

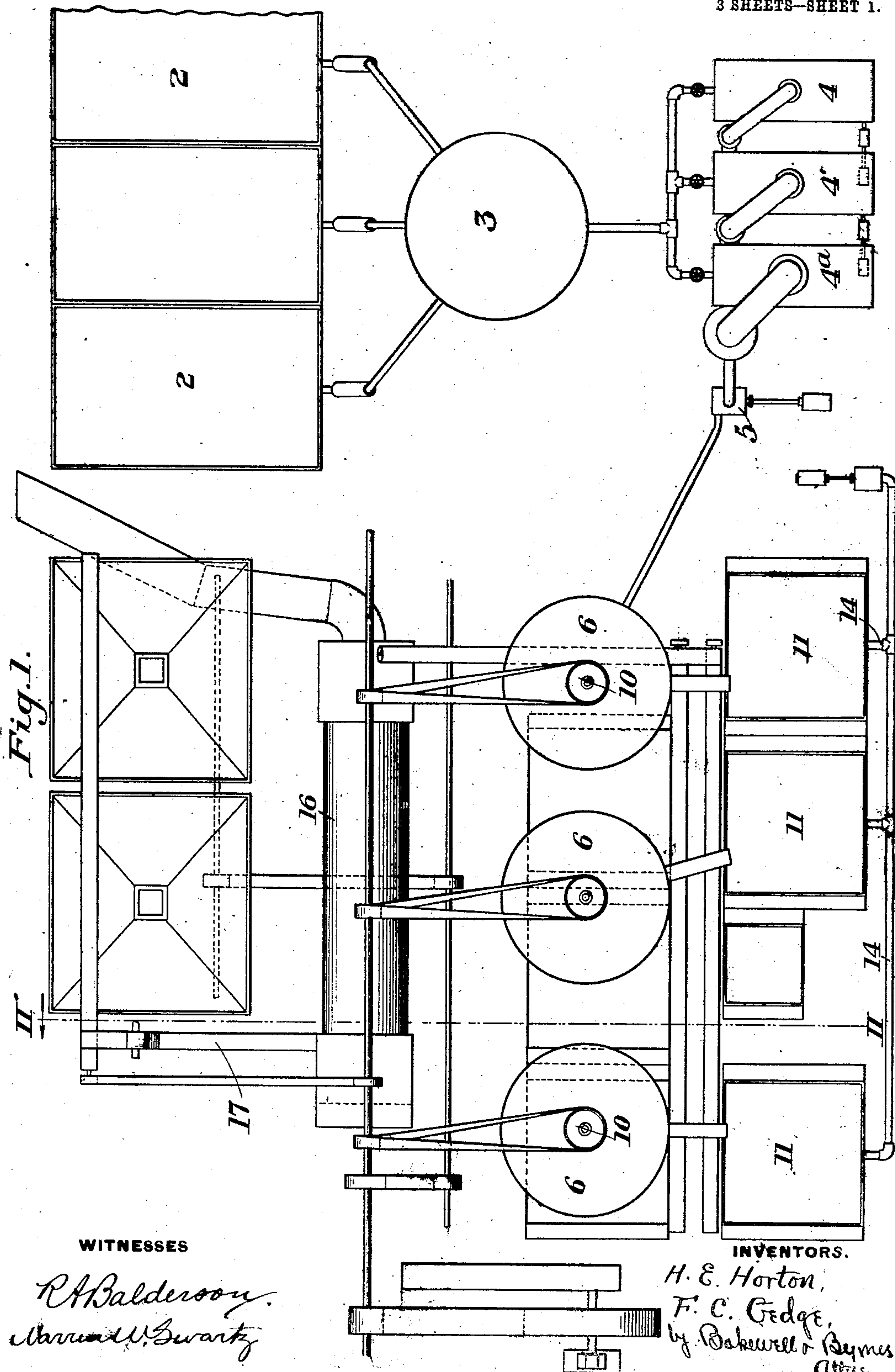
No. 863,279.

