April 12, 1966 C. B. SAWYER 3,245,760

APPARATUS FOR GROWING CRYSTALS

Filed Oct. 31, 1961 4 Sheets-Sheet 1

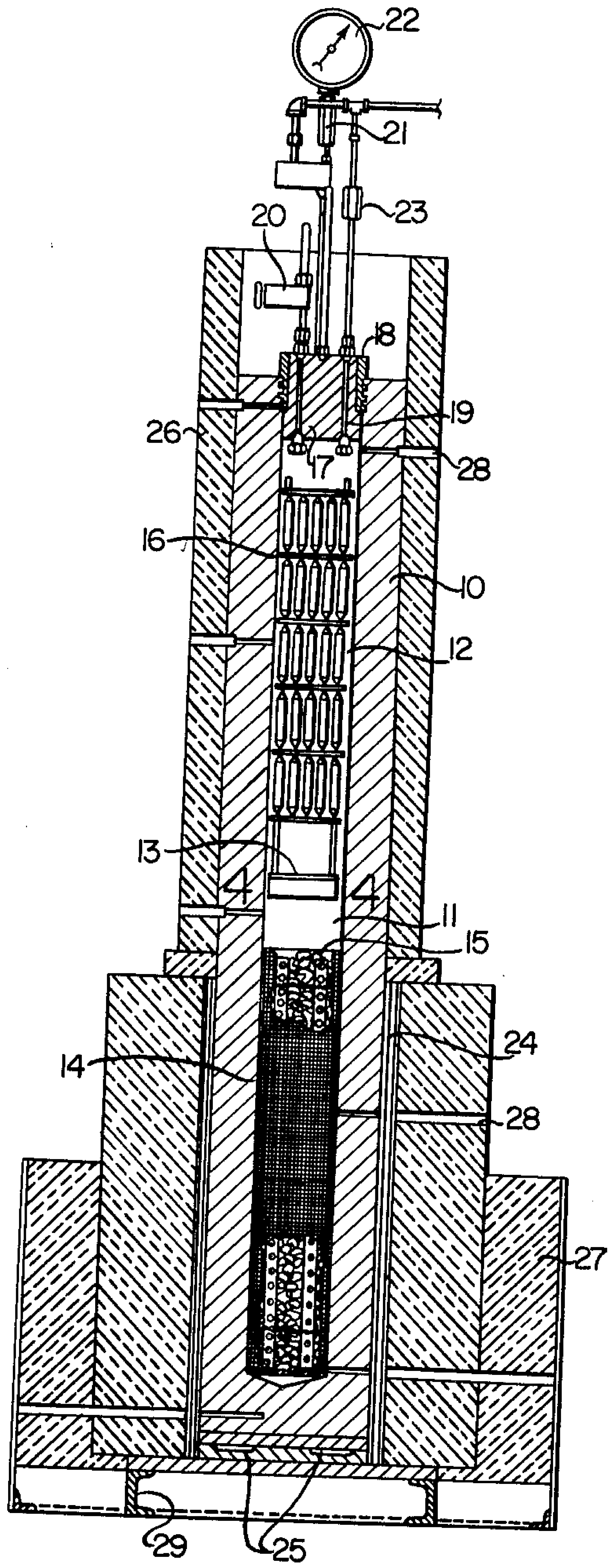

FIG I

INVENTOR.
CHARLES B. SAWYER
BY
Oberlin, Maky & Donnelly
ATTORNEYS

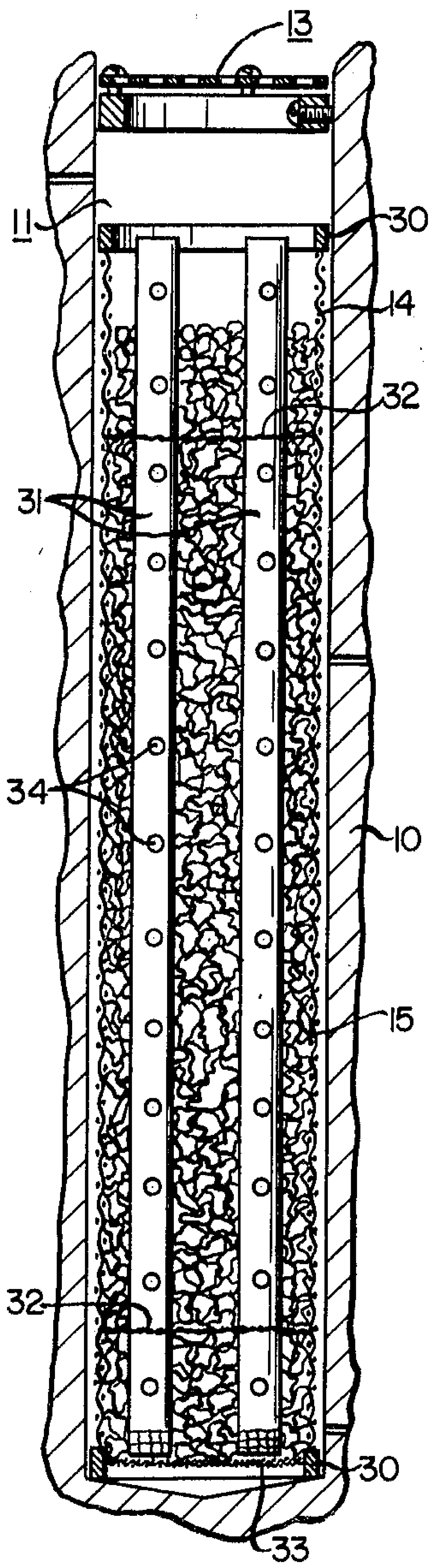

FIG 2

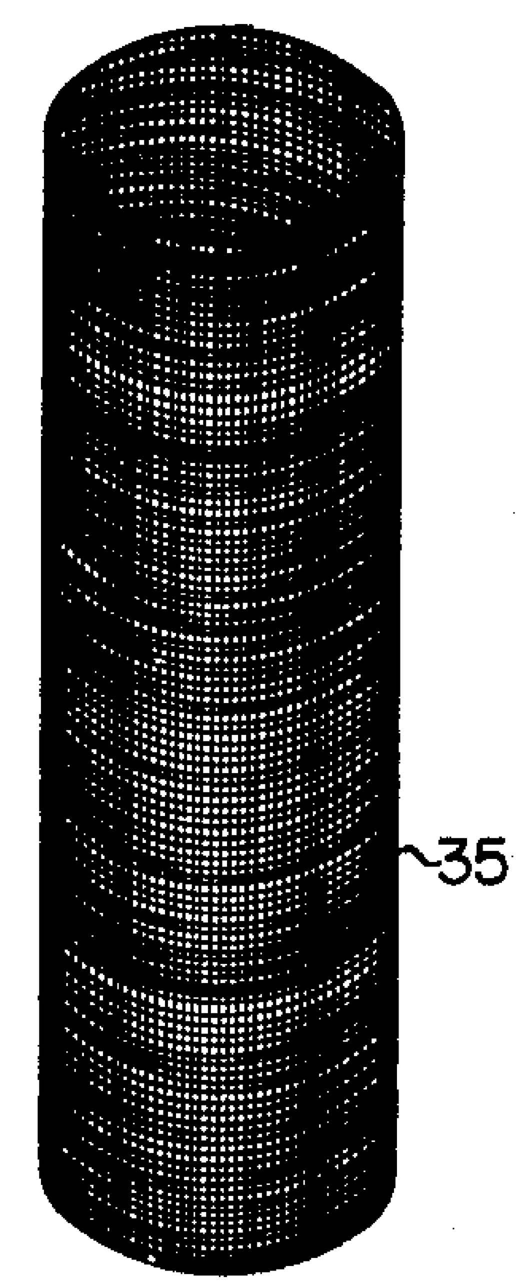

FIG 3

INVENTOR.
CHARLES B. SAWYER
BY
Oberlin, Maky & Donnelly
ATTORNEYS

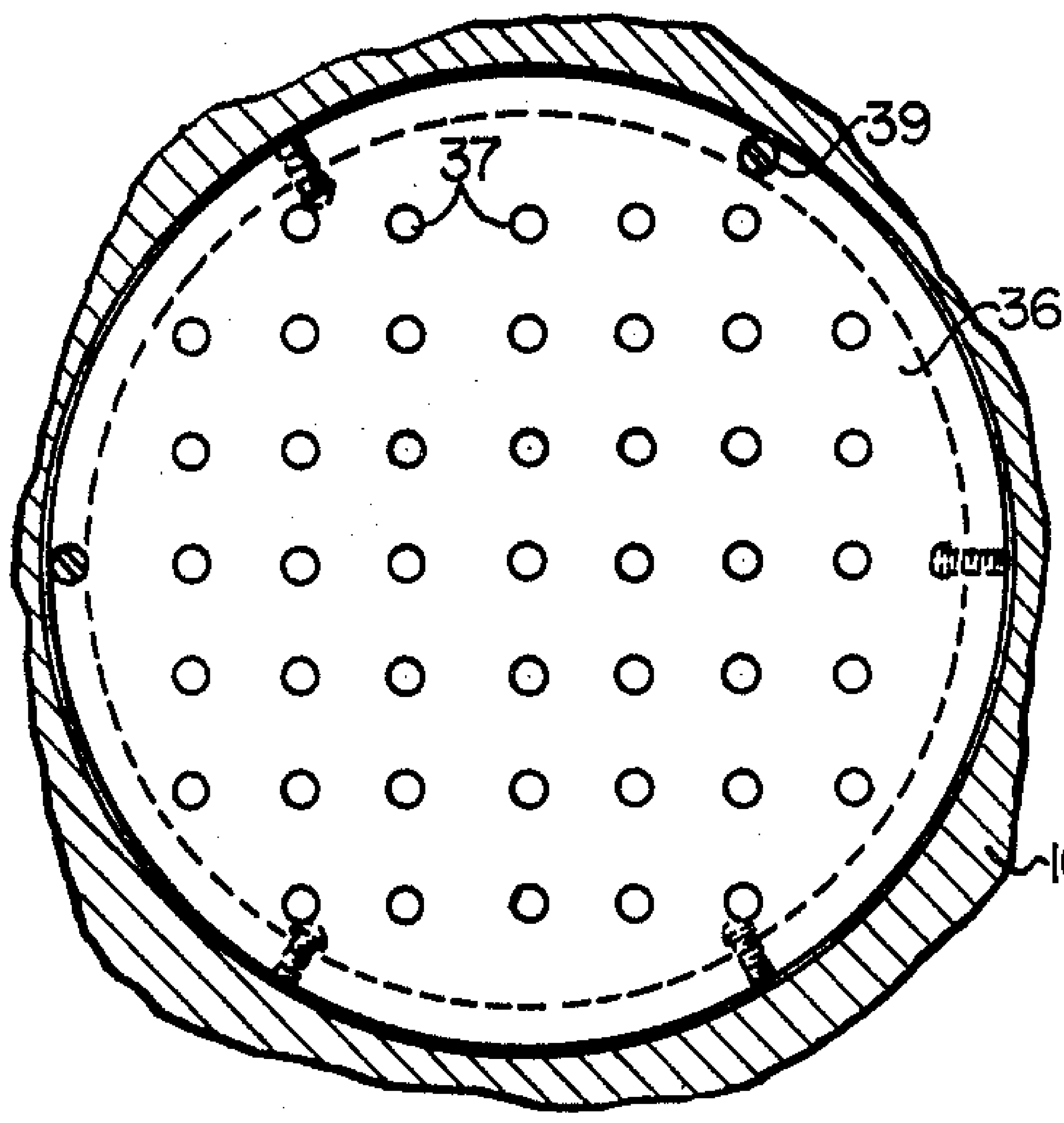

FIG 4

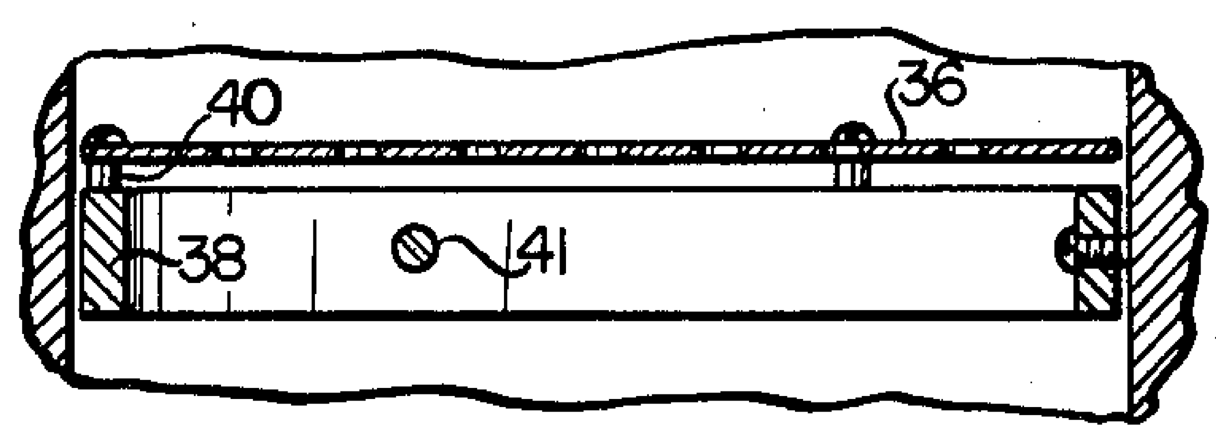

FIG 5

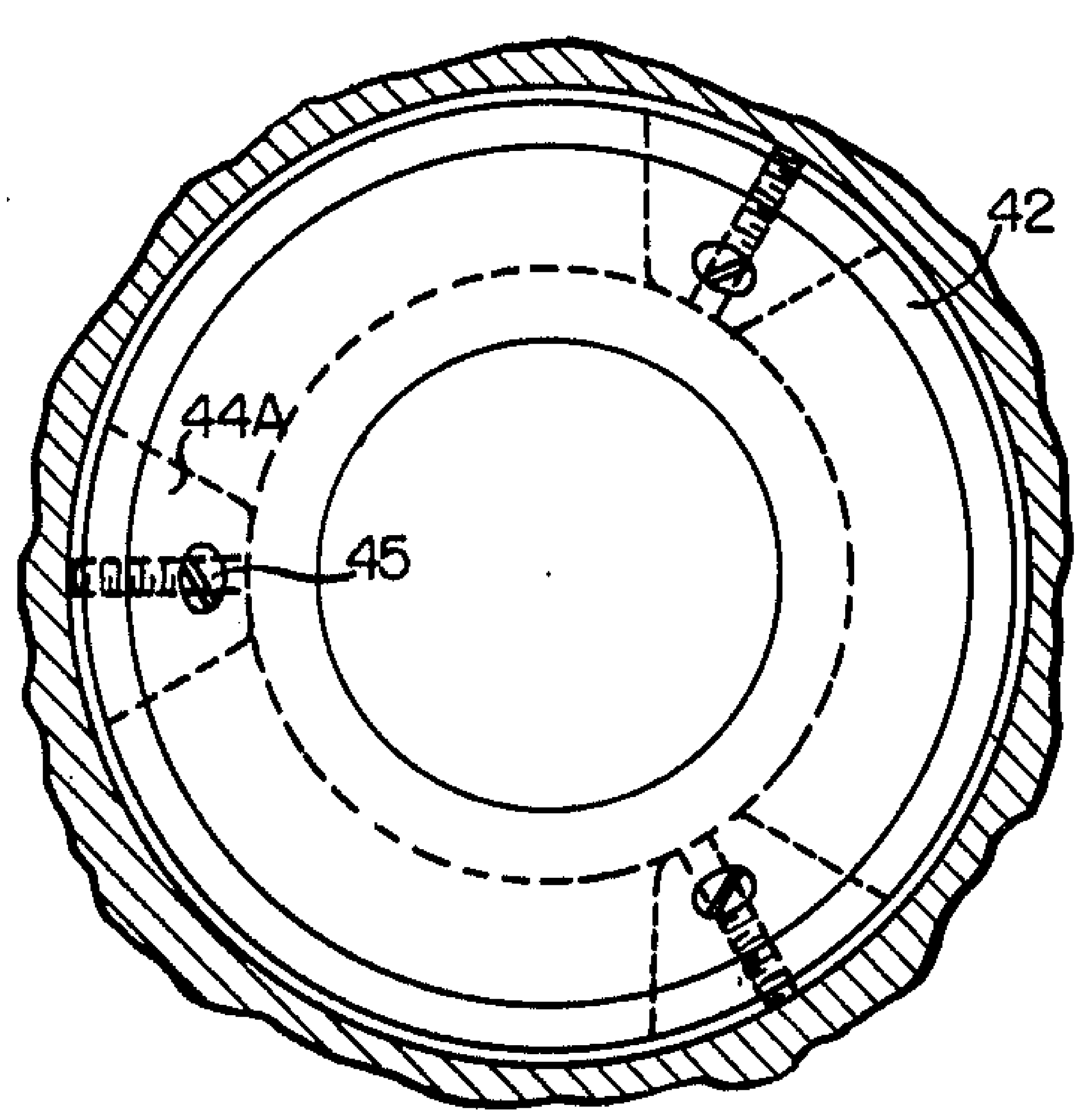

FIG 6

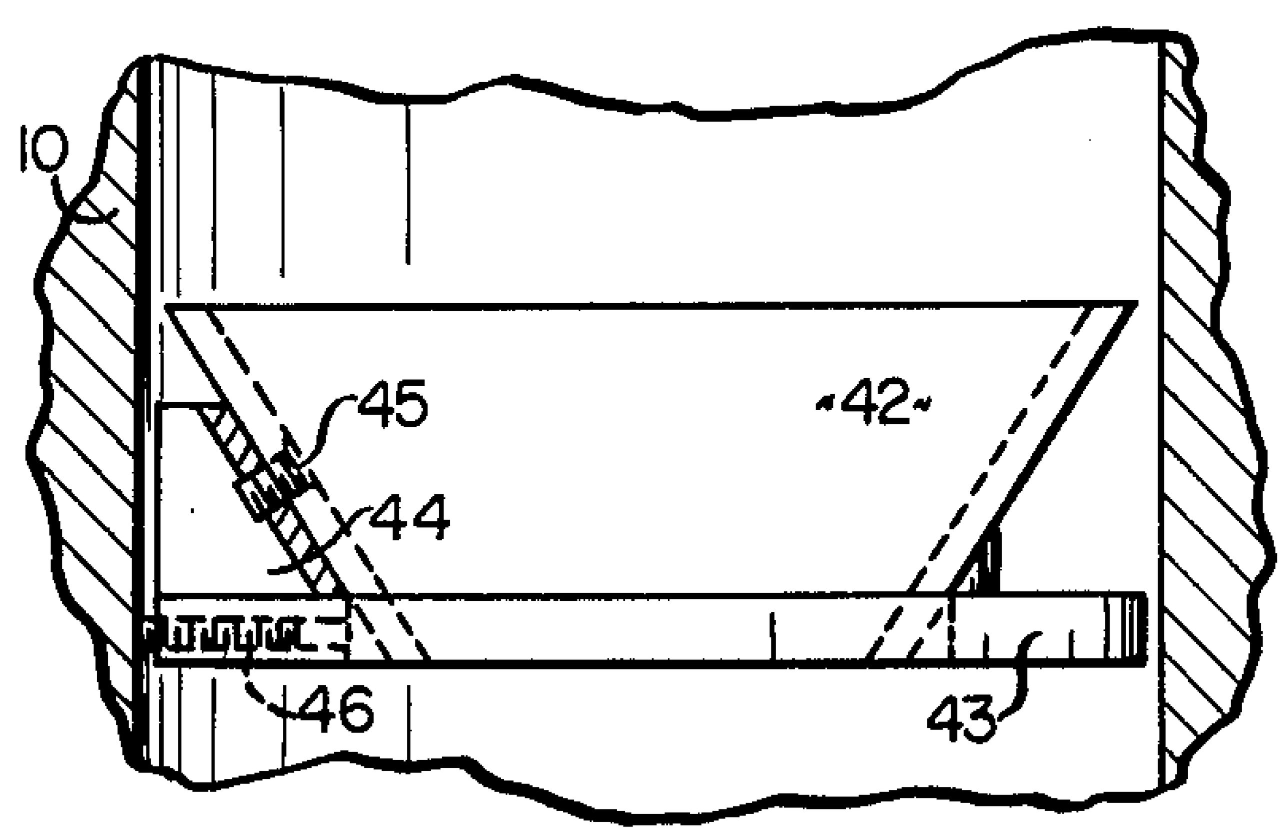

FIG 7

INVENTOR.
CHARLES B. SAWYER
BY
Oberlin, Maky & Donnelly
ATTORNEYS

April 12, 1966 C. B. SAWYER 3,245,760

APPARATUS FOR GROWING CRYSTALS

