Gale & Gattman,

Manf of Sugar of Lead & Acetic Acid.

No 93817. Patented Aug 17. 1869.

Fig: 1.

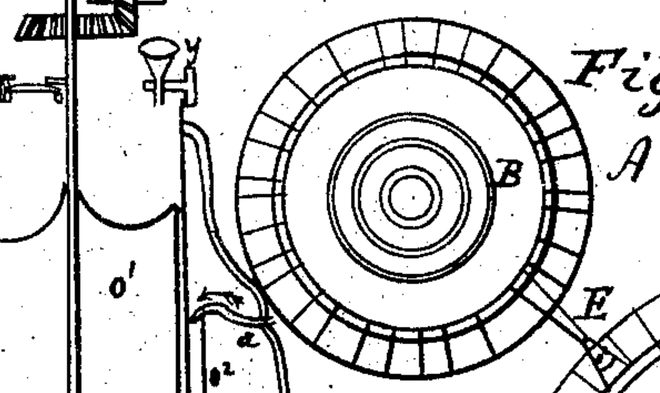
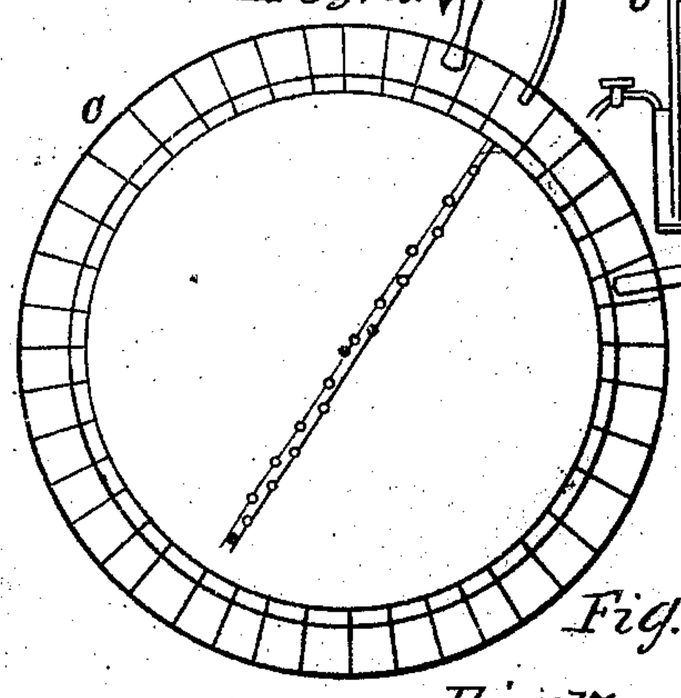


Fig: 2.



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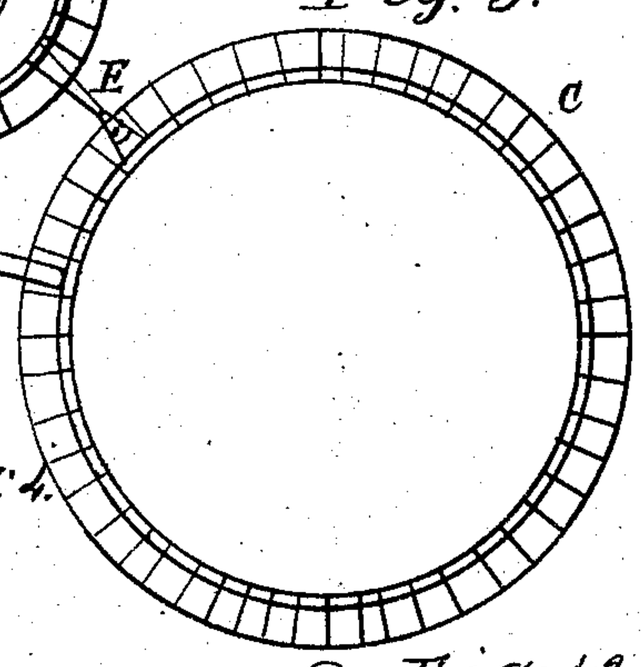