PATENTED AUG. 13, 1907.

H. E. HORTON & F. C. GEDGE.  
METHOD OF RECOVERING SULFATE CRYSTALS.

APPLICATION FILED MAY 11, 1906.

3 SHEETS—SHEET 1.



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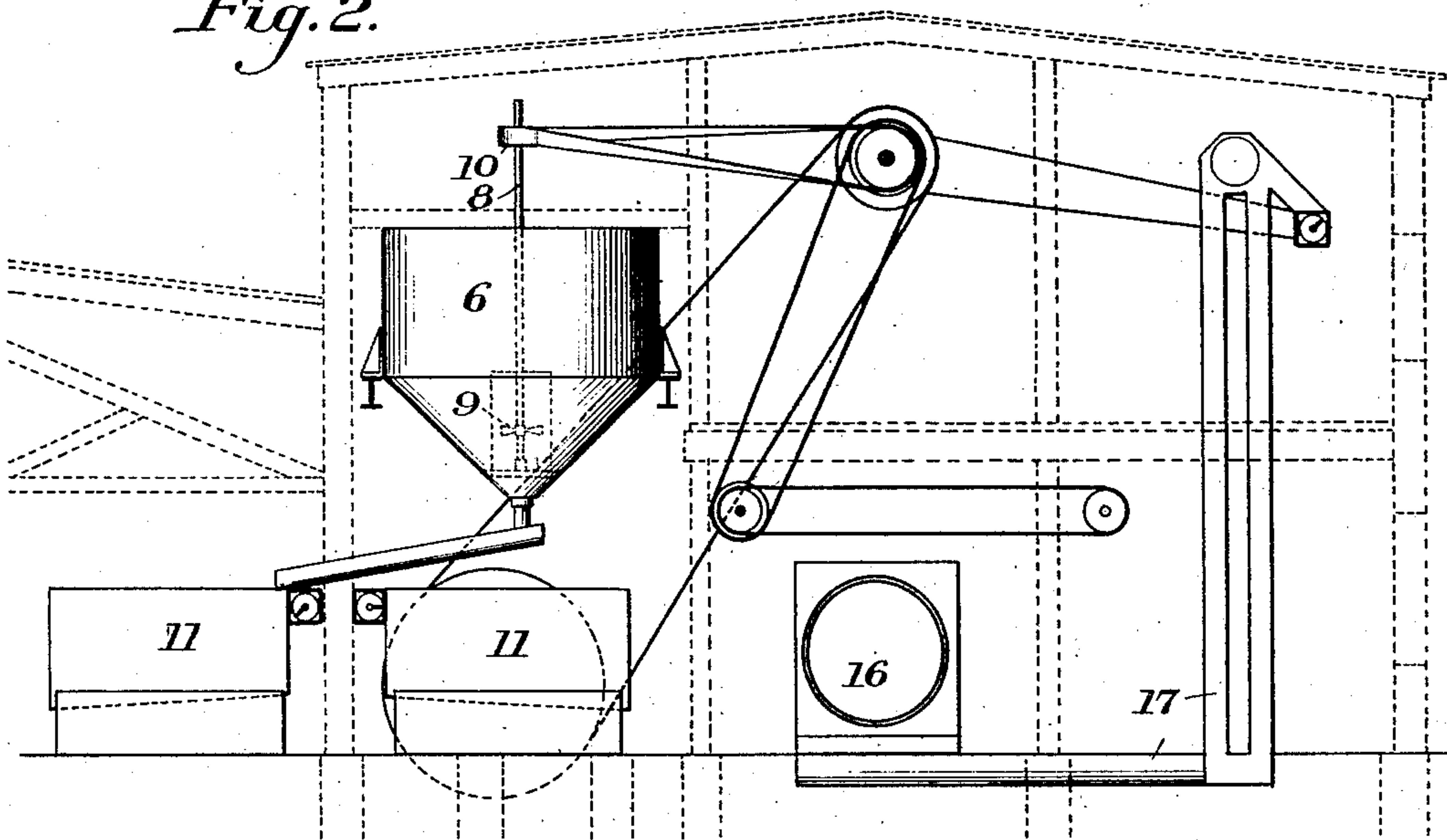
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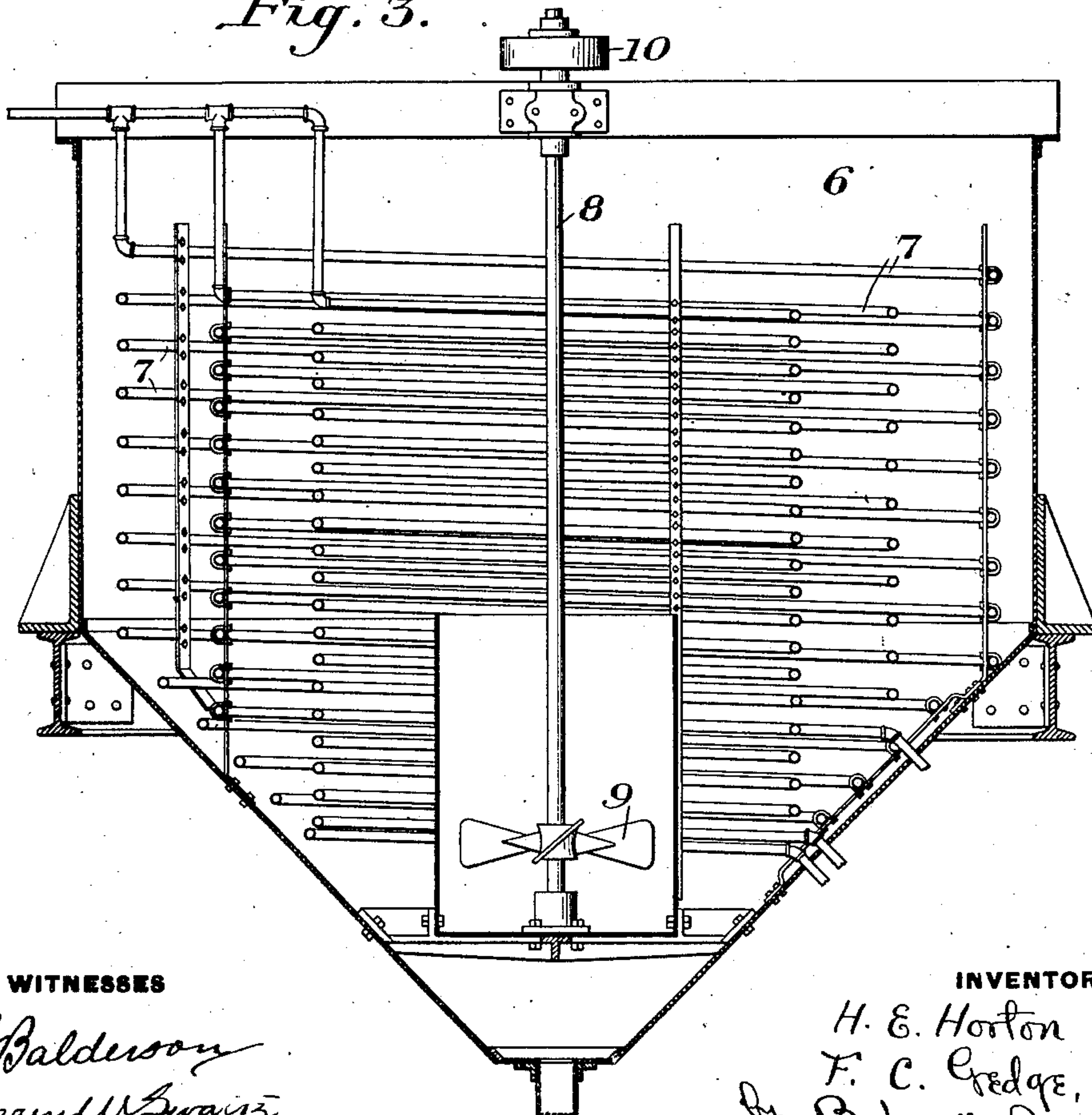
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3 SHEETS—SHEET 2.