Filed Oct. 31, 1961 4 Sheets-Sheet 4

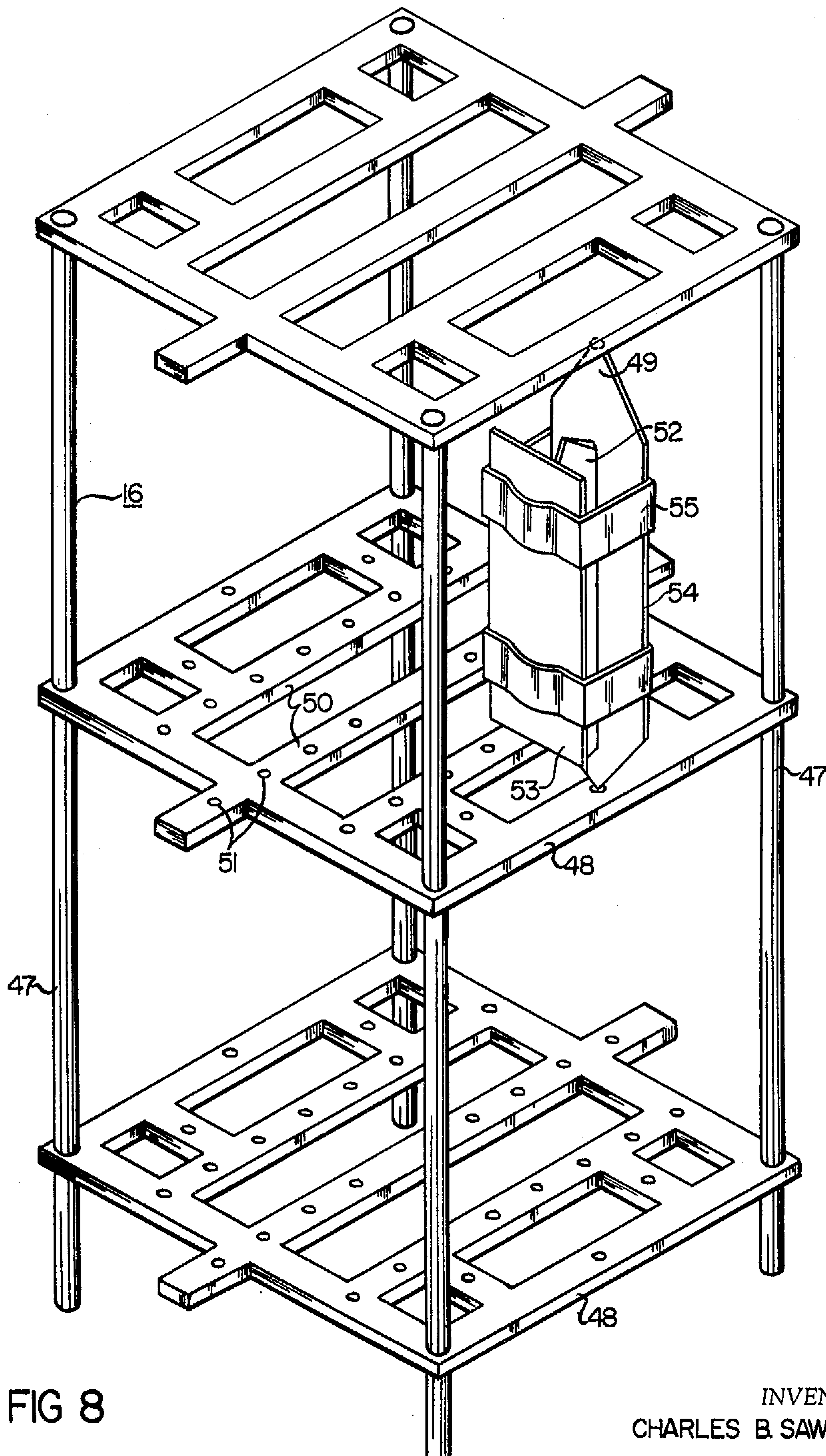

FIG 8

INVENTOR.
CHARLES B. SAWYER
BY
Oberlin, Maky & Donnelly
ATTORNEYS

United States Patent Office

3,245,760
Patented Apr. 12, 1966

3,245,760

APPARATUS FOR GROWING CRYSTALS

Charles B. Sawyer, Cleveland Heights, Ohio, assignor to Sawyer Research Products, Inc., Eastlake, Ohio, a corporation of Ohio

Filed Oct. 31, 1961, Ser. No. 148,948
10 Claims. (Cl. 23—273)

The present invention relates to apparatus for growing crystals and, more particularly, to an improved apparatus for growing cultured crystals, such as cultured quartz crystals, which are substantially immune to the effects of bridging or welding together of portions of the raw material used to beget the cultured crystals.

The present apparatus is adapted for artificially producing various crystals, usually oxides, such as beryllium oxide and aluminum oxide. However, the invention is particularly adapted for the production of pegmatitic crystals, notably quartz crystals, and therefore for convenience of disclosure will be described in connection with such crystals. Quartz crystals are becoming increasingly important commerically not only for optical purposes but because they possess the piezoelectric property of generating an electric potential when subjected to mechanical stress and, conversely, exerting a mechanical stress when subjected to an electric potential.

In the usual manner of artificially growing quartz crystals, a nutrient solution bathes a seed crystal either by mechanically induced means and/or by thermally induced means. The nutrient solution may comprise an aqueous alkaline solution of a siliceous material, such as pure natural quartz, and nurtures the growth of the seed by gradual deposition of the siliceous material on the seed crystal. In the usual operation, the bathing of a seed crystal by the nutrient solution takes place at rather high pressures such as 8,000 p.s.i. to 25,000 and at elevated temperatures such as 350° C. to 400° C. It is accordingly necessary to confine the seeds and mother solution in an airtight autoclave or bomb during crystal growth.

