Nov. 28, 1961 THOMAS C. CHENG 3,010,805

CLASSIFYING CRYSTALLIZERS AND SYSTEMS

Filed Sept. 24, 1959 4 Sheets-Sheet 1

FIG. 1

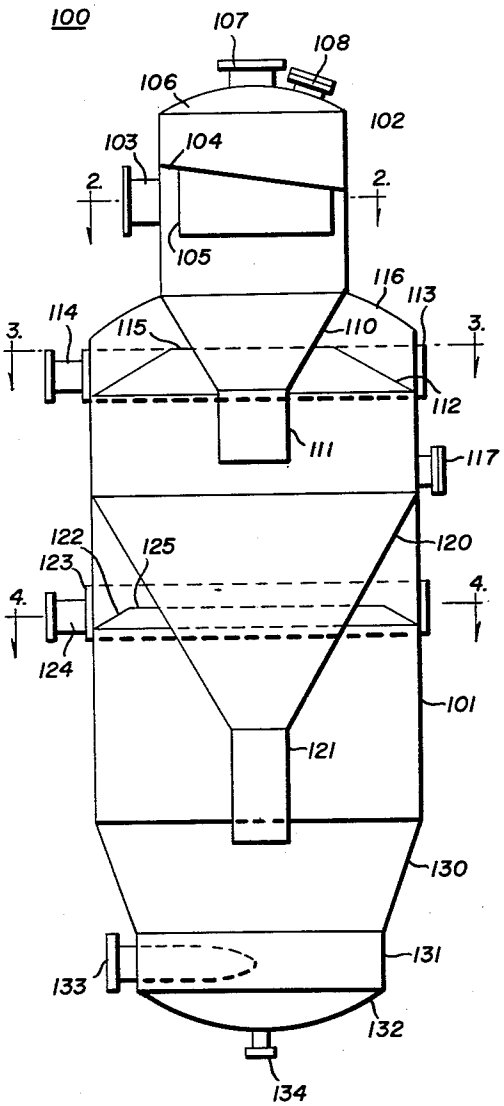


FIG. 2

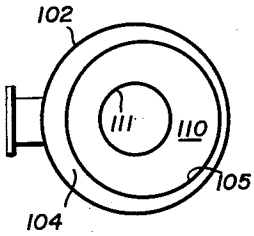


FIG. 3

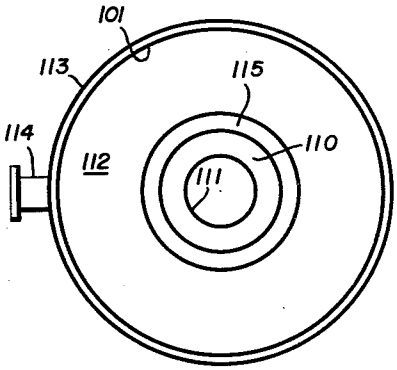
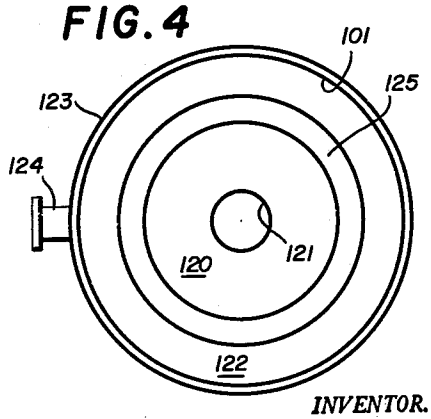


FIG. 4



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FIG. 6

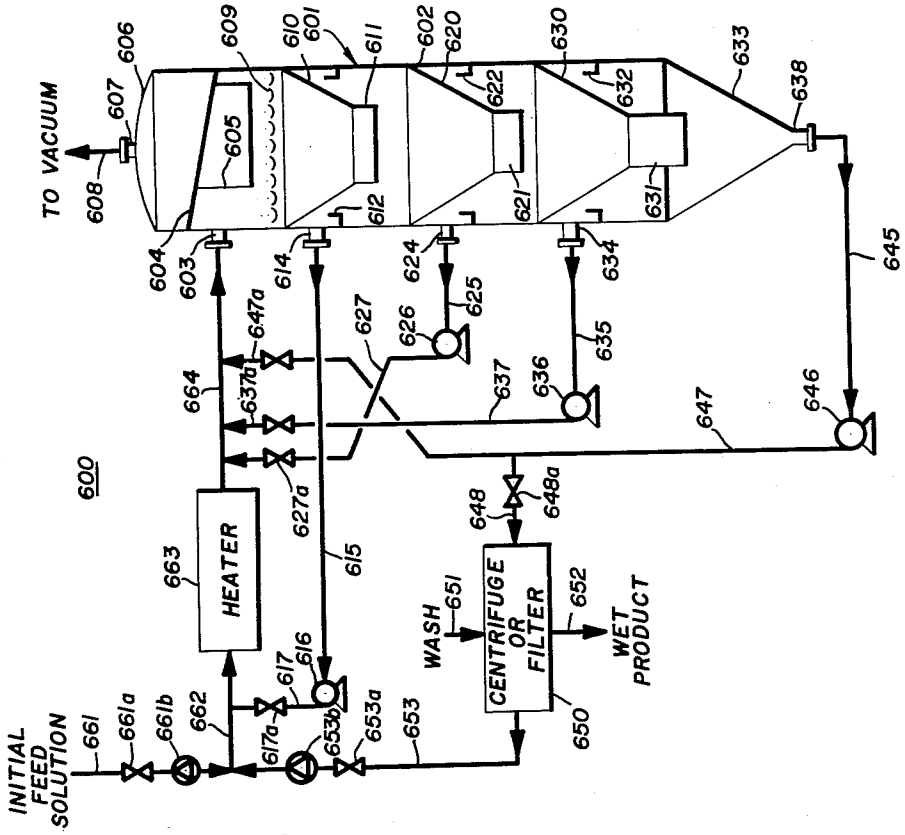
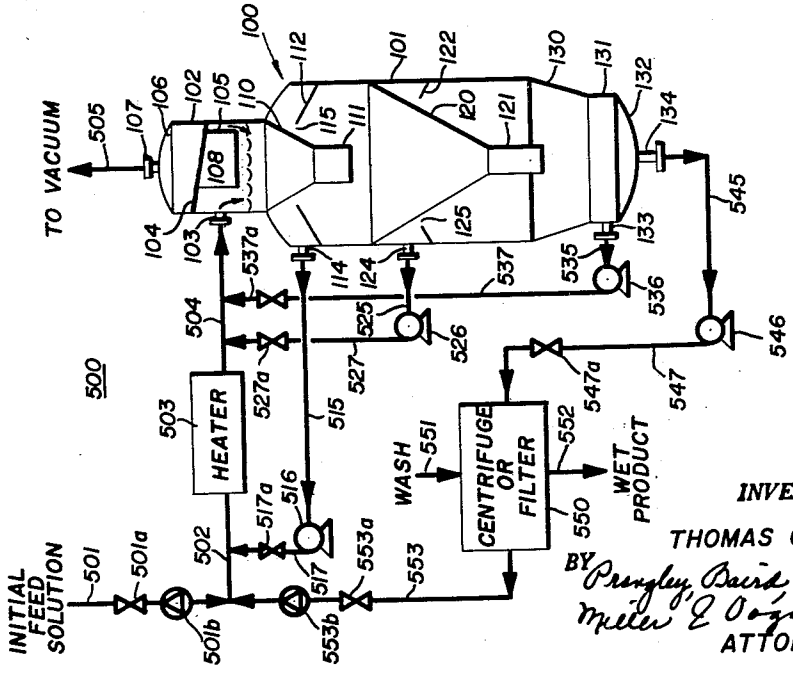


FIG. 5



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FIG. 7

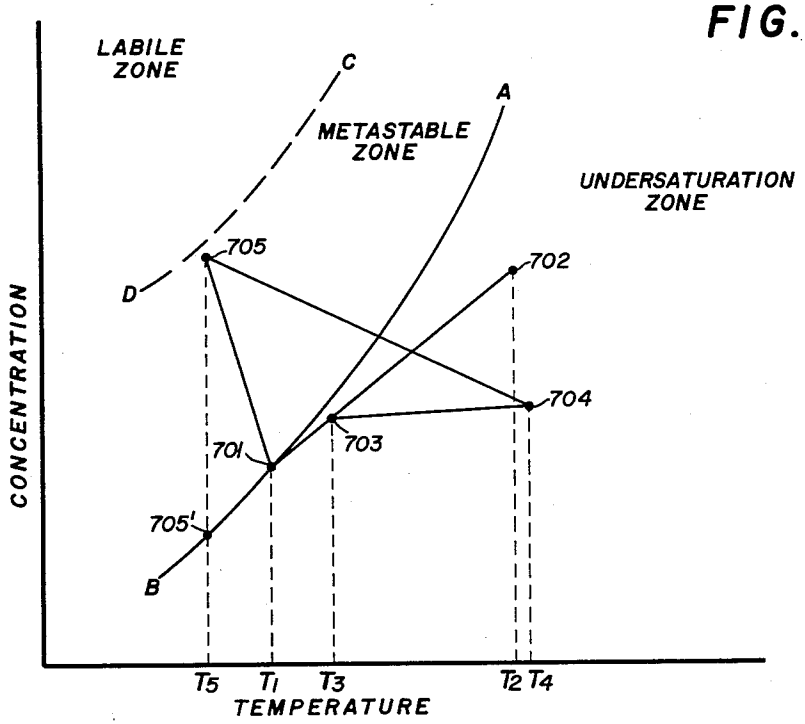
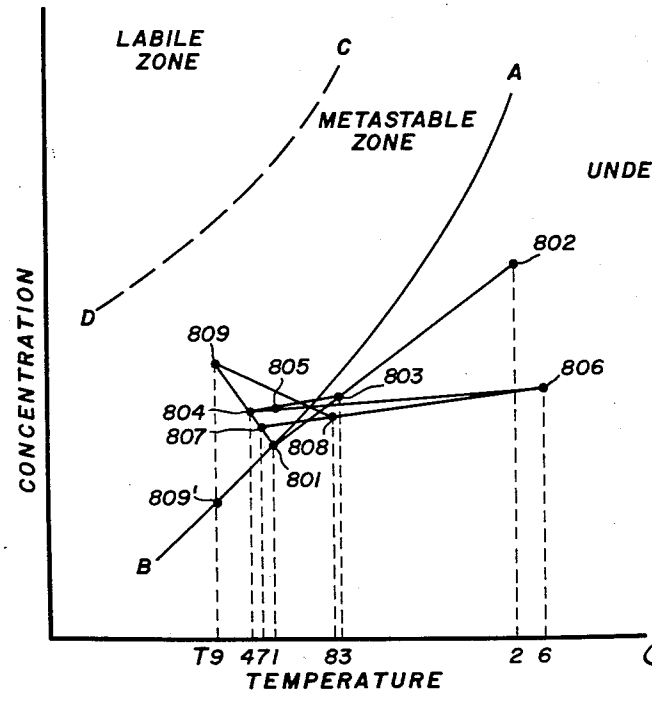


