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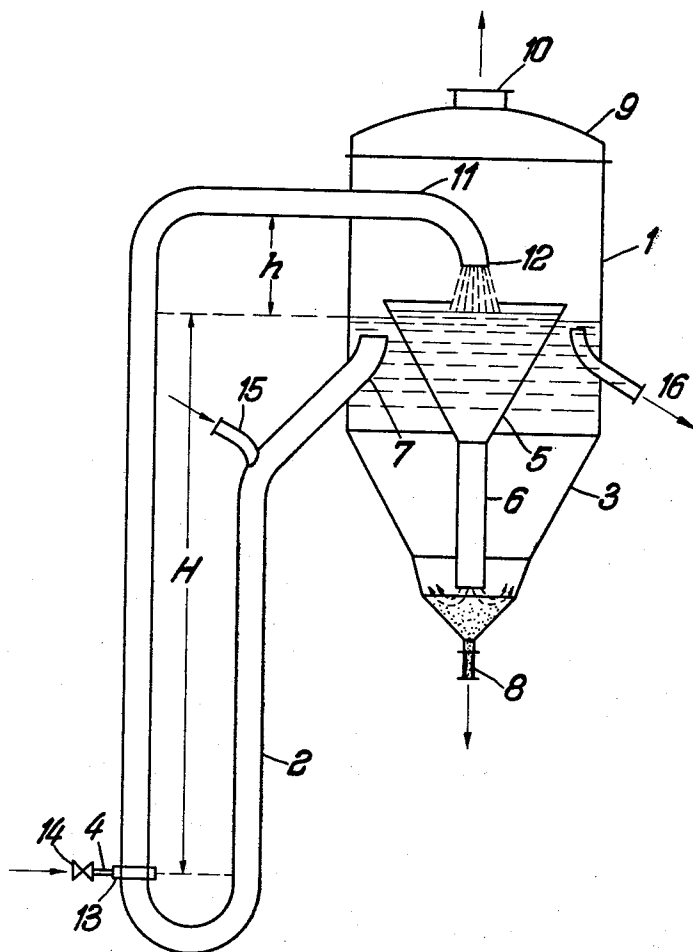
3,137,544

