

[54] <b>CRYSTALLIZATION COLUMN</b>	2,150,608	3/1939	Olier.....23/270 R
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[22] Filed: <b>June 12, 1969</b>	3,389,970	6/1968	Scheibel.....196/14.52
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[21] Appl. No.: <b>832,668</b>	2,667,407	1/1954	Fenske.....196/14.52
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- [51] Int. Cl. ....B01d 9/04, B01d 9/02, B01d 11/04
- [58] Field of Search .....23/273 F, 267, 270, 270.5, 23/310, 309, 295; 62/58, 123; 196/14, 14.52

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[57] **ABSTRACT**

The column is divided into individual mixing chambers by separating walls disposed transversely of the flows of the phases. Each separating wall comprises a passageway for a counterflow of the phases between adjacent mixing chambers and agitating means are disposed in each chamber to agitate the phases in a whirling manner. Transporting means are also provided to effect passage of the crystals between the mixing chambers.

**1 Claim, 11 Drawing Figures**

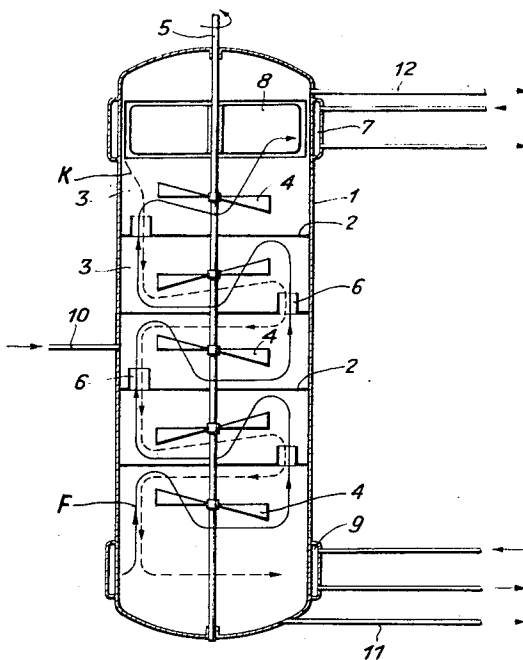


Fig. 1

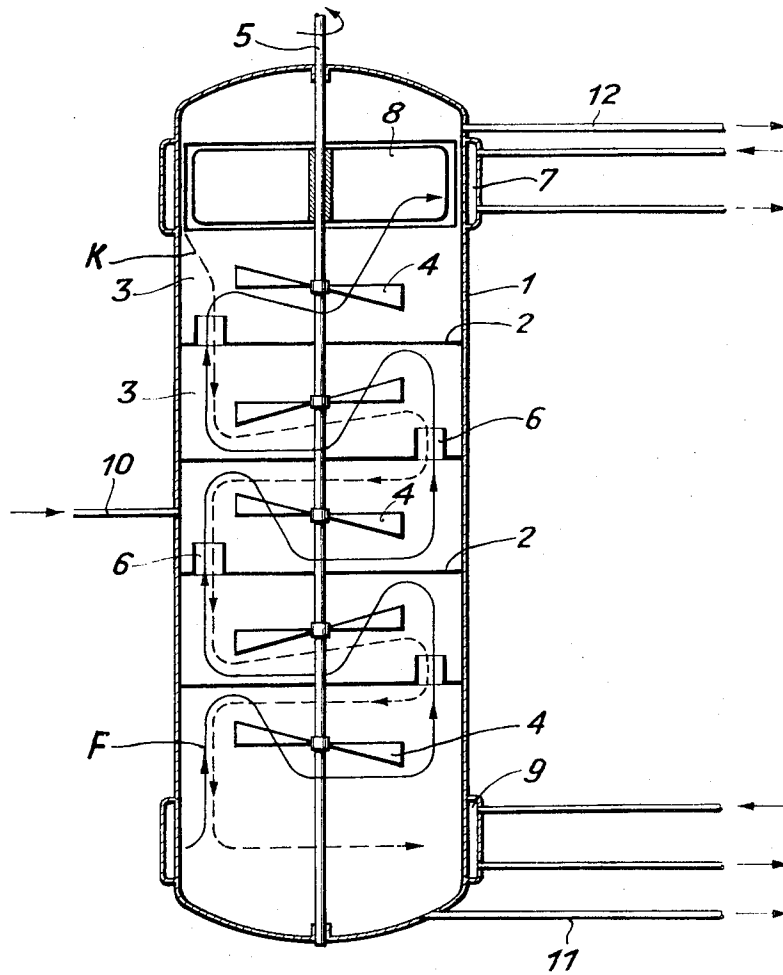
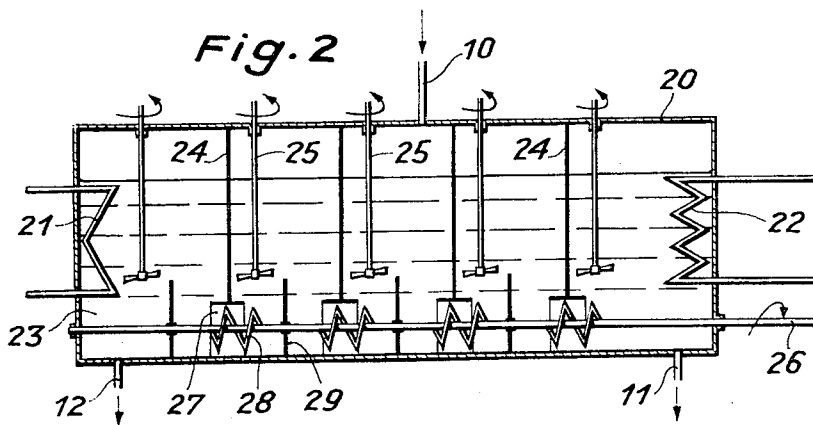


