

B. JUNQUERA.
MANUFACTURE OF CONCENTRATED SOLUTIONS.
APPLICATION FILED JULY 2, 1919.

1,436,809.

Patented Nov. 28, 1922.

3 SHEETS—SHEET 1.

Fig. 1.

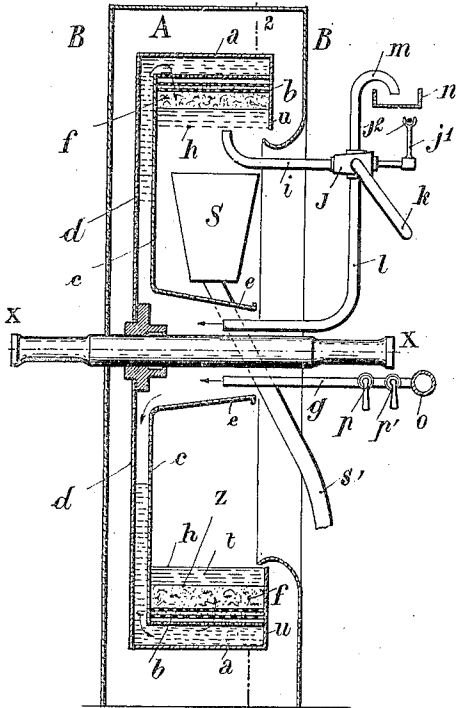


Fig. 2.

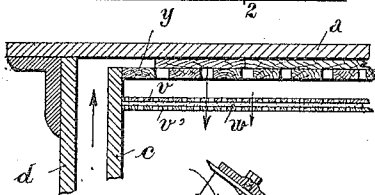
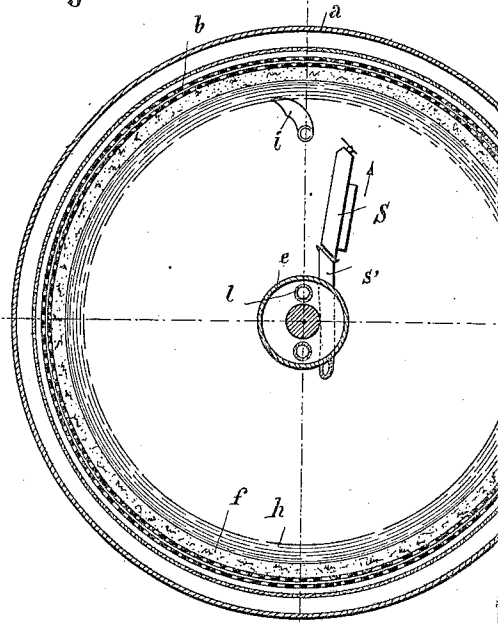


Fig. 5.

Fig. 6.

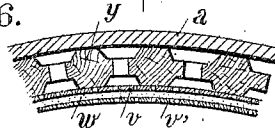


Fig. 7.

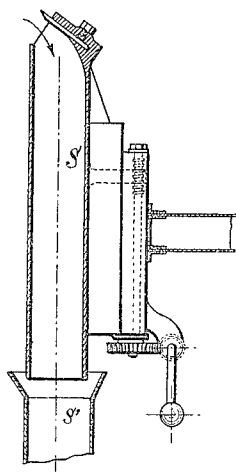
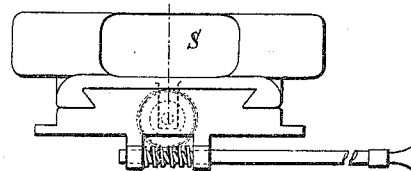


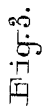
Fig. 8.



Inventor:
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B. JUNQUERA.
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3 SHEETS—SHEET 2.