Ordinarily, the nutrient solution floods over the seed crystal in repetitious waves, as by the rocking arrangement in United States Patent No. 2,675,303 to Sobek et al.; or the nutrient solution is allowed to bathe a seed or seeds wholly by thermal currents in an unimpeded manner as in United States Patent No. 2,785,058 to Buehler. By still another technique, a nutrient solution has been projected from a source onto a seed crystal as through a funnel-shaped transfer medium.

The rate of crystal growth under the best known operating conditions is extremely slow. Morever, the growing process is most sensitive and susceptible to many factors. For instance, crystals are often found to have "blue haze" which is a faint blue milkiness visible under intense illumination and also known as the Tyndall effect. "Blue haze" is caused by scattered microscopic inclusions of roughly two microns in diameter, judging by their Tyndall scattering pattern and is most often present in the minus x growth on Y-bar seeds. These inclusions are not otherwise identified but are suspected to be inclusions of liquid. A further defect often found in such crystals is crevicing. Crevices are defects resembling small cracks in appearance and are thought to be caused by local failures of growth producing small, narrow voids. Attempts to accelerate crystal growth as by raising the operating temperature or by increasing the molarity of the siliceous material in the nutrient solution seem to have limitations in terms of defects such as these.

In general, to grow cultured crystals two chambers or regions are used. One chamber is a supply region for the dissolution of the feed material, and the other chamber is a seed-growing region in which the seed crystals

are bathed by the nutrient solution formed in the supply region. In the more general practice, the two chambers are arranged in tandem fashion, such as in the form of a vertical autoclave. The nutrient solution is heated in the mineral-dissolving or supply chamber to impart a thermal potential causing the solution to flow by thermal convection into the seed-growing region. In this region, the nutrient solution cools and, becoming supersaturated, smoothly deposits its correspondingly supersaturated solute on the seed crystals. The solution, now cooler and denser, returns to the mineral-dissolving region where the process is repeated.

If cultured crystals of acceptable quality are to be grown, it is necessary to control the rate and volume of flow of the nutrient solution as it is interexchanged from chamber to chamber. In practice a divider or separator plate is stationed within the apparatus to define on opposite sides thereof the mineral-dissolving and seed-growing regions. The plate may be suitably shaped with one or more openings so as to baffle the interexchanging flow and preferably to afford an exchange of nutrient solution in such a diffused manner that the circulation of solution in each chamber becomes substantially independent. The size and/or number of openings in such a baffle means are accordingly quite important. If the area of the opening or the sum of a plurality of openings is too large, the situation approaches that in which there is no plate; and the two regions have fairly free interchange such that they lose their identity as herein defined. Also too rapid an interchange of the nutrient solution may result in too low a supersaturation with too slow a growth of the desired crystals. Further, under these conditions of free interchange, the differential temperature between a mineral-dissolving region and a seed-growing region may decrease uncontrollably. On the other hand, if there are too few openings or the openings are too small, the two regions become choked off from each other, and the amounts of both heat and solute carried by the hydrothermal solution into the seed-growing region seriously decrease. Also, the temperature drop between regions may become excessive if there is not a compensating change in heat loss from the seed-growing region. These effects can produce both blue haze and crevicing.

Depending on conditions of a "run" and results sought, a baffle or separating member will be chosen having an opening or total openings of desired total area relative to the size or cross-section of the autoclave in which the baffle is placed. During the course of the run, however, a clogging of the mineral-dissolving chamber can result which operates to replace the baffle means as a control on the rate and volume of exchange of the nutrient solution between the described regions by effectively reducing the area of the baffle plate openings. This clogging is technically referred to as "bridging," a tendency shown by the lumps of supply material, for example, raw quartz crystals, at the top of the supply load to grow and to interlock in such a manner that the lumps do not fall as the supply is dissolved by the nutrient solution. Furthermore, it appears that when these interlocked lumps stay in position, the structure becomes cemented or encrusted to form the "bridge." When a bridge covers most of the autoclave's transverse or cross-sectional area, the bridge acts as a baffle insofar as continued operation is concerned. If the bridge baffle has a smaller percentage opening than the actual baffle, the former becomes the effective baffle for that run and may, consequently, force the run toward operation either at a larger temperature differential between the supply and growing regions than planned, or a smaller power flow, or both. Thus, it is seen that the formation of a bridge can have very serious consequences affecting the outcome of the run, for example, greatly increasing the incidence of crevicing in the grown crystals. The present invention provides means to prevent this occurrence.

It is, therefore, a principal object of the present invention to provide an improved apparatus for producing crystals.

Another object is to provide such apparatus for growing cultured pegmatitic crystals and especially quartz crystals of high quality.

A further object is to provide such apparatus which continues a control afforded by a baffle or like means throughout the course of a run.

A still further object is to provide such apparatus which is substantially immune to bridging in a mineral-dissolving region.

Other objects will become apparent as the description proceeds.

To the accomplishment of the foregoing and related ends, the invention consists of the features hereinafter fully described and particularly pointed out in the claims, the annexed drawing and following disclosure describing in detail the invention, such drawing and disclosure illustrating, however, but one or more of the various ways in which the invention may be practiced.

In the accompanying drawings:

FIGURE 1 is a vertical section of an autoclave embodying the present invention and shows a full complement of grown crystals;

FIGURE 2 is an enlarged fragmentary view of the mineral-dissolving region of FIGURE 1 and illustrates pipes or columns within such region;

FIGURE 3 is a perspective view of a modified form of column or pipe;

FIGURE 4 is a section of FIGURE 1 on the line **4—4** and shows a baffle or separating means that may be used;

FIGURE 5 is a diametric section of FIGURE 4;

FIGURES 6 and 7 are fragmentary views corresponding, respectively, to FIGURES 4 and 5 and illustrate a modified form of baffle or divider; and

FIGURE 8 is a perspective view of the seed rack of FIGURE 1 and for convenience of illustration shows one seed and one seed holder mounted in position.

In accordance with the present invention, the results of bridging and like phenomena can be either effectively reduced or eliminated. Especially in the latter instance, the baffle or divider means remains the sole control on the rate and volume of exchange of the nutrient solution between the described chambers. This is accomplished by assuring a clear open channel or zone in the mineral-dissolving chamber. Effective results are obtained when the open channel or zone has a minimum transverse area substantially equal to at least one-half of the area afforded by the baffle means for the interexchanging flow. In order to ensure that the baffle or like means remains at all times the sole control on the rate and volume of exchange of nutrient solution, the minimum transverse area of the open zone or channel desirably should be approximately equal to the area, as of an opening or openings, afforded by the baffle means for solution flow. The ratio of minimum zone area to the area of baffle openings or flow area may be as high as 2:1, although this is not an upper or critical limit.

