Aug. 16, 1955 R. LEE 2,715,607

KNOCKDOWN DISTILLATION APPARATUS

Filed Sept. 22, 1949 2 Sheets-Sheet 1

FIG. 1

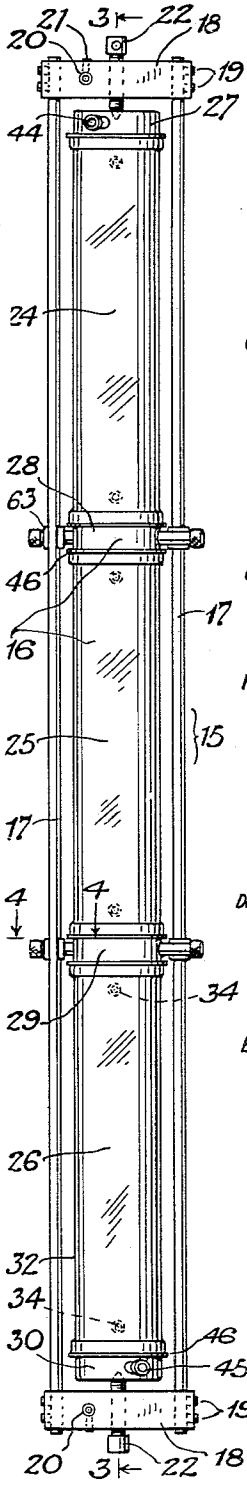


FIG. 3

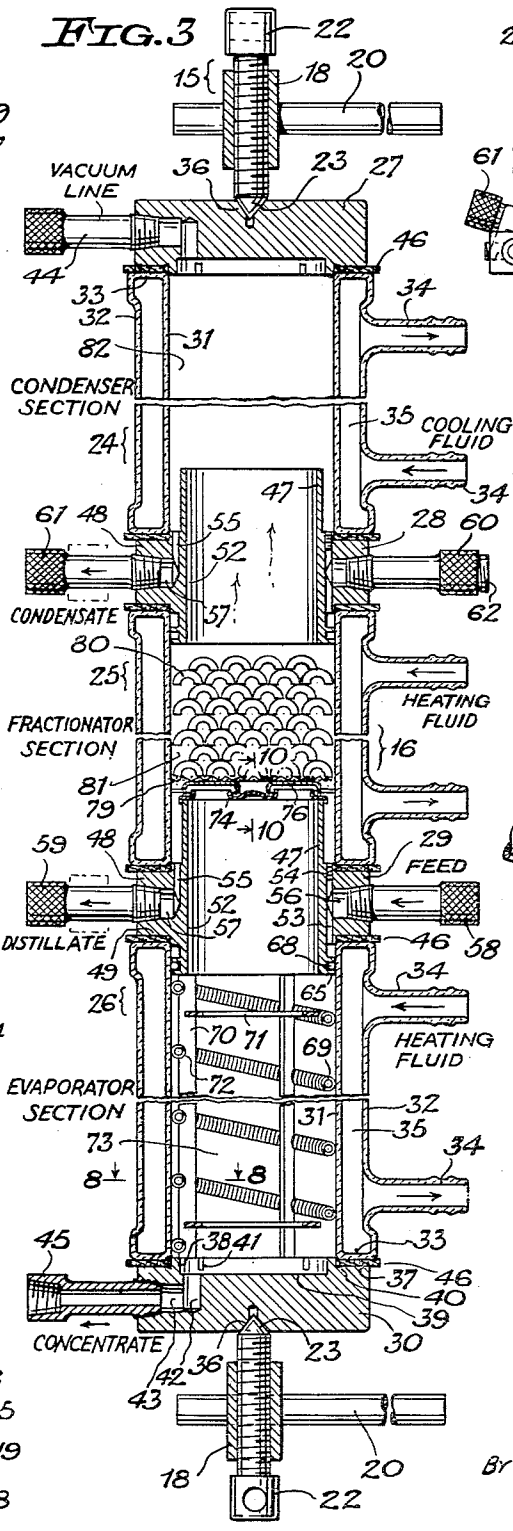


FIG. 2

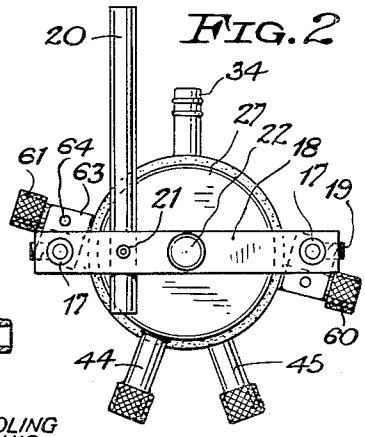


FIG. 4

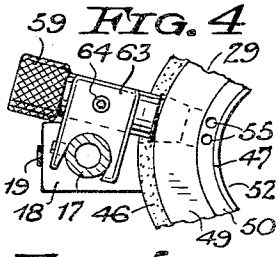


FIG. 5

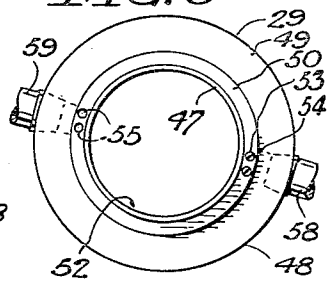
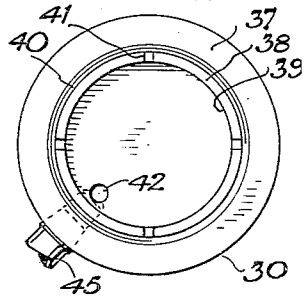


