherwise involve danger of an

explosion from the mixing of the hot gases from below with the said air.

K K, Fig. 1, indicate the apparatus through which the heated gases are introduced into the distilling-chamber. This consists of two cellular structures of refractory brick or other suitable material, contained in two boxes of iron plate with gas-tight joints, one extending horizontally along each longer side of the distilling-chamber. These boxes must be lined with fire brick or tiles or other non-conducting lining.

L L, Fig. 2, indicate heating-coils for the gas, which may be heated by two or more powerful ring gas-burners, M M, or by any other suitable means.

N N, Figs. 1 and 2, indicate one or more perforated distributing-tubes for the heated gas, situated behind the cellular brick-work on both sides, inside the gas-tight iron boxes, and intercommunicating by means of horizontal connecting-tubes whose point of separation is partially indicated at N in Fig. 2.

Q Q is a steam-boiler to take up and utilize the waste heat from the fire which heats the gas-heating coil. Such boiler may be vertical or horizontal.

R is the flue to the chimney-stack.

The lower part of the distilling-chamber, between the cellular gas-chambers and the sole, is lined all round with a continuous solid iron casting, X X, which serves to brace and strengthen the apparatus at this point, and permits of any carbonaceous incrustations being broken or picked off without damage.

S S is the movable sole, which is formed of a cast-iron frame-work filled in with fire-brick, fire-tiles, or any suitable refractory material, which may be covered, in addition, with movable plates of iron to take up incrustations of carbon. T T is the channel all around this sole for holding the fusible metal seal. The alloy of two parts of tin and one part of lead, melting at 360° Fahrenheit, which was before referred to, is quite suitable for this seal; but pure tin, which melts at about 450°, may be used with proper management. The channel for this seal must be of such width at top that no fragment of material that can drop from the cases above will be large enough to wedge in it and prevent the free entrance of the vertical plate of iron T' T', which completes the seal when the sole is elevated to its seat. This part T' T' may be made up of four bars of angle-iron welded together at the four corners of the parallelogram and set into the fire-brick base of the distilling-chamber, as sufficiently depicted in both Figs. 1 and 2. The sole may be run up and down by means of a screw, V, working gas-tight through an iron casting, W W, which constitutes the floor of the distilling-tower, and serves also to brace the walls at this part. Accumulations of dust and fragments which settle on this floor W W may be removed from time to time through a stoppered aperture therein. Under this floor a cellar should extend from end to end of a tier

of distilling-towers, containing the machinery for working the screws, which cellar must be permanently open to the air from end to end to prevent possible accumulation of gas therein, and may be lighted with gas to allow of inspection and care of the machinery. The sole S S may be elevated also by means of a hydraulic apparatus.

The truck Z Z is wholly composed of iron, and serves to transfer the box of coke or other residuum of the distilling process first to the cooling-chamber Y Y, and then, after cooling, to the open air, through the door of the cooling-chamber *d*, which, for that purpose, may open down on a hinge at its base to the position *d'*. The rod *c' c*, which works with proper lubrication gas-tight through an aperture in the wall of the tower, serves both to push the iron truck Z Z, loaded with the iron box B'', from off the sole into the cooling-chamber, and to draw the truck upon the sole again after its next descent from its seat at the base of the distilling-chamber. The latter is accomplished by means of a projection at *c'*, which, on half-turning the rod, falls into a groove in the platform of the truck, and thus establishes an attachment thereto. The tube *a* is for the introduction of gas into Y Y and under the sole S S, when the latter is elevated into place, to displace air from these spaces before lowering the sole and opening communication with the gases in the distilling-chamber.

Mode of operating the apparatus.—New towers may first be dried out and seasoned while empty by passing into them a current of warm air, this being effected by passing such air through the heating-coil heated to a moderate temperature. The whole apparatus may be closed by shutting the outside doors of the antechamber and cooling-chamber, leaving open only the small tube J at the top of the antechamber, from which the current of warm air sent into the tower must therefore be emitted with such moisture as it has taken up. When such moisture has disappeared, as may be shown by tests made by attaching glass tubes that have been dusted internally with dry chloride of calcium, the next step is to introduce one of the boxes or cases in the position B, but empty of material. The door D, which is luted gas tight all around, is then closed, and all air is next removed from every part of the closed tower by passing a current of cold gas into it through the tube J at the top of the antechamber, the air passing out through a tube which should be attached at or near the floor of the cooling-chamber. When the air has been all thus displaced from the apparatus—a point determinable by tests with glass tubes containing asbestos wetted with alkaline pyrogallol, which will then no longer turn brown—it will then be practicable to begin heating up the tower, still void of all materials to be distilled, but containing the empty box B, by passing into it heated gas from the heating-coil. It will be advisable to elevate the movable sole at this stage high enough to