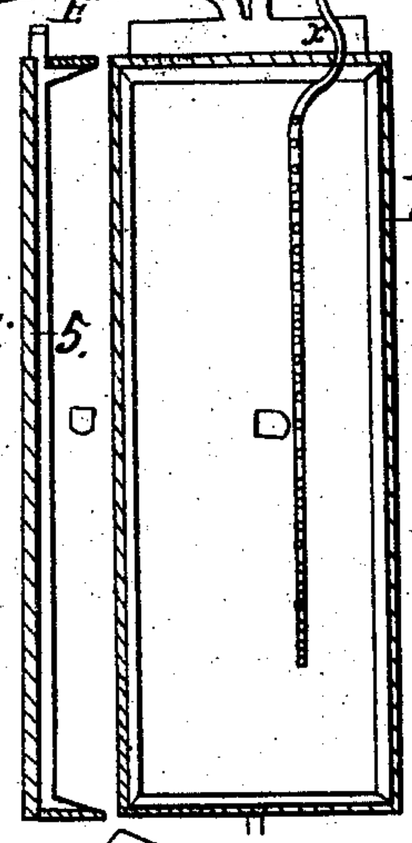


Fig: 4.

Fig: 5.

Fig: 17.

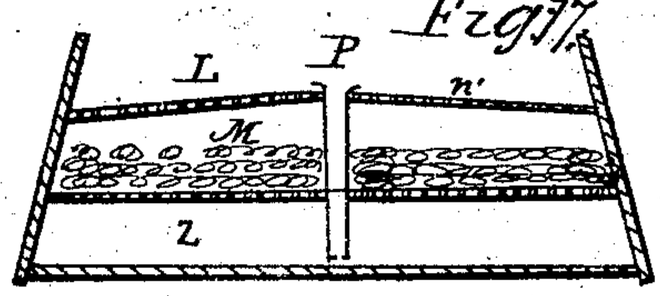
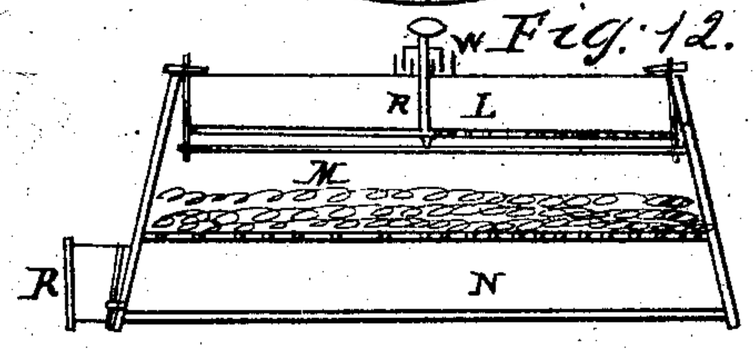


Fig: 12.



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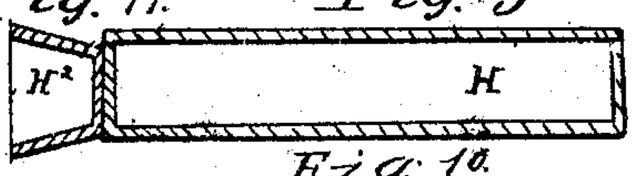
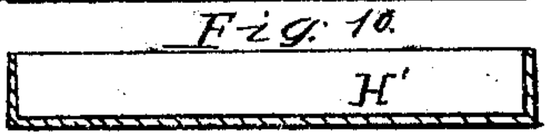


Fig: 9.

Fig: 10.



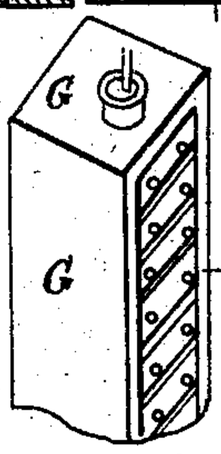
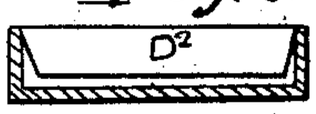


Fig: 8

Fig: 6.