Fig. 2



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Fig. 3a

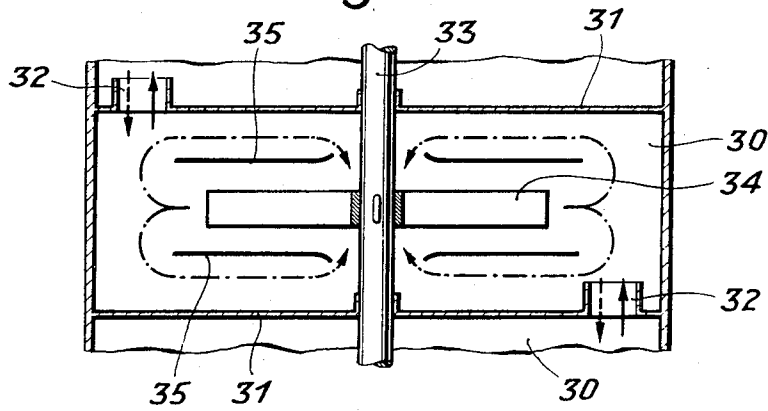


Fig. 3b

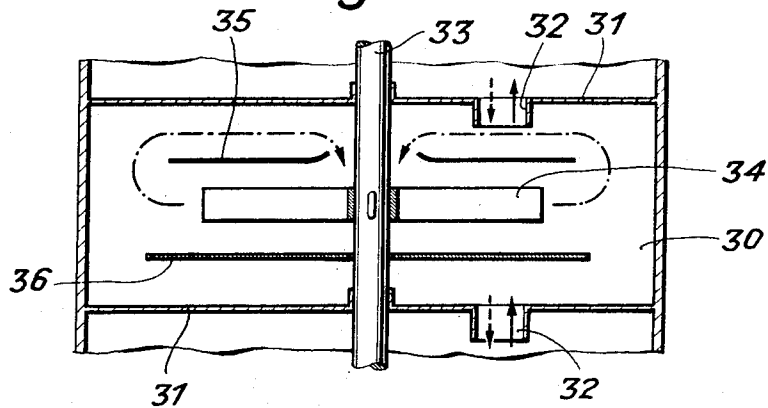
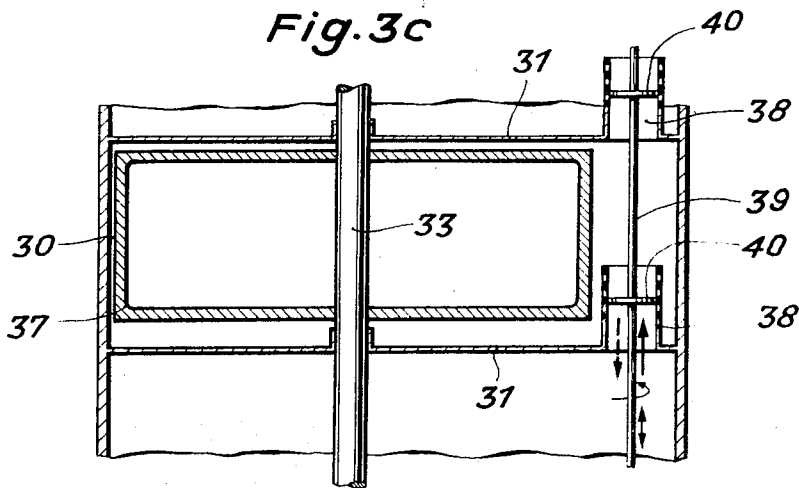