FIG. 8



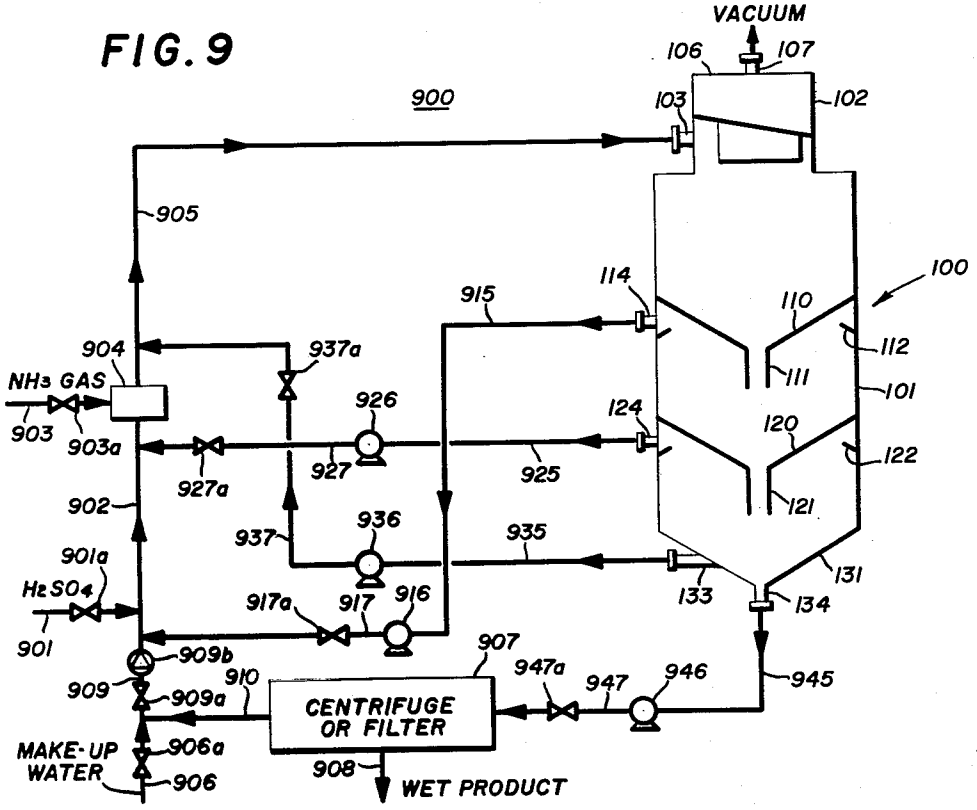
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CLASSIFYING CRYSTALLIZERS AND SYSTEMS

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FIG. 9



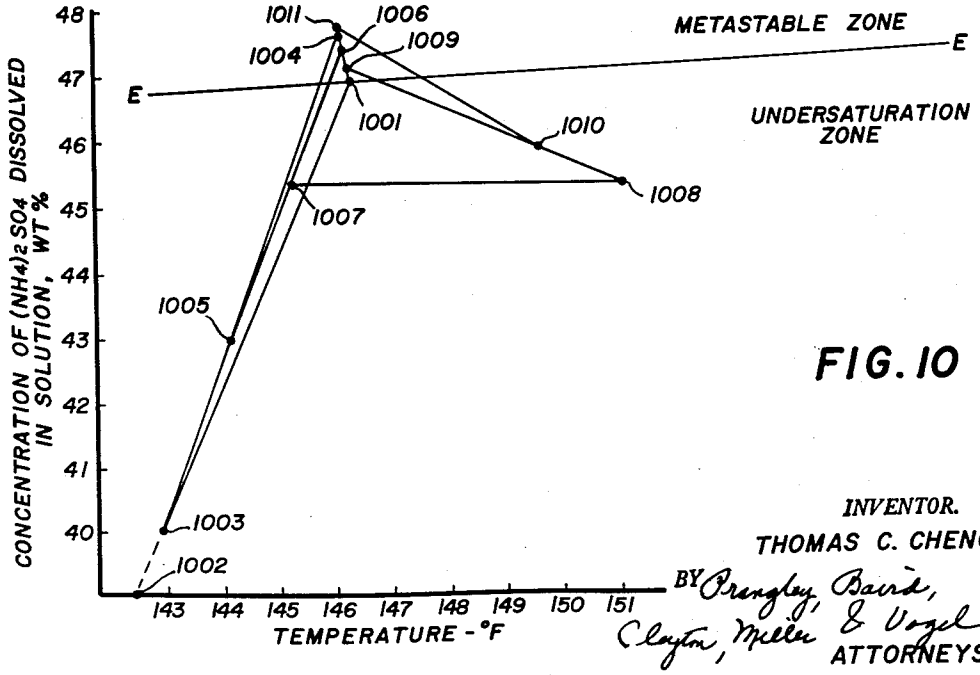


FIG. 10

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United States Patent Office 3,010,805 Patented Nov. 28, 1961

3,010,805 CLASSIFYING CRYSTALLIZERS AND SYSTEMS

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Filed Sept. 24, 1959, Ser. No. 842,099
26 Claims. (Cl. 23—273)

The present invention relates to crystallizing apparatus and systems and particularly to such apparatus and systems employing classification therein to produce uniform, controlled size crystals from crystallizable solutions.

It is a general object of the invention to provide an improved apparatus and system of the character noted wherein desupersaturization of crystallizable solution and crystal growth therein serves to form nuclei and fines and undersized crystals and crystal product in mother liquor, the apparatus comprising a shell having several stages of classification structure therein to remove the nuclei from the solution and to remove crystals smaller than the crystal product from the solution whereby to obtain maximum production of crystal product in mother liquor.

Another object of the invention is to provide an improved classifying crystallizer and system in which crystallizable solution in a main stream undergoes desupersaturization and crystal growth to form nuclei and fines and undersized crystals and crystal product in mother liquor, the crystallizer including structure for withdrawing from the main stream a first side stream containing substantially all of the nuclei shortly after formation thereof, for thereafter heating the first side stream to dissolve the nuclei, and for returning the first side stream to the main stream; and also including structure for withdrawing from the main stream a second side stream containing crystals smaller than the crystal product and for returning the crystals smaller than the crystal product to the main stream for additional crystal growth.

Still another object of the invention is to provide in a classifying crystallizer apparatus and system of the character noted, multiple stages of classification within the crystallizer shell whereby the solution undergoing crystallization encounters successive zones of classification, the structure defining the first classification zone creating a rate of solution flow sufficient to remove nuclei and successive classification zones creating rates of solution flow suitable to remove crystals smaller than the crystal product, the rates of flow in successive crystallization zones being successively greater.

In connection with the foregoing object it is a further object of the invention to provide in crystallizing apparatus and systems of the character noted, successively increasing cross sections of the successive classification zones and successively increasing lengths of the successive classification zones.

Yet another object of the invention is to provide an improved classifying crystallizer apparatus and system which provides more economic production with a large throughput of crystallizable solution per unit length of time, and which gives trouble-free operation.

A further object of the invention is to provide in a classifying crystallizer apparatus and system of the type set forth an improved entry baffle which admits the crystallizable solution in the form of an annular liquid curtain whereby to prevent improper formation of nuclei due to violent turbulence upon entry and to eliminate salt lumps formed by splashing.

A still further object of the invention is to provide an improved entry baffle of the character noted including structure to reduce vapor entrainment of particles and constantly to renew the surface of the crystallizable solution

in the crystallizer by breaking the thin film of crystals thereon, the film being broken and renewed without splashing whereby to increase the free surface exposed for flashing.

Further features of the invention pertain to the particular arrangements of the elements of the apparatus and systems whereby the above-outlined and additional operating features thereof are attained.

The invention, both as to its organization and method of operation, together with further objects and advantages thereof, will best be understood by reference to the following specification taken in combination with the accompanying drawings in which:

FIGURE 1 is a diagrammatic view in vertical cross section through one preferred embodiment of a classifying crystallizer made in accordance with and embodying the principles of the present invention;

FIG. 2 is a view in horizontal section through the crystallizer of FIG. 1 along the line 2—2 thereof;

FIG. 3 is a view in horizontal section along the line 3—3 of FIG. 1;

FIG. 4 is a view in horizontal section along the line 4—4 of FIG. 1;

FIG. 5 is a diagrammatic illustration of a crystallizer system embodying the present invention and incorporating the crystallizer of FIG. 1 therein;

FIG. 6 is a diagrammatic illustration of a crystallizing system embodying the present invention and incorporating therein a crystallizer comprising three classification stages and also embodying the present invention;

FIG. 7 is a concentration-temperature curve for a conventional vacuum crystallization process applied to a common salt;

FIG. 8 is a concentration-temperature curve for the system of FIG. 5 as applied to the common salt for which the concentration-temperature curve is shown in FIG. 7;

FIG. 9 is a diagrammatic illustration of a system embodying the present invention and incorporating therein the crystallizer of FIG. 1, the system being particularly adapted for producing crystalline ammonium sulfate obtained from the reaction of ammonia gas and concentrated sulfuric acid; and

FIG. 10 is a temperature-concentration curve for the classifying crystallizer process carried out by the system of FIG. 9.