FIG. 6



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FIG. 11

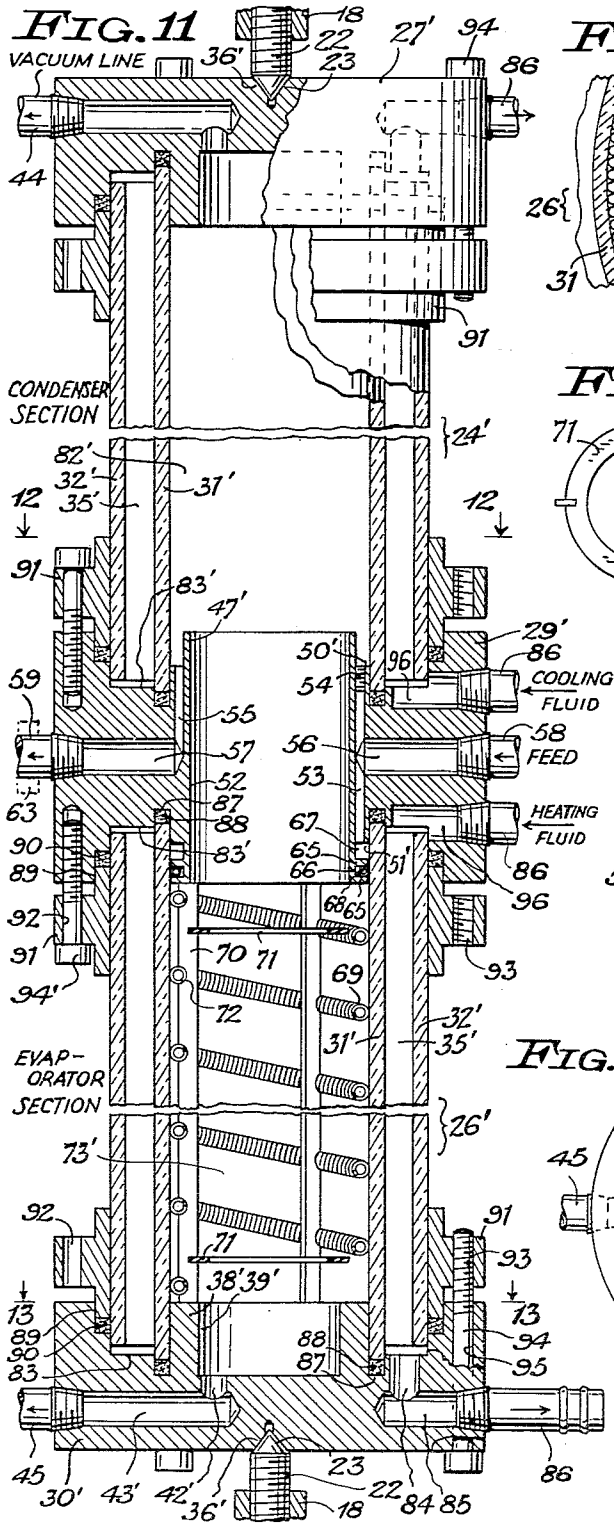


FIG. 8

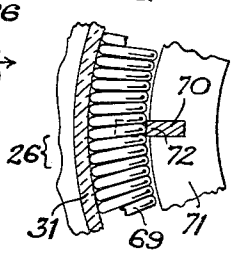


FIG. 7

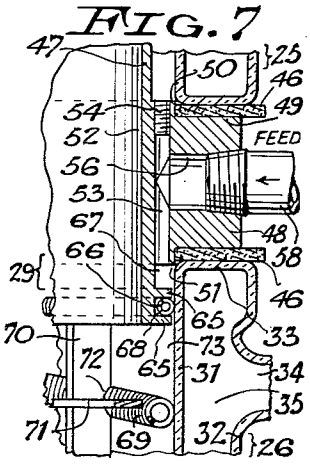


FIG. 9

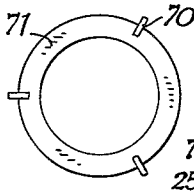
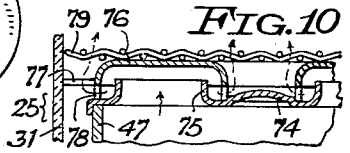


FIG. 10



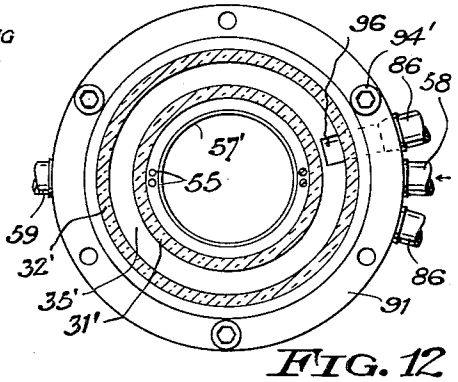
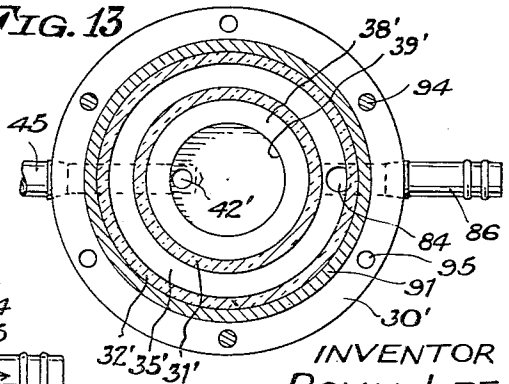


FIG. 12

FIG. 13



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United States Patent Office 2,715,607
Patented Aug. 16, 1955

2,715,607

KNOCKDOWN DISTILLATION APPARATUS

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Application September 22, 1949, Serial No. 117,129

5 Claims. (Cl. 202—153)

The present invention relates to distillation apparatus, and more particularly to stills for laboratory or pilot plant use.

An object of the invention is to provide a still adapted for separating or stripping solvents from solutions or extracts of heat-labile organic or biological material and which is so arranged as to minimize heating time of the heat-labile material.

Another object is to provide a still having simple and inexpensive glassware which can be easily cleaned with ordinary laboratory equipment, and which if broken can be quickly replaced at low cost.

A further object is to provide a still which can be arranged in various ways, which can be quickly assembled and dismantled, which can be placed in full capacity operation in a few minutes, and which includes improved film-type evaporating means.

The invention further consists in the several features hereinafter described and claimed.