CRYSTALLIZING APPARATUS AND METHOD OF OPERATING THE SAME

Filed May 20, 1958

2 Sheets-Sheet 1

Fig. 1



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2 Sheets-Sheet 2

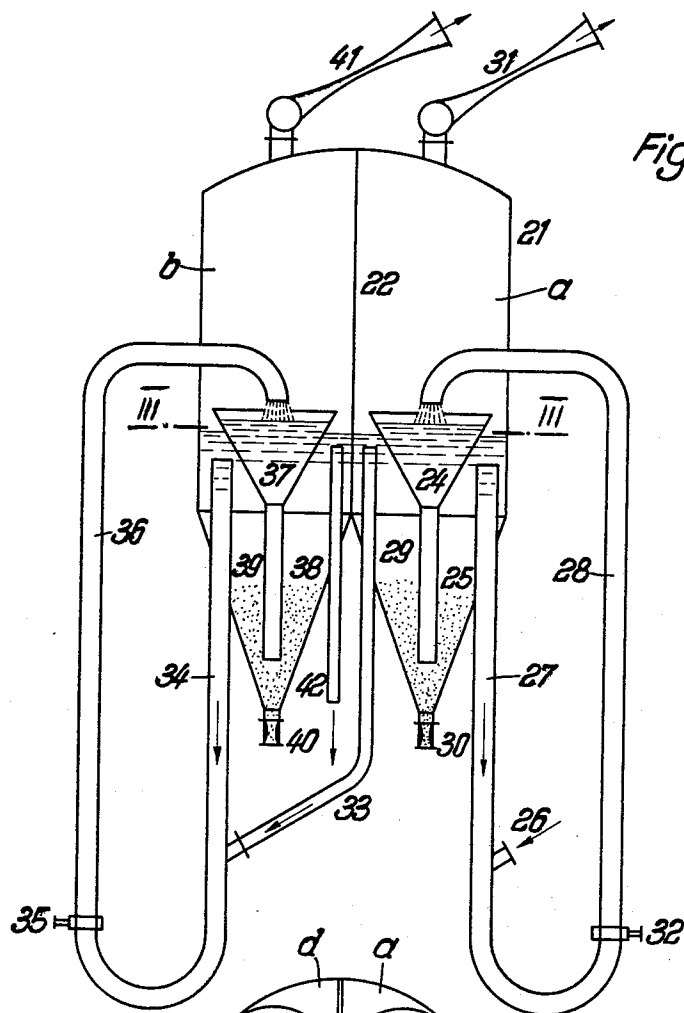


Fig. 2

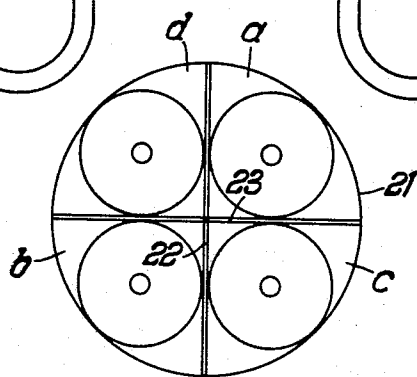


Fig. 3

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3,137,544

CRYSTALLIZING APPARATUS AND METHOD OF OPERATING THE SAME

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When crystallizing solids from solutions, it is mostly desired to obtain crystals which are as far as possible of uniform size and granular in form. If a salt-bearing liquid is cooled by evaporation under pressure with the aid of a vacuum, a very fine grained crystal separates out on the saturation point of the solution being considerably exceeded. Within a small range of temperature below the saturation point, a metastable state prevails in which the crystalline growth takes place. The temperature range of this metastable state is not uniform in extent. It generally becomes greater the higher the molecular weight of the dissolved substance is. The metastable range is considerably reduced by impurities in the solution which cause increased and premature crystal nucleus formation.

If therefore it is desired to crystallize out a product as far as possible in the form of uniform coarse grains from a solution cooled under vacuum, it must be seen that a sufficient quantity of crystals which act as seeding crystals is present in suspension in the solution in uniform distribution, but that the speed of nucleus formation also remains slow, so that the existing crystals and the crystal seeds can grow to the desired grain size.

Therefore, it follows that the construction of the vacuum crystallizing apparatus should be such that the solution is preferably cooled in stages, the reduction in temperature in each stage amounting, for example, to 3° C. or 4° C. The crystals separating out become larger and more uniform if the cooling in each stage is only about 1° C. In the case of vacuum stages connected in series, however, a large number of evaporators will be necessary resulting in a very expensive plant.

Vacuum crystallizing apparatus are known in which crystals can be grown by circulating a large quantity of liquid by means of a pump and only cooling slightly in a vacuum, so that no crystals separate out but only slight oversaturation takes place. This oversaturated solution or a portion thereof is then passed through a layer of already formed crystals mostly arranged outside the actual cooling apparatus, in which layer the existing newly formed crystals grow thereby reducing the oversaturation.

Evaporators with external heating systems have likewise been used for crystallizing salts out of solutions. From the evaporator provided with a conical bottom the separated salts are drawn off through a sluice into a centrifuge in which the liquid carried by the salt is centrifuged off. Underneath a built-in unit of conical shape arranged in the cylindrical portion of the evaporator and connected at its upper edge to the wall of the cylinder, cooled solution from the evaporator is continually drawn off and conducted into the heating element. It returns therefrom re-heated, through a conduit which terminates just above the liquid level in the evaporator. The vapors forming in the evaporator are condensed in a condenser. These evaporators are not suitable for crystal culture and the salt coming from the centrifuge contains a great quantity of moisture.

According to another method of procedure, solution in the range of metastable saturation from a common evaporator was distributed to several crystallizers of different sizes, from which it was then returned into the evaporator.

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The crystal growth took place due to the fact that a mixture of solution and crystals was maintained in turbulent movement in each of the crystallizers and that the crystals, on attaining a certain size, separated out of the solution under the action of the gyrating movement. They then passed into the next larger crystallizer in which they were developed by the same system. Finally the crystals of the desired grain size were drawn off from the largest crystallizer.