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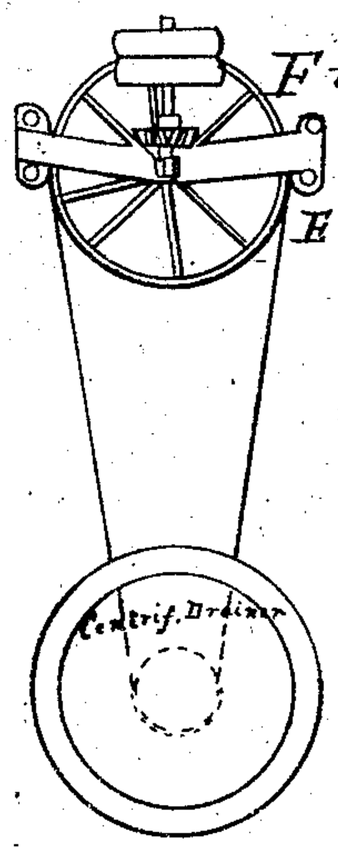
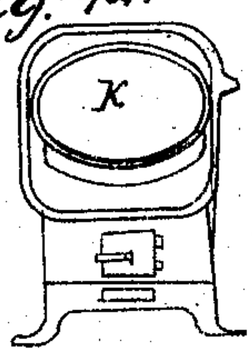


Fig: 18.

Fig: 14.



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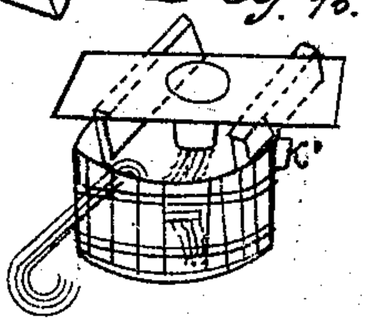
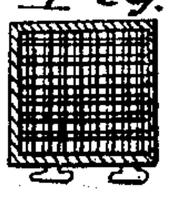


Fig: 13.



Witnesses

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United States Patent Office.

LEONARD D. GALE, OF WASHINGTON, DISTRICT OF COLUMBIA,
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Letters Patent No. 93,817, *dated August* 17, 1869.

IMPROVEMENT IN THE MANUFACTURE OF SUGAR OF LEAD AND ACETIC ACID.

The Schedule referred to in these Letters Patent and making part of the same.

To all whom it may concern:

Be it known that we, LEONARD D. GALE, of Washington, District of Columbia, and ISAAC M. GATTMAN, of the city, county, and State of New York, have invented new and useful Improvements in the Manufacture of Sugar of Lead, which we verily believe have not been known or used before; and we declare that the following is a full and accurate description of the same, reference being had to the annexed drawings which make part of this specification.

Figure 1 represents a boiler, being a vertical view of the wooden shell, upper head removed, to show an end view of the three hollow copper steam-cylinders within, B, the shell being marked A.

Figures 2 and 3 represent a vertical view of a pair of corroding-tubs, seven feet diameter at bottom, the cover removed and tub unfurnished.

Figure 4 represents a vertical view of the copper-evaporator D, in a wooden shell or box.

Figure 5, a longitudinal vertical section, D^1, and showing steam-passage under it.

Figure 6, a transverse vertical section, D^2, of the same.

Figure 7, a vertical view of the centrifugal drainer F.

Figure 8, a perspective view of the centrifugal pump G, over a case of drawers, G′, for drying the sugar of lead.

Figure 9, a top view of a wooden crystallizer, H, made of three-inch plank, of white-wood, well bolted, twelve feet long, eighteen inches wide, and twelve inches deep.

Figure 10, a longitudinal vertical section, H′, of the same.

Figure 11, a transverse section of the same.

Figure 12, a sectional elevation of a corroding-tub, containing a rotary sprinkler.

Figure 13, separate view of one of the drying-drawers, with open bottom.

Figure 14 represents a perspective view of a casting-furnace.

Figure 15, a perspective view of a water-tub, with iron colander for casting wire.

Figure 16, a sectional elevation of the iron vinegar-still, with steam-jacket and rotary agitator, as found on sale.

Figure 17, a sectional elevation of the corroding-tub, containing an overflow-pump, P, sometimes used in place of the sprinkler for wetting down the lead.

Figure 18 represents the double trumpet-pipe E^3.

Fig. 16 shows the connection of the still with the corroding-tub, by means of trumpet-pipe E, or a perforate pipe, o^5 or o^6, for acidifying the liquor in the corroding-tub.

Figs. 4 and 16 show the connection of the still with the evaporator, by means of pipe E^4, to acidify the liquor in the evaporating-pan.

The nature of our invention consists in several new and distinct features, which, when combined together, constitute a complete process of manufacture.