In the accompanying drawings, illustrating certain embodiments of the invention,

Fig. 1 is an elevation of a stripper still constructed in accordance with the invention, and including a fractionator section;

Fig. 2 is a top view of the still;

Fig. 3 is a sectional elevation of the still, taken generally on the line 3—3 of Fig. 1;

Fig. 4 is a detail sectional view taken generally on the line 4—4 of Fig. 1;

Fig. 5 is a top view of an intermediate fitting or flow ring of the still;

Fig. 6 is a top view of a bottom end fitting or header of the still;

Fig. 7 is a detail sectional view of the lower intermediate fitting and associated parts;

Fig. 8 is a detail sectional view taken generally on the line 8—8 of Fig. 3;

Fig. 9 is a top view of a distributor frame;

Fig. 10 is a detail sectional view taken generally on the line 10—10 of Fig. 3;

Fig. 11 is a sectional elevation of a modified form of still omitting a fractionator section;

Fig. 12 is a transverse sectional view taken generally on the line 12—12 of Fig. 11, and

Fig. 13 is a transverse sectional view taken generally on the line 13—13 of Fig. 11.

Referring to the form of apparatus shown in Figs. 1 to 10, 15 designates a vertically extending loop-shaped rectangular frame adapted to receive therein a columnar still assembly 16 hereinafter described. The frame 15 comprises a pair of parallel vertical frame bars or rods 17, preferably of tubular shape, connected at their upper and lower ends by apertured cross bars 18 in which the vertical bars are secured, as by screws 19. Each cross bar carries a laterally projecting horizontal rod 20, preferably of tubular shape, fastened in place as by a screw 21 and adapted to be secured to a suitable support, such as a laboratory lattice frame, not shown.

The cross bars 18 are further provided with respective central vertical clamping screws 22 each having a conically formed inner end 23. The screws 22 are axially aligned and are adapted to detachably clamp and support the still assembly 16 between them as hereinafter described.

The still assembly 16 comprises a series or stack of detachably connected elements and is here shown to include three tubular jacket-forming column members 24, 25, and 26, and four joint members or flow fittings 27, 28, 29, and 30, the column members alternating with the fittings and being disposed in axial alignment. The endmost fittings 27 and 30 form top and bottom end covers or headers, and the intermediate fittings 28 and 29 form spacing rings. The column member 24 is interposed between the top fitting 27 and the upper intermediate fitting 28, the column member 25 is interposed between the upper and lower intermediate fittings 28 and 29, and the column member 26 is interposed between the lower intermediate fitting 29 and the bottom fitting 30. In the present instance, the still is arranged to effect evaporation in the lowermost column member 26, fractionation in the intermediate column member 25, and condensation in the uppermost column member 24.

The three column members 24, 25, and 26 are identical and each is preferably made of heat-resisting glass and has cylindrical inner and outer walls 31 and 32 and flat annular end walls 33. The outer wall 32 has integrally formed thereon a pair of upper and lower side tubes or nipples 34 which communicate with a jacket space 35 formed between the inner and outer walls of the column member, one of the nipples forming a fluid inlet and the other an outlet. Either end of the column member may be placed uppermost. The cylindrical inner wall 31 of each column member is smooth and straight and can be easily cleaned by ordinary laboratory equipment.

The four flow fittings are formed of a metal such as aluminum or an aluminum alloy, which is suitable for all ordinary organic solvents and materials. However, the fittings can be made of stainless steel or other resistant metal for use with acids or other corrosive substances. The end fittings 27 and 30 are identical and each is of disk-like shape and has a central conical recess 36 at its outer face adapted to receive the conically formed end 23 of the adjacent clamping screw 22. The opposite face of each end fitting has a flat annular seat 37 which extends to the periphery of the fitting and is bounded at its inner edge by an annular rib 38 forming therein a circular recess 39. Adjacent to the rib the seat 37 of the bottom end fitting has a shallow annular drain groove 40 which slopes inwardly and which communicates with annular drain slots 41 cut in the rib 38. The recess 39 communicates by way of a vertical passage 42 with a radial bore 43 extending to the peripheral wall of the fitting. A connector nozzle 44 is screwed into the bore 42 of the top fitting, and a similar nozzle 45 is screwed into the bore of the bottom fitting. The seat 37 of each end fitting receives a flat annular gasket 46 against which abuts the flat end of the adjacent column member. In the still illustrated, the nozzle 45 of the bottom fitting forms a discharge for concentrate, and the nozzle 44 of the top fitting is a vacuum connection for removing air or non-condensable gas. The evacuating means, not shown, may be of any suitable type, such as a vacuum pump or water aspirator.