These circulating evaporators are, however, open to the objection that the relatively large pumps which operate against the vacuum are relatively expensive apparatus.

The invention relates to crystallizing plants which are equipped with an evaporating zone and a crystallization zone and means by which the solution from which uniform and coarse grained crystals are to be obtained, is maintained in circulation through the evaporating zone and the crystallization zone. At the same time inlets and outlets are provided by which the hot initial solution is introduced into the device with the conical bottom and by means of which the cooled solution, the vapors produced by self-evaporation and the separated crystals are conducted off from the crystallization process.

The invention consists in that funnel-shaped built-in units are provided in the evaporator vessel substantially concentric therewith. These built-in units are arranged with clearance from the wall of the evaporating vessel. The apparatus according to the invention also comprises a conduit serving for guiding the circulation of the solution, which conduit first descends from the liquid level in the evaporator outside the evaporator and then rises so that its extremity is just above the built-in units. The lowest point of the conduit is located 10 to 20 times the height of lift of the liquid over the liquid level in the evaporator below the level of the liquid in the evaporator and an air feed is arranged at the bottom of the conduit so that the circulating motion of the solution through the conduit takes place on the known air lift principle.

The units built-in the evaporator according to the invention which catch the solution introduced into the evaporator, prevent the liquid scattered by self-evaporation from splashing the walls of the evaporator as such splashes would quickly lead to the formation of large deposits and cause continual operating trouble. At the same time, owing to the fact that the circulation conduit for the solution according to the invention first descends to a low level and then rises, the advantage is derived that very little air is used for the circulation of the solution, so that the vapors produced during the evaporation can be used for heating purposes or, if the evaporation is carried out at low temperatures, these vapors can be condensed in standard condensers with the usual air extracting devices. The combination of the evaporating zone and the crystallizing zone in a vessel having a conical bottom enables very good crystal growth to be obtained, because the liquid which is guided by the built-in units from the circulation conduit right into the lower part of the evaporator, in rising in the conical bottom maintains the fine crystal grains in suspension until they grow into crystals which can sink to the bottom against the flow of the liquid.

According to a special form of construction according to the invention, the growth of crystals when crystallizing salts or similar solid substances out of solutions maintained in circulation in known manner by vacuum evaporation and crystallization, is carried out in several stages in such a manner that the solution is conducted successively through all the stages and the freshly introduced solution is mixed with the solution in circulation in each stage by vacuum evaporation and crystallization. By mixing the

fresh solution introduced in each stage with the solution in circulation a temperature is imparted to the mixture composed of circulating liquid and fresh solution, which lies within the range of metastable saturation in the particular stage and, by the vacuum cooling of the liquid mixture, a portion of the heat is extracted which the freshly introduced solution has introduced and which corresponds to the drop in temperature between the two working temperatures of two successive stages.

Owing to the fact that the crystallization is carried out with circulating liquid in this manner in several stages, the formation of new crystal nuclei during the cooling operation is prevented to a great extent. It has been found that the formation of fresh seed in practice is proportional to the quantity of liquid which is maintained in circulation during a unit of time. If the cooling in one or several stages is to the limit of the available drop in temperature, a relatively large quantity of liquid must be kept in circulation so that the mixture composed of circulating liquid and freshly introduced liquid is brought within the range of metastable saturation. If, on the other hand, the treatment in stages is carried out in such a manner that in each stage the cooling is effected only to a fraction of the drop in temperature available, the necessary quantity of liquid circulating in each stage is reduced in inverse proportion to the number of stages. In the case of three stages, only $\frac{1}{3}$ and in the case of five stages only $\frac{1}{5}$ of the quantity of circulating liquid necessary for single stage crystallization, is required. Now if the solution passes successively through all the circulation stages, the seed crystals newly formed in the preceding stage grow in the following stage and consequently a larger and more uniform grain is obtained than by the process in which, when also working in stages, the solution is cooled in each stage by the whole of the drop in temperature available.