Fig. 3c



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Fig. 4a

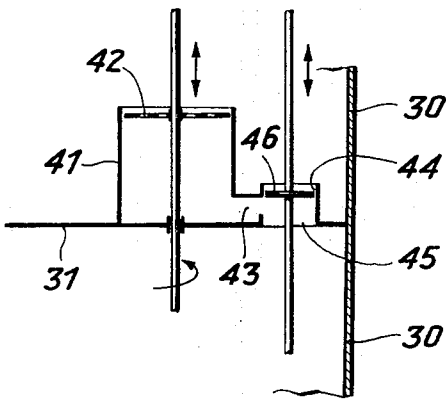


Fig. 4b

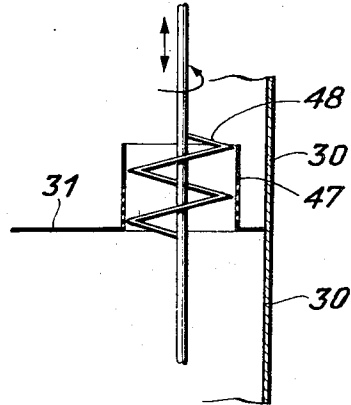


Fig. 4c

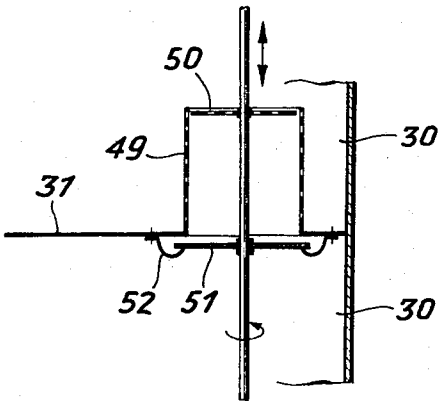
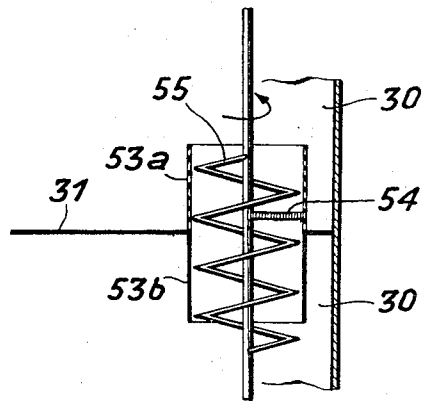
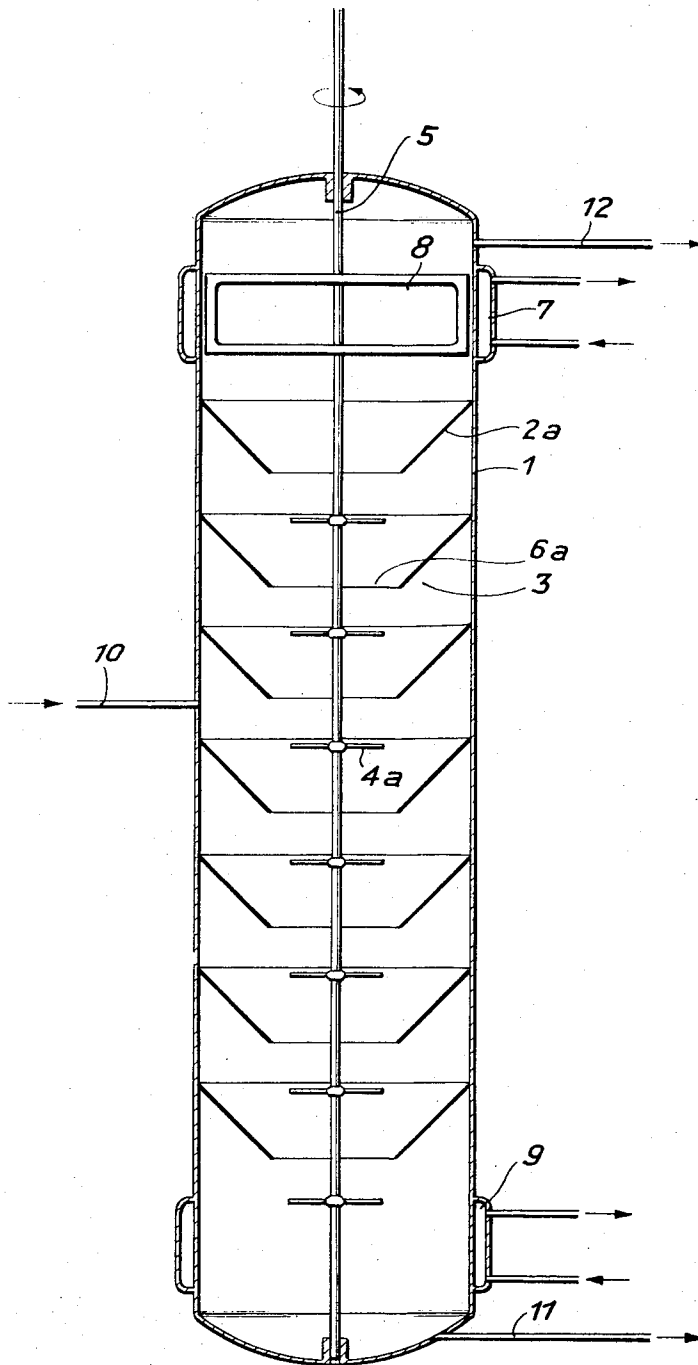