The two intermediate fittings or joint rings 28 and 29 are identical and each comprises a tubular shell 47 having formed thereabout an annular flange 48. The flange has opposite flat annular seats 49 each of which extends to the periphery of the flange and is bounded at its inner edge by upper and lower annular shoulders 50

and 51. The seats 49 receive gaskets 46 against which abut the flat ends of the adjacent column members. The tubular shell 47 projects upwardly and downwardly from the flange 48 and forms therethrough a vertical bore or passage 52 which is nearly as large as the inner diameter of the column members and is coaxial with the column members. At one side of each intermediate fitting two adjacent vertical bores 53 extend between the upper and lower shoulders 50 and 51, and have their upper ends plugged, as by screws 54. At the other side of the intermediate fitting two adjacent vertical bores 55 extend from the upper shoulder 50 to about midway of the flange 48. The vertical bores 53 and 55 communicate with respective radial bores 56 and 57 formed in the flange 48. Connector nozzles 58 and 59 are screwed into the respective bores 56 and 57 of the lower intermediate fitting 29, and similar nozzles 60 and 61 are screwed into the respective bores 56 and 57 of the upper intermediate fitting or joint ring 28. The nozzle 58 of the lower intermediate fitting forms a feed inlet for the solution or extract to be distilled and the corresponding nozzle member 60 of the upper intermediate fitting is closed by a plug 62. The nozzle member 59 of the lower intermediate fitting forms a discharge for high-boiling distillate and the corresponding nozzle member 61 of the upper intermediate fitting forms a discharge for low-boiling distillate. Preferably, the nozzle members of both intermediate fittings are detachably secured to the side rods 17 of the still frame, as by suitable fork-ended clamps 63 with fastening screws 64, Figs. 2 and 4, thus providing lateral support for the columnar still assembly.

The tubular shell of the lower intermediate fitting 29 has formed at its lower end a pair of vertically spaced ribs 65 defining between them an annular groove 66, the outer diameter of the ribs being slightly smaller than the inner diameter of the surrounding column member. For uniformity in construction the upper intermediate fitting 28 is similarly constructed. Between the lower shoulder 51 of the lower intermediate fitting 29 and the upper rib 65 is an annular groove 67 which communicates with the inlet bores 53. An endless coiled distributor spring 68, such as of stainless steel, fits in the annular groove 66 of the fitting 29 and bears against the inner wall 31 of the glass column member 26, conforming to any slight irregularities of the glass. Another coiled distributing spring 69, also of stainless steel, is disposed in helical relation along the inner wall 31 of this column member and is carried by a stainless steel skeleton frame consisting of vertical strips 70 joined by two or more ring members 71, the outer edges of the strips having semi-circular notches 72 to receive the spring. The frame 70, 71, rests on the bottom end fitting 30 and its upper end is near the lower end of the intermediate fitting 29. The distributing spring 69 conforms to any slight irregularities in the inner wall 31 of the glass column member 26. The column member 26 forms therein an evaporating chamber 73, the inner wall 31 of the column member being heated by a suitable heating fluid, such as steam, flowing through the jacket space 35 by way of the nipples 34. The springs 68 and 69 serve to distribute a film of dissolved material on the heated glass wall 31 and thus prevent channeling and insure rapid evaporation of the volatile constituent.

The upper end of the tubular shell 47 of the lower intermediate fitting 29 carries thereon a baffle plate 74 with upwardly flanged ports 75 over which is mounted a baffle ring 76 of inverted channel cross-section to form therewith vapor passages, the baffle ring having positioning and supporting lugs 77 and 78. A screen 79, such as of stainless steel, rests on the baffle ring and supports thereon a mass of packing 80, such as berl saddles and the like, confined in the column member 25, which forms a fractionating chamber 81. The jacket space of the column member 25 is maintained at a suitable temperature by passing therethrough a heating fluid. By way of example, if the dissolved organic material to be recovered is contained in a water-alcohol mixture, the temperature of the packed section is maintained at a point slightly higher than the boiling point of alcohol at the absolute pressure employed. While the fractionating section is shown to be of the packed type, it could equally well be of the bubble plate type.