There is shown in FIGS. 1 through 4 of the drawings a classifying crystallizer 100 embodying the present invention and adapted to be incorporated in the crystallizing system of the present invention, the crystallizer 100 including a shell comprising a cylindrical body 101 on top of which is disposed a cylindrical vapor head 102 and below which is disposed a bottom 131. Mounted on one side of the vapor head 102 and communicating with the interior thereof is a solution inlet connection 103 which is adapted to be connected to a source of crystallizable solution. The solution entering through the connection 103 impinges upon an entry baffle 105 supported in the vapor head 102 by a plate 104. The plate 104 is formed substantially flat and extends across the vapor head 102 above the conduit 103, the plate 104 being inclined at an angle of approximately 10° with respect to the horizontal, the higher side of the plate 104 being disposed adjacent to the conduit 103 and the lowermost portion of the plate 104 being disposed opposite and away from the conduit 103. The baffle 105 is cylindrical in configuration and surrounds a complementarily shaped opening formed in the plate 104, the baffle 105 being disposed eccentrically with respect to the vapor head 102 as may be best seen in FIG. 2, the baffle being positioned farthest away from that portion of the wall forming the vapor head 102 which carries the connection 103 and being disposed nearest to the

portion of the wall disposed away from and opposite the conduit **103**. Because of this arrangement the solution entering the connection **103** impinges upon the baffle **105** and is divided into two streams which flow around the baffle **105** and fall therefrom in the form of a continuous circular "liquid curtain." The upper end of the vapor head **102** is closed by a curved wall **106** carrying a first outlet **107** adapted to be connected to a vacuum or area of low pressure and a second outlet **108** adapted to be connected to monitoring controls and the like.

The lower end of the vapor head **102** joins a connecting wall portion **116** which in turn connects with the body **101** to form a fluid tight connection therebetween. Mounted in the bottom of the vapor head **102** is a first funnel **110** formed as a section of a truncated cone which converges downwardly to connect with a substantially cylindrical neck **111** extending downwardly therefrom. The walls of the funnel **110** are preferably inclined at an angle greater than the angle of repose of the material to be treated in the crystallizer **100** and for most materials can be formed at an angle of 60° with respect to the horizontal. Disposed about the funnel **110** and within the body **101** is a baffle **112** connected to the inner wall of the body **101** completely around the periphery thereof and extending upwardly and inwardly toward the funnel **110**. Preferably the baffle **112** is formed as a section of a cone and the smaller diameter thereof is slightly greater than the external diameter of the funnel **110** at the closest point thereof to provide therebetween an annular passage **115**. Mounted on the body **101** is a collector ring or manifold **113** which is positioned to collect solution which flows upwardly through the passage **115**, the collector ring **113** being provided with an outlet connection **114**.

Disposed within the body **101** and below the funnel **110** is a second funnel **120** having the greater diameter thereof equal substantially to the internal diameter of the body **101** and secured thereto completely around the periphery thereof. The wall of the funnel **120** may be inclined with respect to the horizontal at the same angle as the wall of the funnel **110**. The funnel **120** extends downwardly and terminates in an opening having a smaller diameter than the opening at the bottom of the funnel **110** and is connected to a cylindrical neck **121** having a diameter less than the diameter of the neck **111** and having a length greater than the length of the neck **111**. Disposed within the shell **101** and extending around the inner circumference thereof is a conical baffle **122** which extends upwardly and is provided with an opening therein spaced from the wall of the funnel **120** to provide an annular passage **125** therebetween, the area of the passage **125** being greater than the area of the passage **115** described above. A collector ring or manifold **123** similar to the collector ring **113** is provided to receive solution flowing through the passage **125** and the collector ring **123** is provided with an outlet connection **124**.

The funnels **110** and **120** divide the crystallizer into upper, intermediate and lower chambers providing two crystallization stages and two classification zones, as will be explained more fully hereafter.

The lower end of the body **101** joins a conical wall section **130** which connects with the cylindrical bottom **131**. A peripheral tangential outlet **133** is provided for the bottom **131** and the lower end of the bottom **131** is closed by a dome shaped wall **132**. The wall **132** is provided with an outlet connection **134** at the bottom thereof in general alignment with the necks **111** and **121** on the funnels **110** and **120**, respectively.

Referring to FIG. 5 of the drawings, there is shown a crystallizing system **500** embodying the present invention and incorporating therein the crystallizer **100** which has been described above. Initial feed solution containing dissolved material which is to be crystallized from the solution is fed (from a source not shown) through a pipe **501** containing a control valve **501***a* and a check valve **501***b* which permits flow through the pipe **501** only in the direction of the arrow. The outlet of the check valve **501***b* is connected by a pipe **502** to the inlet side of a heater **503** (the outlet of the heater **503** being connected by a pipe **504** to the conection **103** on the vapor head **102** of the crystallizer **100**. Solution entering through the connection **103** falls downwardly about the baffle **105** and solution is accumulated so that during normal operation of the system **500**, the liquid level in the crystallizer **100** is maintained a short distance below the lower edge of the baffle **105**, the liquid level being indicated by the numeral **108**. The outlet connection **114** is connected by a pipe **515** to the inlet of a pump **516** which pumps solution to a pipe **517** connecting with the pipe **502**, the pipe **517** having a control valve **517***a* therein to control the flow of solution therethrough. The outlet connection **124** is connected by a pipe **525** to the inlet side of a pump **526** which discharges solution under pressure into a pipe **527** conected to the pipe **504**, the pipe **527** having a control valve **527***a* therein to control the flow of solution therethrough. The outlet connection **133** is connected by a pipe **535** to the inlet side of a pump **536** which is connected to pump solution into the pipe **537** connecting with the pipe **504**, the pipe **537** having a control valve **537***a* therein to control the flow of solution therethrough. The bottom outlet connection **134** is connected to a pipe **545** which in turn is connected to the input of a pump **546**. The outlet of the pump **546** is connected by a pipe **547** to apparatus **550** for removing crystal product from mother liquor such as a centrifuge or filter, the pipe **547** having a control valve **547***a* therein. Wash water can be added to the apparatus **550** to wash the crystal product through a pipe **551** and the wet crystal product is removed at **552**. The mother liquor is returned by a pipe **553** to the pipe **502**, the pipe **553** having a control valve **553***a* therein and a check valve **553***b* permitting flow of solution only in the direction of the arrow, i.e., from the pipe **553** to the pipe **502**.

To begin operation of the crystallizing system **500**, initial feed solution is fed via the pipes **501** and **502** under the control of the valve **501***a* to the heater **503** where the solution is heated to a temperature above the equilibrium temperature within the crystallizer **100** so that a portion of the solution will evaporate upon entering the vapor head **102** whereby to render the remaining solution supersaturated. Accordingly, upon entry of the solution into the vapor head **102**, at least a portion of the solution is vaporized and the vapors are withdrawn through the outlet connection **107** and the pipe **505**. The hot solution entering the vapor head **102** strikes the baffle **105** and flows therearound in both directions so that a continuous circular liquid curtain falls from the lower edge of the baffle **105**, and after accumulation of sufficient solution to raise the level to the operating point the curtain falls onto the upper surface **108** of the solution in the crystallizer **100**. The solution in the liquid curtain is exposed to a pressure corresponding to an equilibrium temperature below the temperature of the incoming solution and, accordingly, a portion of the solution will be evaporated or "flashed" and the vapor passes upwardly through the opening in the baffle **105** and the plate **104** to the outlet connection **107**. The falling solution will quickly reach equilibrium temperature due to the character of the stream falling from the baffle **105** and, accordingly, most of the vaporization or flashing takes place while the solution is falling rather than after the solution has reached the surface **108**. As a result there will be little stirring and no splashing action at the surface **108** and the falling curtain of solution will strike the surface **108** substantially perpendicular thereto with no whirling or turbulent action. As a result there will be no improper formation of nuclei due to violent turbulence of solution flow from the baffle **105** and there will be no salt lumps from splashing, thereby to lengthen

the period between wash outs of the crystallizer **100** and to eliminate any problem of clogging in the various pumps and valves. The solution surface **108** is usually covered with a thin film of crystals which will be broken by the falling flow of incoming solution and thus provide a constant renewal of the surface **108** and thus produce additional free surface for flashing of vapor upwardly through the baffle **105**.

The solution continues downwardly under the action of the various pumps from the surface **108** and the main solution stream flows downwardly through the funnel **110** and the funnel **120** to the bottom **131**, this movement of the solution being accompanied by formation of nuclei and fines and undersized crystals and crystal product in mother liquor by desupersaturation of the solution and crystal growth. A first side stream comprising a magma of supersaturated solution with nuclei and crystals therein is drawn from the main stream through the outlet connection **114**. More specifically, a portion of the solution flowing downwardly through the neck **111** is directed upwardly in a direction opposite to its prior direction of flow and through the passage **115** into the outlet connection **114**. The funnel **110**, the neck **111** and the baffle **112** in cooperation with the shell body **101** form a first classification stage, the volume between the bottom of the funnel **111** and the outlet connection **114** providing a first classification zone. The first side stream is removed through the connection **114** via the pipe **515** under the action of the pump **516** and via the pipe **517** under the control of the valve **517***a* to the pipe **502**. The rate of flow of the first side stream is adjusted so that the rate of flow of solution through the first classification zone, i.e., upwardly from the bottom of the neck **111** pass the baffle **112** and into the connection **114**, is slightly greater than the terminal settling velocity of the nuclei therein, whereby the nuclei will eventually be remove from the first classification zone by "floating" or being carried past the baffle **112** and pass outwardly with a portion of the solution through the connection **114** while the large particles "sink" through the first classification zone back to the main stream.