Fig. 4d



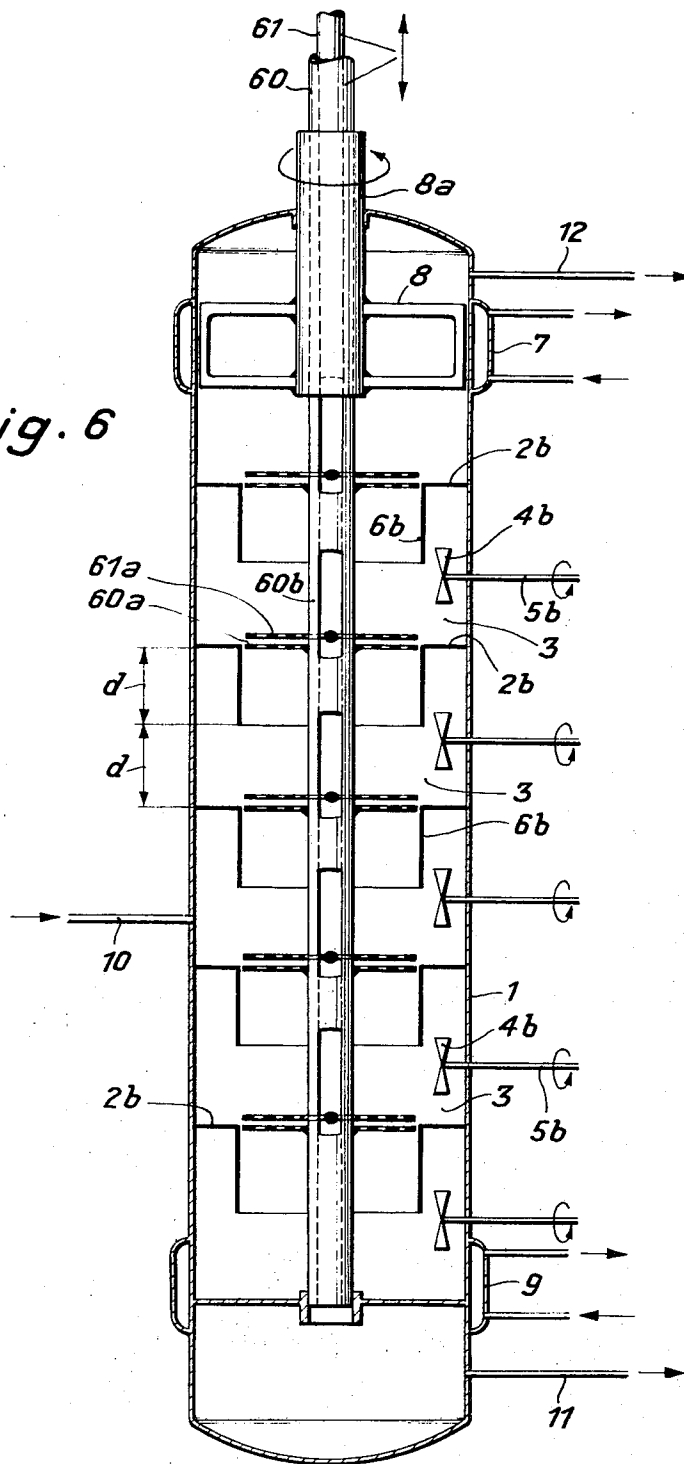
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Fig. 5



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Fig. 6



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**CRYSTALLIZATION COLUMN**

This invention relates to a crystallization column.

As is well known, column crystallization for general separating and purifying processes offers various advantages in comparison with distillation or rectification. Thus, in many cases, it is possible to achieve higher separation factors because the crystallization state is of the highest order. Furthermore, separation through crystallization can be done with a relatively low energy requirement and at a relatively low temperature, which is of considerable importance when it is desired to separate mixtures of substances that are thermally unstable in a higher temperature region. However, one of the chief problems which makes general utilization of column crystallization difficult relates to a controlled transport of the crystals which are formed.

Heretofore, crystallization columns have generally been constructed with a cooling device at one end and a heating device at an opposite end which operate so that a fluid phase and a crystallized phase of a mixture of substances supplied to the column are conducted in counterflow to each other. Because of the phase equilibrium between the two phases, the component having the lower melting point accumulates at one end while the component having the higher melting point accumulates at the other end. For example, one known crystallization column has included an annular gap, in which a coil spring has been rotated so as to contact the walls about the annular gap. The rotation of this coil spring has caused the solid phase to become transported downwardly while the fluid phase of necessity escapes in an upward counterflow. This form of construction, in the case of an extremely small column diameter, achieves a good efficiency; however, any increase in the column diameter creates great difficulties. In fact, up to the present time, such a column has not been known. Furthermore, the cost of the apparatus, relative to the coil spring, would be uneconomically high, and there would also be considerable friction between the spring and the column wall.

Other crystallization columns have also been known to use a piston that moves up and down in the column in order to convey the crystals in the downward direction while the fluid phase is supposed to be transported upwardly positively, in counterflow. However, in these instances, it has not been possible to avoid the formation of channels in the "plug of crystals" which is pushed downwardly by the piston through which the fluid phase can flow. As a result, the separating action of these columns has been poor. Moreover, because there is no agitating action, the exchange of substances across the column cross-section proceeds only extremely slowly.

Accordingly, it is an object of the invention to provide a crystallization column that is suitable for industrial purposes.

It is another object of the invention to provide a rapid interchange of substance between the crystallized and the fluid phases across the column cross-section of a crystallization column.

It is another object of the invention to transport the crystallized phase within a crystallization column in a highly efficient manner.