The upper intermediate fitting 28 opens freely into the uppermost column member 24 which forms therein a condenser chamber 82 closed at its upper end by the top end fitting 27. A suitable cooling fluid, such as cold water or brine, is passed through the jacket space 35 of the column member 24. Any well known means may be provided for recovering any solvent vapor which may pass through the vacuum line 44. The connector nozzle 59 serves to discharge high-boiling distillate from the fractionating chamber, and the connector nozzle 61 serves to discharge condensate from the condenser section. It will be understood that all sections of the still will be under a vacuum when the still is in use. However, for some purposes the still may operate at atmospheric pressure.

In the operation of the still, the lowermost column member 26 is heated to a suitable temperature, as by steam or hot water. The intermediate column member 25 is heated to a lower temperature, and the uppermost column member is cooled. The solution to be distilled, such as an alcoholic extract or solution of an organic or biological material from which the solvent is to be stripped, is admitted to the lower intermediate fitting 29 through the connector nozzle 58 at a suitable rate, as by a pump or by gravity. The solution flows downwardly through the bores 53 of this fitting and into the annular groove 67 from which the solution spreads over the entire length of the endless distributing spring 68 contacting with the inner wall of the lowermost column member 26. The solution then flows through the interstices of the coiled spring 68 and onto the inner wall 31 of the column member 26 in a continuous film and descends to the successive convolutions of the helical distributing spring 69, so that the film formation is maintained and channeling is prevented. The heated inner wall 31 of the column member quickly evaporates the solvent from the film of solution, and the solvent vapor passes upwardly into the fractionating section of the still, while the concentrate is collected by the bottom end fitting 30 and flows out the connector nozzle 45. The tubular shell 47 of the lower intermediate fitting 29 is sufficiently long to prevent any spray from entering the fractionating section. In the fractionating section the higher boiling component of the solvent finds its way out through the connector nozzle 59 of the lower intermediate fitting, and the lower boiling vapor component passes upwardly into the condenser section where it is condensed and passes out through the nozzle 61 of the upper intermediate fitting 28. Air and non-condensible gases are removed through the vacuum nozzle 44.

The still is of such construction that it can be placed in full capacity operation within a few minutes, and the material to be heated is subject to heat for only a short period, thus avoiding damage to heat-labile organic or biological materials, such as extracts of vegetable and animal tissue.

In cases where the solution or extract to be distilled or stripped includes only a single solvent, or a mixture of solvents which need not be separated from each other, the packing material 80 and its supporting means may be omitted, and the two upper column members may form a condenser, or the intermediate column member may be omitted. By way of example, a solution of wheat oil in trichlorethylene can be stripped without utilizing the fractionating section of the still. The still may also be used as a vapor compression type by withdrawing the solvent vapor and compressing it, and then condensing the vapor in the jacket space of the evapor-

ating chamber. In this type of still it is desirable to enlarge the vapor outlet from the still.

It will be seen that the still can be readily dismantled by unscrewing the top screw 22 and lifting off the various parts. The glass column members are of simple and inexpensive construction and can be easily cleaned, and if broken can be replaced at low cost. In some instances, one or more of the column members may be made of metal with or without a glass lining. If the uppermost glass column member 24 should require replacement, the upper clamping screw 22 is backed off, allowing removal of the upper end fitting 27 and the column member and permitting installation of a new column member. This replacement can be effected without disturbing the rest of the still assembly. The lowermost column member can be replaced in a generally similar manner. The intermediate column member can be replaced after removing the top column member.