A second side stream comprising a magma of supersaturated solution and crystals smaller than crystal product is withdrawn through the outlet connection **124** from a second classification stage formed by the funnel **120**, the neck **121**, the baffle **122** and the shell body **101**. The portion of the crystallizer **100** disposed between the lower end of the neck **121** and the outlet connection **124** comprise a second classification zone within which crystals smaller than crystal product can be removed in the second side stream. More specifically, the pump **526** can be operated under the control of the valve **527***a* to draw the second side stream through the second classification zone at a velocity slightly greater than the terminal settling velocities of the fines and all crystals smaller than the predetermined and desired crystal product. As a result fines and crystals smaller than crystal product issuing from the bottom of the neck **121** will be carried upwardly through the second classification zone with the second side stream and pass outwardly through the connection **124** via the pipe **525**, the pump **526** and the pipe **527** to the pipe **504** while product crystals fall back into the main stream and into the bottom **131**,

When first starting up the crystallizing system **500**, there will be no crystals of crystal product size present in the bottom **131** and therefore the remaining solution not withdrawn through the first and second side streams will be withdrawn in a third side stream comprising a magma of supersaturated solution and crystals smaller than crystal product through the connection **133** and fed via the pipes **535** and **537** under the action of the pump **536** back to the pipe **504**, whereby all of the solution will be recirculated. Eventually crystals of product size will be obtained in the bottom **131** and thereafter yet another stream can be withdrawn from the crystallizer **100** through the bottom connection **134**. This stream will comprise a magma of mother liquor having crystal product therein and will be conveyed to the centrifuge or filter **550** via the pipes **545** and **547** under the action of the pump **546**. After separation of the crystal product from the mother liquor, the mother liquor is returned through the pipe **553** and mixed with the initial feed solution in the pipe **501** and thence fed through the pipe **502** to the heater **503**. The system can now be operated in a substantially steady state condition with crystal product being continually withdrawn with mother liquor through the bottom connection **134**, the excess solution being evaporated and the vapors withdrawn through the outlet connection **107**, and new feed solution being added as required to compensate for removal of solids as crystal product.

The various control valves and pumps are adjusted to obtain economic production of product crystals with a large throughput per unit length of time. Economic production is best achieved by providing a low degree of supersaturation of the crystallizable solution entering the first funnel **110** in the first stage, a subsequent slow release of supersaturation, the maintenance of an optimum magma density in the crystallizer **100** and optimum rate of circulation through the crystallizer **100**, providing more seed surface as compared to prior apparatus and systems, and steady solution flow throughout. The rate of growth of crystals is a function of the surface area of existing crystals available for crystal growth and since nuclei proportionately present the greatest surface area for crystal growth, nuclei will preferentially grow to the size of fines thereby exhausting the available supersaturation and preventing undersized crystals from growing to crystal product size. Therefore it is essential to control and prevent the formation of nuclei in the solution within the crystallizer **100** or to remove the nuclei if formed so that the growth of nuclei will not exhaust the available supersaturation of the solution. The rate of formation of nuclei can be suppressed by re-cycling large volues of magma and mother liquor to obtain a small temperature change before and after flashing so as to minimize the supersaturation release per cycle of solution through the crystallizer **100**.

The crystallizer system **500** is operated in this manner, i.e., a substantial portion of the solution is withdrawn through the outlet connections **114**, **124** and **133** to be recirculated and the heater **503** is operated to provide only a small temperature change before and after flashing of the solution as it enters the vapor head **102** through the connection **103**. Such a method of operation minimizes the formation of nuclei as does also the configuration and structure of the entry baffle **105** described above. Any nuclei that are formed are immediately withdrawn in the first side stream from the first classification zone through the pipe **515** to the pipe **502**. The nuclei in the first side stream are dissolved in the heater **503** so that substantially no nuclei are present in the entering stream within the pipe **504**. The heater **503** is of the tube type and is operated to provide a low temperature difference across the surfaces of the tubes thereof and to provide a low temperature change between the inlet pipe **502** and the outlet pipe **504** therefor. The elevation of the heater **503** is preferably chosen relative to the crystallizer **100** so that there will be no evaporation within the heater **503** so that there will be a minimum deposition of crystals within the heater **503** and thereby to preserve the high heat transfer coefficient thereof.

In addition to minimizing the heat added to the solution mix in the pipe **502** by means of the heater **503**, the solution mix in the pipe **504** is further cooled by the addition thereto of the second side stream derived from the second classification zone through the connection **124** via the pipes **525** and **527** and by the addition thereto of the bottom side stream from the connection **133** via

the pipes 535 and 537. As a result of the mixing of these various streams, the magma entering the vapor head 102 through the connection 103 has a temperature only slightly above the equilibrium temperature within the vapor head 102.

As has been mentioned before there will be present within the crystallizer 100 four successive ranges of solid particle size, i.e., nuclei, fines, undersized crystals and crystal product, the nuclei being the smallest in size. The circulation magma in the first side stream from the first flash consists of slightly supersaturated solution, nuclei and a few fines. The circulation magma from the second stage flowing through the outlet connection 124 may consist of a few nuclei, fines, undersized crystals and mother liquor. The side stream drawn through the outlet connection 133 may consist of fines, undersized crystals and mother liquor. The bottom stream withdrawn through the outlet connection 134 consists of crystal product in mother liquor. The second side stream drawn through the outlet connection 124 and the bottom side stream drawn through the outlet connection 133, in addition to cooling the heated solution in the pipe 504, furnish the crystal seeds for subsequent crystal growth in the crystallizer 100. Thus the entry stream flowing into the vapor head 102 will contain crystals with a wide range of sizes, but substantially no nuclei, in a slight undersaturated solution. Flashing in the vapor head 102 will remove sufficient solvent vapor to provide a slightly supersaturated solution which can supply the necessary material for growth of the seed crystals present.

The factors governing the classification of crystals in the first and second classification zones and the elutriation taking place in these zones are the density of the magma and crystals, the content of solids in the magma, the crystal shape, the magma viscosity, the flow rate within the zone, the size of crystals to be removed, and the flow area in the annular openings 115 and 125. Since the flow rate is usually in the steady region, the flow velocity is in the range of 1.5 feet per minute to 5 feet per minute for most solutions. The settling rate of nuclei, fines and crystals decreases as the particle size decreases, i.e., the terminal settling velocity is smaller for the smaller particle size and therefore a relatively low flow velocity through the first classification zone and the annular opening 115 will be sufficient to remove nuclei while returning substantially all larger particles to the main stream and correspondingly higher flow velocities must be maintained in the second side stream through the annular opening 125 to remove the fines and undersized crystals from the second classification zone through the outlet conduit 124. To achieve efficient classification, relatively long settling times are required and therefore a longer classification zone must be utilized with higher flow velocities to obtain the same degree of separation as compared to classification zones utilizing lower flow velocities. Accordingly, the vertical extend of the first classification zone, i.e., the space between the bottom of the neck 111 and the outlet connection 114, is shorter than the height of the second classification zone, i.e., the distance between the lower end of the neck 121 and the second outlet connection 124. As the crystals grow larger the tendency to collide with other crystals of different size is increased, therefore a larger cross section of flow area is desirable in classifying the larger crystals. Accordingly, the cross section of the second or lower classification zone is preferably formed greater than the cross section of the first or upper classification zone.

Preferably the above desired construction and arrangement of the parts in the crystallizer 100 is accomplished by providing like angularity of the walls of the funnels 110 and 120, and changing the size and shape of the necks 111 and 121. Preferably the diameter of the upper neck 111 is greater than the diameter of the lower neck 121 and conversely the length of the upper neck 111 is preferably less than the length of the lower neck 121. The greater diameter of the upper neck 111 facilitates and accommodates the greater total flow of solution therethrough since all of the solution introduced into the vapor head 102 and not evaporated must flow therethrough. The neck 121 on the other hand can have a smaller diameter since the total volume to be passed therethrough is less than that through the neck 111 and more particularly the volume through the neck 121 is reduced by the amount drawn through the connection 114. The length of the necks 111 and 121 are determined by (1) the rate of ratio of solution flow through the neck to the solution flow of the side stream taken therefrom through the connection 114 or 124 (the ratio Q/F), (2) the magma solids content of the solution through the neck, and (3) the size of the crystals in the solution falling through the neck. In the lower classification zone, the Q/F ratio is the smallest, and both the crystals content and size are the highest, and therefore the length of the neck 121 must be greater than the neck 111. The longer neck 121 provides a longer classification zone in the second stage and also provides a classification zone having a greater cross sectional flow area whereby to reduce the probability of collision and to minimize the rubbing effect between the crystals in the heavy magma present in this zone. The classification efficiency is rendered maximum by the above described proportion of the parts.

Since the flow rate in each stage is controlled by means of the valves 517*a*, 527*a*, 537*a* and 547*a*, the magma solids content can be kept constant throughout the crystallizer 100. The solids content in the magma will be the lowest at the upper or first stage and the highest in the bottom or second stage while the degree of supersaturation is highest at the top or first stage and lowest at the bottom or second stage. Because the rate of crystal growth is dependent upon the exposed surface area present, the rate of release of supersaturation will be substantially the same in the first stage as in the second stage and substantially the same throughout the crystallizer 100. This will insure that the crystallization process will proceed at a constant and steady rate.

The above described principles are equally applicable to crystallizers having more than two stages and in fact are applicable to crystallizers having any desired number of stages, the number of stages employed in any particular crystallizer depending upon the particular performance desired and the requirements placed upon the crystal product, such as purity, crystal size, the range of crystal size, etc. There is shown in FIG. 6 of the drawings a crystallizer system 600 incorporating therein a three stage crystallizer 601. The crystallizer includes a shell 602 having three funnels 610, 620 and 630 dividing the shell into an upper vapor head, a first crystallization stage disposed between the funnels 610 and 620, a second crystallization stage disposed between the funnels 620 and 630 and a third crystallization stage disposed between the funnel 630 and the bottom 633, each of the crystallization stages providing a corresponding classification zone. The vapor head is provided with an inlet connection 603 which is positioned to direct incoming undersaturated solution against a baffle 605 identical in construction to the baffle 105 described above. A portion of the solution flashes and the vapor formed passes upwardly through the opening in the baffle 605 through an outlet connection 607 in an upper wall 606 closing the upper end of the vapor head. The connection 607 is in communication with a pipe 608 which in turn is connected to an area of reduced pressure.

The three classification stages are provided with outlet connections 614, 624 and 634, respectively from top to bottom, and with weir rings 612, 622 and 632 in association therewith and spaced from the corresponding funnels 610, 620 and 630 to provide an annular flow passage therebetween. Each of the funnels is provided with a cylindrical neck, the funnel 610 having a neck

611, the funnel 620 having a neck 621 and the funnel 630 having a neck 631. The diameter of the necks progressively decreases from top to bottom and the length of the necks progressively increases from top to bottom, whereby the vertical extent of the classification zones increases from top to bottom and the cross sectional flow area in the classification zones increases from top to bottom. Similarly the flow rates in the classification zones are regulated so that the lowest flow rate is maintained in the upper or first classification zone and the highest flow rate is maintained in the third or lowermost classification zone.