Briefly, the invention provides a crystallization column of vertical or horizontal disposition in which a

cooling device and a heating device are disposed at respective ends and in which the interior of the column is divided into a plurality of mixing chambers by means of separating walls which are set transversely of the flow direction of the phases therein. Each mixing chamber is also provided with at least one agitating device for agitating the phases therein and is connected via passages in the respective separating walls to the adjacent mixing chambers. In this way, at least the main amount of the fluid phase and the entire amount of the crystallized phase are conducted from mixing chamber to mixing chamber while contacting each other in counterflow. In addition, spatially separated devices are provided for the mixing of the phases in the individual mixing chambers and for their transport from mixing chamber to mixing chamber.

Because of the good mixing of the two phases obtained in accordance with the invention in the individual mixing chambers, the temperature gradients matching phase equilibrium is reached relatively rapidly, and the concentration differences in mixing chambers become equalized rapidly. Further, as at least the chief amount of the fluid phase and the entire amount of the crystallized phase contact one another in counterflow from mixing chamber to mixing chamber, the fluid is prevented to a great extent from flowing over with the crystallized phase from one mixing chamber into the next mixing chamber. That is, the counterflowing fluid passing out of one mixing chamber forces the fluid in the other chamber to remain in that mixing chamber. Thus, an extremely efficacious washing action on the crystals is exerted by the counterflowing fluid.

In one embodiment of the invention, the separating walls connect the mixing chambers in fluid-tight relation with the column wall, while the passageways for any structural elements such, for example, as the shafts for the agitating devices, pass through the separating walls in fluid-tight relation so that the entire phases are conducted through one and the same passageway.

These and other objects and advantages of the invention will become more apparent from the following detailed description and appended claims taken in conjunction with the accompanying drawings in which:

FIG. 1 illustrates a schematic representation of one form of construction of a vertical crystallization column according to the invention;

FIG. 2 illustrates a crystallization column made in the form of a horizontal tank according to the invention;

FIGS. 3a to 3c each illustrate different constructions for mixing chambers with connected passage channels in accordance with the invention;

FIGS. 4a to 4d each illustrate different constructions of passage channels and transportation devices disposed in the channels according to the invention;

FIG. 5 illustrates a modified crystallization column according to the invention; and

FIG. 6 illustrates another modified crystallization chamber according to the invention.

Referring to FIG. 1, the crystallization column consists of a cylindrical, vertically disposed tube 1, which is closed at its top and bottom, and which is divided by horizontal separating walls 2 into individual mixing chambers 3. Each mixing chamber has an agitating

device disposed therein which consists of vaned wheels 4, driven by a rotating shaft 5 which passes through the column. Alternatively, each mixing chamber can be provided with an agitator which passes through the column wall or with a plurality of agitators. Each of the separating walls 2 have tubular nozzles 6 therein which serve as passage channels, and which in two adjacent walls are offset relatively to one another. Alternatively, instead of these tubular nozzles 6, the passage channels may also be made as segments which are partitioned off from the remainder of the column cross-section by a vertical wall. In any event, the cross-section of a passageway advantageously amounts to approximately 10 to 30 percent of the total column cross-section at the level of the passageway.

A cooler 7 is disposed at the upper end of the column and has a surface which is continuously cleared of crystals by a scraper 8 fastened to the shaft 5. Depending on the melting point of one component, or on the melting point of an eutectic mixture of a mixture of substances to be separated or purified, an appropriate cooling medium which on occasion can be cooling water, flows through the cooler. In principle, it is also possible to dispose the cooler outside the column and to conduct the head product out of the column into the cooler for crystallization and subsequent return of the crystals into the column. A heater 9, for example, a steam heater, is also disposed at the lower end of the column. On occasion, the column may also be heated electrically.

In operation, in order to separate or purify a mixture of substances, for example, a mixture of hydrocarbons, the mixture is fed into the column through a conduit 10 in the side of the tube 1. This mixture may be fluid, or under certain circumstances, may already be partly crystallized.

At the beginning of the process, the column becomes filled with the mixture of substances, for example, up to a level above the cooling device 7. Then the cooling device is put into operation. Thereafter, crystals of the component having the higher melting point become formed on the exposed cooler surfaces and are subsequently shaved off the surfaces by the scraping device 8. The loose crystals are then whirled by the agitator 4 in the uppermost mixing chamber 3 in a generally radial plane with the fluid in such a way that these crystals continuously come into contact with new fluid, and an intense interchange of substances takes place. Assuming that the crystals are of greater specific weight than the fluid after being whirled thoroughly in the mixing chamber, a following separation under the influence of gravity takes place and the crystals sink to the bottom. Further, under the influence of gravity, the crystals flow through the first passage channel 6 without further mixing but with a quieting down of the whirling movement, and pass downwardly into the next mixing chamber. At the same time, a corresponding quantity of fluid necessarily passes out of the lower mixing chamber through the passage channel 6 into the upper mixing chamber. Because of this, the fluid in the upper mixing chamber is prevented to a great extent from penetrating into the crystals and into the lower mixing chamber. Once in the lower mixing chamber 3, the crystals again become whirled and brought into contact with the fluid therein. Then, these crystals are

again transported through a passage channel 6 into the next lower mixing chamber. A corresponding quantity of fluid is forced out of that chamber into the mixing chamber above, and so forth. As soon as the crystals, which during their passage through the column have gained a great degree of purity of one component, have reached the lower part of the column, they are melted by the heater 9 into a fluid state. A part of the fluid substance then flows perpendicularly to the crystals upwardly while another part can be removed through a discharge conduit 11 in the lower end of the column. The upper part of the column is provided with a second discharge conduit 12 for the removal of the product having the lower melting point, or the eutectic mixture product.