*Fig. 2.*



*Fig. 3.*



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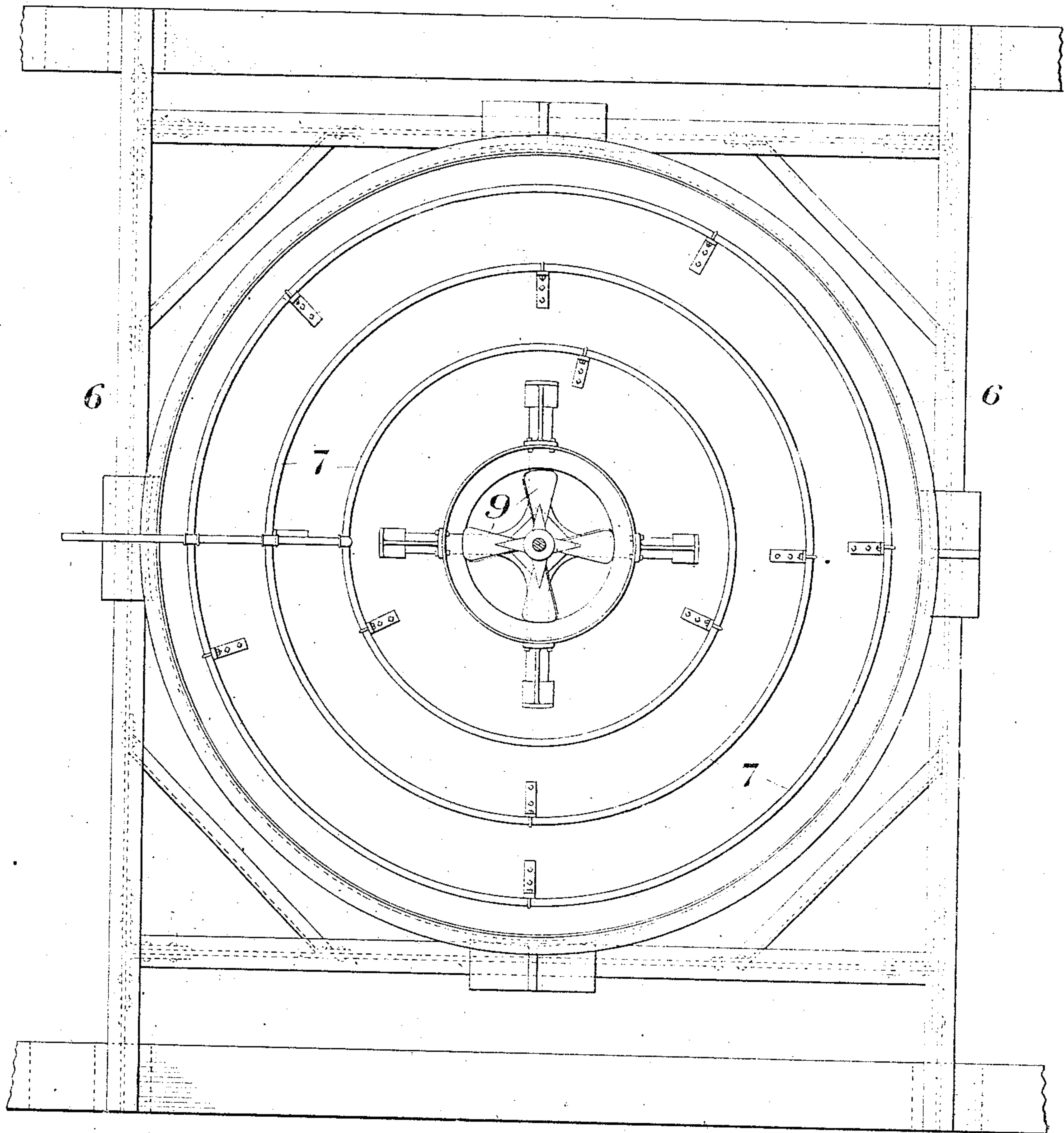
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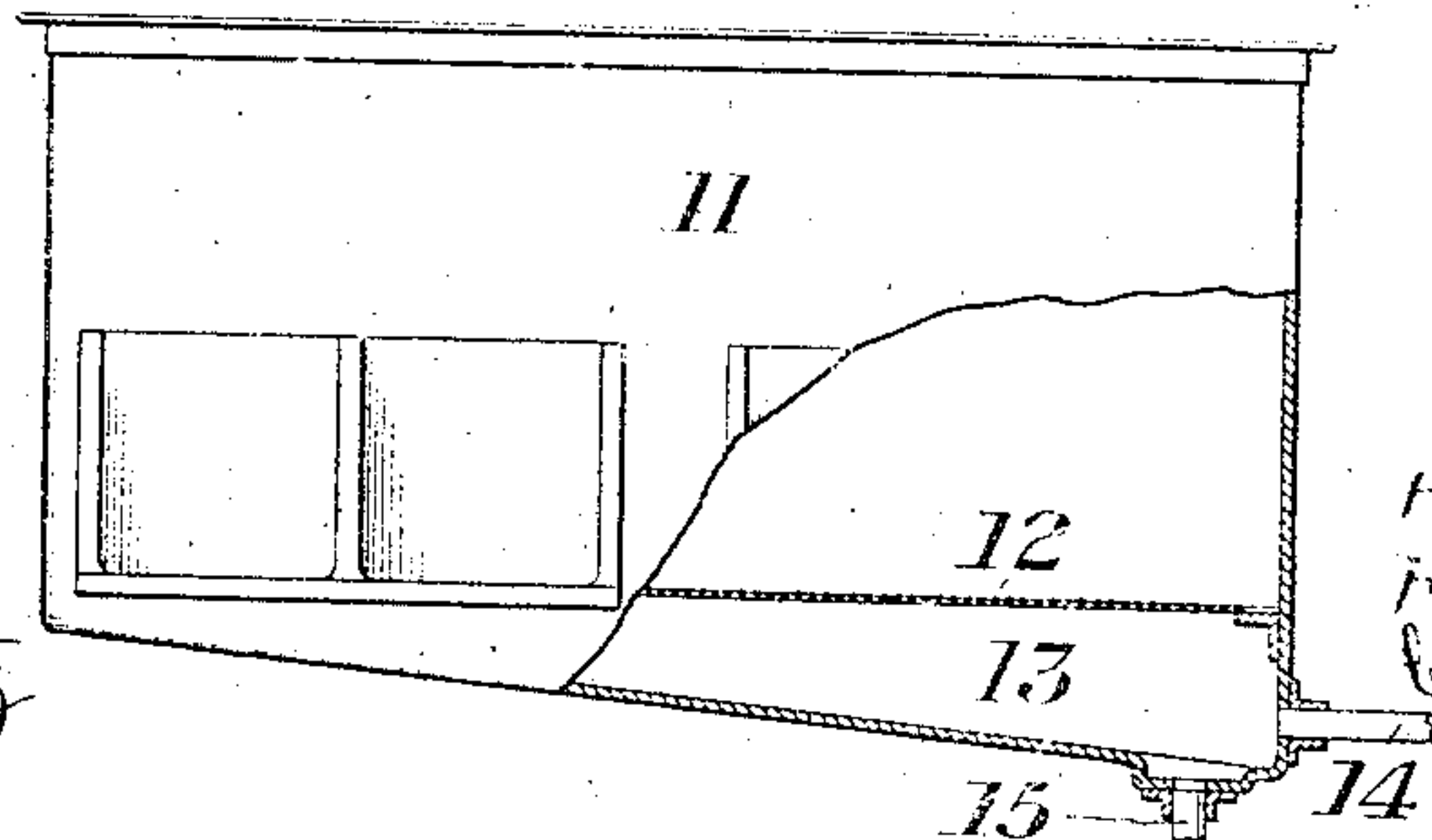
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3 SHEETS—SHEET 3.

*Fig. 4.*



*Fig. 5.*



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# UNITED STATES PATENT OFFICE.

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## METHOD OF RECOVERING SULFATE CRYSTALS.

No. 863,279.

Specification of Letters Patent.

Patented Aug. 13, 1907.

Application filed May 11, 1906. Serial No. 316,297.

*To all whom it may concern:*

Be it known that we, HORACE E. HORTON, of Waukegan, county of Lake, State of Illinois, and FREDERICK C. GEDGE, of Evanston, county of Cook, State of Illinois, have invented a new and useful Method of Recovering Sulfate Crystals, of which the following is a specification, reference being had to the accompanying drawing, in which—

Figure 1 is a plan view of apparatus suitable for the practice of our invention; Fig. 2 is a vertical sectional view on the line 2, 2 of Fig. 1; Fig. 3 is a vertical section on a larger scale, showing the crystallizing tank; Fig. 4 is a plan view of Fig. 3, and Fig. 5 is a sectional view showing the means which we employ for separating the water from the crystals.

The purpose of our invention is to provide means for recovering sulfate of iron from the liquor which results from the pickling of iron wire, rods, etc. in an acid bath.

Our invention provides means by which a valuable commercial product can be obtained in this way, and also provides for obtaining the product in the form of small crystals or granules of substantially uniform size and substantially uniform purity and much drier than the larger crystals heretofore obtained so that the process is available for crystals to be used in the arts for the purification of water and for other purposes for which such sulfate is employed.

The invention is also applicable, with suitable changes in the materials of which the apparatus is made, to the recovery of sulfate of copper from the liquor resulting from the pickling of copper wire, rods, etc.

In the practice of our invention, we take the sulfuric acid liquor from the pickling vats and deliver it to suitable storage tanks 2 in which it is neutralized by the addition of waste iron wire or other iron or steel scrap. These tanks are preferably provided with steam-coils to heat the solution somewhat in order to prevent premature crystallizing in the tanks.

When the solution becomes neutralized, that is to say, when the free acid has been substantially satisfied by the iron with which it comes in contact, the solution is removed by pumps into a settling-tank 3 in which the mechanical impurities are settled by gravity, and from this settling-tank the solution is drawn successively into vacuum evaporators 4, 4', 4'', known in the art as multiple effects. A suitable number of these effects may be employed. Each one contains tubes preferably made of copper, and the effects are provided with vacuum-producing apparatus.

The dilute neutralized liquor is supplied to the first effect, where it comes into contact with and circulates

around the copper tubes, which are filled with a heating medium, and in this effect a part of the water passes off in the form of vapor. The liquor then passes to the second effect, in which it is further evaporated, and in the same manner to the other effect or effects, where it is finally concentrated. The steam supplied to the coils of the first effect may be exhaust steam or live steam from an outside source, and the steam for the heating-coils of the second effect may be obtained by utilizing the vapor generated by the boiling of the liquor in the first effect. In the same manner the vapor from the second effect may be passed to the coils of the third effect, and so on, the vapors from the liquor in the final effect being passed into a suitable condenser equipped with a vacuum pump.