Initial feed solution is fed (from a source not shown) through a pipe 661 including a control valve 661*a* and a check valve 661*b* to a pipe 662 communicating with a heater 663. The output of the heater is fed through a pipe 664 to the inlet connection 603 and the solution not vaporized in the shell 602 falls downwardly through the several funnels in a main stream whereby nuclei, fines, undersize crystals and crystal product in mother liquor are formed by desupersaturation and crystal growth. A first side stream is taken from the main stream in the first classification zone through the outlet connection 614 and is conveyed by a pipe 615 to a pump 616 and from the pump 616 by a pipe 617 to the pipe 662, the pipe 617 having a control valve 617*a* therein. A second side stream is derived from the main stream drawn through the outlet connection 624, a pipe 625, a pump 626 and a pipe 627 to the pipe 664, the pipe 627 having a control valve 627*a* therein. A third side stream is drawn from the main stream through the third classification zone from the outlet connection 634 through a pipe 635, a pump 636, and a pipe 637 to the pipe 664, the pipe 637 having a control valve 637*a* therein. The remaining portion of the main stream flows downwardly through a bottom outlet connection 638, a pipe 645, a pump 646 and a pipe 647 also connecting with the pipe 664 and having a control valve 647*a* therein. A side pipe 648 is also connected to the pipe 647 to withdraw mother liquor with crystal product therein from the pipe 647, the pipe 648 having a control valve 648*a* therein to regulate the flow of the mother liquor with crystal product therethrough. The pipe 648 is connected to suitable apparatus 650 for removing the crystal product from the mother liquor, the apparatus 650 being, for example, a centrifuge or a filter. Wash water, if required, is supplied through a pipe 651 to the apparatus 650 and the wet product is removed therefrom through a conduit 652. The mother liquor leaves the apparatus 650 through a pipe 653 which connects with the pipe 652, the pipe 653 having a control valve 653*a* and a check valve 653*b* therein.

The crystallizer system 600 operates in substantially the same manner as the system 500 described above. However, better control of the product and process is obtained from the system 600 because of the third classification zone and associated side stream.

The principle of operation of the crystallizer systems 500 and 600 illustrated in FIGS. 5 and 6 of the drawings can be best illustrated by the reference to the concentration-temperature curves set forth in FIGS. 7 and 8 of the drawings. As is usual in such curves, the concentration shown therein is the concentration of dissolved salt in the solution and does not include any solids or crystals carried by, but not dissolved in, the solution. The curve shown in FIG. 7 is typical of prior art systems of crystallizing common salts, for example, magnesium sulfate, the line AB therein representing the solubility curve for the salt separating the "undersaturation zone" below the curve from the "metastable zone" above the curve. The dashed line CD is the so-called "supersolubility curve" or the hypothetical margin line dividing the metastable zone beneath the curve CD from the "labile zone" above the curve CD. In the undersaturation zone, there is no supersaturation in the solution available for crystal growth and in fact, there may be a net loss of crystals through dissolution thereof. In the metastable zone, there is supersaturation present in the solution available for crystal growth but not sufficient to cause spontaneous formation of nuclei. In the labile zone, there is sufficient supersaturation present to cause spontaneous formation of nuclei with substantially no external cause therefor.

Point 701 on the curve AB represents the concentration of the re-cycling mother liquor in prior systems at the final crystallization temperature T_1, this being an equilibrium point fixed by the operating conditions such as the pressure within the crystallizer. Point 702 represents the concentration of the initial feed solution at the feeding temperature T_2, and the point 703 represents the resultant concentration achieved by mixing the re-cycling mother liquor and the initial feed and the temperature T_3 resulting from the mixing of the two streams of different temperatures, the point 703 being positioned on a straight line connecting the points 701 and 702 and the position thereof along the line being determined by the relative weight of the individual component streams. The point 703 will move closer to the point 701 if more re-cycling mother liquor is mixed with a given weight of initial feed solution and conversely the point 703 will move closer to the point 702 if less re-cycling mother liquor is mixed with a given weight of initial feed solution. In a typical prior art crystallization process, the solution represented by the point 703 is passed through a heater and is heated to a temperature T_4 represented by the point 704. If there are no appreciable solids to dissolve in the mother liquor, there will be no significant change in concentration and the line interconnecting the points 703 and 704 will be substantially horizontal; on the other hand, if there is dissolution of solids, then the concentration will slightly increase in going from point 703 to point 704. The heated solution represented by the point 704 is then fed into the crystallizer and it will flash to an equilibrium temperature T_5 determined by the pressure within the crystallizer, the flashing of the solution removing the solvent as vapor and causing supersaturation of the remaining solvent. There is supersaturation present when changing the temperature from T_4 to T_5 along the line interconnecting the points 704 and 705 as soon as the temperature crosses the curve AB and the supersaturation will increase until the temperature T_5 is reached at which time no further flashing will take place. As soon as there is any supersaturation in the solution, there will be crystal growth on any crystal surfaces existing in the solution. Care must be taken in the correlating of the temperature T_4 with the pressure within the crystallizer to insure that the point 705 lies in the metastable zone and not the labile zone since there will be an undersired spontaneous formation of nuclei as soon as the labile zone is reached.

In the desupersaturation or crystal growth process, the temperature of the solution will increase as the solution flows downwardly through the crystallizer if there is no way to dissipate the heat of crystallization evolved. Accordingly, crystal growth and desupersaturation is accompanied by a slight rise in temperature from T_5 to T_1 and a corresponding reduction in concentration from point 705 to 701. The temperature difference between T_5 and T_1 must be minimized since the yield per cycle is decreased as the temperature difference increases due to the fact that the curve AB goes upwardly indicating that less supersaturation is utilized in crystal growth as the temperature T_1 increases.

The degree of supersaturation is the difference between the concentration at the point 705 and the concentration at the same temperature T_5 on the curve AB, namely, that represented by the point 705'. Preferably, the degree of supersaturation is minimized in order to achieve a crystal product of the desired size, and in the conventional process illustrated in FIG. 7, this is accomplished by resorting to re-cycling a large volume of mother liquor. This procedure has serious disadvantages in that

the mother liquor withdrawn from the crystallizer will contain only small amounts of solids and consequently more facilities will be required to remove the crystal product therefrom. As the supersaturation in the solution is relieved, the concentration moves along the line interconnecting the points 705 and 701 toward the point 701 until equilibrium is reached in the recycled mother liquor withdrawn from the bottom of the crystallizer.

The crystallization process of the present invention is illustrated in FIG. 8 where the process has been shown applied to the crystallization of the same solution as in FIG. 7, the temperatures T_1, T_2 and T_3 and the corresponding concentrations in FIG. 8 being the same as the like temperatures and concentrations in FIG. 7. More specifically, the point 801 represents the concentration and temperature of mother liquor in the pipe 553 of FIG. 5, the point 802 represents the temperature and concentration of the initial feed solution in the pipe 501 and the point 803 represents the first mixing of the mother liquor and the initial feed solution. This first mix is further joined by the first stage circulation solution which has a concentration and temperature represented by the point 804, this solution being contained in the pipe 517 in FIG. 5. The solution at the point 804 is still supersaturated and has a concentration lying on the line interconnecting the points 809 and 801. The mixing of the solutions in the pipes 501, 517 and 553 provides a solution in the pipe 502 having the temperature and concentration represented by the point 805. That solution is then fed into the heater 503 where it is heated to the temperature T_6. The nuclei contained in the first side stream solution appearing in the pipe 517 are dissolved as are a portion of any crystals remaining in the mother liquor in the pipe 553 and, accordingly, there is a slight increase in concentration in going from the point 805 to the point 806. The temperature T_6 in FIG. 8 is identical to the temperature T_4 in FIG. 7 and is sufficiently high to dissolve the nuclei in the solution provided that sufficient time is permitted to achieve dissolution. Those crystals having the greater proportional surface area, i.e., the nuclei, will be preferentially dissolved since the rate of dissolution is proportional to the surface area and in fact the nuclei will be substantially completely dissolved before the larger crystals start to dissolve. The larger crystals thus saved provide seeds for crystal growth in the crystallizer 100 and increase the economy of the crystallizing process.

The heated solution represented by the point 806 is fed to the pipe 504 where it is mixed with the two side streams in the pipes 527 and 537 which comprise the recirculation magma from the second stage and the bottom of the crystallizer 100. The combined recirculation streams in the pipes 527 and 537 have a concentration and temperature represented by the point 807, these solutions carrying a small amount of supersaturation at a temperature lower than the temperature T_1. The resultant stream entering the connection 103 has a temperature and concentration at the point 808, the position of the point 808 on the line connecting the points 806 and 807 being determined by the ratio between the volume of solution leaving the heater 503 and the volume of solution in the pipes 527 and 537. Preferably this ratio is arranged so that the recirculation streams in the pipes 527 and 537 cool the solution at the point 808 into the undersaturation zone but at a temperature T_8 only slightly less than that required to enter the metastable zone.

Entry of the stream through the connection 103 into the area of reduced pressure in the crystallizer 100 causes a portion of the solution to flash at a temperature T_9 which is identical to the temperature T_5 in FIG. 7. The slope of the line interconnecting the points 704 and 705 is the same as that interconnecting the points 808 and 809 provided the solvents are the same. As a result of the flashing, the remaining solution will be cooled to the temperature T_9 and will have a concentration such that it is supersaturated, the degree of supersaturation being indicated by the difference of concentrations between the points 809 and 809′ at T_9. The supersaturated solution will desupersaturate along the line 809–801 as the solution falls through the crystallizer 100. The completely desupersaturated solution in dynamic equilibrium with the solid crystals, i.e. the mother liquor represented by the point 801, is present at the bottom connection 134 and the mother liquor is removed and recirculated through the pipe 553 as described above.