In the modified form of still shown in Figs. 11 to 13, the glassware is further simplified in that each column section includes a pair of cylindrical concentric inner and outer tubes 31′ and 32′ made of strong heat-resisting glass and forming an intervening jacket 35′. The still is here shown to comprise an upper condenser section 24′ and a lower evaporating section 26′, and includes metal end fittings 27′ and 30′ and an intermediate fitting 29′, which are somewhat similar to the corresponding fittings of the still of Fig. 3 and are adapted to form seals with the ends of the jacket-forming tubes. The columnar still assembly is mounted in a supporting frame as in the device of Fig. 1, the disk-like metal end fittings or headers 27′ and 30′ having central conical recesses 36′ at their outer faces receiving the clamping screws 22 of the frame. The opposite face of each end fitting has a stepped annular recess 83 receiving the adjacent ends of the glass tubes 31′ and 32′. The recess 83 is bounded at its inner side by an annular rib 38′ forming therein a central recess 39′. The central recess communicates by way of a vertical passage 42′ with a radial bore 43′ extending to the periphery of the fitting. As in the still of Fig. 3, a connector nozzle 44 is screwed into the bore of the top end fitting, and a similar nozzle 45 is screwed into the bore of the bottom end fitting. The bottom of the annular recess 83 communicates by way of a vertical passage 84 with a radial bore 85 extending to the peripheral wall of the fitting. Nipples 86 are screwed into the bores 85 of the top and bottom end fittings. The annular recess 83 of each end fitting includes an inner groove 87 confining therein a packing ring 88, such as of cork or other suitable material, against which the adjacent end of the inner glass tube 31′ abuts. The annular recess further includes a counterbore 89 forming a seat for a similar packing ring 90 surrounding the end portion of the outer glass tube 32′. The packing ring 90 for the outer glass tube is held under compression against the tube by a gland ring 91 surrounding the tube and having a circular series of alternately plain and screw-threaded openings 92 and 93, the gland ring being urged against the packing ring by three screws 94 passing through openings 95 in the end fitting and engaged in the screw-threaded openings 93 of the gland ring.

The intermediate fitting 29′ is of annular shape and has stepped upper and lower annular recesses 83′ generally similar to the annular recesses 83 of the end fittings and receiving ends of the glass tubes 31′ and 32′. The recesses 83′ are bounded at their inner sides by a tubular shell 47′ with surrounding upper and lower annular shoulders 50′ and 51′. The upper end of the shell projects above the shoulder 50′, and the lower end is peripherally grooved like the shell 47. The shell 47′ has a cylindrical bore 52 therethrough nearly as large as the bore of the inner glass tube 31′. Each annular recess 83′ includes an inner groove 87 with a packing ring 88, and a counterbore 89 with a packing ring 90 and gland ring 91, as in the end fittings, the gland ring being

secured by screws 94′ tapped into the intermediate fitting and passing through the plain bores 92 of the gland ring.

Like the intermediate fitting 29 of Figs. 3 and 7, intermediate fitting 29′ has vertical fluid-conducting inlet and outlet bores 53 and 55 communicating with respective radial bores 56 and 57 provided with respective inlet and outlet connector nozzles 58 and 59, the nozzles being clamped to the still frame as in the device of Fig. 3. Also, the lower end of the tubular shell 47′ has annular ribs 65 and annular grooves 66 and 67, the latter communicating with the vertical inlet bores 53.

The bottoms of the annular recesses 83′ communicate with respective radial bores 96 into which nipples 86 are threaded. These nipples and the similar nipples of the end fittings 27′ and 30′ permit the flow of fluid through the jacket spaces 35′ of the evaporator section and the condenser section. A suitable heating fluid, such as steam, is passed through the jacket space of the evaporator section to heat the inner tubular wall thereof, which defines an evaporating chamber 73′ A suitable cooling fluid, such as cold water or brine, is passed through the jacket space of the condenser section to cool the inner tubular wall thereof, which defines a condensing chamber 82′. The tubular shell 47′ provides a free vapor passage between the evaporating chamber and the condensing chamber. As in the device of Fig. 3, the solution or extract to be distilled is distributed on the heated inner wall of the evaporating chamber by coiled distributing springs 68 and 69, the latter being carried on a supporting frame 70, 71. The nozzle member 44 in the upper end fitting 27′ permits connection to a vacuum pump, not shown.

In assembling the still, the several stacked parts are placed between the clamping screws 22 which seat the ends of the inner glass tubes 31′ firmly against the packing rings 88. The outer glass tubes 32′ are sealed by the packing rings 90 and gland rings 91 which permit these tubes to "float" so as not to interfere with the application of sealing pressure to the inner glass tubes. The glass tubes are relatively inexpensive and easily cleaned and can be of sufficient thickness to provide considerable strength.