We propose to manufacture sugar of lead by the combined and simultaneous action of concentrated acetic-acid vapors and atmospheric air on metallic lead; and

Our improvements relate not only to the production of sugar of lead, but also to the production of pure and concentrated acetic acid, employed in its manufacture.

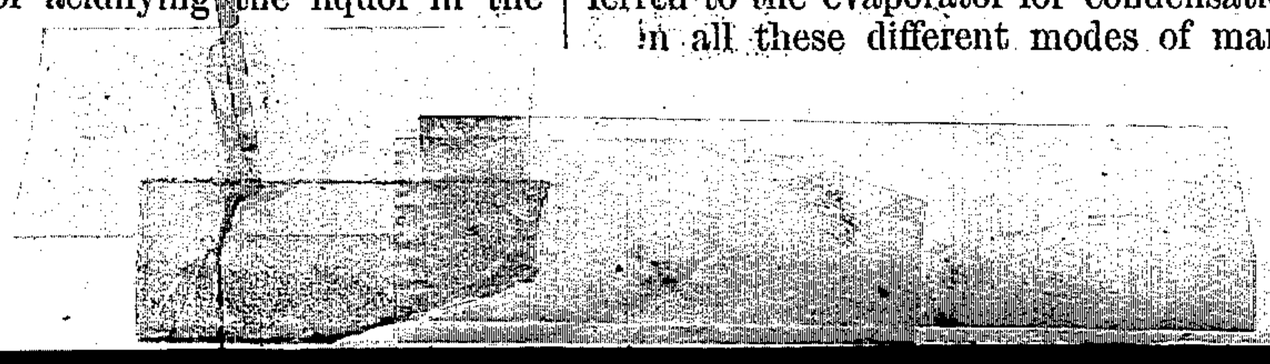
In the manufacture of sugar of lead, either one or the other of the following processes has heretofore been employed:

First. Protoxide of lead (litharge) was dissolved in whiskey, or malt-vinegar, of a strength of from thirty-two to forty grains of bicarbonate of potash to the ounce of the vinegar, equal to about four to five per cent. of hydrated acetic acid. The solution thus obtained was evaporated, or boiled down to the point of crystallization, under addition of fresh vinegar, to prevent the formation of basic salt. In this process the cost of evaporation, and the unavoidable loss of vinegar, besides the additional time, fuel, and labor, enhance the cost of production considerably.

Second. Whiskey, or malt-vinegar of the above strength, was put into a still, and heated over a furnace or by steam. The weak vinegar-vapors passing over were brought in contact with the protoxide of lead, placed in a convenient vessel over a perforated bottom. The vinegar-vapors acting on the protoxide of lead, the weak solution of sugar of lead collected at the bottom, and was transferred into the evaporating-pans for condensation. The same objections are applicable to this mode of procedure as to that of No. 1, with the single exception that the solution of sugar of lead, as obtained under No. 2, was more free from coloring-matter than that obtained under No. 1.

Third. A series of suitable tubs was placed in a range, one above the other, and filled with lead spangles or lead-wire. Vinegar of the above-mentioned strength was run into the upper tub, left for some time in contact with the lead, then run off into the next, &c., until it reached to the lower tub. By this time the lead in the upper tubs was exposed to the action of the atmosphere for the purpose of oxidation, the liquor was raised from the lowest to the upper tub to make a second circuit, and was then transferred to the evaporator for condensation.

In all these different modes of manufacture only



the first crop would yield white merchantable crystals of sugar of lead, the mother-liquor retaining all the coloring-matter originally contained in the vinegar, or absorbed during the process of evaporation.

To bleach the liquor for the purpose of yielding white sugar of lead, it was boiled up with ground animal-charcoal, (bone-black,) and filtered, by which process the carbonate of lime in the bone-black decomposed an equivalent proportion of the sugar of lead in the solution, thereby effecting a considerable loss, besides that lost by the absorbing nature of the bone-black itself.

When the different liquors were evaporated to the crystallizing-point, they were drawn off into the crystallizing-pans, left in repose for twenty-four to thirty-six hours, the mother-liquor removed, the pans raised on end for the purpose of draining, and the crystals placed on drying-boards in a room slightly heated, and, when dry, broken up and packed for market.

This mode of drying requires considerable room, and about a week's time, when no loss from heat shall be sustained.

All the different difficulties, obstacles, and disadvantages, in the processes above described, we obviate by our new improvements, which we will now describe, to enable others skilled in the art to use the same.