Generally, the two regions are vertically superposed in tandem fashion, so as to aid in the exchange of the stronger and weaker solutions between the described regions by hydrothermal currents. By a "strong solution" is meant that nutrient liquid which effects the transfer of the siliceous material, in the case of quartz crystals and the described vertical arrangement, from the lower region to the upper region by dissolving the material in the mineral-dissolving region and then depositing such material on the seed crystals in the seed-growing region. In contrast, the "weak solution" moves from the upper region to the lower region. Preferably also, the temperature in each region is substantially constant throughout the bath of liquid circulation therein. However, there is a rather sharp temperature drop between such regions, the lower temperature being in the seed-growing region. This condition not only provides the thermal potential which forces the infiltration of the solution of the mineral-dissolving region into the seed-growing region and vice versa, but it causes the solution to become supersaturated within the latter region so that deposition of the solute is readily accomplished.

It will be understood that the paths of flow of the hydrothermal currents may vary in force and direction from time to time and also experience momentary fluctuations. However, the general direction of movement for the "strong solution" is primarily upwardly in the mineral-dissolving region along the walls of the autoclave (which are receiving heat energy), across the baffle means while some of the solution simultaneously infiltrates into the seed-growing region, and then downwardly of the mineral-dissolving region substantially centrally thereof. The direction of movement of the "weak solution" is primarily downwardly in the seed-growing region along the walls of the autoclave (which are radiating heat energy), across the baffle means while some of the solution simultaneously infiltrates into the mineral-dissolving region, and then upwardly of the seed-growing region substantially centrally thereof. Of course, the solution which infiltrates a given region from a companion region tends to join the general current flow of that region.

Referring to the drawings and particularly to FIGURE 1, an autoclave in which the present apparatus and method may be used includes a vertical tubular chamber **10** divided into a mineral-dissolving region or supply chamber generally indicated at **11** and a seed-growing region generally represented at **12** by a separator or baffle means **13** located generally midway of the height of the chamber **10**. This chamber may be fabricated from steel of high creep strength, such as steel containing 2.25 percent chromium and 1 percent molybdenum. Within the mineral-dissolving region **11** lies a wire mesh feed basket **14** containing crystalline quartz **15**, such as Lascas grade natural quartz, which is to be dissolved to form a nutrient solution and carried thereby to the seed-growing region **12** where a rack, generally indicated at **16**, supports a plurality of seed crystals.

A plug **17** tightly seals the upper end of the chamber **10** and has a wear-resistant collar **18** fixed to the plug **17** and threadably meshing with a threaded upper terminus of the chamber. Narrow passages **19** extend through the plug **17** to expose suitable equipment to conditions extant within the interior of the chamber **10**. Such equipment may include, for example, a bleeder valve **20**, a surge check valve **21**, a pressure gauge **22**, a rupture disk **23**, and other desired testing and control equipment.

Side and bottom strip heaters **24** and **25**, respectively, are secured to the lower portion or mineral-dissolving region **11** of the chamber. These strip heaters are electrically energized as through potentiometer-type controllers operating in conjunction with a thermocouple and a saturable core type transformer.

Suitable high temperature insulation **26** encompasses the length of the chamber **10** and may comprise, for instance, magnesium oxide block insulation. Additional insulation **27** such as expanded mica (Vermiculite) embraces the lower end in order economically to effect in combination with the strip heaters **24** and **25** a higher temperature at that end of the chamber **10** and thereby induce thermal currents in the nutrient solution which travel upwardly and longitudinally of the chamber toward the seed-growing region **12**. A series of thermocouple wells **28** spaced vertically along the autoclave penetrate to desired areas of the chamber **10** and receive thermocouples to indicate the temperature at such areas in a known manner. Standard beams **29** support the entire autoclave structure.

Referring now more in detail to the mineral-dissolving chamber and especially FIGURES 2 and 3, the wire mesh basket **14** is of sufficient rigidity to be self-supporting and terminates in rings **30** to which the wall and ends of the wire basket are suitably secured. One or more hollow columns **31**, each having at least two openings communicating with its interior, are supported longitudinally of the chamber **11**. Although the pressure in an autoclave during a run may be of the order of 11,000 p.s.i. or more, the pressure is everywhere extant so that quite simple means can be used to position the columns or pipes **31** as needed. Indeed, it is necessary only to ensure that the pipes are in a substantially upright position. To this end, twisted wire **32** fixed to the sides of the basket **14** loops around or otherwise embraces the pipes **31** for a slip fit. Ordinarily, such a wire support is used adjacent the top and the bottom of the pipes as illustrated and may be of the same gauge as that used for the basket **14**.

It is important that the minimum internal transverse or cross-sectional area of a pipe **31** (or cumulative cross-sectional areas where more than one pipe is used) be equal to at least one-half of the area of the openings of the baffle means hereinafter described. Although pipes or columns **31** of uniform cross-sectional dimension are ordinarily used, should a pipe have a varying cross-sectional dimension, the minimum dimension is the effective dimension insofar as the present purpose of a pipe is concerned. Generally, the ratio of the minimum internal area of a pipe or cumulative minimum internal area of pipes to the area of a baffle opening or cumulative area of the baffle openings is approximately one, but such ratio can be within the range of about 0.5:1 to 2:1, respectively. Thus, in an autoclave having an internal diameter of eight inches, one to two pipes have been used each having an internal diameter of one to 1.5 inches; in an autoclave having an internal diameter of 10 inches, two pipes of such size have been used; and in an autoclave having an internal diameter greater than 10 inches, two to four pipes of such size have been used. In these instances, baffle plate means have been used having percentage openings within the described ratio range hereinafter specified.

Variations in position, length and nature of openings in a pipe **31**, itself, are of course possible. It is necessary that the top of a pipe **31** be out of reach of the raw crystal lumps **15**, that is, higher than the lumps in the case of a vertical autoclave, since bridging tends to occur at the top of the bed of raw crystals. Thus, the top of a pipe **31** may be stationed at a point between the top of the bed of quartz **15** and the baffle means **13**. The length a pipe **31** extends from the top of a crystal bed away from the baffle means and into a mineral-dissolving region (and thus through the top portion of the crystal supply) need be only long enough to pass any area of possible bridge formation. This distance may be less than half the height of the vertical dissolving chamber. As a practical matter, however, the pipes **31** usually extend substantially the length of the mineral-dissolving region **11** since the brunt of the weight of the pipe can then be carried at the bottom of the chamber. Similarly, although a pipe **31** must have at least two openings to permit circulation therethrough, the pipe can have any number of additional openings or perforations. The only precaution is to prevent entry of the raw crystal lumps **15** into the pipe or column. In this manner, the pipes of FIGURE 2 may have wire mesh caps **33** fitted about their lower ends, as well as their upper ends if desired, and also perforations **34** spaced along their sides. Indeed, an entire pipe **31** may be replaced with a wire mesh screen **35** of FIGURE 3 which has been rolled on itself to form a retaining cylinder of fluid-permeable wall. The mesh of the screen **35** is as indicated of sufficient size to keep out the raw crystals employed.