For example, if, according to the invention, the crystallization of the salt or the like from the saturated or nearly saturated running solution is carried out in three or four or even more stages, the temperature of which drops by about 4 to 7° C., e.g. 5° C. from stage to stage in the direction of liquid flow, before entering each stage the inflowing solution entering the liquid circulation of the solution present in the particular stage which is quantitatively measured so that the mixture composed of the hot freshly introduced solution and the cold solution of the particular stage, assumes a temperature which is about 1 to 2° C. higher than the temperature of the liquid content in this stage. The mixture is introduced into the evaporating space of the particular stage where by partial evaporation its temperature is again reduced by 1 to 2° C., that is, by the amount by which the temperature of the mixture exceeds that of the stage. The liquid mixture is now passed into this stage through an accumulation of salt in which the oversaturation of the solution caused by the cooling is released in such a manner that the salt crystals of the accumulation of salt grow. The accumulation is maintained in suspension in such a manner that the grains of salt only settle when they have attained a certain size. The water vapor generated in the stage is fed into the condensation by means of injectors, for example into a common condenser, so as to keep the steam consumption of the plant as low as possible.

This embodiment of the invention possesses the further advantage that the steam consumption is far lower than in the known processes and that apparatus can be used which is relatively inexpensive. The stages can be produced by dividing a cylindrical vessel into chambers by radial partition walls, through which chambers the solution to be treated is fed successively. The chambers can be so equipped with the aid of conical bottom parts and funnel-shaped built-in units from which the circulating liquid is conducted by a pipe into the lower part of the chamber, so that a suitable quantity of salt is maintained in suspension in the lower part in which the crystallization takes place. Heavy grain separated out can, when it has attained

the desired size, be continuously or periodically drawn off at the bottom of the chamber by means of known devices.

It has also been discovered that the crystal growth according to the invention can be still further improved by conducting the liquid in circulation by means of gases which are introduced into the uptake of the liquid circulation. By this means, the apparatus necessary for carrying out the method according to the invention, is simplified still further; moreover the crystals produced retain their original shape, that is the edges and corners of the crystals are not rubbed off as is the case, due to impact and friction, when other propelling devices such as rotary pumps are used.

The invention is illustrated diagrammatically and by way of example in the accompanying drawings, in which:

FIG. 1 is a vertical section through a single stage apparatus according to the invention;

FIG. 2 shows a multi-stage apparatus, also in vertical section, and

FIG. 3 is a section on line III—III of FIG. 2.

The apparatus illustrated in FIG. 1 comprises an evaporator 1 with vapor outlet and vacuum connection 10, a discharge device 8 for removing the formed crystals, a funnel-shaped built-in unit 5, 6 acting as guide surface and a circulating device 2. The evaporator 1 consists of a cylindrical upper part and a lower part 3 conically tapering, if desired in steps, and which leads into the discharge sluice 8 for removing the crystals. The evaporator is closed at its upper end by means of a cover 9 which is preferably domed and in which the connection piece 10 for producing the vacuum and drawing off the vapors is arranged preferably centrally.

The funnel-shaped guide surface is preferably concentrically mounted in the evaporator with its upper aperture higher than the liquid level adjustable in the evaporator and with a pipe 6 extending from its lower aperture to within close proximity of the discharge sluice 8.

The circulating device consists of a pipe 2, the main portion of which is bent in U-shape, one end of this pipe being bent out laterally and passing through the wall of the evaporator. The mouth of this pipe length 7 which is located within the evaporator lies below the liquid level in the evaporator. The other end is bent over horizontally and passes through the evaporator wall, terminating in a downwardly directed pipe section 11 over the centre of the funnel-shaped built-in unit 5. The mouth 12 of the pipe section 11 may be of nozzle shape.

A throttle air admission inlet 4 is provided at the lower end of the long arm of the U-shaped pipe. It may, for example, be so shaped that the pipe wall is provided with holes in its circumferential circle and the pipe surrounded at this point with a sleeve or jacket 13 carrying a pipe connection piece with throttle valve 14. An air feed pipe may be also arranged in the jacket of the U-shaped pipe and provided on its outer end with a throttle valve and at its inner end with one or more nozzles. The air feed may, however, also be effected by means of a pipe passing through the wall of the U-shaped pipe and bent in the direction of flow in the interior of the pipe. This air-admission pipe is fitted with a throttle valve on its end outside the pipe and with an outlet nozzle on its inner end.