The still of Fig. 11 is generally similar in operation to that of Fig. 3 with the exception that the solvent evaporated in the evaporating chamber passes directly to the condensing chamber. The solvent vapor condenses on the cold surface of the inner glass tube of the condensing chamber and flows through the bores 55 and 57 and out through the discharge nozzle member 59.

The still construction of Fig. 11 is also useful in vapor compression systems where the evolved vapor is drawn off and compressed and then passed into and condensed in the jacket space of the evaporating chamber.

What I claim as new and desire to secure by Letters Patent is:

1. In a still, a columnar still assembly including aligned upper and lower column members and a plurality of separately mounted flow fittings one intermediate said column members and others at the upper end of said upper column member and at the lower end of said lower column member, said lower column member forming an evaporating chamber and said upper column member forming a condensing chamber, said intermediate fitting forming a joint ring between said column members and having a feed inlet for liquid material to be distilled and further having a vapor passage between said chambers and a liquid outlet from the condensing chamber, said ring-forming intermediate fitting further having an annular portion projecting downwardly into said lower column member and provided with an outwardly facing peripheral liquid-receiving groove communicating with said feed inlet and disposed adjacent to the inner wall of said lower column member, and said lower fitting having a liquid outlet for said evaporating chamber.

2. In a still having upper and lower column members forming fluid-treating chambers therein, the lower column member having a heating surface and forming an evaporating chamber, a flow fitting having a peripheral flange interposed between said column members in sealing engagement therewith, said flow fitting forming a joint ring with upper and lower portions extending into said column members and further having a central vapor passage therethrough, said fitting also having a lateral liquid feed inlet with a circumferential bottom discharge adapted to introduce liquid into the lower column member, and said fitting having a lateral liquid outlet communicating with said upper column member to conduct liquid from the upper column member.

3. In a still having upper and lower column members forming fluid-treating chambers therein and each provided with jacket spaces, the lower column member having a heating surface and forming an evaporating chamber, a flow fitting adapted to be interposed between said column members in sealing engagement therewith, said flow fitting forming a joint ring between said column members and having a central vapor passage therethrough, said fitting also having a lateral liquid feed inlet with a circumferential bottom discharge adapted to introduce liquid into the lower column member, said fitting having a lateral liquid outlet adapted to conduct liquid from the upper column member, and said fitting further having lateral passages adapted to communicate with said jacket spaces.

4. In a still, a columnar still assembly including a lower column member with heating means and forming an evaporating chamber and an upper column member with cooling means and forming a condensing chamber, closure-forming fitting means at the lower end of said lower column member having an outlet for liquid separated in said evaporating chamber, closure-forming fitting means at the upper end of said upper column member having a vacuum passage communicating with said condensing chamber, intermediate ring-forming fitting means between said column members having a vapor passage connecting said chambers and having a feed inlet for liquid to be evaporated and an outlet for condensed liquid, said intermediate fitting means including a tubular portion forming therethrough said vapor passage and forming a condensate-confining wall at the upper end of said tubular portion, and supporting means for holding said column members and fitting means in assembled relation under axial compression, said supporting means centrally engaging said upper and lower closure-forming fitting means.

5. In a still, a columnar still assembly including a plurality of at least three aligned column members and fittings between adjacent column members and at the ends of the uppermost and lowermost column members, said lowermost column member having heating means and forming an evaporating chamber, said uppermost column member having cooling means and forming a condensing chamber, and the intervening column member forming a fractionating chamber, the fitting between said evaporating column member and the fractionating column member forming a joint ring and having a feed inlet for liquid material to be distilled and further having a vapor passage between the evaporating chamber and the fractionating chamber and a liquid outlet from said fractionating chamber, said lower end fitting having an outlet for liquid separated in said evaporating chamber, the fitting between said fractionating column member and condensing column member having an outlet for condensate and having an upstanding tubular condensate-confining portion projecting upwardly into said condensing chamber and spaced from the inner walls of said condensing chamber, and the upper end fitting having a vacuum connection communicating with said condensing chamber.

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