For the better exemplification of our process, we divide the specification under the following headings:

I. The sugar-of-lead solution;
II. The evaporation and crystallization;
III. The draining and drying;

and as our improvements relate to the use of whiskey and malt-vinegar, as well as to wood-vinegar, we will describe both modes of manufacture under these three different headings.

I. *The Sugar-of-Lead Solution.*

We make the sugar-of-lead solution from the metallic lead. To prepare the metal for our purpose, we cast it either into spangles or wire, and place it into a corroding-tub, figs. 12 and 17, arranged in the following way:

The bottom, inside, is seven feet in diameter; the staves three and a half feet high, made of three-inch plank, of white oak or white poplar, the former preferred.

The tub is, by means of two perforate bottoms, divided into three chambers, the lower, N, for the lead-liquor, the middle one, M, for the metallic lead, and the upper one, L, containing the rotary sprinklers, with feed-funnel, or, in place thereof, the overflow-pump P, fig. 17.

The tub is tightly covered with a well-fitting cover or lid, into which a copper pipe, four inches wide, is fitted, E^1, figs. 2 and 3, for the escape of the surplus steam, to be utilized either for the evaporation and concentration of the solution of sugar of lead, or for some other purpose, as will be shown hereafter.

The whiskey, or malt-vinegar, of from four to five per cent. of hydrated acetic acid, (equal to thirty-two to forty grains of bicarbonate of potash to the ounce,) is put into a still, fig. 1, heated by steam by means of three concentric hollow copper steam-cylinders B, capable of sustaining a pressure of twenty pounds to the square inch, by which the vinegar-vapors are driven through the double trumpet-pipes E^3 into the lower chamber of the corroding-tub.

This double trumpet-pipe is best seen in fig. 18, E^3, and consists of two conic copper pipes, brazed together at their small ends.

The current of vinegar-vapors enters the pipe in the direction of the arrow. Small holes are made into this pipe, beyond its smallest diameter, for the admission of atmospheric air.

The pipe has its induction-end in the still, fig. 1 or fig. 16, and the opposite, or eduction-end, enters the corroding-tub just below the perforate bottom.

By this arrangement atmospheric air rushes in through the small holes in the pipe, simultaneously with the current of the acetic-acid vapors, whereby oxidation and acetification take place in one continuous operation, and the metal is rapidly corroded and dissolved.

The solution of sugar of lead is continually falling, in drops or streams, into the lower chamber of the corroding-tub. At the same time a large surplus of heat is developed, partly from the chemical action of the acid and lead, and partly from the surplus heat of the vinegar-vapors, which surplus heat is, by the four-inch copper pipe, E^1, figs. 2 and 3, conducted from the upper chamber of the corroding-tub into the steam-chamber of the evaporator, figs. 4, 5, and 6, to concentrate the weak solution of the sugar of lead.

To facilitate the corrosion of the metallic lead, it is necessary to keep the metal always clean, so as to offer a bright surface to the action of the atmospheric air and the acid-vapors.

We accomplish this in the most efficient and convenient way, by means of the rotary sprinkler, R, fig. 12, in connection with an endless belt or chain of copper buckets—a contrivance well known by its use in flouring-mills and grain-elevators, and therefore not necessary to describe here.

The rotary sprinkler, made of copper, with funnel at the top, has on the hollow vertical shaft two hollow horizontal arms, one on each side of the central shaft, that on the right having a row of small holes in the front, that on the left an equal number of holes on the rear; so, when the central shaft is supplied with liquor from the lower chamber of the corroding-tub, the reaction of the liquid in the horizontal arms *z z'* will cause the machine to rotate rapidly, and wash down the metal in the most perfect manner.

The same purpose we accomplish by a lifting-pump, with equal overflow over the second perforate bottom, *n'*, fig. 17.

But vinegar of the above-mentioned strength, of from thirty-two to forty grains of saturation to the ounce, will only yield a very weak solution of sugar of lead, which, for the purpose of crystallization, must be evaporated or condensed.

To save the additional expense of this operation, and to gain time, we have made an improvement in the distillation of such kind of vinegar, which enables us to produce a concentrated solution of sugar of lead without evaporation, which object we accomplish by the use of common salt, (chloride of sodium,) in the proportion of thirty-three pounds of salt to every one hundred pounds, or twelve gallons of the vinegar.