bring the solidified metal in the channel for the seal into contact with the vertical plates T' T'. The temperature should be then elevated by degrees until it is found that the metal seals at the top and bottom of the distilling-chamber have liquefied, so that the doors G G' can be shut down into place and the sole S S can be brought up completely to its seat. Before closing the doors G G', however, the empty box B is lowered one step, so as to bring it into the position B'. The doors G G' may then be closed by crank-handles outside, the door D of the antechamber opened, and the steel rod F F introduced through the link C' to support the box B'. By slightly lowering the vertical rod B° it will unhook itself from the link C' and may be drawn up to the top of the tower. The platform E E E having then been put into place, a charged case is brought up on the truck, slid into the antechamber upon the platform, linked to B°, slightly raised to admit of the removal of the platform, and then linked below to C'. The door D is again shut, which may be done after due adjustment thereof of two rods (not shown in the figures) which open valves in the gas inlet tubes J and *a*. When the antechamber has become filled with gas the rod F F may be drawn out, the doors G G' opened, B then let down to the position of B', the latter falling a step lower, the doors G G' again closed, and the rod F F again introduced. Distillation at moderate temperature will now begin in B', and may be allowed to proceed for one or two hours, as desired, another charge being in the meantime introduced by opening D, unlinking C', raising B°, introducing platform E E E, sliding in B, linking C, slightly raising B°, removing E E E, linking C', closing D, turning on the gas at J, and when the antechamber is filled with gas removing F F and opening G G', lowering the new charge, closing G G', and reinserting F F. After another suitable interval another charge is brought in by dint of the same routine of manipulations. It may here be pointed out that when distillation is proceeding in the distilling-chamber, and a new charge has been shut into the antechamber, which has also been freed from air and filled with gas by means of J and J', it will be advisable to close J', but not J, leaving the latter in communication with a gas-holder or other source of gas, in which a pressure is maintained uniformly somewhat higher than that in the distilling-chamber. If, then, any leakage occur through the joints of the doors G G', it will be from without inward, and will hence be immaterial. When a charge or (as in the initiation of the action of the tower) the first empty case reaches the position of B''—the number of steps to attain which depends of course on the height of the tower—the routine required for the removal of B'' from the tower must be put in practice. This is as follows: The truck Z Z being in the cooling-chamber and the door *d* being closed, gas must be made to flow in through tube *a* till the whole space is filled, as

determined by precautions such as specified in the case of the antechamber at the summit of the tower. The sole S S is then lowered by means of the screw V until its upper surface is on a level with the floor of the cooling-chamber. By pushing in and then half-turning the rod *c' c* the truck Z Z is next caught hold of and drawn upon the sole in the position Z' Z'. At this stage the doors G G' between the antechamber and the distilling-chamber should be opened, and the whole chain of cases being lowered one step will bring the case B'' down to the position B''', so that it stands upon the truck. The next proceeding is a momentary elevation of the sole S S again for about six inches, which forces the link C''' off the hook of box B''' in the manner previously explained, so that it is thrown over into the position shown by dotted lines in Fig. 2 at C'''. Case B''' is thus entirely detached from the chain, and is immediately wheeled out into the cooling-chamber by pushing with the rod *c' c*, which is then drawn back into its recess, and the sole is screwed up without delay into its place at the base of the distilling-chamber. In the meantime, the valves G G' having been shut down into place, the distillation proceeds in the closed distilling-chamber. The next proceeding is the cooling of the mass of carbonaceous residuum in box B''', which is now in the cooling-chamber—an operation effected sometimes by passing through this space cold gas, which may be afterward conveyed to the heating-coils L L, or to the burners M M for heating these, or to both. Such cooling may also take place simply by radiation and conduction, in case a portion of the walls of the cooling-chamber be suitably composed of metal. When cooled to a point at which the residuum will no longer kindle into combustion in the air—which is determinable by observation of the pyrometer at *b*—the door *d* may be opened and the truck, with the box, wheeled out of the tower altogether.

I do not in this application present claims for the novel processes of distillation and improved methods of operation which are in part or in whole set forth and implied in the foregoing specification, as I reserve these to be made subjects of distinct applications for Letters Patent. I confine myself now, therefore, to the following claims for novel combinations of parts and novel devices in construction and arrangement of apparatus and materials, to wit:

I claim—

1. A vertical distilling-chamber of an oblong horizontal section, without external fire space or flues, provided with lids, doors, or valves, closing both over the top and under the bottom, containing a series of receptacles or cases of corresponding oblong section linked or hooked together with interspaces between,

forming a connected chain suspended constantly from the upper end of the chamber, with means of arresting their descent at each interspace, said chamber being connected by pipes at its lower end with a tubular gas-heater or coil external to the said chamber and inclosed in a distinct highly-heated fire-space, all constructed, combined, and operating substantially as set forth.

2. A vertical distilling-chamber of oblong horizontal section, provided with lateral recesses in its two broader sides, at their lower ends, furnished with perforated or cellular refractory brick walls, forming part of the internal walls of the said distilling-chamber itself, said recesses also containing horizontal tubes which convey heated gas behind said perforated or cellular walls from an external tubular gas-heater or coil, all combined, constructed, and operating substantially as specified.

3. In the series or chain of vertical cases in the distilling-chamber herein described, the combination of the laterally-movable link above each case, with the hook rigidly fixed to the case immediately above, and the vertically-movable sole, combined and operating together in the manner and for the purposes substantially as set forth.

4. The combination, with a retort or distilling-chamber, of an antechamber or vestibule situated at a higher level, and provided with a gas-tight door, through which to introduce into said antechamber a freshly-charged case, and furnished with one or more horizontally-binged valves or doors, closing gas-tight at the base of the antechamber and between it and the distilling-chamber below, and furnished with a gas-induction pipe at top, together with means, as set forth, of attaching and suspending said charged case from the crown or apex of the said antechamber internally, all combined and constructed in the manner and for the purposes substantially as set forth.

5. The combination of the gas-induction pipe J at the top with the air-education pipe J', Fig. 2, at the bottom of the antechamber, by which said antechamber is emptied of air before opening communication with the distilling-chamber below, the whole arranged substantially in the manner specified.

6. In combination with a retort or distilling-chamber, a lateral chamber, Y Y, at a lower level, provided with a gas-tight door, and a vertically-movable sole, forming part of the said distilling-chamber, all constructed, combined, and operating together substantially as set forth.

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