The solution to be crystallized is introduced into the short arm of the U-shaped pipe through a lateral connection piece 15. Excess mother lye is removed from the evaporator vessel through the lateral connection 16, the inner end of which is located near the level of the liquid.

So as not to hamper the maintenance of the vacuum and to restrict as far as possible the dilution by air of the vapors generated during the evaporation, the quantity of air entering through the valve 14 should be kept at a minimum.

This is attained according to the invention by making the feed conduit as long as possible in relation to the lift. The lift is the distance h from the liquid level to the high-

est point of the feed conduit. The liquid circulation which is attainable by a given quantity of air is so much better the lower the air admission is below the liquid level. This distance is designated by H on the drawing. According to the invention the ratio $h:H$ is taken at 1:10 to 1:20. The air before entering the ring conduit is preferably brought to saturation temperature corresponding to the temperature of the circulated liquid.

The mouth of the circulation conduit is arranged so high above the funnel-shaped built-in unit 5 that the liquid in the distribution existing on this path is only cooled about 1°C .

The liquid reaches the narrowest zone of the conical lower part 3 of the evaporating vessel through the funnel 5 and the pipe 6 extending therefrom.

Owing to the suction effect at the commencement of the circulation conduit 2 an upward flow sets in in the vessel outside the funnel-shaped built-in unit 5. The liquid passing out from the pipe 6 is thus deflected upwards and then rises at a speed which becomes slower as the cross-section of the conical lower part of the vessel 3 increases.

At the relatively high speed of flow in the neighborhood of the lower aperture of the pipe 6 crystals which have attained a minimum grain size settle. Smaller crystals are carried along by the current and, according to their size, are held in suspension in the zones of the larger cross-section of the lower part of the vessel. They there grow to a grain size which enables their sedimentation.

The deposited crystals with uniformly well and regularly grown grain size collect over the discharge 8 and form a bed of crystals from which a portion is continually withdrawn by the discharging device mixed with as much mother lye as is advantageous for the subsequent separation of the crystals in a centrifuge.

In the conical lower part of the vessel a sedimentation zone forms above the bed of crystals and is followed by a clearing or settling zone in the cylindrical part of the vessel. The solution entering the circulation is generally clear and practically free from crystals.

The sedimentation zone in the lower part of the vessel may, as shown in the drawing, be constructed with another conically tapering part as the part directly adjoining the cylindrical wall. If this conical lower part is made slimmer, speeds diminishing more slowly along a longer path will be obtained than with a cone which is not so steep. As a result the crystals remain in suspension along a longer path of travel in the slimmer lower part. Therefore it is possible to allow the oversaturated lye to act for a longer or shorter period on the crystals held in suspension. In the case of saline solutions which have a lesser tendency to oversaturation it is therefore possible to keep the sedimentation zone small and in the case of saline solutions with a high degree of oversaturation to make this zone longer.

As, however, the circulation takes place without pulsation and sudden deflections, such as always occur when using rotary pumps, it is of no significance if smaller crystals also enter the circulation conduit.

To avoid deposits of crystals within the apparatus, which would finally result in the formation of turbulent flow, the funnel-shaped built-in 5, 6, the discharge sluice 8 and the pipe length 7 of the circulating conduit 2 located within the vessel, can be made of soft rubber.

Mother lye is drawn off continually or periodically through the discharge 16 in order to avoid the concentration of impurities in the solution in circulation. This drawn off lye can, preferably after undergoing an intermediate treatment in a cyclone separator or the like, be again further concentrated and crystallized in one or several crystallizing apparatus according to the invention.

The apparatus illustrated in FIGS. 2 and 3 consists of a cylindrical wall 21 which is divided, for example, into

four chambers by partition walls 22 and 23. Each of these chambers accommodates a funnel-shaped built-in unit with a cylindrical extension projecting into the funnel-shaped lower part of the chamber.