From the above description of the cycle illustrated in FIG. 8, it is clear that a minimum degree of supersaturation is utilized in the present system, namely that represented by the difference in concentration between the points 809 and 809′ and that this degree of supersaturation is substantially less than that of the conventional system illustrated in FIG. 7 when utilizing like operating conditions, i.e., the same temperature and concentration of the mother liquor, the same temperature and concentration of the initial feed solution, the same temperature of the solution leaving the heater, and the same flashing temperature. The initial crystallization point 809 in the process of FIG. 8 is also further removed from the labile zone than is the corresponding point 705 in the process of FIG. 7 and consequently there will be a minimum of spontaneous formation of new nuclei in each cycle.

There is shown in FIG. 9 of the drawings a slightly modified system 900 embodying the present invention and illustrating the crystallizer 100 utilized to crystallize ammonium sulfate from a solution obtained by reacting concentrated sulfuric acid and ammonia gas. In the system 900, concentrated sulfuric acid is fed in a pipe 901 (from a source not shown) through a control valve 901*a* to a mixing pipe 902. The pipe 902 connects with a reactor 904 which has connections to receive ammonia gas through a supply pipe 903 (connected to a source not shown) through a control valve 903*a*. The concentrated sulfuric acid and the ammonia gas chemically react in the apparatus 904 to form an ammonium sulfate solution which is fed through a pipe 905 to the inlet connection 103 of the crystallizer 100. The first side stream taken from the crystallizer 100 through the outlet connection 114 is conveyed through a pipe 915 under the action of a pump 916 through a pipe 917 having a control valve 917*a* therein and communicating with the pipe 902 ahead of the sulfuric inlet pipe 901 so that the concentrated sulfuric acid can be injected into a stream for dilution therein. The second side stream taken from the crystallizer 100 through the outlet connection 124 is conveyed through a pipe 925 under the action of a pump 926 to a line 927 connecting with the pipe 902 after injection of the sulfuric acid therein, the pipe 927 having a control valve 927*a* therein. The third side stream derived from the crystallizer 100 through the outlet connection 133 is conveyed through a pipe 935 to a pump 936 and then to a pipe 937 connecting with the pipe 905, the pipe 937 having a valve 937*a* therein. Mother liquor containing crystal product is withdrawn through the bottom connection 134 and conveyed by a pipe 945 to a pump 946 and by a pipe 947 having a control valve 937*a* therein to a crystal separation apparatus 907 which may be a centrifuge or filter as described above. The wet product from the apparatus 907 is removed through a conduit 908 and the mother liquor is conveyed through a pipe 910 to a junction with a make-up water pipe 906 having a control valve 906*a* therein. The resulting mixed stream is fed through a pipe 909 having a control valve 909*a* and a check valve 909*b* therein to the pipe 902.

In the system 900 the crystallizer 100 operates as has been explained above with respect to FIG. 5 but the various side streams therefrom are utilized in a somewhat different manner to accommodate the particular needs of the system in manufacturing ammonium sulfate from the con-

centrated sulfuric acid and ammonia gas. The three streams in the pipes **906**, **910** and **917** are mixed together to form a relatively large stream to which is added a relatively small stream of concentrated sulfuric acid whereby to dilute the sulfuric acid, the dilution of the sulfuric acid being accompanied by some evolution of heat. The stream in the pipe **917** may contain substantial nuclei and these are at least partially dissolved by the heat evolved by the dissolution of the sulfuric acid in the stream within the pipe **902**. The heated solution is then partially cooled by the second side stream from the pipe **927** before the ammonia gas is added thereto. Dissolution of the ammonia gas in the solution from the pipe **902** serves substantially to heat the solution whereby further to destroy nuclei therein. The third side stream in the pipe **937** is admitted to the pipe **905** to cool the solution coming from the reactor **904** and the resultant stream constitutes the feed for the crystallizer **100**.

There is shown in FIG. 10 of the drawings a concentration-temperature diagram of the crystallization process carried out by the system **900** of FIG. 9, the curve EF being the solubility curve of ammonium sulfate in water, the temperatue being plotted in degrees Fahrenheit along the horizontal axis and the concentration of ammonium sulfate dissolved in solution, in weight percent, being plotted along the vertical axis. The point **1001** represents the equilibrium solution of the mother liquor in the pipe **910** and corresponds to the point **801** in FIG. 8. The initial feed solution in FIG. 10 (corresponding to the point **802** in FIG. 8) is actually a mixture of make-up water and sulfuric acid which has an ammonium sulfate concentration of 0% at 122° F. which is off of the graph in FIG. 10 but is symbolically represented thereon at **1002**. The mixing of the mother liquor in the pipe **910** with the sulfuric acid and the make-up water produces a diluted mother liquor having a concentration and temperature represented by the point **1003** (corresponding to the point **803** in FIG. 8). To this is added the first side stream containing nuclei from the pipe **917** having a concentration and temperature represented by the point **1004** (corresponding to the point **804** in FIG. 8) and the resulting mixture has a temperature and concentration represented by the point **1005**, this being the solution in the pipe **902** before the addition of the stream from the pipe **927** (corresponding to point **805** in FIG. 8). To this is then added the second side stream through the pipe **927**, the side stream having a temperature and concentration represented by the point **1006**. The resulting mixture entering the reaction chamber **904** has the temperature and concentration represented by the point **1007**.

The reaction of the ammonia gas with the sulfuric acid solution evolves substantial heat and raises the temperature of the solution to that represented by the point **1008** (corresponding to the point **806** in FIG. 8) and slightly increases the concentration of ammonium sulfate in solution by dissolving the nuclei therein. To the heated solution is then added the third side stream from the pipe **937** which has the composition and temperature represented by the point **1009** (corresponding to the point **807** of FIG. 8) and the resultant mixture has a temperature and concentration represented by the point **1010** (corresponding to point **808** in FIG. 8), this solution being fed through the pipe **905** to the inlet connection **103**.

Flashing of the solution as it enters the crystallizer **100** removes solvent from the solution whereby to raise the concentration of ammonium sulfate in the remaining solution and to reduce the temperature to that represented by the point **1011** (corresponding to point **809** in FIG. 8). The remaining solution is now supersaturated since point **1011** lies in the metastable zone and immediately desupersaturation begins accompanied by crystal growth on the seed crystals fed into the crystallizer **100** through the pipe **905**. As desupersaturation proceeds, the concentration of ammonium sulfate in the solution falls and the temperature slightly increases along the line **1011**–**1001**. The first side stream is removed at the operating point **1004** through the connection **114**, the second side stream is removed at the operating point **1006** through the outlet connection **124**, the third side stream is removed at the operating point **1009** through the outlet connection **133** and the mother liquor with the crystal product therein is removed from the bottom connection **134** at the operating point **1001**. This completes a cycle of operation of the system **900**.

The above desired operation of the system **900** is achieved by utilizing apparatus having the following dimensions and operating under the conditions here set forth. It is to be understood that this specific example is given only by way of illustration and is not intended in any way to limit the present disclosure.

The system **900** can be designed to operate utilizing as raw materials 98% by weight of sulfuric acid at the rate of 12,800 pounds per hour and anhydrous ammonia at 50° F. and 75 pounds per square inch gage pressure in the amount of 4,370 pounds per hour. The mother liquor drawn from the bottom outlet connection **134** has a temperature of 146° F. and an ammonium sulfate concentration of 47% by weight (corresponding to point **1001** in FIG. 10) and the crystallizer is operated at a pressure of 25 inches of mercury vacuum. When operated in the above manner, the system **900** will produce 203 tons per day of ammonium sulfate (assuming a 1.5% over-all loss) having a crystal size such that 60% will be maintained on a 16 mesh screen and 90% retained on a 28 mesh screen.

The crystallizer **100** has the following dimensions when utilized in the system **900**: the diameter of the vapor head **102** is 9 feet and the height thereof is 9 feet; the diameter of the body **101** is 16 feet and the height thereof is 24 feet; the slope of the sides of the funnels **110** and **120** is 60°; the diameter of the neck **111** is 3 feet, 6 inches and the length thereof is 3 feet, 6 inches; the diameter of the neck **121** is 2 feet, 8 inches and the length thereof is 5 feet, 4 inches; the baffle **112** is inclined at an angle of 30° with respect to the horizontal and has an opening therein of 7 feet, 3 inches and the radius of the passage **115** is 7½ inches; the angularity of the baffle **112** is also 30° and the diameter of the opening therein is 10 feet, 7 inches and the radius of the annular passage **125** is 3½ inches; the manifold or collector ring **113** has ten 6 inch diameter openings therein communicating with the interior of the crystallizer and the collector ring **123** has fourteen 6 inch diameter openings therein; the entry baffle **105** has a diameter of 7 feet, 10 inches and is placed 9 inches away from the inlet connection **103** and 5 inches away from the nearest wall of the vapor head **102**, the lower edge of the baffle **105** being disposed 3 feet above the juncture of the vapor head **102** and the wall section **116**.

The crystallizer having the dimensions described above has a suspension volume for solution of 5,950 cubic feet. The circulation rate from the first stage through the outlet connection **114** is adjusted to be 2900 gallons per minute, the circulation rate from the second stage through the outlet connection **124** is 3800 gallons per minute and the circulation rate from the bottom through the outlet connections **133** and **134** is 4,855 gallons per minute. The flow velocity at the inlet connection **103** will be 7.4 feet per second. The pump **916** and the control valve **917***a* are adjusted so that the flow velocity through the outlet connection **114** for the first stage is 7.8 feet per second which provides a superficial flow velocity for nuclei in the first stage classification zone of 0.034 foot per second. The pump **926** and the control valve **927***a* are adjusted to provide a flow velocity of 8.5 feet per second through the second stage outlet connection **124** which results in a superficial flow velocity of fines in the second stage classification size of 0.043 foot per second. The bottom outlet connection **134** has a flow velocity

therethrough of 8.1 feet per second. The above flow rates result in a flow velocity of two feet per second through the first stage neck 111 and a flow velocity of two feet per second through the second stage neck 121.

The cross sectional area of the classification zone in the first stage is 191 square feet and the distance from the bottom of the funnel 111 to the passage 115 is 6 feet. The cross sectional area of the classification zone in the second stage is 194 square feet and the distance from the bottom of the neck 121 to the passage 125 is 12 feet.