The arrows K and F as shown, trace in a purely schematic way, the counterflow of the crystallized and fluid phases through the column without the flow direction of the whirling phases in the mixing chambers being shown in detail.

This example of operation relates to a continuous column crystallization; however, it is self-evident that batch operation of such a column is also possible.

Referring to FIG. 2, the crystallization column can also be made as a horizontal tank 20. In this case, the cooler 21 and heater 22 are disposed at opposite ends of the column and the supply conduit 10 is disposed on the top side of the tank 20 with the discharge conduits 11, 12 disposed on the opposite side. The column is also divided, perpendicularly to the flow direction of the phases, by separating walls 24 into individual mixing chambers 23, in which separately driven propeller type agitators 25 are disposed. In addition, a rotating shaft 26 passes through the column and mounts worms 28 in the region of the passage channels 27 for transporting the crystals from the cooling zone of the column to the heating zone. By means of the fixed wall elements 29, a quieting down zone is created following the whirling zone.

The operation of the horizontal tank 20 is similar to the vertical tube of FIG. 1 and need not be further described. While the transporting worms 28 are suitable for a separation of substances in the case where the crystals are of greater specific weight than the fluid phase, the horizontally disposed column can, through a suitable design of its transporting means, also be constructed so that it is possible to separate mixtures of substances in which the crystals are lighter than the fluid phase. Examples of such transporting means are described below.

Referring to FIG. 3a, a mixing chamber 30 has horizontal separating walls 21 through which passage channels 32 constructed as tubular nipples pass so as to communicate with the adjacent chambers (not shown). By means of a vaned rotor agitator 34 in the chamber 30 which is driven by a shaft 33, the phases are intensively mixed, and the crystals are continuously brought into contact with new fluid. In order to produce a systematic circulation of the phase mixture, as indicated schematically by dot-dash lines, fixed guide plates 35 are disposed above and below the rotor agitator 34. These guide plates 35 are connected to the separating walls 31, for example, by bridge pieces or to the column walls.

Referring to FIG. 3b, wherein like reference characters as above have been used to indicate like parts, the fixed guide plates 35 are disposed only above the rotor agitator 34 while a fixed horizontal plate 36 is positioned below the agitator 34 so as to form a quieting down space, separated from the mixing space, in which whirling of the phases no longer occurs, and out of which (as described for FIG. 1) the crystals, under the influence of gravity, and counter to the fluid forced upward, pass through a passage channel 32 into the lower adjacent mixing chamber.

Referring to FIG. 3c, wherein like reference characters as above have been used to indicate like parts, the agitator, which is driven by a shaft 33, consists of an opened stirring vane 37 which fills the greater part of the mixing chamber, and which, for example, in order to improve the whirling action may have slanting webs (not shown) in its opening or may have its periphery slanted from the vertical plane. Because the edge zones of the agitating vane are arranged at a small spacing from the channel wall, the crystals are prevented from accumulating on the channel wall, because the crystals are scraped off.

The passage channels 38 are still made as tubular nipples but, however, have a perforated upper side wall so as to allow fluid through while being impermeable to crystals. A shaft 39 which moves up and down is piloted through all the tubular nipples and may also have a superposed rotary motion. The shaft 39 serves to carry pistons 40 which function as transporting means at the locations of the tubular nipples that periodically open and close the channels formed by the nipples. The provision of such a transporting means is particularly advantageous when the crystals are lighter than the fluid phase in substance exchanging contact with them. In use, when the piston 40 is in a position above the tubular nipple 39, the light crystals collect below the piston and form a porous plug. During the downward movement of the piston 40, these crystals become transported into the lower mixing chamber, and the fluid becomes forced through the perforations in the channel walls back into the mixing chamber or out of the next lower mixing chamber into the upper mixing chamber.

Referring to FIG. 4a, wherein like reference characters as above have been used to indicate like parts, each mixing chamber 30 can be provided with a passage channel formed as a sluiceway or lock whose mode of operation corresponds to that of a volumetric pump which, independently of the specific weights of the crystals and of the fluid, forwards a definite volume of the solid phase and of the fluid phase downwardly or upwardly. The passage channel in this case consists of a tubular nipple 41 in which a supplementary transporting means such as a porous piston 42 is movably mounted for reciprocal up and down movement. The 42 is formed as is known so as to be permeable only for the fluid. In addition, the piston 42 can also be mounted for superposition of a rotary movement thereon. The tubular nipple 41 is closed off from the lower mixing chamber at the lower end by a closure piece and is connected to an overflow valve 43 which is provided with an opening 44 at its upper side and with an opening 45 at its opposite lower side spaced from the nipple 41. In addition, a suitable valve disk 46 is

disposed in the overflow valve 43 so as to alternately open and close the openings 44, 45.