The baffle or separator means may take various forms and shapes, as long as it effects an interexchanging flow of nutrient solution from chamber-to-chamber as described. Here and in the claims, the terms "openings" or "apertures" of the baffle or like means are taken to mean all avenues of flow afforded by the baffle means, including openings or apertures in the baffle itself and any peripheral or annular opening it may define with respect to the wall of an autoclave. The percentage of openings may vary widely depending on operating conditions, for instance the composition of the mother liquor, since various solvents such as sodium carbonate or sodium hydroxide may be used. Usually the combined openings or apertures constitute from about 2.5 percent to about 30 percent of the area through which the nutrient solution is exchanged and preferably from about 5 percent to about 15 percent. As the percentage of openings in the baffle increases, the tendency toward bridging likewise increases and becomes more serious. The openings or apertures in a baffle or separator may be of any configuration including all polygonal or arcuate configurations. Likewise, the openings in a given baffle plate or like means need not all be of the same configuration nor necessarily form a uniform pattern over a plate although normally the latter is followed. As the particle size or dimensions of lumps of quartz in the feed diminishes, the tendency to bridge increases, and may even take place with pipes present. The bad effects however are mitigated by the preservation of the channels through the supply by means of the pipes.

FIGURES 4 and 5 illustrate a preferred baffle or separator means. The baffle of this embodiment includes a plate **36** having a plurality of openings or apertures **37** and is fixed to a support ring **38** by screws **39** which pass through intervening spacers **40**. Set screws **41** engage threaded openings in the ring **38** and bear against the sides of the wall of the chamber **10** to hold the plate assembly in position. In one particular installation in a pilot plant autoclave having an internal diameter of 8 inches, a circular exchange baffle plate also 8 inches in diameter of 16 gauge low carbon steel was used. This plate had 45 holes uniformly distributed over its surface. These holes were of the same diameter and comprised in total amount about 7.5 percent of the surface area of the plate.

FIGURES 6 and 7 illustrate a further type of baffle or divider that may be used. This baffle includes a truncated cone **42** disposed in a ring **43**. Three support members **44**, fixed to the ring **43** at 120° from each other, have flange portions **44***a* which overlie the exterior of the cone section **42** and are secured thereto by set screws **45**. Additional set screws **46** radially pass through the ring **43** and bear against the inside wall of the chamber **10** to hold the assembly in position.

The type of seed used and the manner of mounting it in the seed-growing chamber are not at all critical to the present invention. The seed rack shown in FIGURE 8 is intended only to illustrate one techinque that may be used. This rack includes four corner steel posts **47**, the lower ends of which may rest on the baffle means previously described, to which vertically spaced plates **48** are suitably secured. The plates **48**, which may form as many tiers as permitted by the size of the chamber **10** and the length of the seed holders, support triangular ends indicated at **49** of the seed holder, or the ends of a seed crystal itself may be directly supported in a similar manner. In order to provide for the circulation of a nutrient solution around and between the seed holders, each rack or plate **48** has spaced bar members **50**, and each bar member has chamfers or recesses **51** which are vertically aligned with other chamfers on the facing side of an adjacent plate **48**. The chamfers **51** thus readily receive the pointed ends **49** of a seed holder to position seed crystals **52** carried in the holders in a vertical position with respect to the chamber **10**. The bar members **50** of each rack are arranged to support the holders so as to dispose them in a substantially circular cross-sectional pattern similar to the cross-sectional pattern of the chamber **10**. A seed holder may comprise a pair of substantially parallel plates **53** and **54** adapted frictionally to engage the opposing minor surfaces of the seed crystal **52** of any desired cut. The plates **53** and **54** may be made of iron or low carbon alloys of iron, although other materials such as silver, titanium, and even graphite have been used. Tension means secure the plates **53** and **54** in relation to the seed crystal **52** and may take the form of one or more extensible metal bands **55** which snugly embrace the exterior of the plates **53** and **54** to clamp them in a desired frictional engagement with the edges of the seed crystal **52** as described. By the arrangement illustrated in FIGURE 8, the growth of the seed crystals is in a transverse direction of the vertical chamber **10**.

It will be apparent that the use of the present method and apparatus is not critical to any particular set of operation conditions. However, the following data are submitted as exemplary of one autoclave and operation conditions therefor which may be used in carrying out the invention.

Inside diameter	8 inches.
Volume	79 liters.
Design pressure	10,000 p.s.i.
Design temperature	450° C.
Steel	Croloy 2¼.
Closure	Modified Bridgeman.
Manufacturing method	Bored.
Inside height	8 feet.
Outside height	106⅝ inches.
Outside diameter	14 inches.
Number of pipes in supply chamber	2, thin wall steel.
Size of pipes	I.D. 1.5 inches.
Percent of open area in baffle means	7.5 percent.

"Croloy" is a tradename for steel-containing chromium. By "Modified Bridgeman" is meant that conventional high pressure seal known in the art.

The operating conditions of a typical run for an autoclave of the type just described are:

Solution	0.5 to 0.3 molar sodium carbonate in water.
Degree of initial filling	80%.
Operating temperature of seed-growing region	350° C.
Operating temperature of mineral-dissolving region	365° C.
Pressure	10,000 p.s.i.
Number of seeds	180 to 200 (depending on lengths).
Length of run (including clean out and restart)	40 to 45 days.
Heat input	4 kilowatts.
Quartz transport	Average 2 pounds per day.

A normal procedure is to load the autoclave through the opening of the plug **20** and after sealing to commence heating the autoclave. When the temperature of the autoclave reaches about 150° C. as indicated by thermocouples, the air and some steam within the autoclave is bled through the bleeder valve **23**, which is thereafter closed. Heating is then continued through the heater strips **27** and **28** until a control point is reached which is normally about 365° C. for the mineral-dissolving region **11**. Subsequently, the temperature is maintained at the control point as by standard automatic electrical equipment. At the operating elevated temperature and pressure, the aqueous sodium carbonate solution dissolves some of the quartz crystal material in the basket **14** of the mineral-dissolving region **11** thus approaching saturation. Due to the temperature differential between the seed-growing region and the mineral-dissolving region, controlled by adjusting heat loss, there are upward thermal currents of solution into the seed-growing region **12**. Here the solution is cooled and becomes supersaturated with respect to the dissolved quartz crystals, again because of the difference in temperature, and deposits the quartz on the seed crystals.

The cooled solution passes again through the baffle means into the mineral-dissolving chamber. The two pipes or tubes are supported substantially vertically and entirely within the supply chamber at about mid-radius and extend from the initial level of the supply quartz nearly to the bottom (about 38 inches). These pipes were open ended thin wall steel pipes to the lower ends of which wire mesh caps were welded. The pipes formed an assured open channel for the cooled nutrient solution to flow to the bottom of the autoclave where the solution was diverted and heated once again as it traveled upwardly through the supply quartz. Thus, even if a bridge is formed, a sufficient opening is assured penetrating through the bridge baffle, so that the actual metal baffle remains the controlling one.