Recapitulating: the classifying crystallizer of the present invention includes at least two, and if desired, three or more stages contained within a shell provided with inlet and outlet connections so that a main stream of crystallizable solution can be fed through the various stages in succession. The crystallizable solution may contain supersaturation or the crystallizer can be provided with a connection to an area of reduced pressure to flash a portion of the solution and cause supersaturation of the remainder thereof whereby the supersaturated solution as it flows through the various stages has formed therein nuclei and fines and undersized crystals and crystal product in mother liquid by desupersaturation of the solution and crystal growth. An improved entry baffle has been provided to minimize disturbance and splashing of the supersaturated solution thereby substantially to reduce the number of nuclei formed. Each of the stages in the crystallizer includes a classification zone through which a side stream is withdrawn, each side stream containing a predetermined size range of solids, i.e., the first side stream from the first stage (the stage nearest the inlet) carrying substantially only nuclei from the first classification zone, and the second and successive side streams from the second and successive stages carrying crystals smaller than crystal product and of increasing size in successive stages. To accomplish this, each stage comprises a funnel extending downwardly and cooperating with an outlet conduit spaced upwardly from the lower end of the funnel to provide therebetween a classification zone and preferably a baffle or a weir ring is provided between the lower end of the funnel and the outlet conduit to provide a passage of controlled area. The rate of flow in the classification zones increase successively from the inlet to the outlet of the crystallizer, and the length and cross sectional area of the classification zones increase from the inlet to the outlet.

The crystallizer as described above is further incorporated in systems of the present invention which include means such as a heater to remove the nuclei from the first side stream; the first side stream, the mother liquor obtained from crystal product recovery and the initial feed solution preferably are all heated preferentially to dissolve the nuclei and the colder side streams from the second and subsequent stages are mixed with the heated stream to provide an inlet stream for the crystallizer having the desired temperature and salt concentration. Suitable controls are provided to obtain the desired characteristics of operation of the system and particularly the desired rate of flow of the inlet streams, the various outlet or side streams, and the product stream.

In view of the foregoing, it is apparent that there has been provided an improved classifying crystallizer and an improved crystallizing system incorporating the improved classifying crystallizer therein which satisfies all of the objects and advantages set forth above.

While there has been described what is at present considered to be certain preferred embodiments of the invention, it will be understood that various modifications may be made therein, and it is intended to cover in the appended claims all such modifications as fall within the true spirit and scope of the invention.

What is claimed is:

1. A classifying crystallizer system for separating crystals from solutions, comprising a crystallizer shell, an inlet pipe communicating with said shell for supplying solution from which crystals are to be crystallized, an outlet pipe communicating with said shell and supplied with mother liquor containing crystal product, whereby passage of the solution from said inlet pipe in a main stream through said shell to said outlet pipe serves to form nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation of the solution and crystal growth, first conduit means communicating with said shell for separating a first side stream containing substantially only nuclei from the main stream adjacent to said inlet pipe, second conduit means communicating with said shell between said first conduit means and said outlet pipe for separating a second side stream containing crystals smaller than the crystal product from the main stream between said first conduit means and said outlet pipe, means connected to said first conduit to remove the nuclei from the first side stream, and means connecting said first and second conduit means to said inlet pipe to return the first and second side streams to the main stream through said inlet pipe.

2. The classifying crystallizer system set forth in claim 1, wherein the means connected to the first conduit means comprises means to heat the first side stream to dissolve the nuclei therein.

3. A classifying crystallizer system for separating crystals from solutions, comprising a crystallizer shell, an inlet pipe communicating with the upper portion of said shell for supplying a solution from which crystals are to be crystallized, a vapor outlet communicating with the upper portion of said shell above said inlet pipe for connecting to an area having a pressure lower than that in said shell, a plate mounted in said shell and extending thereacross and disposed between said inlet pipe and said vapor outlet and having an opening therein, a substantially cylindrical baffle mounted on said plate and surrounding the opening therein and extending downwardly past said inlet pipe, said baffle being disposed nearer the wall of said shell opposite said inlet pipe to provide more flow space on the inlet side of said baffle, whereby the solution entering from said inlet pipe imginges upon said baffle to flow therearound and downwardly therealong and to fall therefrom in a continuous liquid curtain, an outlet pipe communicating with said shell and supplied with mother liquor containing crystal product, whereby passage of the solution from said inlet pipe into said shell and around and down said baffle is accompanied by evaporation of a portion of the solution to form vapor which passes upwardly through the opening in said plate and through said vapor outlet to provide supersaturation in the remaining portion of the solution which flows through said shell in a main stream in which are formed nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation and crystal growth, conduit means communicating with said shell below said baffle for separating a side stream containing substantially only nuclei from the main stream, means connected to said conduit to remove the nuclei from the side stream, and means connecting said conduit to said inlet pipe to return the side stream to said shell through said inlet pipe.

4. The classifying crystallizer system set forth in claim 3, wherein the upper portion of the shell is substantially circular in cross section, the plate is inclined downwardly away from the inlet pipe, and the baffle is substantially circular in cross section and is arranged eccentrically with respect to the upper portion of said shell and farthest away from said inlet pipe.

5. A classifying crystallizer system for separating crystals from solutions, comprising means for heating a solution having an inlet and an outlet, a supply pipe inter-

connecting said inlet and a source of initial feed solution, a crystallizer shell, an inlet pipe interconnecting said outlet and the upper portion of said shell, a vapor outlet communicating with the upper portion of said shell above said inlet pipe for connecting to an area having a pressure lower than that in said shell, an outlet pipe communicating with said shell and supplied with mother liquor containing crystal product, whereby passage of the solution from said inlet pipe into said shell is accompanied by evaporation of a portion of the solution to form vapor which passes upwardly through said vapor outlet to provide supersaturation in the remaining portion of the solution which flows through said shell in a main stream in which are formed nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation and crystal growth, first conduit means communicating with said shell for separating a first side stream containing substantially only nuclei from the main stream adjacent to said inlet pipe, second conduit means communicating with said shell between said first conduit means and said outlet pipe for separating a second side stream containing crystals smaller than the crystal product from the main stream between said first conduit means and said outlet pipe, means connected to said outlet pipe for removing crystal product from the mother liquor therein, and means connected to said first and second conduit means and said crystal product removing means to return the first and second side streams and the mother liquor to the main stream through said inlet pipe, at least said first conduit means being connected to said supply pipe whereby the first side stream passes through said heating means to dissolve the nuclei therein.

6. The classifying crystallizer system set forth in claim 5, wherein the second conduit means is connected to the inlet pipe, and the crystal product removing means is connected to the supply pipe.

7. The classifying crystallizer system set forth in claim 5, wherein a third conduit means is connected to the outlet pipe to return a portion of the mother liquor with crystal product therein to the main stream through the inlet pipe.

8. The classifying crystallizer system set forth in claim 7, wherein the second and third conduit means are connected to the inlet pipe, and the crystal product removing means is connected to the supply pipe.

9. The classifying crystallizer system set forth in claim 7, wherein the third conduit means is connected to the inlet pipe, and the second conduit means and the crystal product removing means are connected to the supply pipe.

10. A classifying crystallizer system for separating crystals from solutions, comprising a source of initial feed solution, a heater having an inlet and an outlet, a supply pipe interconnecting said source and said heater inlet, a crystallizer shell, means dividing said shell into upper and intermediate and lower chambers, an inlet pipe interconnecting said heater outlet and said upper chamber, a vapor outlet communicating with the upper portion of said shell above said inlet pipe for connecting to an area having a pressure lower than that in said shell, an outlet pipe communicating with the lower portion of said lower chamber, whereby passage of solution from said inlet pipe into said shell is accompanied by evaporation of a portion of the solution to form vapor which passes upwardly through said vapor outlet to provide supersaturation in the remaining portion of the solution which flows through said chambers in a main stream in which are formed nuclei and fines and undersized crystals and crystal product in mother liquor by desupersaturation and crystal growth, means in said intermediate chamber providing a first classification zone and means in said lower chamber providing a second classification zone, first conduit means communicating with said intermediate chamber for withdrawing a first side stream containing substantially only nuclei from the main stream through said first classification zone, second conduit means communicating with said lower chamber for withdrawing a second side stream containing crystals smaller than crystal product from the main stream through said second classification zone, means connecting said outlet pipe to said heater inlet to return the mother liquor thereto, means connecting said first conduit means to said heater inlet to return the first side stream thereto, and means connecting said second conduit means to said inlet pipe to return the second side stream thereto.

11. The classifying crystallizer system set forth in claim 10, wherein adjustable flow control means are provided in said supply pipe and in said first and second conduit means and in said outlet pipe, whereby the rate of flow of the first and second side streams through the first and second classification zones can be adjusted to remove thereby a predetermined size range of nuclei and fines and crystals.

12. A classifying crystallizer system for separating crystals from solutions, comprising an upstanding shell, a first downwardly extending funnel disposed in said shell and a second downwardly extending funnel disposed in said shell below and spaced from said first funnel, said funnels dividing said shell into upper and intermediate and lower chambers, said first funnel having a first opening in the lower portion thereof communicating with said intermediate chamber and said second funnel having a second opening in the lower portion thereof communicating with said lower chamber, means for heating a solution, a supply pipe interconnecting said heating means and a source of initial feed solution, an inlet pipe interconnecting said heating means and said upper chamber, a vapor outlet communicating with the upper portion of said shell above said inlet pipe for connecting to an area having a pressure lower than that in said shell, a first outlet in the upper portion of said intermediate chamber and spaced vertically above said first opening to provide therebetween a first classification zone, a second outlet in the upper portion of said lower chamber and spaced vertically above said second opening to provide therebetween a second classification zone, a third outlet in the lower portion of said lower chamber and spaced vertically below said second opening, whereby passage of the heated solution from said inlet pipe into said shell is accompanied by evaporation of a portion of the solution to form vapor which passes upwardly through said vapor outlet to provide supersaturation in the remaining portion of the solution which flows through said chambers in a main stream and to said outlets to form nuclei and fines and undersized crystals and crystal product in mother liquor by desupersaturation and crystal growth, first conduit means connected to said first outlet for withdrawing a first side stream containing substantially only nuclei from the main stream through said first classification zone, second conduit means connected to said second outlet for withdrawing a second side stream containing crystals smaller than the crystal product from the main stream through said second classification zone, means connected to said third outlet for removing crystal product from the mother liquor, and means connected to said first and second conduit means and said crystal product removing means to return the first and second side streams and the mother liquor to the main stream through said inlet pipe, at least said first conduit means being connected to said supply pipe whereby the first side stream passes through said heating means to dissolve the nuclei therein.