In operation, the piston 42 is brought into its lowest position, so as to rest on the closure piece of the tubular nipple 41, and the opening 45 in the separating wall 31 is closed by the valve disk 46. Then, the piston 42 is moved upward, and the mixture is sucked through the opening 44 into the overflow valve 43 and the interior of the nipple 41. In order to transport the crystals into the next lower mixing chamber, the piston 42 is then moved downwardly, and the valve disk 46 is moved upwardly until the opening 44 is closed. This causes the crystals to be pushed into the lower mixing chamber. At the same time, a corresponding quantity of fluid becomes forced out of the lower mixing chamber through the overflow valve 43 and the tubular nipple 41, through the porous piston 42, and into the upper mixing chamber.

Referring to FIG. 4b, the passage channel 47 can alternately be constructed with a tubular wall which is perforated in the lower region and through which a transporting means in the form of a shaft 39 is mounted for reciprocal vertical as well as rotary movement. The shaft 39 further carries a worm 48 in the region of the passage channel which conveys the mixture of fluid and crystals downwardly. During operation, the fluid passes through the perforations of the passage channel wall back into the mixing chamber, while the crystals remain suspended and become forced by the worm 48 downwardly. At the same time, the quantity of fluid forced out of the lower mixing chamber is also transported through the perforated wall into the upper mixing chamber.

Referring to FIG. 4c, the passage channel 49 can also be constructed with a porous or perforated wall in which a transporting means in the form of a piston 50 fastened to a shaft 39 is moved up and down. The shaft 39 which also rotates carries a valve disk 51 which, by means of one or more springs 52, is pressed from below against the underside of the separating wall 31. When the piston 50 is positioned above the channel 49 the channel 49 becomes filled with a mixture of fluid and crystals. During the downward movement of the piston 50, the crystals become forced downwardly while the valve disk 51 (as shown) is forced open. The crystals are thus directed into the next lower mixing chamber while fluid emerges through the porous channel wall and through the piston 50 into the upper mixing chamber.

Referring to FIG. 4d, the tubular passage channel can also be constructed to pass through a separating wall 31 with an upper wall 53a which is perforated and a lower wall 53b which is solid. As in the case of the transporting means of FIG. 4b, a rotating worm 55 is positioned in the channel to provide for the transport of the crystals out of an upper mixing chamber into the next lower mixing chamber, while the counter flowing fluid passes out of the lower mixing chamber through the perforated wall 53a and into the upper mixing chamber.

A brush 54 may also be advantageously disposed on the rotating shaft 39 within the worm 55 so that the inner wall of the channel is continuously freed of any crystals clinging thereto.

In order to prevent crystals from accumulating on the various transporting means described above, it may under certain conditions be advantageous to heat the mechanical parts of such means. For example, an electric current could be sent through the shaft 39 of the transporting means, or in certain cases, the heat insulation could be removed from the column in the region of the transporting means when the temperature in the column is substantially below the temperature surrounding the column.

Referring to FIG. 5, wherein like reference characters as above have been used to indicate like parts, a vertical crystallization column is provided with a plurality of funnel-like separating walls 2a which end in outlets 6a situated centrally on the longitudinal axis of the column 1. This formation of the separating walls 2a facilitates the transport of the crystals from one mixing chamber 3 into the adjacent lower mixing chamber 3 because the crystals slide down the sloping separating walls 2a into the outlets 6a under the influence of gravity. This form of construction further largely prevents the formation of dead spaces in the mixing chambers in the region where the separating walls 2a connect to the column wall. The term "dead spaces" is here to be understood to mean those spaces in which the crystals collect outside the mixing zone proper and do not come into the desired contact with the fluid phase.

If desired, the separating walls may also be heated, for example, through electric heating wires, for the purpose of positively preventing an accumulation of crystals on the separating walls.

In addition, the agitating devices include disk elements 4a which are mounted in the individual mixing chambers and driven by a common shaft 5. The remainder of the column corresponds with that of the column shown in FIG. 1 and therefore no further description is necessary.

Referring to FIG. 6, wherein like reference characters as above have been used to indicate like parts, a crystallization column 1 is constructed with a transporting means in the form of a pair of piston rods 60, 61 which are coaxially displaceable relative to each other and which pass through passageways formed as tubular nipples 6b in the separating walls 2b of the mixing chambers 3b. Each piston rod 60, 61 has a plurality of radially extending porous fluid permeable piston plates 60a, 61a which are connected thereto in order to periodically open and close the tubular nipples 6b in chronological relationship.

The pair of coaxial piston rods 60, 61 are constructed so that the outer piston rod 60 is formed as a hollow shaft with elongated coaxial slots 60b in the side walls while the inner piston rod 61 is formed of hollow or solid cross-section and is piloted within the outer piston rod 60. In addition, the outer piston rod 60 is piloted within a hollow drive shaft 8a of a scraper 8 at the top end of the column 1. The lower piston plates 60a of each set of piston plates 60a, 61a are each fixed, as by welding, to the outer peripheral surface of the outer piston rod 60 while the upper piston plates 61a are connected, as by welding, to the inner piston rod 61 by suitable extensions which pass through the slots in the outer piston rod 60. In this way, the piston plates need have only one opening for the passage of the two piston rods. This is also the case when the two piston

rods consist of two solid elements which are able to slide on one contact surface on one another. The piston plates 60a, 61a are of porous construction, for example, of woven wires, of sintered material or of filters for organic materials.

It is advantageous for the piston plates, in addition to their reciprocating movement, to also have a rotary movement during operation. This prevents crystals from being able to adhere to the piston plates because such would be thrown off outwardly by the rotary motion. It is moreover possible, if desired, for the rotating piston plates 60a, 61a to act as agitating devices for moving the crystallized and fluid phases round in the mixing chambers, so that supplementary mixing devices, such as propellers for example, may be dispensed with.

The tubular nipples 6b of the crystallization column which itself consists of a cylindrical vertical tube 1 closed at its top and bottom are of any suitable cross-section such as circular or polygonal cross-section. The cross-section of such a tubular nipple 6b may, on occasion, amount to the greater part of the cross-section of the column.

Depending on the cross-sectional shape of the tubular nipples 6b, the piston plates 60a, 61a are correspondingly shaped to slide through the nipples while substantially sealing one side of the plate to the other to the passage of crystals.

In order to agitate and circulate the different phases together within the mixing chambers 3, one or more propeller-like agitating devices 4b are disposed in each chamber 3 and are driven by shafts 5b which pass in fluid-tight relation through the walls of the column 1. As above, an efficient mixing together of the phases is obtained with a correspondingly good interchange of the elements of the phases.

During operation, in order to transport the crystallized phase downwardly through the column, assuming such is of greater specific weight than the fluid phase, the piston plates 60a, 61a are initially positioned tightly upon one another at the level of an associated separating wall 2b so as to close off the nipple 6b passing through the wall 2b to the passage of crystals. Next, the upper plate 61a is moved a distance *d* upward, so as to close the tubular nipple 6b of the adjacent upper mixing chamber 3. Then, while retaining the distance *d*, the two plates 61a, 60a are moved downward a distance *2d* equal to twice the distance of the previous upward movement so that the major part of the crystal content of the mixing chamber 3 becomes transported into the adjacent lower mixing chamber. At the end of this movement, the plates are below the end of the nipple 6b so that the crystals can pass into the lower mixing chamber. At the same time, a corresponding volume of fluid is forced out of this lower mixing chamber into the adjacent upper mixing chamber; this fluid flowing through the openings or pores in the piston plates which are permeable only to the passage of the fluid. The lower plate 60a is then moved upward the distance *d*, that is, until the plate 60a closely adjoins the upper plate 61a. Finally, the two plates 61a, 60a, in close contact, are moved back into the initial position after which, either after a desired interval of time or else immediately, the stages of movement for the transporting means are repeated.

In the above example of construction, the distance *d* corresponds to the length of a tubular nipple *6b* which in turn is half the height of a mixing chamber *3*. Although this form of construction is advantageous, the invention is also intended to include such designs with which these dimensions deviate from the illustrated form of construction.

While the piston rods *60, 61* are described above as making only translatory movements, it is however possible, as previously described, for the piston rods to make a supplementary rotary movement particularly for the purpose of throwing off crystals that may cling to the piston plates *60a, 60b*.

This above column construction is independent of whether the column is used as a vertical, horizontal, or sloping column. Furthermore, the speed of transportation of the crystallized phase in the column is independent of the physical characteristics of the crystalline sludge composed of the crystallized and fluid phases, and is also, for example, independent of the density or of the viscosity of the sludge.

In the event that there are spaces between the passage channels and the column wall which are large in comparison with the mixing space, then under certain conditions, it is advantageous to close up these spaces, or to fill them with a filler material, for the purpose of preventing a large accumulation of crystals in such spaces.

What is claimed is:

1. A crystallization column for counterflow of a crystal phase and a fluid phase of a mixture therein comprising:

- a cooler at one end of the column for crystallizing the mixture thereat;
- a heater at an opposite end of the column for melting crystals of the mixture thereat;

a plurality of separating walls spaced along and disposed transversely across the column, and defining a plurality of mixing chambers longitudinally of the column for mixing of the mixture phases therein;

one passageway in each separating wall communicating adjacent mixing chambers with each other for a counterflow of the mixture phases between said adjacent mixing chambers;

at least one agitating device disposed in each mixing chamber for agitating and circulating the crystal phase and liquid phase of the mixture together in each mixing chamber; and

a supplementary transporting means in said column for transporting the phases between said mixing chambers, said transporting means including a pair of piston rods relatively displaceable with respect to each other and passing through said passageways, and a first plurality of transverse radially extending porous piston plates connected to one of said piston rods, each of said porous pistons being in radially disposed relation to pass through one of said respective passageways in said separating walls, and a second plurality of transverse radially extending porous piston plates connected to the other of said piston rods wherein said piston rods are disposed in coaxial arrangement and wherein the outer piston rod is hollow and includes a plurality of elongated slots therein for passage of said plates connected to the inner piston rod, and whereby said piston rods are reciprocally movable in chronological sequence to open and close the passageways into a respective mixing chamber while transporting the phases between pairs of adjacent mixing chambers.

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