The solution from which the salt or other solid substances are to be crystallized out, is passed successively through all the chambers and in each chamber is cooled by partial evaporation under vacuum. For example, the solution passes in saturated or nearly saturated state through the conduit 26 into the liquid circulation in the conduits 27, 28. Liquid having a temperature corresponding to that of the vacuum prevailing in the chamber *a* flows through the conduit 27 in downward direction out of the chamber *a*. It returns, mixed with the warmer solution fed through the conduit 26, via the conduit 28 into the chamber *a* dropping into the funnel-shaped built-in unit 24. The quantity of liquid led off from the chamber *a* through the conduit 27 amounts to a multiple of the quantity of the liquid flowing in at 26. It is so calculated that the mixture composed of cold circulating liquid and the warmer freshly arrived liquid is given a temperature which is higher by about 1 to 2°C . than the temperature maintained by the vacuum evaporation in the liquid content of the chamber *a*. On entering the chamber *a* the liquid is again cooled about 1 to 2°C . to the temperature of the liquid in this chamber. During the cooling process no salt crystals separate out. The crystals first form in the lower funnel-shaped part of the chamber *a* into which the solution flowing out at 28 oversaturated by cooling, passes through the cylindrical extension 25. When the crystals have attained a sufficiently large size they settle in the part 29 of the chamber *a* and can be withdrawn continually or periodically, for example, through a throttle element 30. The vacuum in the chamber *a* is produced by a steam jet apparatus 31 which feeds into a steam condenser (not shown). The circulation of the liquids through the conduits 27 and 28 is effected with the aid of known conveying arrangements, for example, by means of a small quantity of air which is introduced into the conduit 28 through a ring-shaped nozzle 32.

From the chamber *a* the liquid which has cooled therein through part evaporation in a vacuum to a temperature which is, for example, 5°C . lower than that of the liquid arriving through 26, passes through the conduit 33 into the liquid circulation 34, 36 of the next chamber *b* which is maintained in the same way as the liquid circulation through the conduits 27 and 28 coordinated to the chamber *a*, by known conveying arrangements such as the introduction of air by means of a device, preferably a ring nozzle 35. The liquid circulation and the guiding of the liquid into and through the chamber *b* are carried out in the same way as in the case of chamber *a*, in that the liquid coming out of the conduit 33 cooled to a temperature about 1 to 2°C . above the temperature prevailing in the chamber *b* by mixing with a many times greater quantity of liquid flowing through the conduit 34, drops through 36 into the funnel 37 and is guided downwards by the cylindrical extension 38 thereof into the conical part of the chamber *b* where the crystallizing out from the liquid cooled by about 1 to 2°C . on entering the chamber *b*, takes place. The large crystals obtained by this crystal growth in chamber 39 can be led off through a throttle element 40 which can be constructed and operated, for example, in a similar manner to the throttle element 30 in the chamber *a*. The vacuum is produced in the chamber *b* by steam jet apparatus 41 which, just like the steam jet 31, conducts the steam to a steam condenser (not shown). The liquid passing out of the chamber *b* is led off through the conduit 42 to be further treated in the chambers *c* and *d* in the same way as in the chambers *a* and *b*, these chambers *c* and *d* being equipped with built-in units, operated and evacuated by steam jet apparatus into steam condensers, just as in the case of the chambers *a* and *b*. From the last chamber *d*—there

can also be more than four chambers connected in series—the liquid passes out of the crystallizing apparatus at the desired final temperature. By cooling in stages in the series-connected chambers *a, b, c, d* a particularly uniform crystal grain is obtained. The steam consumption of the jet apparatus effecting the cooling, is also low because these have to overcome a considerably smaller drop in temperature than in the known crystallizers. If the series-connected chambers are formed by dividing a cylindrical space by partition walls, the partition walls can be thin because the difference in pressure between the individual stages is slight. The chambers are preferably insulated from each other, which can be effected, for example, by making the partitions 22, 23 double walled when the space between the walls is either filled with heat insulating substances or maintained under a good vacuum. By the heat insulation of the chambers from one another, deposits of crystal on the partition walls are prevented.