In this manner, although the present method and apparatus do not actually prevent bridge formation, the invention does render a crystal growing operation substantially immune to the effects of bridging so that desired circulation of solution and growth of crystals of fine quality may still be realized.

When the seeds are grown to a desired size as indicated, for example by gammagraph measurements, the electrical power is turned off. The autoclave is permitted to cool and then may be opened for removal of the grown crystals.

Other forms embodying the features of the invention may be employed, change being made as regards the features herein disclosed, provided those stated by any of the following claims or the equivalent of such features be employed.

I therefore particularly point out and distinctly claim as my invention:

1. In apparatus for growing a crystal wherein a nutrient solution is exchanged between a supply chamber having a charge of raw crystal-growing material and a seed-growing chamber through a divider separating said chambers and having an opening to pass the nutrient solution, the improvements comprising: a hollow column having at least two communicating openings stationed within the supply chamber said column being spaced from the divider and extending in a direction away therefrom, said column having an internal minimum transverse area substantially equal to at least one-half of the area of such divider opening, and said communicating openings being of a size to prevent entry of the crystal-growing material into the column, whereby said column prevents bridging in the supply chamber from superceding the divider and opening as a control on such exchanged nutrient solution.

2. In apparatus for growing a quartz crystal wherein a nutrient solution is continuously exchanged between a heated supply chamber having a charge of raw quartz material and a cooler seed-growing chamber through openings in baffle means stationed at an interconnecting area common to both chambers, the accumulative area of the openings in the baffle means amounting to a predetermined desired value affecting the quality of the quartz crystal to be grown, the improvements comprising: at least one hollow column having at least two openings communicating with the interior thereof stationed in the supply chamber spaced from said baffle and extending from a point between said baffle means and charge of raw quartz material in a direction away from the baffle means and into said raw quartz material, said at least one columnar having a minimum cumulative cross-sectional columnar area substantially equal to at least one-half of the cumulative area of such baffle openings, said communicating openings being of a size to prevent entry of the raw quartz material in said at least one hollow column, whereby regardless of bridging in the supply chamber the bridging cannot supercede the baffle means as a control on the rate and volume of flow of the nutrient solution through the openings of the baffle means.

3. Apparatus as claimed in claim 2 wherein said supply chamber contains a basket for holding the raw quartz material, and said at least one hollow column is fixed with respect to the basket to support said at least one column.

4. In apparatus for growing a quartz crystal wherein a seed-growing chamber is disposed above and separated from a mineral-dissolving chamber containing a raw quartz material by apertured baffle means and wherein the mineral-dissolving chamber is heated to induce circulation of a nutrient solution from the mineral-dissolving chamber through the apertures of the baffle means into the seed-growing chamber and back through the baffle means into the mineral-dissolving chamber, the accumulative area of the apertures of the baffle means amounting to a predetermined desired value affecting the quality of the quartz crystal to be grown, the improvements comprising: a plurality of substantially tubular members each having at least two communicating openings supported substantially vertically in the supply chamber with the upper ends thereof beneath and spaced from the baffle means but higher than the raw quartz material, said members having a cumulative minimum cross-sectional area that is substantially at least equal to the cumulative area of the apertures of the baffle means, said communicating openings for each member being of a size to prevent entry of the raw quartz material into the tubular members, whereby bridging in the mineral-dissolving chamber cannot replace the baffle means as a control on the rate and volume of the circulation of the nutrient solution between said chambers.

5. In a vertical autoclave for growing a quartz crystal having a seed-growing chamber disposed over a mineral-dissolving chamber containing a bed of raw quartz material and separated therefrom by apertured baffle means through which a nutrient solution passes from chamber-to-chamber, the accumulative area of the apertures of the baffle means amounting to a predetermined desired value affecting the quality of the quartz crystal to be grown, and wherein the mineral-dissolving chamber is heated to induce continuous vertical circulation of the solution from such chamber-to-chamber to effect growth of the crystal, the improvement of preventing bridging in the mineral-dissolving chamber from effectively replacing the baffle means as a control on the rate and volume of flow of the nutrient solution during operation of the autoclave comprising: a plurality of generally pipe-shaped members having a cumulative minimal cross-sectional area bearing a ratio to the cumulative area of the apertures of the baffle means of about 0.5:1 to about 2:1, respectively, said members being spaced from said baffle means and substantially vertically disposed in the mineral-dissolving chamber and extending downwardly from a point between the top of the quartz bed and the baffle means and substantially through the vertical extent of said raw quartz bed, and permeable means fixed over the lower ends of said

pipe-shaped members to prevent entry thereinto of the raw quartz material.

6. The apparatus of claim 4 wherein the accumulative area of the apertures of the baffle means constitutes approximately from about 2.5 percent to about 30 percent of the area through which the nutrient solution circulates.

7. In apparatus for growing a crystal wherein a nutrient solution is exchanged between a supply chamber having a charge of raw crystal-growing material and a seed-growing chamber through a divider separating said chambers and having an opening to pass the nutrient solution, the improvements comprising: tube means stationed within the supply chamber to maintain a cross-sectional area thereof free of the raw material charge for an appreciable length of the supply chamber, said tube means being spaced from said divider and extending in a direction away from the divider and into said chamber, said cross-sectional area being substantially equal to at least one-half of the area of said divider opening, whereby bridging in said supply chamber cannot replace said divider and opening as a control on such exchange of nutrient solution.

8. The apparatus of claim 7 wherein said tube means also has openings spaced longitudinally thereof and of a size to pass nutrient solution and solute without passing said raw crystal-growing material.

9. The apparatus of claim 7 wherein said divider has a plurality of openings and said openings are spaced from the periphery of the divider to prevent a peripheral exchange of nutrient solution from occurring between said chambers.

10. Apparatus for growing pegmatitic crystals wherein a nutrient solution is exchanged between a supply chamber having a charge of raw pegmatitic material and a seed growing chamber through openings in a separator positioned in an interconnecting region common to both of said chambers, said separator means positioned transversely of said chambers in said interconnecting region, and at least one hollow column within the supply chamber spaced from said separator and having at least two openings communicating with the interior of said supply chamber, one end of said column terminating between said separator and such charge of raw material.

References Cited by the Examiner

UNITED STATES PATENTS

187,468	2/1877	Johnson	23—272.5
2,204,180	6/1940	Gerlach	23—301
2,895,812	7/1959	Kohman	23—301
3,013,867	12/1961	Sawyer	23—301
3,051,558	8/1962	Jost	23—301

NORMAN YUDKOFF, *Primary Examiner.*

ROBERT F. BURNETT, MAURICE A. BRINDISI, *Examiners.*