By the addition of the salt to the vinegar, the boiling-point of the latter is raised several degrees above the boiling-point of water or weak vinegar. The vinegar-vapors, as being more volatile, will therefore pass over below the boiling-point of the mixture of salt and vinegar, whereby more concentrated vapors of vinegar are brought in contact with the metallic lead; hence a more concentrated solution of sugar of lead, obtained at once.

The solution of salt, deprived of vinegar-vapors, may be evaporated to dryness by means of the surplus heat of the corroding-tub, by pipe E^1, figs. 2 and 3, without additional expense for its repeated use.

The sugar-of-lead solution will always be in a basic state as long as there is any lead in excess.

For the purpose of forming the marketable crystallized sugar of lead, the liquor must have an acid reaction on litmus paper, and for this reason it is necessary to acidulate the liquor during evaporation, by the addition of fresh vinegar, whereby a great portion of

the vinegar is not only lost, but also coloring-matter from the vinegar is added to the solution.

To remedy this evil we have contrived the following improvements: When the lower space N, figs. 12 and 17, of the corroding-tub, is filled up so high with the basic solution of the sugar of lead as to cover the opening of the trumpet-pipe E, the rotary sprinkler or pump is stopped, whereby the corrosion of the lead ceases. The vinegar-vapors coming over from the still are condensed in the basic solution of sugar of lead, and the latter acidulated thereby, which will be tested by its reaction on litmus paper.

When the solution is brought to the acid state, it is immediately transferred into the crystallizing-pans; or the basic solution might be first transferred into the evaporator, and a branch-pipe, E^4, Figure 20, from the still, enters into the basic solution, wherein the acid-vapors are condensed, and the solution acidified thereby free of coloring-matter.

Since the introduction of the revenue-tax on distilled spirits and malt-liquors, the use of whiskey or malt-vinegar in the manufacture of sugar of lead is out of question. We were, therefore, compelled to resort to the use of pyroligneous acid, or wood-vinegar.

But here we had to meet with another difficulty. Pyroligneous acid, free from odor and coloring-matter, is not any cheaper than malt or whiskey-vinegar, and when not free from both, it cannot be used.

Perfectly colorless and odorless pyroligneous acid has heretofore been made only by repeated distillations of the product of decomposition of acetate of lime with hydrochloric acid or diluted sulphuric acid, by which process the cost of the pure acid becomes too high for its application in the manufacture of sugar of lead.

By repeated experiments we have invented a new process for the production of pure acetic acid from the acetate of lime in one operation, by which process we are enabled to apply it profitably in the manufacture of sugar of lead.

The nature of this improvement consists in decomposing dry acetate of lime by concentrated sulphuric acid, of no less than 65° to 66° Baume, in an apparatus provided with an agitator, by which the adhesion of the resulting sulphate of lime in course of operation is prevented, the volatile oily matter of the lime-salt solidified, and the passing over of pure, colorless, and odorless acetic-acid gas effectually secured.

An iron still, fig. 16, with steam-jacket for heating, and an agitator for stirring the contents, cylindric in form, and capable of working five hundred pounds of acetate of lime with convenience, is charged with dry acetate of lime and concentrated sulphuric acid, of 66° Baumé, in the proportion of one hundred pounds of the former to sixty pounds of the latter, and the agitator set in motion.

By the chemical reaction, enough heat is evolved to liberate over one-half of the acetic acid, in a pure state, without the application of artificial heat. Only when the action begins to slacken, steam is gradually employed, and continued until the acid-vapors are all driven off through the trumpet-pipe E into the corroding-tub. The flow of sulphuric acid is regulated by the funnel *y*, fig. 16.

In working the still, the heat from the chemical action accumulates in the first part of the process, while, in the latter part, it diminishes greatly. To avoid the excess of the former, we throw cold water into the jacket and ply the agitator vigorously; and, to avoid the want of heat in the latter, we throw steam into the jacket.

In all other respects the arrangements are the same as those in the application of whiskey or malt-vinegar.

II. *Evaporation and Crystallization.*

By the use of acetate of lime, a concentrated color-less solution of sugar of lead is obtained, ready, without evaporation, to be run off immediately into the crystallizers.