13. The classifying crystallizer system set forth in claim 12, wherein the flow velocity of the first side stream through the first classification zone is less than that of the second side stream through the second classification zone, and the length of the flow path in the first classification zone is less than that in the second classification zone.

14. The classifying crystallizer system set forth in

claim 13, wherein the first opening in the first funnel has a larger area than the second opening in the second funnel, and the cross sectional area of the first classification zone is less than that of the second classification zone.

15. The classifying crystallizer system set forth in claim 12, wherein a first baffle is mounted in the shell between the first opening in the first funnel and the first outlet to provide a first predetermined flow passage, and a second baffle is mounted in the shell between the second opening in the second funnel and the second outlet to provide a second predetermined flow passage.

16. A classifying crystallizer system for separating crystals from solutions, comprising an upstanding shell, a first downwardly extending funnel disposed in said shell and a second downwardly extending funnel disposed in said shell below and spaced from said first funnel and a third downwardly extending funnel disposed in said shell below and spaced from said second funnel, said funnels dividing said shell into upper and upper intermediate and lower intermediate and lower chambers, said first funnel having a first opening in the lower portion thereof communicating with said upper intermediate chamber and said second funnel having a second opening in the lower portion thereof communicating with said lower intermediate chamber and said third funnel having a third opening in the lower portion thereof communicating with said lower chamber, means for heating a solution, a supply pipe interconnecting said heating means and a source of initial feed solution, an inlet pipe interconnecting said heating means and said upper chamber, a vapor outlet communicating with the upper portion of said shell above said inlet pipe for connecting to an area having a pressure lower than that in said shell, a first outlet in the upper portion of said upper intermediate chamber and spaced vertically above said first opening to provide therebetween a first classification zone, a second outlet in the upper portion of said lower intermediate chamber and spaced vertically above said second opening to provide therebetween a second classification zone, a third outlet in the upper portion of said lower chamber and spaced vertically above said third opening to provide therebetween a third classification zone, a fourth outlet in the lower portion of said lower chamber and spaced vertically below said third opening, whereby passage of the heated solution from said inlet pipe into said shell is accompanied by evaporation of a portion of the solution to form vapor which passes upwardly through said vapor outlet to provide supersaturation in the remaining portion of the solution which flows through said chambers in a main stream and to said outlets to form nuclei and fines and undersized crystals and crystal product in mother liquor by desupersaturation and crystal growth, first conduit means connected to said first outlet for withdrawing a first side stream containing substantially only nuclei from the main stream through said first classification zone, second conduit means connected to said second outlet for withdrawing a second side stream containing fines and crystals smaller than the crystal product from the main stream through said second classification zone, third conduit means connected to said third outlet for withdrawing a third side stream containing crystals larger than those in said second side stream and smaller than the crystal product from the main stream through said third classification zone, means connected to said fourth outlet for removing crystal product from the mother liquor, and means connected to said first and second and third conduit means and said crystal product removing means to return the first and second and third side streams and the mother liquor to the main stream through said inlet pipe, at least said first conduit means being connected to said supply pipe whereby the first side stream passes through said heating means to dissolve the nuclei therein

17. A classifying crystallizer for separating crystals from solutions, comprising an upstanding shell, a first downwardly extending funnel disposed in said shell and a second downwardly extending funnel disposed in said shell below and spaced from said first funnel, said funnels dividing said shell into upper and intermediate and lower chambers, said first funnel having a first opening in the lower portion thereof communicating with said intermediate chamber and said second funnel having a second opening in the lower portion thereof communicating with said lower chamber, an inlet in said upper chamber for solution from which crystals are to be crystallized, a first outlet in the upper portion of said intermediate chamber and spaced vertically above said first opening to provide therebetween a first classification zone, a second outlet in the upper portion of said lower chamber and spaced vertically above said second opening to provide therebetween a second classification zone, and a third outlet in the lower portion of said lower chamber and spaced vertically below said second opening, whereby passage of the solution from said inlet through said chambers and said openings to said outlets serves to form nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation of the solution and crystal growth, said openings and said outlets being arranged to remove nuclei in said first classification zone through said first outlet and to remove crystals smaller than the crystal product in said second classification zone through said second outlet and to supply mother liquor containing crystal product through said third outlet.

18. The classifying crystallizer set forth in claim 17, wherein the distance from said first opening to said first outlet is less than the distance from said second opening to said second outlet.

19. The classifying crystallizer set forth in claim 18, wherein the diameter of said first opening is greater than the diameter of said second opening, and the cross sectional area of the first classification zone is less than the cross sectional area of the second classification zone.

20. A classifying crystallizer for separating crystals from solutions, comprising an upstanding shell, a first downwardly extending funnel disposed in said shell and a second downwardly extending funnel disposed in said shell below and spaced from said first funnel, said funnels dividing said shell into upper and intermediate and lower chambers, said first funnel having a first opening in the lower portion thereof communicating with said intermediate chamber and said second funnel having a second opening in the lower portion thereof communicating with said lower chamber, an inlet in said upper chamber for solution from which crystals are to be crystallized, a first outlet in the upper portion of said intermediate chamber and spaced vertically above said first opening, a first baffle mounted in said shell between said first opening and said first outlet to provide a predetermined flow passage and to define a first classification zone between said first opening and said first baffle, a second outlet in the upper portion of said lower chamber and spaced vertically above said second opening, a second baffle mounted in said shell between said second opening and said second outlet to provide a predetermined flow passage and to provide a second classification zone between said second opening and said second baffle, a third outlet in the lower portion of said lower chamber and spaced vertically below said second opening, whereby passage of the solution from said inlet through said chambers and said openings to said outlets serves to form nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation of the solution and crystal growth, said openings and said outlets and said baffles being arranged to remove the nuclei in said first classification zone through said first outlet and to remove crystals smaller than the crystal product in said second classification zone through

said second outlet and to supply mother liquor containing crystal product through said third outlet.

21. The classifying crystallizer set forth in claim 20, wherein each of the baffles is mounted on the shell around the periphery thereof and is inclined upwardly toward the associated funnel and is spaced therefrom to provide therebetween an annular flow passage of predetermined flow area.

22. The classifying crystallizer set forth in claim 20, wherein each of the baffles is mounted on the shell around the periphery thereof and comprises a weir ring surrounding the associated funnel and spaced therefrom to provide a passage therebetween of predetermined flow area.

23. A classifying crystallizer for separating crystals from solutions, comprising an upstanding shell, a first downwardly extending funnel disposed in said shell and a second downwardly extending funnel disposed in said shell below and spaced from said first funnel, said funnels dividing said shell into upper and intermediate and lower chambers, an inlet in said upper chamber for solution from which crystals are to be crystallized, a vapor conduit communicating with the upper portion of said chamber above said inlet for connecting to an area having a pressure lower than that in said shell, a plate mounted in said upper chamber and extending thereacross and disposed between said inlet and said vapor conduit and having an opening therein, a substantially cylindrical baffle mounted on said plate and surrounding the opening therein and extending downwardly past said inlet, said baffle being disposed nearer the wall of said upper chamber opposite said inlet to provide more flow space on the inlet side of said baffle, whereby the solution entering from said inlet impinges upon said baffle to flow therearound and downwardly therealong and to fall therefrom in a continuous liquid curtain, a first outlet in the upper portion of said intermediate chamber and spaced vertically above said first opening to provide therebetween a first classification zone, a second outlet in the upper portion of said lower chamber and spaced vertically above said second opening to provide therebetween a second classification zone, and a third outlet in the lower portion of said lower chamber and spaced vertically below said second opening, whereby passage of the solution from said inlet through said chambers and said openings to said outlets serves to form nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation of the solution and crystal growth, said openings and said outlets being arranged to remove nuclei in said first classification zone through said first outlet and to remove crystals smaller than the crystal product in said second classification zone through said second outlet and to supply mother liquor containing crystal product through said third outlet.

24. The classifying crystallizer set forth in claim 23, wherein the upper chamber of the shell is substantially circular in cross section, the plate is inclined downwardly away from the inlet, and the baffle is substantially circular in cross section and is arranged eccentrically with respect to said upper chamber and farthest away from said inlet.

25. A classifying crystallizer for separating crystals from solutions, comprising an upstanding shell, a first downwardly extending funnel disposed in said shell and a second downwardly extending funnel disposed in said shell below and spaced from said first funnel and a third downwardly extending funnel disposed in said shell below and spaced from said second funnel, said funnels dividing said shell into upper and upper intermediate and lower intermediate and lower chambers, said first funnel having a first opening in the lower portion thereof communicating with said upper intermediate chamber and said second funnel having a second opening in the lower portion thereof communicating with said lower intermediate chamber and said third funnel having a third opening in the lower portion thereof communicating with said lower chamber, an inlet in said upper chamber for solution from which crystals are to be crystallized, a first outlet in the upper portion of said upper intermediate chamber and spaced vertically above said first opening to provide therebetween a first classification zone, a second outlet in the upper portion of said lower intermediate chamber and spaced vertically above said second opening to provide therebetween a second classification zone, a third outlet in the upper portion of said lower chamber and spaced vertically above said third opening to provide therebetween a third classification zone, and a fourth outlet in the lower portion of said lower chamber and spaced vertically below said third opening, whereby passage of the solution from said inlet through said chambers and said openings to said outlets serves to form nuclei and fines and undersize crystals and crystal product in mother liquor by desupersaturation of the solution and crystal growth, said openings and said outlets being arranged to remove nuclei in said first classification zone through said first outlet and to remove crystals smaller than the crystal product in said second classification zone through said second outlet and to remove crystals larger than the crystals removed in said second classification zone and smaller than the crystal product in said third classification zone through said third outlet and to supply mother liquor containing crystal product through said fourth outlet.

26. A classifying crystallizer as set forth in claim 25, wherein the vertical extents of the classification zones become successively greater from the first classification zone toward the third classification zone, the diameters of the funnel openings become successively smaller from the first classification zone to the third classification zone, and the cross sectional flow areas of the classification zones become successively greater from the first classification zone to the third classification zone.

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