 Inventor:
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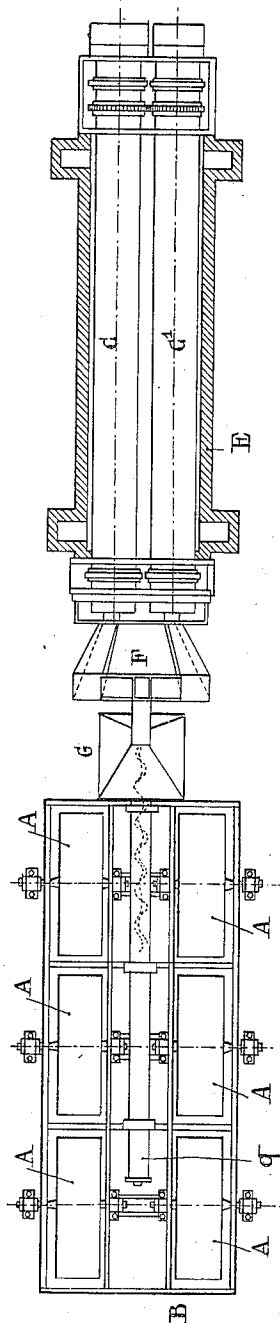
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3 SHEETS—SHEET 3.

Fig. 4.



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UNITED STATES PATENT OFFICE.

BUENAVENTURA JUNQUERA, OF OVIEDO, SPAIN.

MANUFACTURE OF CONCENTRATED SOLUTIONS.

Application filed July 2, 1919. Serial No. 308,219.

To all whom it may concern:

Be it known that I, BUENAVENTURA JUNQUERA, subject of the King of Spain, residing at Oviedo, Spain, have invented certain new and useful Improvements in the Manufacture of Concentrated Solutions, of which the following is a specification.

This invention relates to an improved process and apparatus for the manufacture of concentrated solutions.

It is old to use solution processes in which the current of the solvent passes through the substances to be treated with accelerated speed because of the action of centrifugal force. However, in these old processes the current of the solvent circulates in the same direction as the centrifugal force so that the particles of the material to be treated, being subjected to two forces acting in the same direction, are liable to be carried away by the current of the solvent. Therefore, only substances in large pieces can be practically treated by currents of a solvent at a low speed, if the solid particles are not to be carried off by the current of the solvent.

Now, my invention consists in a new solution process whereby the said drawbacks are avoided. This process essentially consists in causing a rapid and continuous current of a solvent to circulate through the finely pulverized material while the same is held against a false perforated bottom by the action of centrifugal force, the current of the solvent circulating through the material to be treated in a direction opposite to that of the centrifugal force, the resultant speed of circulation thus being accelerated by the action of the centrifugal force.

This process can moreover be improved by a preliminary heating to a temperature suitable to the material to be treated and to the solvent. This temperature is then maintained as constant as possible by heating jackets surrounding the apparatus wherein the solution is effected. One can still further improve the process by providing a systematic circulation of the current of the solvent and the solid material in a series of elementary apparatuses, the arrangement being such that the current of the liquid solvent, when it has reached the state of purity, enters the almost exhausted layers of the material to be treated, where-

upon, after having dissolved more and more of the soluble elements, it passes through elementary apparatuses charged with richer material to be treated.

This process is more particularly applicable to the extraction of the nitrates or chlorides of sodium or potassium, or of any soluble salts; it may be also employed for concentrating any solutions and even for extracting metals such as gold, silver, copper, and the like by the wet process.

In order to rapidly dissolve in any suitable liquid any soluble substance two essential conditions must be fulfilled to wit: firstly, the material to be dissolved must be crushed or reduced to as fine a condition as possible, and secondly, the liquid solvent must circulate through the material to be dissolved, continuously and rapidly.

These conditions, which are easily fulfilled in a laboratory, for small quantities of products to be dissolved, are not so easily fulfilled in dissolving substances on an industrial scale for instance the separation of the salts of potassium, sodium and the like, as well as the metals, gold, silver, copper and the like from their natural ores.

The processes at present employed for dissolving the salts and even metals, are attended by the difficulty, if fine crushing be adopted of causing the liquid solvents to circulate through these masses. Even if coarsely crushed the material contains large quantities of fine dust which completely obstruct the passage of the liquid, if the circulation takes place vertically downwards, and the circulation is very irregular and incomplete when taking place vertically upwards; further, the final separation of the liquids from these large pasty masses is very difficult and costly, in spite of the use of filter presses, vacuum filters, or other like apparatus.

The apparatus and process forming the object of this invention obviate all these difficulties and enable any suitable solutions to be obtained at the most suitable degree of concentration in each case, with regularity and in a practically continuous manner.

By the present invention the material is subjected to a series of successive and rapid washings in a series of rotary apparatus, with liquids at high or low temperatures by an ascending circulation (i. e., a circulation

directed from the periphery of the apparatus toward its axis of rotation, or, in other words, in a direction contrary to the direction of the centrifugal force,) and utilizing centrifugal force on the one hand for maintaining the material in the apparatus in a condition of fixation and absolute homogeneity (whether the material be coarsely or finely crushed) and on the other hand for producing by this same centrifugal force, a powerful circulating and dissolving liquid current throughout the pulverized mass.

The apparatus on this system of dissolution is composed of a series of groups of rotary washing apparatus, mounted in pairs on each axis of rotation so as to form a continuous circuit. The number of groups is determined in each case by the greater or lesser number of washings necessary for the complete exhaustion of the material treated.

The invention is more particularly described with reference to the accompanying drawings, in which:

Figure 1 is a section through the apparatus in a vertical plane passing through its axis of rotation: Figure 2 is a section through the apparatus in a vertical plane at right angles to the one of Figure 1 as indicated by the line 2—2 in said figure: Figure 3 is a detail view of the filter in section in a vertical plane at right angles to that of Figure 1; Figure 4 is a detail view of the filter in section in a vertical plane at right angles to that in Figure 2; Figure 5 is a detail view in section of the discharge arrangement in a plane perpendicular to the axis of rotation of the turbine A; Figure 6 is a plan view of the same as seen from below; Figure 7 is a diagrammatic view partially in section of an installation for the discharge of the material treated; Figure 8 is a plan view thereof.

The elementary apparatus (unit) as shown diagrammatically in Figures 1 and 2 comprises principally a rotatable turbine A, a fixed casing B and a system of pipes for the distribution of the liquid solvent.

The turbine A principally comprises a circular casing *d* of sheet iron secured at its center on a rotary shaft *x x*, a circular casing *u* concentric with the said shaft and of the same outer diameter as the circular casing *d*; a cylindrical sheet iron connecting *d* and *u*, a circular annular plate *c* concentric with the shaft *x x*, secured to *u* and provided with a conical extension *e*, a false perforated cylindrical bottom *b*, disposed between the plates *u* and *c*, the false bottom consisting of two metal cloths *v v'* (Figures 5 and 6) of fine mesh of about one to two millimeters, between which there is interposed a good quality fabric cloth *w*, a wooden frame *y* bearing against the inner wall of plate *a* and supporting the metal cloths

v v' and the fabric *w* and provided with openings for the circulation of the liquid.

The casing B which surrounds the rotary turbine A is stationary and rests on the base plate of the apparatus. On the side adjacent the plate *u*, it has large openings so as to provide space for the pipe system above referred to.

The arrangement for the removal of the material treated shown in detail in Figures 3 and 4 principally consists in a hopper G provided in its upper portion with an adjustable scraper and capable of sliding by means of a dove-tailed connection on a fixed frame. A crank is provided operating an endless screw, and a pinion secured to a threaded shaft with which said hopper engages enables the hopper to be vertically displaced. A tube *s'* is also provided for receiving the material treated as it falls from said hopper.

The pipe system principally consists in a pipe *g* for admitting the liquid to the apparatus, said pipe being connected by a valve *p* and a regulating valve *p'* to a general distributing conduit *o* and entering the conical extension *e*; a collecting pipe *i* for the liquids connected to a four-way cock *j*, a return pipe *l* also entering the conical extension *e*; a pipe *m* for the removal of the concentrated liquid in the channel *n*; a pipe *k* for establishing communication between the four-way cock *j* of one apparatus and the conical extension *e* of the next apparatus. The four-way cock *j* is controlled by a crank *j'* connected by a link *j''* to some motor, e. g., a servo-motor.