When malt or whiskey-vinegar is used, with the addition of salt, or chloride of sodium, as stated above, the same result will be obtained. But when whiskey or malt-vinegar is used, without salt, only a weak solution of sugar of lead is the result, which must be evaporated or condensed for the purpose of crystallization. This we accomplish by the surplus heat of the corroding-tub, by the escape-pipe E^1, figs. 2 and 3, without additional cost.

The evaporator consists of a wooden shell or box, figs. 4, 5, 6, twenty-five feet long, five feet wide, and twelve inches deep, lined with copper.

The copper bottom of this pan rests on ten longitudinal bearing-strips, one and a half inch high, fastened to the bottom of the wooden shell or box, forming a steam-chest.

The four-inch copper pipe E^1, leading from the upper chamber of the corroding-tub, enters the steam-box *x*, leading under the copper bottom of the evaporator, the condensed steam and water escaping through pipe E^2. This escape-pipe is to be frequently examined, to guard against the waste of acetic-acid vapors, whereby a loss would be sustained.

The solution of sugar of lead, although clear and colorless at first, often assumes a darker hue during the process of evaporation, and must be clarified.

This has heretofore been done by bone-black and filtration, which process involves a loss, as stated above.

We therefore substitute, for the bone-black, the sulphuretted-hydrogen water as decolorizing-agent, by mixing one pint of water, saturated with sulphuretted-hydrogen gas, to every eighteen or twenty pounds of sugar of lead held in solution, forming thereby a precipitate of sulphuret of lead, which is a most powerful agent of decolorization, and can be easily reduced again into metallic lead, that no loss is sustained, although the quantity of sulphuret of lead is in itself insignificant, amounting only to about one hundred and eighteen grains for every eighteen or twenty pounds of sugar of lead contained in the solution.

The process of making sulphuretted-hydrogen water is so simple and well known by chemists, that we omit a description of the same.

When the liquor is sufficiently concentrated to the crystallizing-point, it is transferred into the crystallizers, figs. 9, 10, 11, and left until a sufficient quantity of crystals is formed, when the mother-liquor is run off and the crystals are ready for

III. *Draining and Drying.*

In place of the old process of draining and drying on shelves, in a very slightly heated room, we have devised the following plan:

We place the damp crystals in a rotary vertical cylinder, fig. 7, having a wire-gauze or other equivalent periphery, and set the machine in rapid motion.

The liquor in the crystals soon gravitates toward the periphery, and escapes outside. Air will supply its place until all the moisture is driven out.

Instead of finishing the work with this machine, we sometimes transfer the crystals to the drying-box, fig. 8, to be finished there.

This drying-box G^1, fig. 8, consists of a well-made frame, containing a series of drawers, arranged vertically. The drawers fit the case very accurately on all the vertical sides, while the bottoms of the drawers are made of open cane-work, not unlike the cane-seats of chairs, G^2.

An opening in the lowest portion of the drying-box admits atmospheric air, while a rotary pump or fan is connected with the top of the case G, fig. 8, to exhaust continually the entire series of drawers, by which

means the damp crystals are easily deprived of moisture, and fitted for the market.

Having stated the nature of our invention, and the methods of using the same,

What we claim as our invention, and desire to secure by Letters Patent, is—

1. The manufacture of sugar of lead, by the process of corroding the metal by vapors of vinegar mixed with atmospheric air, substantially as described.

2. Applying the surplus heat from the steam of vinegar, and the chemical action of the acid on the lead, for concentrating the solution of sugar of lead, and for other purposes.

3. Concentrating the vapors of malt or whiskey-vinegar by means of common salt, substantially as described.

4. Bleaching the solutions of sugar of lead by means of sulphuretted hydrogen, substantially in the manner herein set forth.

5. Acidifying the basic solution of sugar of lead, substantially in the manner herein described.

6. Draining and drying sugar of lead, by centrifugal action, substantially as described, as an improvement on the mode now used of drying on shelves.

7. The use of the double trumpet-blowing pipes, or their equivalent, for mixing the vinegar-vapors with air, substantially as described.

8. Washing down the corroded lead by means of the rotary sprinklers, or their equivalents, as herein set forth.

9. Generating acetic acid, free from pyroligneous odor and color, by simple distillation of acetate of lime with sulphuric acid, substantially as herein described.

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ISAAC M. GATTMAN.

Witnesses:
A. E. GALE,
C. A. GRAVES.