

Dec. 2, 1958

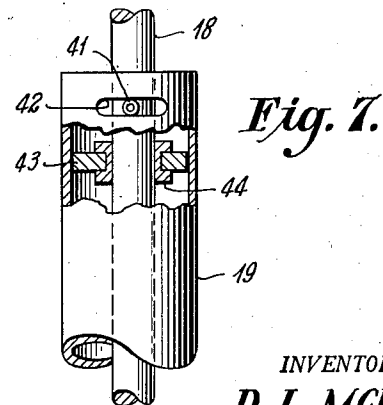
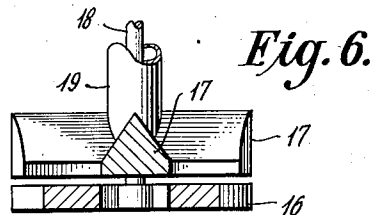
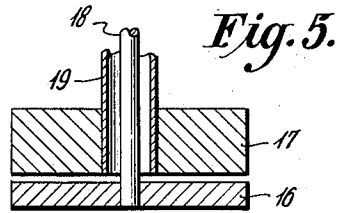
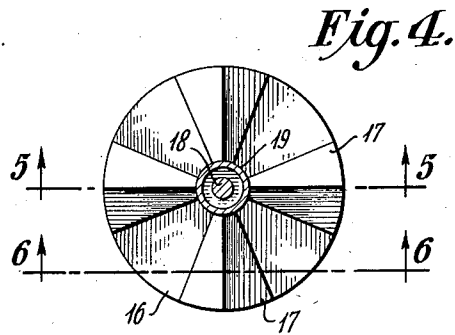
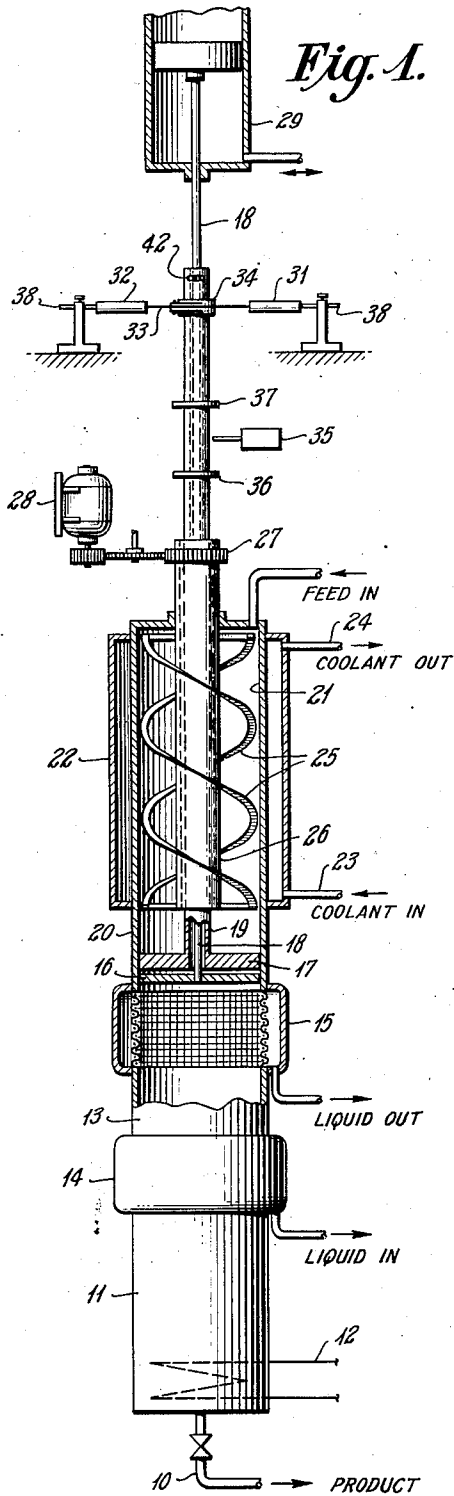
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2,862,797

CRYSTAL PURIFICATION APPARATUS

Filed Oct. 5, 1953

4 Sheets-Sheet 1



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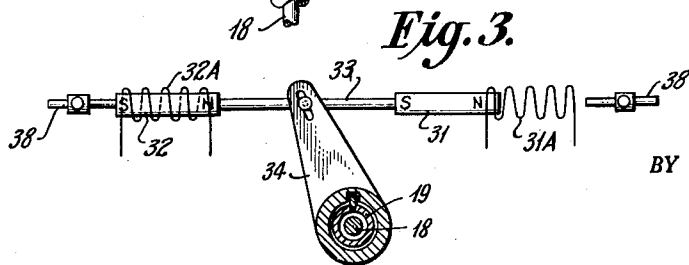
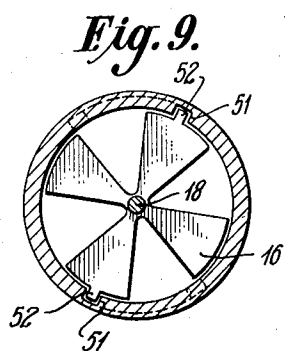
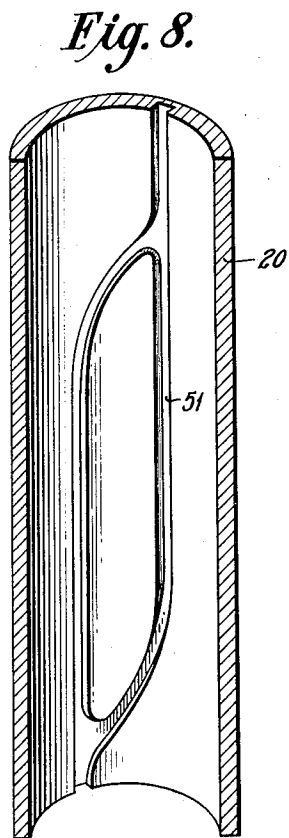
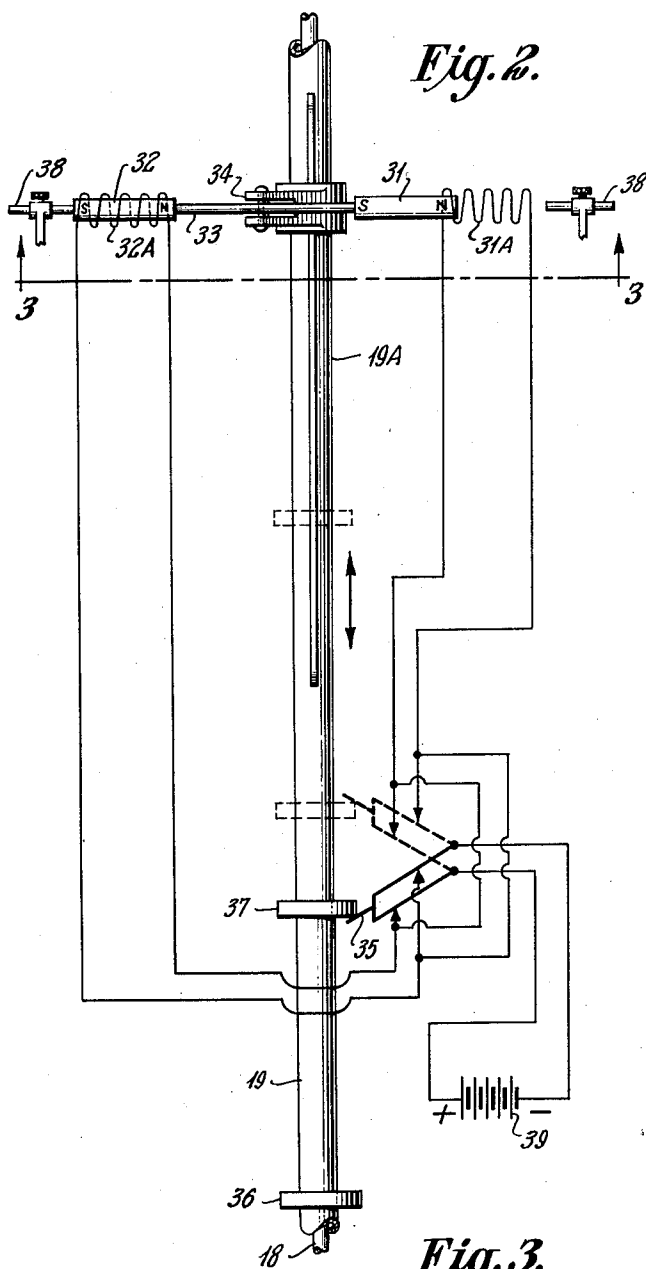
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4 Sheets-Sheet 2



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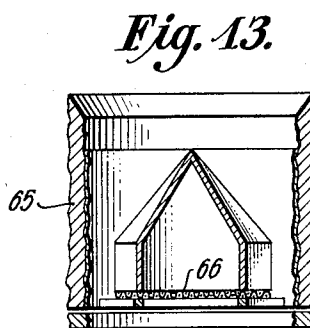
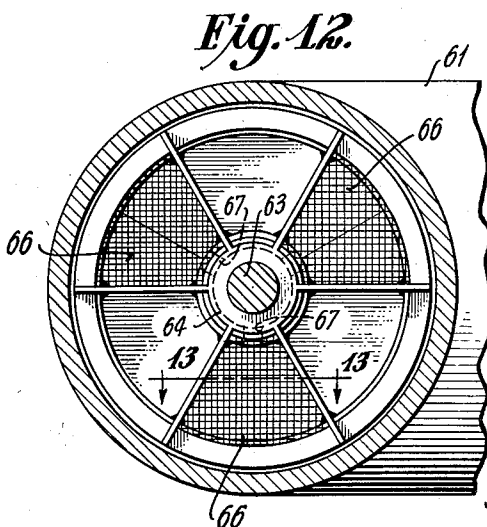
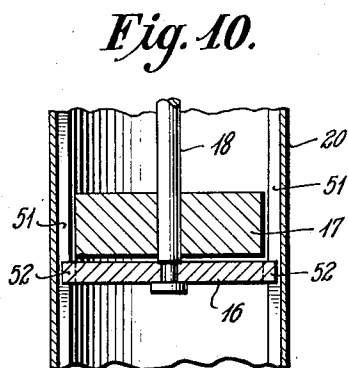
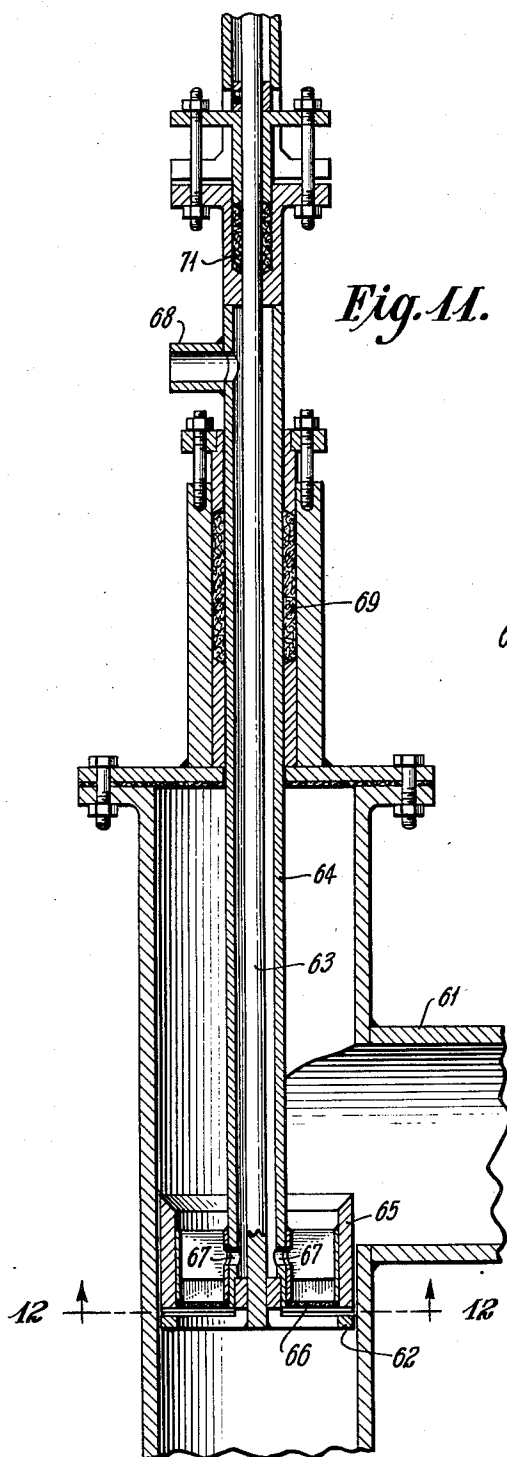
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CRYSTAL PURIFICATION APPARATUS

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4 Sheets-Sheet 3



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CRYSTAL PURIFICATION APPARATUS

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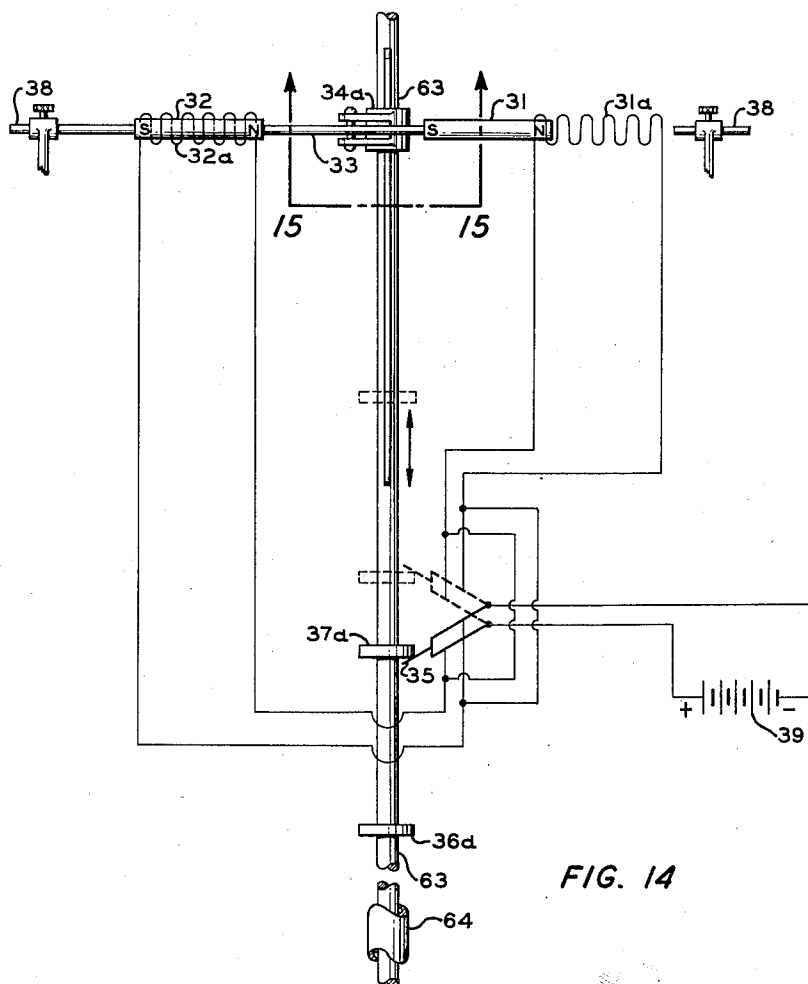


FIG. 14

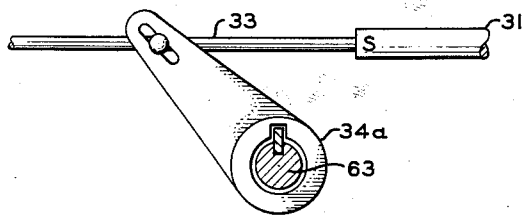


FIG. 15

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2,862,797

CRYSTAL PURIFICATION APPARATUS

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9 Claims. (Cl. 23—273)

This invention relates to crystal purification apparatus. In one aspect, it relates to an apparatus for the fractional separation of mixtures by crystallization. In one of its more specific aspects, it relates to an improved apparatus for moving a granular mass of crystalline material through a crystal purification zone.

In the separation of mixtures of compounds by fractional crystallization, various types of apparatus have been constructed for establishing contact between a mass of the crystallized material moving in one direction and a mother liquor moving in the opposite direction. Usually, these devices employ an auger or a piston for moving the crystals toward the heating section of the purification apparatus. One of the principal difficulties with the auger type crystal movers is the tendency of the auger to become clogged by bridging over of the crystals between vanes of the auger so that a revolving plug of crystals results and no forward movement of the crystal slurry is obtained.

In a piston type crystal purification apparatus, having filters in the wall of the vessel for the removal of mother liquor, a solid piston is usually employed. One of the principal difficulties with the solid piston type apparatus is that the reduction of pressure in the melting section of the apparatus caused by the return stroke of the piston disturbs the crystal mass, and in some instances cause a reverse movement of the crystal mass in the heater section of the apparatus. Disturbance of the crystal mass is particularly troublesome when a vaporizable material such as carbon dioxide is present in the mixture so that the reduction in pressure caused by the return stroke of a solid piston results in the formation of gas bubbles. Disturbance of the crystal mass encourages channeling of the liquid through the crystals.

I have invented a crystal purification apparatus which operates efficiently on any slurry of crystals and mother liquor and leaves the compacted crystal mass in the heating section undisturbed by the piston on its return stroke. The apparatus of my invention can be employed in any position such as horizontal, inclined, inverted, or vertical. However, I prefer to use my invention in a vertical crystal purification apparatus. Of particular importance are the features of a piston head valve which assures positive movement of the crystal mass and positive means for opening the valve on the return stroke of the piston and closing the valve on the forward stroke of the piston. Another important feature of my invention is that the cooling zone, filtration zone and purification zone can be located in line making possible the handling of slurries of high solids content. It has been found that plugging of high solids content slurries tends to occur when a change of direction of flow of the slurry is necessitated as in a side feed crystal purification apparatus. Economies in construction are also possible in an in line crystal purification apparatus.

Another important feature of my improved crystal purifier is that the compacted crystal mass in the heating section is not disturbed by the return stroke of the piston

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and channeling of the liquid through the crystal mass is discouraged.

Each of the following objects will be attained in at least one aspect of the invention.

5 It is an object of this invention to provide an improved crystal purification apparatus.

It is another object to provide a piston for moving a slurry of crystals through a crystal purification apparatus wherein the pressure in the heating section of the apparatus is not reduced on the return piston stroke.

10 It is another object to provide a valved piston head and positive means for opening and closing the valve.

Other objects and advantages of my invention will be apparent to one skilled in the art upon reading the disclosure of this invention and the attached drawing.

15 Figure 1 is a longitudinal sectional view of a vertically disposed fractional crystallizer showing a preferred specific embodiment of my crystal purification apparatus in elevation.

20 Figure 2 is a diagrammatic sketch of one preferred specific embodiment of the valve actuation device of my invention.

Figure 3 is a view of Figure 2 along lines 3—3.

25 Figure 4 is a plan view of the valved piston of my invention.

Figure 5 is a view along lines 5—5 of Figure 4.

Figure 6 is a view along lines 6—6 of Figure 4.

Figure 7 is a longitudinal view, partially in section, of the bearing assembly of Figure 1.

30 Figure 8 is a longitudinal sectional view of a portion of the crystal purification apparatus showing a second preferred embodiment of the valve operating device of my invention.

35 Figure 9 is a cross-sectional view of the embodiment of Figure 8.

Figure 10 is a sectional view of a modification of the piston assembly wherein the forward plate is free to rotate.

40 Figure 11 is a longitudinal sectional view of a crystallizer showing another preferred specific embodiment of my invention.

Figure 12 is a view along lines 12—12 of Figure 11.

Figure 13 is a view along lines 13—13 of Figure 12.

45 Figure 14 is a modification of the valve actuation device as applied to Figure 11.

Figure 15 is a view of Figure 14 along lines 15—15.

Figure 1 shows a vertical crystal purification apparatus wherein the bottom product is taken out at 10. A heating section 11 having a heater 12 is situated in the lower portion of the apparatus. In the filtering section 13 high melting point product contaminated with occluded mother liquor is removed through a filter surface indicated at 14 and mother liquor is removed through a filter surface indicated at 15.

50 A piston assembly composed of forward piston valve plate 16 and rear piston valve plate 17 is caused to reciprocate by piston shaft 18 concentrically disposed within tube 19. Piston valve plates 16 and 17 are formed of segments, which when superimposed one upon the other, leave open spaces between the segments and thus comprise the piston valve in open position. When the piston valve plates are rotated so that the segments are not superimposed, the piston valve is in closed position. In one preferred modification of the invention the segments of plate 16 are composed of a filter surface such as a screen so as to provide free passage of liquid on the compression stroke. The piston travels in cylinder section 20 of the apparatus. The upper portion of the crystallizer apparatus comprises a chilling section for freezing the material to be purified. A ribbon type spiral scraper 25 is rotated by tube 26 and scrapes the crystals from the surface of chilling section 21. A coolant is

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circulated through the jacketed section of the chiller 21 and is admitted through line 23 and removed through line 24. The feed enters at the top of the chilling section and flows by gravitation down to the piston. A gear 27 is secured to tube 26 and is actuated by motor 28 so as to cause rotation of the ribbon scraper 25.

Figure 2 shows one preferred method for actuation of the valved piston. The piston valve is caused to open and close by rotation of the rear piston valve plate resulting from rotating tube 19. Alternatively the forward piston plate 16 can be caused to rotate and the rear plate 17 caused to reciprocate without rotation. Tube 19 has trip members 36 and 37 externally of the crystal purification cylinder for actuating switch 35 at each extreme position of the piston. Trip 37 operates switch 35 so as to complete an electrical circuit energizing coil 31A so as to repel permanent magnet 31 and energizing coil 32A so as to attract permanent magnet 32 thus rotating arm 34 through linkage 33 and turning tube 19 and rear piston valve plate 17 so as to superimpose the segments of the piston valve plates and open the piston valve. Trip 36 actuates switch 35 so as to reverse the electrical circuit and cause coil 31A to attract permanent magnet 31 and coil 32A to repel permanent magnet 32 and thus move arm 34 through linkage 33 so as to rotate tube 19 and piston valve plate 17 in the opposite direction and thus close the piston valve.

Figure 4 shows a detailed view of one modification of the valved piston of the invention.

In the modification shown in Figures 8, 9 and 10 the tube 19 is eliminated and rear piston valve plate 17 is secured rigidly to piston shaft 18. Forward piston valve plate 16 is secured to a portion of piston shaft 18 projecting through valve plate 17 by a bearing and is free to rotate about piston shaft 18 as shown in Figure 10. Plate 16 has projections 52 in its circumference adapted to fit in groove 51 of the cylinder portion 20 of the crystal purifier apparatus. Groove 51 as shown in Figure 8 provides an endless track in cylinder 13. This track is composed of two parallel paths each of which is joined to the other by a curved path so that piston plate 16 is caused to rotate a predetermined amount near the end of a piston stroke and is caused to rotate an equal amount in the opposite direction at the other end of the piston stroke. This rotation of plate 16 opens the piston valve at the completion of the compression stroke, maintains the valve open during the return stroke, closes the valve at the completion of the return stroke, and maintains the valve closed during the compression stroke.

In the modification of my invention shown in Figure 1, piston shaft 18 and tube 19 are caused to reciprocate together by the bearing assembly shown in Figure 7. Tube 19 has slot 42 which engages a pin 41 through shaft 18. Thus, tube 19 is free to rotate about shaft 18 a predetermined amount but is prevented from longitudinal movement. A sealing ring 43 is secured to the inside of tube 19, and packing material 44 provides a sealing contact with piston shaft 18 and tube 19, thus preventing leakage of liquid through the annular space between piston shaft 18 and tube 19. The bearing assembly can be situated near the piston plates if desired. Other means for preventing longitudinal movement of tube 19 with respect to shaft 18 can be employed, for example a pin through shaft 18 directly above plate 17 will serve to prevent longitudinal movement of tube 19 with respect to shaft 18.

A feature which contributes to the successful operation of the crystal purification apparatus of my invention is the configuration of the solid segments of rear piston plate 17. This configuration is shown in Figures 4, 5 and 6. The height of the solid segments of plate 17 is the same at the junction of tube 19 at the center as at the periphery of plate 17. The width of the base of each segment increases from the center to the periphery of the plate. Thus the angle of the peak of the segments is

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greater at their periphery than at the center. As a result of the smaller angle at the center the change of direction of the flow of slurry at the center is less and the rate of flow is greater than at the periphery where the angle of the segment peak is greater and the change of direction of the flow of slurry is greater. This results in an even flow of slurry without plugging because the flow rate is greatest at the center where the solid segments are nearest to each other and the flow rate is less at the periphery where the solid segments are farthest apart.

In carrying out crystal purification in the apparatus of my invention, as shown in Figure 1, the liquid material to be crystallized is introduced into the top of the chilling section of the apparatus and as the crystals are formed they are conveyed downwardly to the purification section of the apparatus. As the piston returns upwardly from a compression stroke, the piston valves are opened and the crystals pass downwardly through the ascending piston valve. At the top of the return stroke of the piston, the valves are closed and the crystals are compressed downwardly toward the melting section of the apparatus. The crystals upon being pressed downwardly displace liquid so as to cause an upward flow of crystal melt and occluded mother liquor. Excess mother liquor in the slurry of crystals is removed through the wall filters during the compression stroke of the piston. Product is removed from the heated section of the column as a crystal melt.

Figure 11 shows a vertical crystal purification apparatus wherein the feed enters at the side. In this figure the crystal slurry enters the column at 61. The piston assembly is comprised of forward plate 62 having solid and open segments. Piston shaft 63 rotates the plate 62 and tube 64 does not rotate. The sloping segments of rear piston plate 65 are hollow and the open forward side or base of each segment is covered with a screen 66. Ports 67 communicate between the interior of the segments of plate 65 and the annulus between shaft 63 and tube 64. Liquid passing through screens 66, and ports 67 is withdrawn through outlet 68. Tube 64 reciprocates through packing gland 69. Packing gland 71 seals the annulus between shaft 63 and tube 64.

Figure 12 shows the screen bases of the segments of valve plate 65.

Figure 13 shows the interior of a segment of valve plate 65.

The operation of the apparatus of Figure 11 is essentially the same as the operation of the apparatus of Figure 1. The slurry of crystals and mother liquor comprising the feed is introduced through side entry 61 by an auger or other suitable means.

The piston valves can be operated by the solenoid arrangement of Figure 2 or by an air cylinder, in which case trips 36 and 37 operate a valve instead of a switch. Other means can be employed to rotate the valves at the completion of each stroke.

Figure 14 shows a modification of the valve actuation device as applied to shaft 63 of Figure 11 so as to rotate plate 62 with respect to plate 65. Trips 36a and 37a actuate switch 35 at the completion of each piston stroke to reverse the polarity of solenoids 31 and 32 so as to rotate shaft 63.

The screened segments of the rear valve plate permit handling a greater amount of liquid and relieve the load on the filters downstream from the piston.

Variations and modifications are possible within the scope of this invention, the essence of which is that an improved crystal purification apparatus has been devised employing a solid piston containing piston-head valves which open on the return stroke of the piston to allow a crystal slurry to pass therethrough and which close on the compression stroke to drive the crystal slurry forward.

I claim:

1. An apparatus comprising a conduit and means for

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moving a slurry of crystals and liquid through said conduit said means for moving a slurry comprising a piston shaft; a piston assembly comprising a plate having openings therethrough rigidly secured at its center to said shaft and a plate having matching openings therethrough rotatably secured at its center to said shaft; a projection extending from the edge of said rotatable plate; said conduit having a groove in its wall adapted to receive said projection, said groove forming an endless track comprising two spaced grooves parallel with the longitudinal axis of the conduit, each groove being joined to the other by one end of each groove converging into the other groove whereby said rotatable plate is caused to rotate a predetermined amount with each piston stroke so as to match said openings at one end of the stroke and to unmatched said openings at the other end of the stroke.

2. In an apparatus for the separation and purification of crystals from a slurry of crystals and mother liquor comprising an elongated closed vessel having a heating section and product outlet near one end, a feed inlet and cooled section near the other end, and a liquid outlet intermediate the two ends, the improvement comprising a valved piston assembly comprising a first piston plate having sloping rearwardly projecting solid portions and identical adjacent openings, a tube coaxially secured to the rearward side of said first plate, a piston shaft disposed within said tube having rigidly secured thereto a second piston plate having openings therethrough spaced identically with the openings in said first plate; trip means located on said tube; an electrical circuit comprising a plurality of solenoids and switch means operatively connected to said tube so as to rotate said tube a predetermined amount; said switch in said electrical circuit actuated by said trips so as to change the direction of current flow in said circuit so as to change direction of rotation of said tube.

3. In an apparatus for the separation and purification of crystals from a slurry of crystals and mother liquor comprising an elongated closed vessel having a heated section and product outlet near one end, a feed inlet and cooled section near the opposite end, a liquid outlet intermediate two ends, and a cylinder portion positioned intermediate the cooled section and the liquid outlet, the improvement which comprises a valved piston assembly positioned so as to reciprocate in said cylinder portion comprising a piston shaft, a first piston plate having openings therethrough and rigidly secured at its center to said shaft; a second piston plate having openings therethrough matching those in the first plate rotatably secured at its center to said shaft; a projection extending from the edge of said rotatable plate; said cylinder portion of said apparatus through which said piston assembly reciprocates, having a groove in its wall adapted to receive said projection, said groove forming an endless track comprising two spaced grooves parallel with the longitudinal axis of the vessel, each groove being joined to the other by one end of each groove converging into the other groove whereby said rotatable plate is caused to rotate a predetermined amount with each piston stroke so as to align said openings at one end of the stroke and to place the openings out of alignment at the other end of the stroke.

4. An apparatus comprising an elongated chamber and means for moving a slurry of crystals through said elongated chamber said means for moving slurry comprising a first piston plate having openings therethrough and having sloping rearwardly projecting hollow portions with open forwardly directed faces; filter surfaces covering the open faces of said hollow portions; conduit means leading from said hollow portions to the exterior of the chamber; a second piston plate adjacent said first piston plate and having openings therethrough spaced identically with the openings in said first piston plate; means for driving said piston plates; and means for rotating one of said plates so as to place the openings out of alignment

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on the compression stroke and to place the openings in alignment during the return stroke.

5. An apparatus comprising a chamber and means for moving a slurry of crystals and liquid through said chamber said means for moving slurry comprising; a piston assembly comprising a rearward piston plate having longitudinal openings therethrough and having sloping rearwardly projecting solid portions and a forward piston plate having longitudinal openings therethrough, spaced identically with the openings in said rearward piston plate; driving means to reciprocate said piston assembly in said chamber; and means to rotate one of said piston plates so as to place the openings out of alignment with the beginning of the compression stroke and to rotate said plate in the opposite direction so as to place the openings in alignment at the beginning of the return stroke.

6. In an apparatus for the separation and purification of crystals from a slurry of crystals and mother liquor comprising an elongated, closed vessel having a heated section and product outlet near one end, a feed inlet and cooled section near the other end and a liquid outlet intermediate the two ends, the improvement comprising a valved piston assembly reciprocally positioned in said vessel and comprising a forward piston section having longitudinal openings therethrough and a rearward piston section having identically spaced openings longitudinal therethrough; driving means for reciprocating said piston assembly in said cylinder; means to rotate one of said sections so as to place said openings out of alignment at the beginning of the compression stroke of said piston assembly; and means to rotate said section in the opposite direction so as to place the openings in alignment at the beginning of the return stroke of said piston assembly.

7. Apparatus for crystallization of at least one component in a liquid mixture and purification of the crystals produced comprising an elongated column, closed at each end and having an inlet at one end and an outlet at the other end; a jacketed section of said column adjacent said inlet for circulation of coolant to freeze crystals to be purified; an auger positioned in said jacketed section to move crystals toward the outlet of said column; a first power source operatively connected to said auger so as to turn said auger; a liquid pervious, crystal impervious filter communicating between the exterior of the column and the interior of the column at a point intermediate the jacketed section and the outlet of the column; a heater positioned in said column adjacent said outlet intermediate said outlet and said filter; a valved piston assembly reciprocally positioned in said column intermediate said jacketed section and said filter in sealing contact with the column wall comprising a first piston plate having a plurality of openings communicating between the interior of the column upstream and downstream of said piston assembly with respect to the flow of crystals; a piston shaft operatively connecting said first plate to a second source of power to reciprocate said piston; a second piston plate positioned adjacent and upstream to said first plate and having openings therethrough matching those of said first plate; a tube connected to said second plate at its upstream side encircling said piston shaft and having a circumferential slot therethrough; a pin secured to said shaft and projecting through said slot; a third power source operatively connected to said tube to rotate said tube a partial turn so as to place the openings in said plates in alignment and out of alignment; a pair of projections extending from said tube and spaced apart substantially equal to the length of stroke of said reciprocating piston; a trigger positioned so as to be actuated by said projections and operatively connected to said third power source to rotate said tube so as to place the openings of said plates in alignment when the piston is travelling countercurrent to the flow of crystals and out of alignment when the piston is travelling concurrent to the flow of crystals.

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8. The apparatus of claim 7 wherein the third power source is an electrical circuit containing a pair of solenoids connected through a linkage so as to rotate said tube; and said trigger is a switch in said circuit to reverse the flow of current through the solenoids at the completion of each piston stroke.

9. In an apparatus for the separation and purification of crystals from a slurry of crystals and mother liquor which comprises an elongated closed vessel having a heated section and product outlet at one end, a feed inlet at the other end, and a liquid outlet intermediate the two ends, the improvement comprising a valved piston assembly comprising a first piston plate having a plurality of openings therethrough and a tube coaxially secured to said plate and communicating with the exterior of said vessel at the feed inlet end; a piston shaft coaxially disposed within said tube and said first plate having one

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end operatively connected to a source of reciprocating power externally of said vessel at the feed inlet end thereof and having rigidly secured to the other end a second piston plate having openings therethrough spaced identically with the openings in the first plate; and means to rotate said tube to place the openings in said plate out of alignment at the beginning of the compression stroke and to align said openings at the beginning of the return stroke.

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