The operation of the apparatus is as follows:

If this apparatus be assumed to be revolving on its shaft *x—x* at a suitable speed and a pulverized material be poured on to the bottom *b* in a uniform and continuous manner, such pulverized material under the action of centrifugal force will be retained and uniformly distributed over this bottom *b*.

The material which is thus distributed over the bottom *b* (which generally has a density of about 2) by the effect of the rotation and centrifugal force may be considered as having a changed condition and its density will virtually increase in a proportion corresponding to this centrifugal force; its virtual density will pass from 2 to 5, 10 and even beyond this. In this condition a liquid ascending current may be caused to circulate through this material as strongly and rapidly as may be desired, without this material being able to be carried away with it; that is to say, that the liquid which passes through it will absorb the soluble substances without carrying away the solid matters in suspension.

The material will fill the bottom of the

apparatus to a certain height this mass being indicated at *f* whereupon a liquid is introduced or poured into the cone *e* by means of a tube *g*, and this liquid, projected by centrifugal force, will fill the double bottom *a—b* passing through the double walls *c—d*. The difference of level in the space between *c, d*, and the fixed level *h* above the perforated false bottom *b*, as well as centrifugal force, will force the liquid to pass through the pulverized mass deposited at *f* and, in its passage, this liquid will carry away in solution the whole or part of the soluble materials which this mass contains.

15 An analogous circulation of liquids would result, if one arranged two stationary communicating vessels at different heights, the lower vessel being provided with a perforated false bottom supporting a material to be dissolved, while the higher vessel contains the liquid solvent, the communicating pipe between the two vessels terminating below the perforated false bottom in the lower vessel. But in the apparatus according to the present invention, under the action of centrifugal force, the difference of level between the liquid level in the space *e, d*, and the fixed level *h* of the liquid above the material at *f* will be multiplied by 5, 10, 20 or more according to the speed of rotation, which will produce differences of pressure of 1, 2 or more atmospheres. In other words, the liquid will easily pass through the mass at *f* because the current circulates from the periphery of the apparatus towards its axis, i. e., from the perforated false bottom *b* towards the surface *h* and it will pass through rapidly because of the high pressure produced by the centrifugal force without this current, however rapid, being able to carry with it the smallest particle of solid materials owing to the assumed high density determined by the centrifugal force and which with the most frequently used materials and at the usual speed of rotation exceeds two or three times the actual density of lead, because the centrifugal force applied to the solid particles opposes their being carried away by the solvent. The liquid rising to the surface of the mass *f* will be clear and limpid, it will only contain soluble substances which it will have dissolved out.

If liquid be continued to be poured into the cone *e*, this liquid will continue to pass through the mass *f*, and will rise above its level *z* and fill the space *t* between the material to be treated and the level *h*. The liquid will continue to fill the space *t* up to the level *h* of a ladle collector *i* which will collect all the liquid exceeding this maximum fixed level.

The collector *i* has a four-way cock *j* which feeds the pipes *k l m*. The pipe *l* discharges the liquid into the cone *e* and produces a local circulation; the pipe *m*

which can advantageously be closed by an auxiliary valve not shown serving for discharging the concentrated liquid which is conveyed to any desired place by a channel *n*.

When the material to be treated is exhausted and freed of its soluble particles which it contained it is discharged by means of a hopper arrangement and a discharge pipe *s'*. For this purpose it is sufficient to turn the controlling crank of this hopper so as to elevate it to the interior of the rotary drum A so that its upper end with its scraper engages the mass *f* of the exhausted material. As the drum A continues to rotate, the exhausted material is collected by the hopper and discharged.

In practice the normal operation of dissolution, washing, or filtration takes place in a series of apparatus similar to the one described. The pipe *k* discharges the liquid into the cone *e* of the apparatus next in the series. In the second apparatus this liquid passes through a fresh layer of material and then passes into a third, and so forth, according to the number of washings held necessary for the concentration or exhaustion of the material to be treated.

An apparatus for the treatment of materials containing soluble substances and intended for dissolving sodium nitrate, may be composed for instance of four groups of two washing drums A.