From the final effect, the concentrated liquor is delivered by a pump 5 to the crystallizing-tanks 6, 6. As shown in Figs. 2, 3 and 4, these crystallizing-tanks are provided with coils of pipe 7 through which cold water or brine is caused to circulate so as to cool the liquor contained therein. The crystallizing-tank has a rotating shaft 8 provided with an agitating-screw 9, rotated by suitable driving gear 10 which causes the liquor to circulate up and down, bringing the hot liquor into contact with the cooling surfaces of the pipes and thereby hastening the crystallization. By reason of this combined agitation and cooling the crystals form in small granules in the liquor, and when the crystallization has proceeded to the desired point, the contents of the tanks are withdrawn by gravity into vacuum dripping tanks 11. These are preferably constructed as shown in Fig. 5, each tank having at the bottom a screen 12, beneath which is a space 13 communicating by a pipe 14 with a vacuum pump and having a liquor-discharging outlet 15. The suction produced by the vacuum pump aids in removing the liquor from the crystals in the tank 11. The mother liquor passes out through an outlet 15 and the crystals remain on the screen 12. These crystals are then removed, preferably by shoveling, into a drier, preferably a rotary drier, 16, in which the crystals are thoroughly and evenly dried. From thence the crystals are discharged to a conveyer 17 by which they may be removed to storage bins or disposed of in any convenient way.

The process above described is simple and extremely efficient. The crystals which are produced are of convenient size; they contain a minimum of moisture, and are of great value in the arts.

The temperature to which we prefer to bring the solution in the crystallizing-tanks is preferably about fifty degrees Fahrenheit, but this may be varied as desired. Roughly speaking, the amount of salt which is deposited from the solution in the crystallizing-tanks will be



governed by the temperature of the solution, as the solubility of the salt in water varies at different temperatures.

Instead of bringing the neutralized liquor into contact with a metal, we may employ any suitable metallic hydrate, oxid or carbonate in any suitable manner.

Within the scope of our invention as stated in the claims, changes may be made in the apparatus and in the steps of the process. Some of the steps of the process may be employed without others, or may be employed in other combinations, since

What we claim is:

1. A process of obtaining in a continuous manner, a metallic sulfate in the form of small crystals, which consists in at least partially neutralizing an acid solution of a metallic sulfate by adding the same metal as the base of the sulfate, transferring the solution to a separate vessel where the impurities separate, then withdrawing and evaporating the solution in a vacuum, to the crystallizing point, then transferring it to a crystallizing tank and simultaneously agitating and artificially cooling the solution in successive tanks; substantially as described.

2. A process of obtaining in a continuous manner a crystalline metallic sulfate, which consists in at least partially neutralizing an acid solution of a metallic sulfate by adding the same metal as the base of the sulfate, maintaining such solution at a temperature above the crystallizing point, then evaporating the solution in a vacuum to the crystallizing point, and then transferring it to a crys-

tallizing tank and simultaneously agitating and artificially cooling the solution in the tank to produce crystals of small granular form; substantially as described.

3. A process of obtaining in a continuous manner a crystalline metallic sulfate, which consists in at least partially neutralizing an acid solution of a metallic sulfate by adding the same metal as the base of the sulfate, continuously withdrawing the treated solution and concentrating it to the crystallizing point in successive steps under vacuum while maintaining a substantially constant current in the solution, then transferring the concentrated solution continuously to crystallizing tanks and successively and simultaneously agitating and artificially cooling the liquor in the successive crystallizing tanks to which it is supplied; substantially as described.

4. A process of obtaining in a continuous manner a crystalline metallic sulfate, which consists in at least partially neutralizing an acid solution of a metallic sulfate by adding the same metals as the base of the sulfate, continuously withdrawing and hot concentrating the solution under vacuum, transferring the concentrated solution to crystallizing tanks in a substantially continuous manner, and simultaneously artificially cooling and agitating the hot solution successively in the successive crystallizing tanks to which it is fed; substantially as described.

In testimony whereof, we have hereunto set our hands.

HORACE E. HORTON.  
FREDERICK C. GEDGE.

Witnesses:

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