Example 1

From a sodium sulphate solution with 23% Na_2SO_4 and 10% H_2SO_4 , 300 g. of Glauber's salt per litre of the solution were to be obtained in coarse crystalline form. The equipment illustrated in FIG. 1 of the drawing was used for the crystallization. The solution to be treated was introduced at 15 at a temperature of 15° C., mixed in flowing through the pipes 2 and 11 with the circulating solution flowing out of the vessel 1 at 7 and passed out at 12 at a temperature of 13° C. The solution in the evaporator had a temperature of 10° C. The precipitated salt was led off from the crystallizing evaporator with a quantity of the solution and fed into a centrifuge. It left the centrifuge with a moisture content of only 3%. The evaporator was operated under a vacuum of 6.5 torr. With a diameter of 1.6 m. and a cylindrical height of 2 m. it produced about 1000 kgs. Glauber's salt per hour. The vapors which left the evaporator through the vapor discharge 10 had such a small air content that the condenser (not shown) in which the vapors were condensed, could be operated with ordinary ventilation and supplementary means were not required for eliminating the transporting air introduced at the bottom of conduit 11.

Example 2

A potassium chloride solution saturated at 95° C. was cooled to 45° C. in a crystallizing apparatus according to the invention. In the apparatus 2.5 m. in diameter operating with a solution flow of 15 cb. m. per hour, about 1300 kgs. of salt were obtained per hour with a grain

	Percent
Exceeding 0.5 mm.-----	About 90
Between 0.2 and 0.5 mm.-----	About 5 to 7
Less than 0.2 mm.-----	About 3 to 5

The salt was in the lower part of the crystallizer according to the invention in loose state and could easily be removed without choking. On the other hand in a known 24-stage vacuum cooling plant a crystallate grain was obtained of which

About 9% exceeded 0.5 mm.,
About 69% was between 0.2 and 0.5 mm., and
About 22% less than 0.2 mm.

Example 3

A potassium chloride solution saturated at about 90° C. was cooled in one stage in a crystallizer which was equipped with liquid circulation. From this circulation lye was extracted at the bottom of the crystallizing vessel and fed onto the surface of the liquid in the crystallizing vessel by air which was introduced into the uptake of the circulation conduit. The flow of lye amounted to 15 cb.

m./hr.; whereby potassium chloride, partly also sodium chloride, crystallized out with the following sieve analysis:

	Percent
Exceeding 1 mm.-----	7
From 0.75 to 1 mm.-----	5
From 0.2 to 0.75 mm.-----	73
Less than 0.2 mm.-----	15

The same solution was worked in two stages at in all the same temperature range and the drop of temperature according to the invention maintained in the stages; as a result, from the same solution with the same lye flow a crystal grain of the following grain sizes was obtained:

	Percent
Exceeding 1 mm.-----	10
From 0.75 to 1 mm.-----	20
From 0.2 to 0.75 mm.-----	60
Less than 0.2 mm.-----	10

The mean grain size which in the one stage treatment was in the neighbourhood of 0.45 mm., therefore attained the surprising value of almost 0.7 mm. by the two-stage treatment according to the invention.

I claim:

1. An apparatus for obtaining large and uniform crystals from solutions which comprises a closed vessel having an inwardly and downwardly tapering bottom portion, said bottom portion terminating in a crystal discharge outlet, said vessel being divided into an upper evaporation zone and a lower crystallization zone, means defining a funnel-shaped chamber being open at both its upper and lower ends completely disposed within said vessel and spaced from the sides thereof at both said upper and lower ends, the upper end of said funnel-shaped chamber extending into said evaporation zone and the lower end of said funnel-shaped chamber extending into said crystallization zone, said vessel being capable of containing a solution at a level at least below the upper end of said funnel-shaped chamber, means for removing vapors from the top of said vessel, circulating means for withdrawing solution from said vessel to a lower level and thence to an upper level and terminating in a discharge opening above and spaced from said funnel-shaped chamber, said lower level being below the level of said solution a distance of from about 10 to 20 times the distance said upper level is above the level of said solution, an air inlet means connected to said circulating means at said lower level, and means for adding fresh solution to said apparatus.