Each apparatus is constructed as described in connection with the elementary apparatus shown in Figures 1 to 4. A screw conveyor *q* brings the material to be treated to the drums A by way of a pipe *r*.

In the dissolution of nitrates which should generally be effected by aid of heat and at a temperature above 100° C., the washing apparatus must be protected against cooling; for this purpose they are enclosed in a chamber B (Figs. 3 and 4) in the interior of which hot gas circulates (generally residual gases of combustion from apparatus for heating water and the material to be treated.)

For this heating, a rotary furnace shown diagrammatically in Figs. 3 and 4, may be used. This furnace is composed of two groups of revolving cylinders C D and C' D' contained in a heating chamber E and comprising hoppers for feeding the material; an elevator F and a distributing hopper G suitably arranged.

It is evident from the foregoing that with this kind of apparatus, raw material may be treated which contains substances to be dissolved (salts or metals), as finely pulverized as may be necessary to rapidly attain complete exhaustion and the removal of all the soluble parts.

The liquid solvent passes successively from one apparatus to the next; it may also be caused to circulate several times in each

apparatus so that it becomes saturated to the desired degree; these two periods of work are carried out simultaneously in all the apparatus because all the cocks j may be operated simultaneously and mechanically. To this end, the link or rod j^2 connects the operating arms j' of all the cocks j of the same series, and the rod j^2 is moved by one and the same motor.

10 The first period of circulation is operated in the two end apparatus of the series; one of them is being charged with fresh material and the next being emptied of exhausted material. The extraction of the liquid corresponding to the charging of the apparatus is also done during this period and in the apparatus preceding the one which is being charged.

The duration of these periods depends on the greater or lesser solubility of the product treated, but in general and more particularly for the salts of sodium and potassium, the time may be fixed at about fifteen minutes for each period, say half an hour interval between the loading of one apparatus and the following one.

The forty-eight charges may be made in twenty four hours, which will make for an installation having a capacity of from 10,000 to 12,000 kilograms for each apparatus, a total of 450 to 500 tons of raw material treated per twenty-four hours.

If the number of washing apparatus be eight for example (six only are shown in Figs. 3 and 4) there will be seven apparatus at work washing and each charge will be subjected to the circulating action of the liquid solvent during seven half hours or three hours and a half.

40 The connections between the elementary apparatus can be arranged in such a way that the work of solution is methodical, i. e., that the liquid solvent arrives in a state of purity in the elementary apparatus charged with material to be treated almost exhausted and then after having dissolved more and more soluble material circulates in the apparatus charged with richer and richer material to be treated.

50 The volume of liquid which each apparatus contains is approximately 3,400 litres, and seeing that this liquid circulates every

fifteen minutes, that will give 47,600 litres of liquid which will have circulated at different concentrations through the 12,000 55 kilogrammes of material or the charge of an apparatus and that for three hours and a half.

The process which has just been described may be employed effectively for the dissolution of all metals contained in rocks or ores, in which case a liquid must be employed which is capable of absorbing these metals when in solution.

For the extraction of gold and silver, 65 cyanide is employed.

As this process allows of numerous vigorous and rapid washings with constantly renewed liquids and with a dissolving power which is greater in proportion as the richness of the material diminishes, a complete exhaustion of the ore is thus obtained in a comparatively short time.

Similarly, very poor copper ore (1% or less) may be treated by this process by means of chloride solutions clear and limpid liquids being obtained which may be subjected directly to electrolysis.

In all these cases, by this process, a regular concentration to a desired degree is reached, which in many cases is indispensable for the ultimate operations of crystallization or electrolysis.

What I claim is:

The process of separating by solution the soluble products from the solids in which they are contained, which consists in causing a rapid continuous current of a solvent to circulate through the finely pulverized solids maintained applied against a false perforated bottom by the action of centrifugal force so that the current of the solvent circulates through the material to be treated in a direction opposite to that of the centrifugal force, the speed of the resultant circulation being accelerated by the action of the centrifugal force.

In testimony whereof I have affixed my signature in presence of two witnesses.

BUENAVENTURA JUNQUERA.

Witnesses:

LOUIS GARDET,
LOUIS FAUTRAT.