

No. 724,477.

PATENTED APR. 7, 1903.

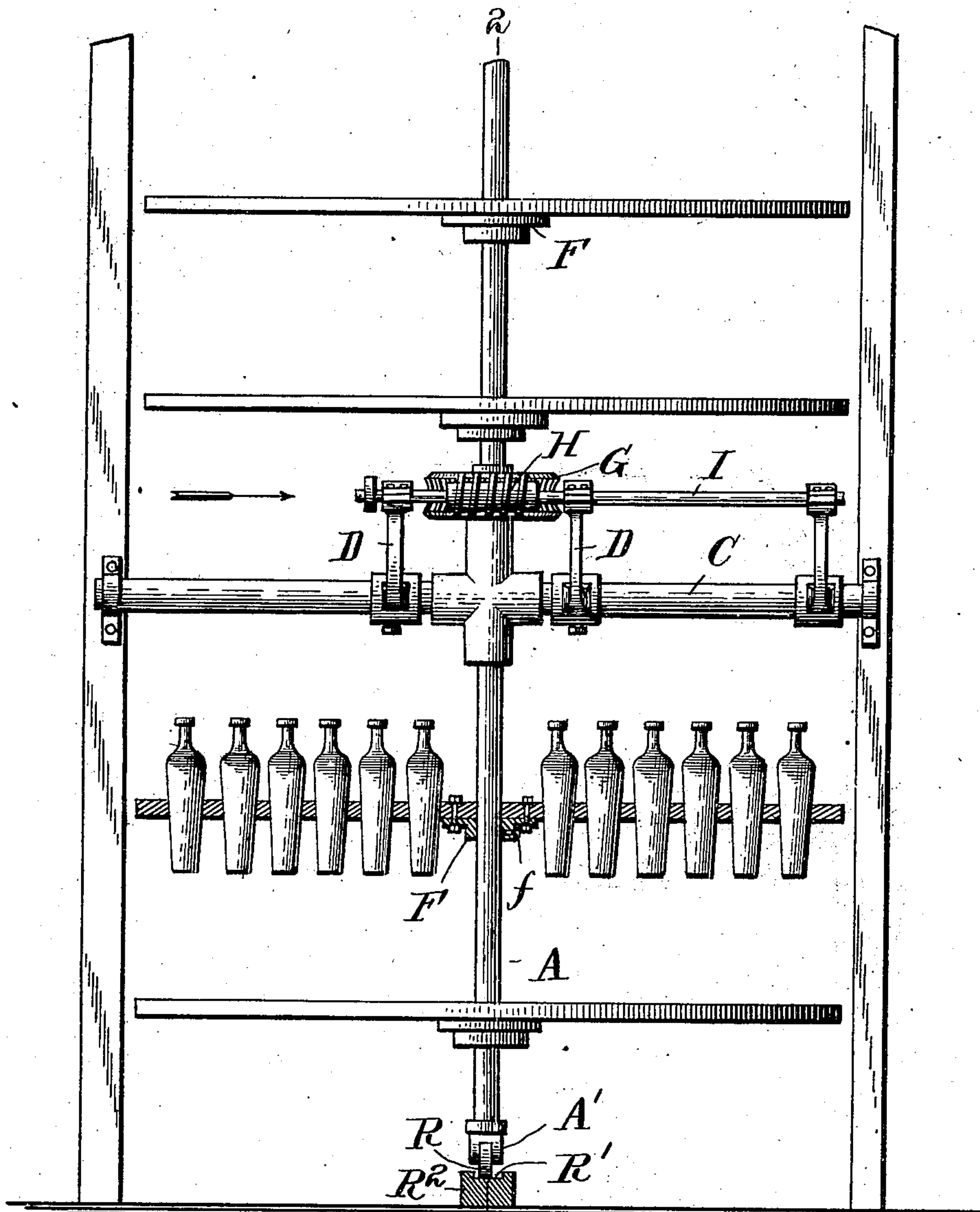
C. HITZL.  
PROCESS OF CRYSTALLIZATION.

APPLICATION FILED MAR. 1, 1902.

NO MODEL.

3 SHEETS—SHEET 1.

Fig. 1.



WITNESSES:

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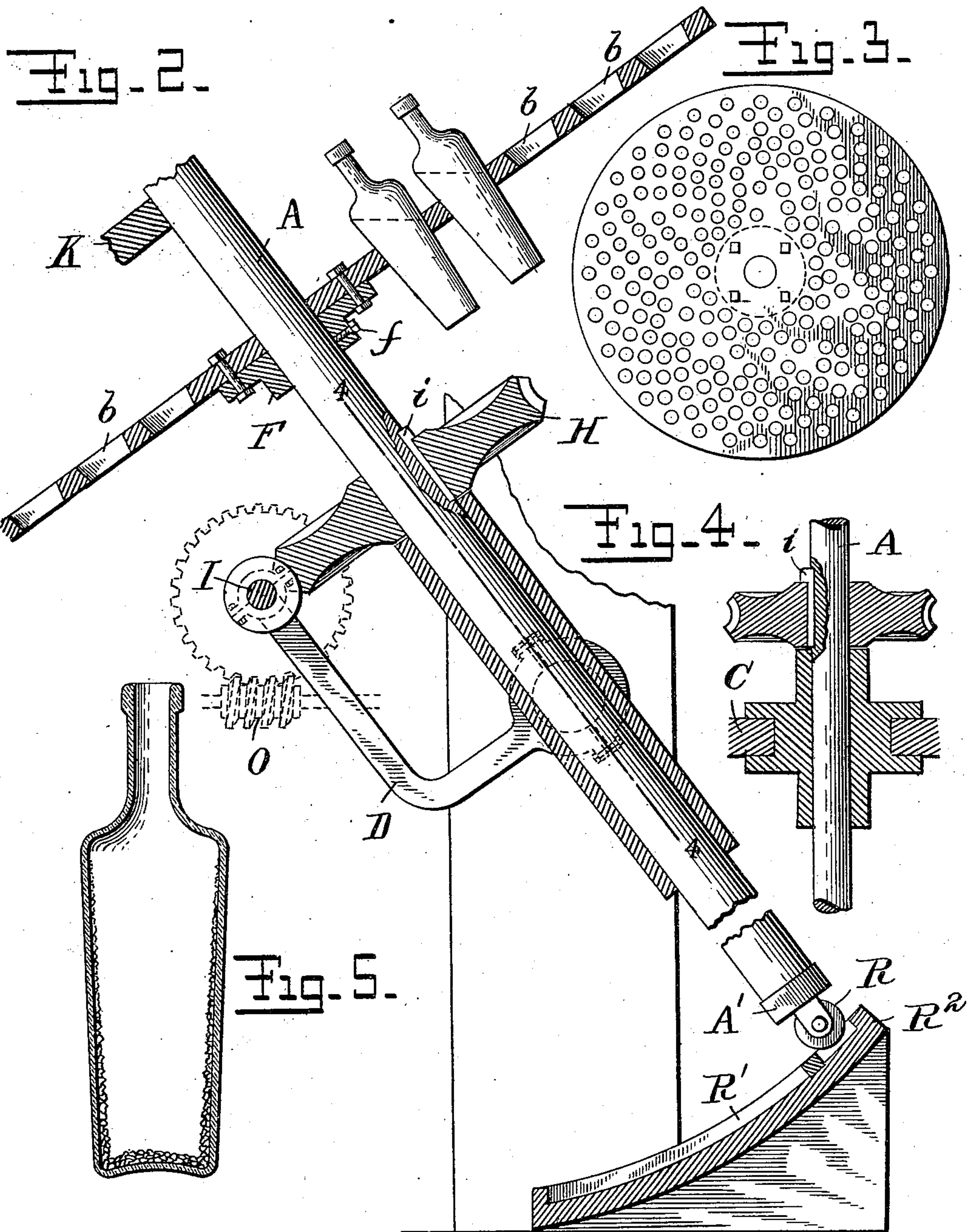
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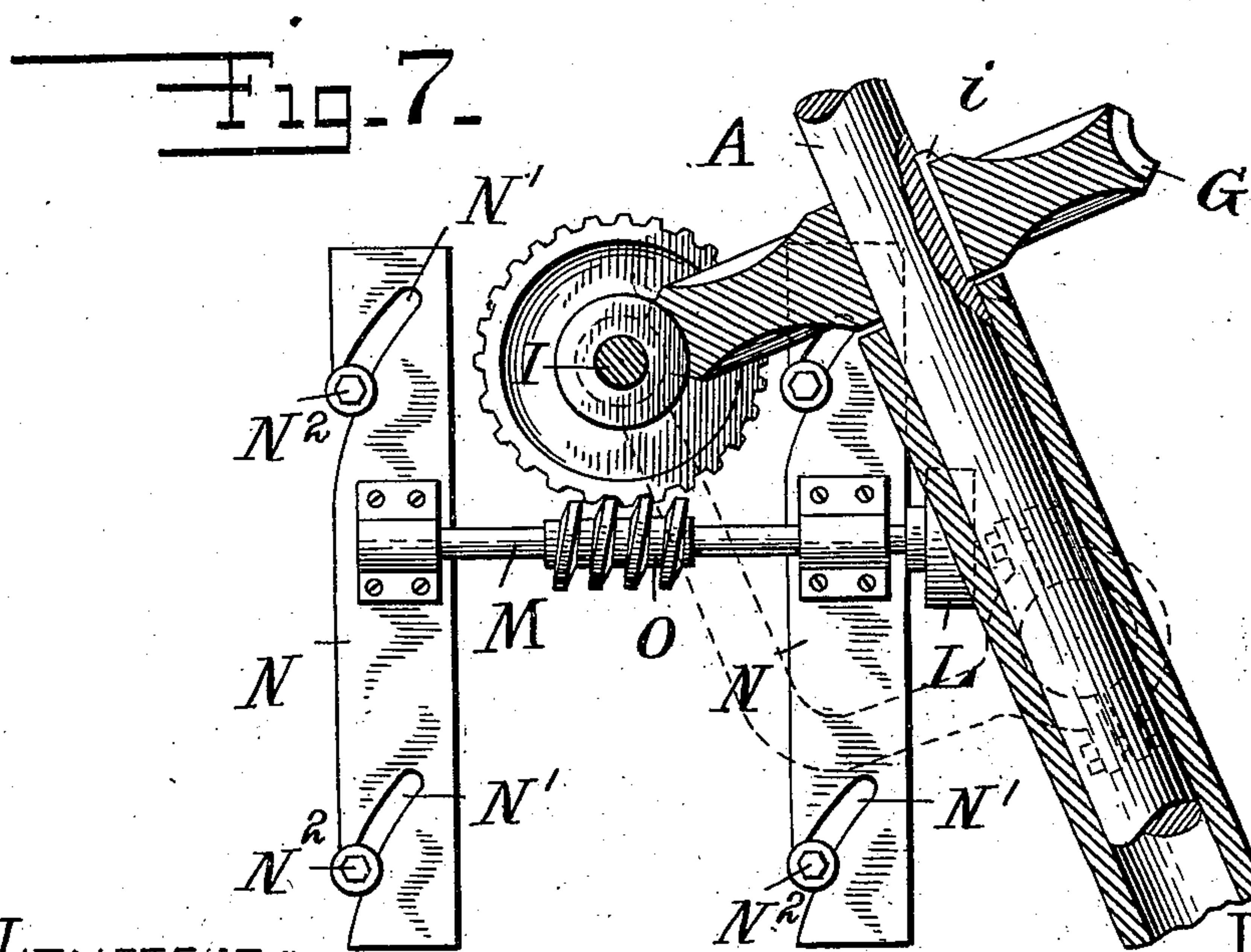
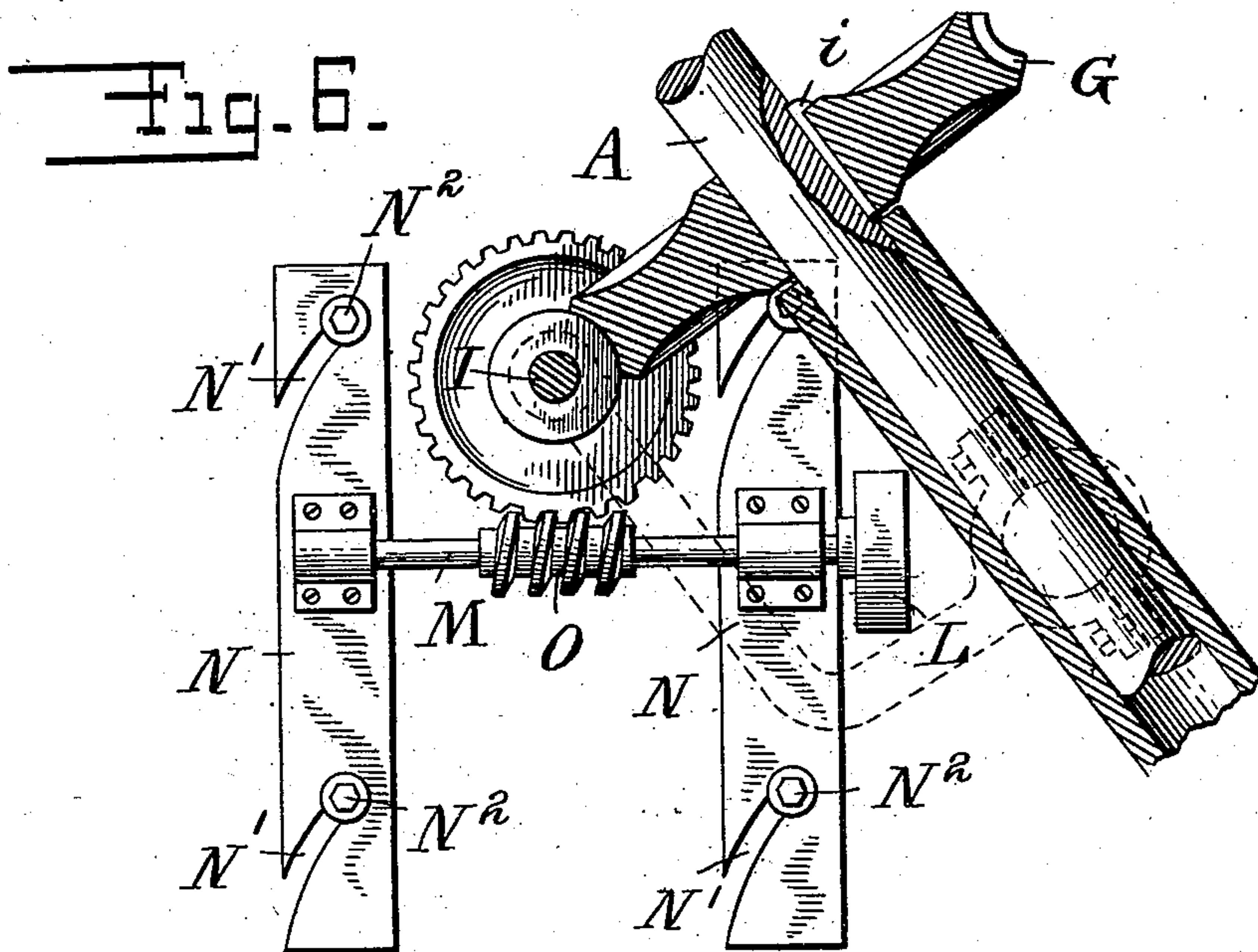


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3 SHEETS—SHEET 3.



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# UNITED STATES PATENT OFFICE.

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## PROCESS OF CRYSTALLIZATION.

SPECIFICATION forming part of Letters Patent No. 724,477, dated April 7, 1903.

Application filed March 1, 1902. Serial No. 96,226. (No specimens.)

*To all whom it may concern:*

Be it known that I, CARL HITZL, a citizen of the United States, residing in the borough of Brooklyn, in the city of New York, in the  
5 county of Kings and State of New York, have invented a new and useful Improvement in Processes of Crystallization, of which the following is a specification.

My invention relates to a process of crystallization, and has for its object the production within a bottle of a layer of crystals distributed over a selected portion of its surface and surrounding the liquid contained in the bottle; and its novelty consists in the several  
15 successive steps of the said process.

The liquids which I treat by my process consist, essentially, of a supersaturated solution of sugar in water, to which are added alcohol and chosen flavoring materials, such as  
20 whisky, peppermint, orange, rose, kummel, honey, or any fruit flavor.

The present mode of causing crystals to be distributed on the inside of the bottles consists in filling the bottles with a supersaturated saccharine solution, tightly corking the  
25 bottles, and placing them in rows in bins with their necks higher than their bottoms. About twice a day an attendant turns each bottle one-half or one-quarter way around, so as to  
30 present a new surface upon which the crystals precipitated out of the solution may fall. This process is crude, unsatisfactory, and expensive, and, moreover, by it an even deposit on the inner surface of the bottle is not secured, and it frequently happens that there  
35 is no deposit at all, as is well known. To avoid these objections is the aim of the present invention.

In carrying out my process the first step  
40 consists in the manufacture of the saturated syrup. This is accomplished by dissolving granulated sugar in water in the presence of heat, so that a heavy uniform syrup is the result. In doing this I use an ordinary steam-jacketed kettle, such as is commonly employed by a confectioner or in similar arts,  
45 and I heat the mass of water and sugars slowly to boiling, constantly stirring during the heating operation in order to make the mixture  
50 more uniform and the solution more perfect. As soon as the syrup is made the heat is shut

off and the syrup permitted to cool down to about 190° Fahrenheit and the alcohol and flavoring material are added, the addition of the alcohol increasing the tendency of the sugar subsequently to crystallize out of the solution. The kettle is now covered, so that the radiation is very slow and the cooling of the mass correspondingly small. In fact, practically the solution is kept at substantially the same temperature during the bottling operation presently to be described. The warm liquid, which, it is now seen, contains water, sugar in excess, alcohol, and the chosen flavoring material, is then drawn off into bottles. The bottles are placed in an oblique position and are slowly and continuously rotated while cooling, and the cooling is very gradual.

It is not essential to this invention that any particular apparatus be employed for carrying into practical effect the last-mentioned step of filling the bottles in an oblique position and slowly and continuously rotating them, and, in fact, many different apparatuses by which the essentials of said step of the process may be performed may readily suggest themselves or be devised for the purpose. One form of apparatus suitable for the purpose is illustrated in the drawings hereto annexed and will be described here in order that the process may be most clearly understood.

In the drawings, Figure 1 is a front elevation and partial section of the apparatus. Fig. 2 is a transverse vertical central section on the plane of the line 2 2 in Fig. 1. Fig. 3 is a plan view, on a small scale, of one of the bottle-supports. Fig. 4 is an enlarged sectional detail showing the manner of connection of the horizontal shaft with the shaft of the bottle-supports. Fig. 5 is a central vertical section of a filled bottle. Figs. 6 and 7 are detailed views of the driving mechanism and adjustable bracket to which the same is secured viewed in the direction indicated by the arrow in Fig. 1.

In the drawings, A is a shaft, to which is secured at intervals the bottle-supports B by means of flanges F and set-screws f or any other suitable means. The bottle-supports are preferably made of wood or metal, are circular in form, and are provided with a cen-



tral aperture to receive shaft A and numerous smaller apertures *b b b b*, &c., to receive the bottles. The precise form and constructions of the bottle-supports are immaterial, and variations from the form shown and equally well adapted for my purpose are quite within the skill of an ordinary mechanic to make. The shaft A is provided at its lower extremity with a shoe A' and caster R, by means of which latter it easily oscillates up and down in a curved groove R' in a guide R<sup>2</sup>, supported upon the floor. The shaft A is also supported on a horizontal shaft C, on which it is adapted to tilt or swing. The shaft C is mounted in bearings in the walls of the chamber in which the apparatus is placed or in some other framework provided for the purpose.

K is a stop to prevent the tilting of the shaft too far in one direction.

One or more hangers or brackets D are secured to the shaft C by flanges and set-screws or otherwise. These brackets in the preferred form of my apparatus are curved at right angles and at their other extremities support a shaft I, upon which is mounted a worm H, adapted to mesh with a circular gear-wheel G, rigidly secured to the shaft A by a key or other similar means, and which wheel when turned causes the shaft to rotate. In the construction shown in Figs. 6, 7, and 8 this mechanism consists of the pulley L on the end of the shaft M, journaled on the adjustable brackets N, and has a worm O intermediate the said brackets, which meshes with the gear-wheel P on the end of the shaft I. The curved slots N' in the brackets N receive the bolts N<sup>2</sup>, by which the said brackets are secured to the frame of the machine, so that the shaft and the worm O may be adjusted to correspond with the movement of the shaft I as the shaft A is tilted. The shaft I is thus maintained in continuous engagement with the driving mechanism.

The rotation of the shaft A and the bottle-supports attached to it upon the shaft C, which is suspended at points distant from the point at which the shaft A is supported, produces a slight but continuous vibration in all of the parts, which I have found to be advantageous in carrying out my process.

The liquids which I propose to treat in this apparatus are composed practically of water, to which has been added sugar in excess to form a thick heavy syrup, alcohol, and some other chosen flavoring material, such as whisky, kummel, peppermint, honey, rose, or any fruit flavor. These materials are incorporated into one mass in an ordinary steam-jacketed kettle, the sugar being added to the water and slowly stirred while being heated. The heating is continued to boiling, and this temperature is continued for a few minutes to insure thoroughness, a thick uniform syrup being the result. After the syrup has been formed and permitted to cool to about 190° Fahrenheit the alcohol and flavoring mate-

rial are added, the kettle being then covered to prevent excessive radiation. The bottles are then filled with the warm liquid, for example, by allowing said liquid to flow through a tube and by means of a suitable nozzle, provided with a check-valve of a nature readily understood by those skilled in the art, introducing it into the bottles which have been placed into the bottle-supports, each aperture in the bottle-supports being filled by an empty bottle. The machinery is started and the shaft A is caused to be slowly and continuously rotated. While this continues the operator introduces the nozzle into the neck of the bottles and allows the liquid to flow in until it has obtained the desired height. He may do this with one hand and use the other to cork the bottle as soon as the filling operation is completed. I have found by experience that for most of the liquors which I treat a period of twenty minutes for one complete rotation of the shaft is sufficient for my purpose. This period for a rotation is also about sufficient to permit a skilled operator to fill the bottles as they are presented before him in turn. The temperature of the room in which the crystallizing operation now takes place should be such that the bottle and the liquor contained therein are permitted slowly to cool. As the cooling and continual rotation of the bottles proceeds the crystals of sugar separate out and acted upon by gravity fall to the lower portion of the liquor and successively deposit upon the inner surface of the bottle, forming, as this deposit proceeds, a coating of crystals upon such surface. Nine or ten rotations are usually sufficient to secure the desired thickness of deposit.

It will readily be seen that the degree of obliquity of the shaft, and consequently the degree of obliquity of the bottles, will control the height within the bottles of the layer of crystals deposited. Other factors, such as the relative specific gravities of the ingredients in the liquor, the change of temperature of the liquor while rotating, and the initial temperature of the crystal-containing mass, all together, serve to control the nature and extent of the deposit which the operator desires to secure.

After the apparatus has been running the desired length of time the shaft A is tilted to a vertical position. This places all of the bottles in an upright position, and while so placed they are permitted to cool further, the process of crystallization continuing somewhat, this depending upon the degree of saturation of the liquor.

I have found by experience that the vibration of the parts of the apparatus tends somewhat to secure greater rapidity in the process of crystallization.

While the bottles are being filled the heavy character of the ingredients of the liquid tends to secure the retention of some of the air within the bottle, and consequently at times bubbles of the air, usually of small size,



are apt to be caught at points between the layer of crystals deposited and the inner surface of the bottle. These, however, do not detract from the appearance of the finished article.

Owing to the fact that gravity is an important element in filling the bottles, the layer of crystals is thickest toward the bottom and gradually decreases in thickness as it progresses upward in the bottle.

The advantages of my invention are numerous. It entirely does away with the manual labor of intermittently rotating the bottles. It enables a very perfect control of the extent of the layer of crystals and their nature to be secured. The apparatus in which the process is carried out occupies a small space and requires little power to run it, and I have found that on the whole the production of the desired layer of crystals within the bottle by my process has been much more economical than any former process by which a similar result could therefore be secured.

What I claim as new is—

1. In the process herein described of causing the deposition of crystals upon the interior surface of a bottle, the following steps, namely, first, supplying said bottle with a heated supersaturated saccharine solution, and then placing said bottle in inclined position and causing the liquid to cool very gradually and slowly and continuously rotating the bottle while the liquid is cooling and the bottle is in said inclined position, substantially as described.

2. The process of producing crystals upon the interior surfaces of bottles filled with liquids, consisting of first placing in the bottles a warm liquid containing water, alcohol flavoring material and an excess of sugar in solution and second slowly and continuously rotating and simultaneously vibrating the filled bottles in an oblique position while cooling.

3. In the process of producing crystals upon the interior surfaces of bottles filled with liquids the following steps, namely first dissolving sugar in water in the presence of heat to form a heavy saturated syrup, second adding a suitable amount of alcohol and flavoring material, third introducing the same into the bottles while still warm and fourth placing the bottles in an oblique position and slowly and continuously rotating them while cooling.

4. The process of producing crystals upon the interior surfaces of bottles filled with liquids consisting of first dissolving sugar in water in the presence of heat to form a heavy

saturated syrup second adding a suitable amount of alcohol and flavoring material, third introducing the same into the bottles while still warm and fourth slowly and continuously rotating and simultaneously vibrating the bottles while they are slowly cooling.

5. The process of producing crystals upon the interior surfaces of bottles filled with liquids, consisting of first placing in bottles a warm liquid containing water, alcohol, flavoring material and an excess of sugar in solution, second slowly and continuously rotating the filled bottles in an oblique position while cooling and third subsequently placing the bottles in a substantially vertical position.

6. The process of producing crystals upon the interior surfaces of bottles filled with liquids consisting of first placing in bottles a warm liquid containing water, alcohol, flavoring materials and an excess of sugar in solution and second slowly and continuously rotating and simultaneously vibrating the filled bottles in an oblique position while cooling and third subsequently placing the bottles in a substantially vertical position.

7. The process herein described of causing the deposition of crystals upon the interior of a bottle, consisting in supplying said bottle with a heated supersaturated saccharine solution and subjecting the liquid while cooling to slow and continuous agitation while the bottle is inclined.

8. The process herein described of causing the deposition of crystals upon the interior of a bottle, consisting in supplying said bottle with a heated supersaturated saccharine solution, subjecting the liquid while cooling to slow and continuous agitation while the bottle is inclined, and varying the angle of inclination of said bottle during a portion of the agitating step of the process.

9. The process herein described of causing the deposition of crystals upon the interior of a bottle, consisting in supplying said bottle with a heated supersaturated saccharine solution, subjecting the liquid while cooling to slow and continuous agitation while the bottle is inclined, varying the angle of inclination of said bottle during a portion of the agitating step of the process, and finally continuing said deposition while the bottle is vertical.

Witness my hand this 24th day of February, 1902, in the presence of two subscribing witnesses.

CARL HITZL.

Witnesses:

MABEL K. WHITMAN,  
ERNEST H. BOYCE.