2. An apparatus for obtaining large and uniform crystals from solutions which comprises a closed vessel having an inwardly and downwardly tapering bottom portion, said bottom portion terminating in a crystal discharge outlet, said vessel being divided into an upper evaporation zone and a lower crystallization zone, means defining a funnel-shaped chamber being open at both its upper and lower ends completely disposed within said vessel and spaced from the sides thereof at both said upper and lower ends, the upper end of said funnel-shaped chamber extending into said evaporation zone and the lower end of said funnel-shaped chamber extending into said crystallization zone, said vessel being capable of containing a solution at a level at least below the upper end of said funnel-shaped chamber, means for removing vapors from the top of said vessel, means for removing excess solution from said vessel, circulating means including inlet means disposed within said vessel below the level of said solution for withdrawing solution from the upper portion of the crystallizing zone of said vessel, exterior conduit means for passing said solution first to a lower level and thence to an upper level and outlet means disposed directly above and spaced from said upper end of said funnel-shaped chamber for discharging said solution thereinto, said lower level being below the level of said solution a distance of from 10 to 20 times the distance said upper level is above the level of said solution, an adjust-

able air inlet means connected to said conduit means at said lower level, and means for adding fresh solution to said conduit means.

3. An apparatus according to claim 1 wherein the bottom portion of said closed vessel tapers inwardly and downwardly in stages to define corresponding settling zones.

4. An apparatus according to claim 1 wherein a plurality of said vessels are arranged in series, said apparatus having conduit means for passing solution from each vessel to the next subsequent vessel.

5. An apparatus for obtaining large and uniform crystals from solutions which comprises a plurality of closed vessels each having an inwardly and downwardly tapering bottom portion, each said bottom portion terminating in a crystal discharge outlet, said vessels being divided into an upper evaporation zone and a lower crystallization zone, means defining a funnel-shaped chamber being open at both its upper and lower ends completely disposed within each said vessel and spaced from the sides thereof at both said upper and lower ends, the upper end of said funnel-shaped chambers extending into the corresponding evaporation zone and the lower end of each said funnel-shaped chamber extending into the corresponding crystallization zone, each said vessel being capable of containing a solution at a level at least below the upper end of the corresponding funnel-shaped chamber, means for removing vapors from the top of each said vessel, circulating means for withdrawing solution from each said vessel to a lower level and thence to an upper level and terminating in a discharge opening above and spaced from the corresponding funnel-shaped chamber, each said lower level being below the level of said solution a distance of from about 10 to 20 times the distance each said upper level is above the level of the corresponding solution, an air inlet means connected to each said conduit means at said lower level, and means for conducting solution from the upper portions of the crystallizing zone of each said vessel to the circulating means of the next.

6. In an apparatus for obtaining large and uniform crystals from supersaturated solutions capable of yielding such crystals, including a closed vessel having an inwardly and downwardly tapering bottom portion terminating in a crystal discharge outlet and being divided into an upper evaporation zone and a lower crystallization zone, means for removing vapors from the top of said vessel and means for adding fresh solution to, for removing excess solution from and for circulating at least a

portion of the solution by withdrawing said solution from and returning the same to said vessel, the improvement which comprises providing completely disposed within said closed vessel means defining a funnel-shaped chamber open at both its upper and lower ends and spaced from the sides thereof at both said upper and lower ends, the upper end of said funnel-shaped chamber extending into said upper evaporation zone and the lower end of said funnel-shaped chamber extending into said lower crystallization zone, said vessel being capable of containing a solution at a level at least below the upper end of said funnel-shaped chamber, said means for circulating including inlet means therefore disposed within said vessel below the level of said solution for withdrawing solution from the upper portion of the crystallization zone of said vessel, exterior conduit means for passing said solution first to a lower level and thence to an upper level, outlet means disposed directly above and spaced from said upper end of said funnel-shaped chamber for discharging said solution downwardly thereinto, said lower level being below the level of said solution a distance of from 10 to 20 times the distance said upper level is above the level of said solution and an adjustable air inlet means connected to said exterior conduit means at said lower level.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,137,544

June 16, 1964

Karl Ebner

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 7, line 29, for "an" read -- and --; column 9, line 36, for "conduit" read -- circulating --; line 38, for "portions" read -- portion --; column 10, line 13, for "therefore" read -- therefor --.

Signed and sealed this 27th day of October 1964.

(SEAL)

Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents