Aug. 29, 1950 G. A. DALIN 2,520,494

DISTILLING COLUMN, AND THE LIKE

Filed July 23, 1948 2 Sheets-Sheet 1

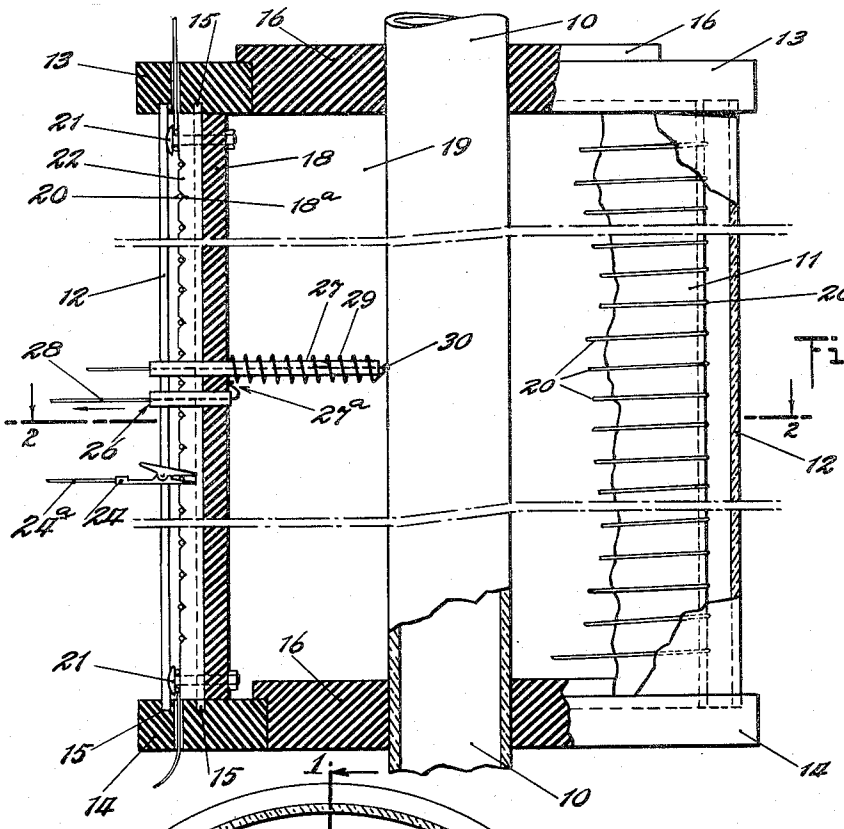


Fig. 1.

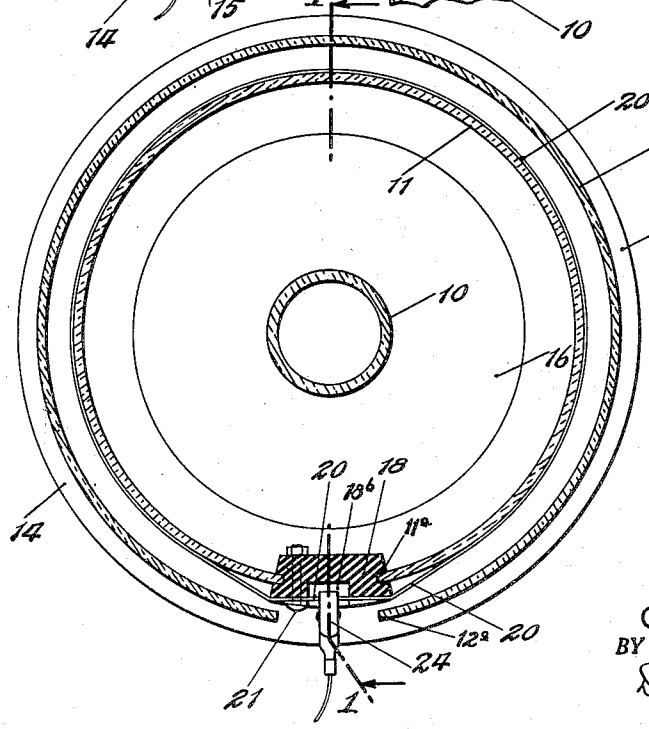


Fig. 2.

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Filed July 23, 1948 2 Sheets-Sheet 2

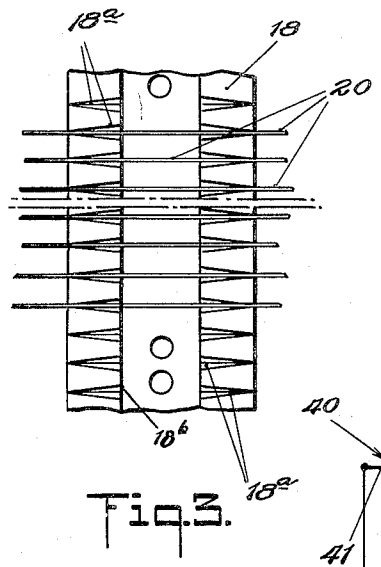


Fig. 3.

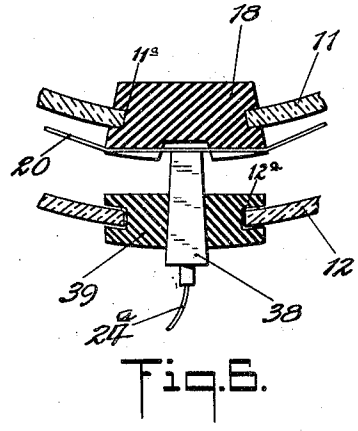


Fig. 6.

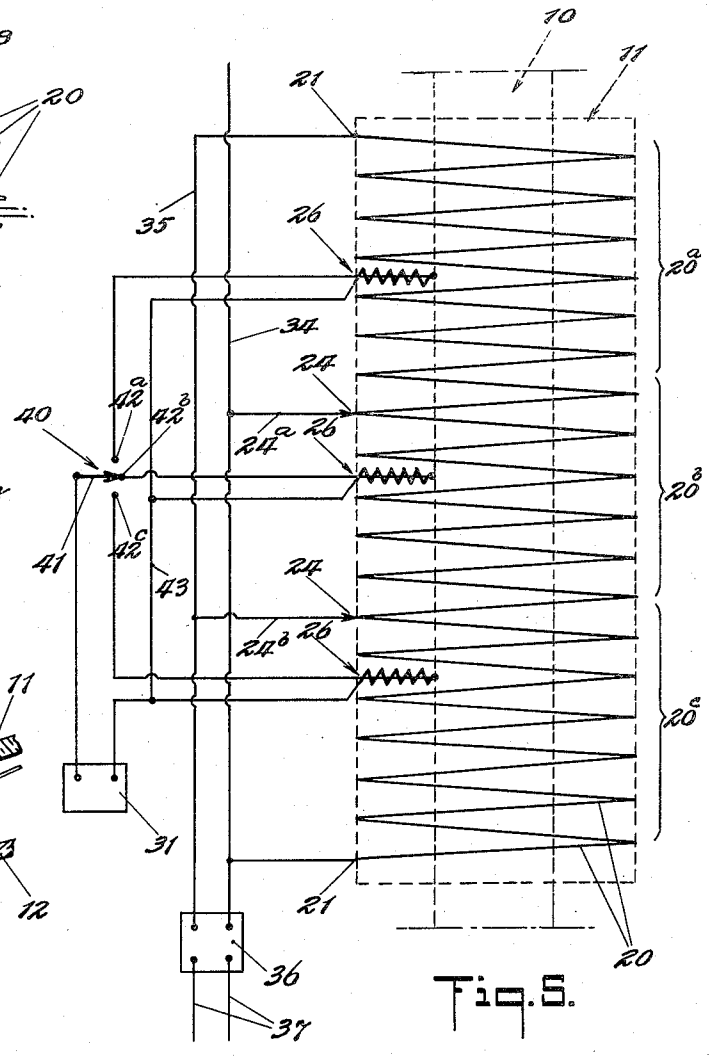


Fig. 5.

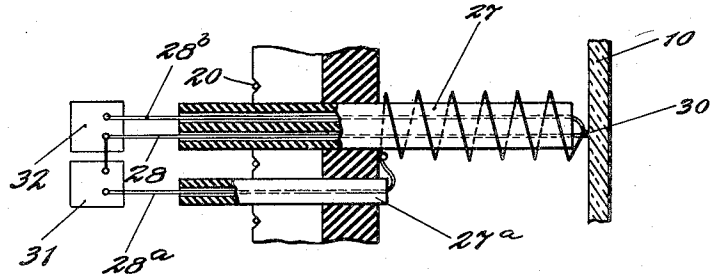


Fig. 4.

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DISTILLING COLUMN AND THE LIKE

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Application July 23, 1948, Serial No. 40,302

16 Claims. (Cl. 219—19)

1

This invention relates to columns or towers in which it is desired to maintain a predetermined temperature condition for distillation, condensation, or chemical reaction purposes.

In the operation of a fractionating or distilling column, it is often important to prevent heat exchange between the vapors in the column and the surrounding atmosphere, so that the column can be operated adiabatically or with a more or less uniform temperature gradient between inlet and outlet. Provision of insulation or a vacuum jacket around the column reduces the heat losses, but such expedients do not always suffice, particularly when the column throughout is small, as in laboratory apparatus, or when even small heat losses cannot be tolerated in larger columns. In such cases, it has been the practice to heat the column externally to offset any heat losses, and it is with externally heated apparatus of this type that the invention is especially concerned.

It has been proposed to maintain the desired temperature conditions in distilling columns, and the like, by surrounding the column with a jacket and heating the intervening enclosed space electrically by one or more wires disposed around the heating jacket from top to bottom. Such apparatus as constructed heretofore leave much to be desired, largely because they do not lend themselves to easy and precise adjustment of both the amount and the distribution of external heat supplied along the column, which adjustments often are critical and are needed during the distilling operation as well as in changing over from one operation to the next. Control of the total amount of heat can be had quite easily by external regulation of the current supplied to the electrical heating element or elements. However, in prior apparatus of this type the facilities for adjusting the distribution of the total heat supply along the column are limited, in spite of the fact that a simple means for making such adjustments accurately and in small increments over a wide range is recognized as highly desirable.

Another difficulty with prior apparatus of this type is in determining the temperature conditions in the annular space between the heating jacket and the column. In some instances, it has been necessary for this purpose to mount thermometers at the desired locations in the annular space. Due to the inaccessibility of this space, which is closed at the top and bottom by supports for the column and jacket, it is a troublesome procedure to change the location of the thermometers in

2

order to determine the proper heat input along different parts of the column or for any other purpose.

The principal object of the present invention is to provide a distilling column, or the like, which overcomes the above-mentioned difficulties inherent in prior apparatus of this type.

In order to realize the advantage of visibility of the column, it is the practice to use glass as the material for the heating jacket and also for the usual draft jacket surrounding the heating jacket. Since the jackets extend continuously around the column and are fragile, access to the heating wire between the jackets, and to the temperature indicators between the heating jacket and the column, has been had only at the top and bottom of the jackets where they are closed in the assembled apparatus by the supports or end caps, which have openings for the conductors leading to the heater. This characteristic of the prior apparatus, inaccessibility of the heater and indicators except from the top and bottom of the jackets, imposes limitations upon the apparatus, as pointed out above, but has been regarded as necessary in view of the nature and arrangement of the jackets.

I have discovered that in apparatus of this type the heating wire and indicators can be made accessible from the side of the apparatus which, at the same time, can be made sufficiently strong even though the jackets are constructed of glass. According to the invention, the heating jacket carries a series of turns of heating wire energized by one or more conductors extending outwardly from the side of the heating jacket and, if a surrounding draft jacket is used, through an opening or vertical slot in the latter. Also, the heating jacket may be provided with a series of vertically spaced openings between the wire turns and through which temperature indicators can be inserted into or removed from the space around the column. Thus, the wire turns on the heating jacket can be easily tapped from the side of the apparatus at any desired vertical location along the jacket so as to enable variations in the heat distribution, and the indicators can be adjusted vertically in the heating jacket in accordance with changes in the heat distribution.

In the preferred construction, the heating jacket is provided with a vertical slot which receives a retaining post aligned with a vertical slot in a draft jacket. The post is made of insulating material adapted to withstand the high temperatures; and it holds the wire turns in position on the heating jacket and is held in its

slot by the tension in the wire. In this way, even though the heating jacket is made of glass, it has sufficient strength and resistance to breakage in spite of the slot. The wire turns extending across the face of the retaining post are readily accessible through the slot in the draft jacket. Also, by providing the retaining post with openings offset from the wire turns, suitable temperature indicators can be easily inserted at the desired locations in the space between the heating jacket and the column.

For a better understanding of the invention, reference may be had to the accompanying drawings, in which:

Fig. 1 is a vertical sectional view of one form of the new apparatus, the view being taken on the line 1—1 in Fig. 2;

Fig. 2 is a horizontal sectional view of the apparatus, taken on the line 2—2 of Fig. 1;

Fig. 3 is an elevational view of the upper part of the retaining post illustrated in Figs. 1 and 2, showing the manner in which the heating wire is held by the post;

Fig. 4 is an enlarged vertical sectional view of a modified form of the temperature indicating device;

Fig. 5 is a schematic view of the apparatus, showing the electrical connections, and

Fig. 6 is a horizontal sectional view of part of a modified form of the apparatus.

For illustrative purposes, the apparatus is shown in a form suitable for laboratory use in fractional distillation. However, it will be understood that the invention can be utilized in production installations and for purposes other than distillation, such as effecting chemical reactions under controlled temperature conditions, in which case the column will constitute a chemical reactor tube. In fact, the apparatus can be used to control the heating of any object within the heating jacket.

In the apparatus as illustrated, the distilling or fractionating column 10 is of the usual hollow cylindrical form made of glass and adapted to receive at its lower end the volatile components from a flask (not shown) in which the mixture to be distilled is boiled. The volatile components passing up through the column are withdrawn at the upper end through a suitable outlet (not shown). A cylindrical heating jacket 11 surrounds the column and in turn is surrounded by a cylindrical draft jacket 12. As is customary in apparatus of this type, the two jackets are made of glass, which allows the operator to see the interior of the column during the distilling operation. The parts 10, 11 and 12 are held in concentric relation by top and bottom caps 13 and 14 mounted above the flask on a suitable support (not shown). Each cap has concentric grooves 15 for receiving the adjacent ends of the two jackets, and also has a central opening for receiving a removable collar 16 closely surrounding the column, the collars 16 having peripheral flanges resting upon the surrounding caps. Thus, the column can be easily replaced by one of different size by substituting collars having openings of different size.

The draft jacket 12 has a vertical slot 12a opposite a vertical slot 11a in the heating jacket, the slots extending from the top to the bottom of the jackets. Slot 11a is somewhat wider than slot 12a and receives a retaining post 18 made of asbestos or other electrical insulating material capable of withstanding the temperatures encountered in the operation. The post is fitted closely in the slot 11a and projects outwardly therefrom toward the draft jacket and also inwardly into the annular space 19 between the column and the heating jacket. The interengaging edges of the post and the slot 11a lie in vertical planes extending radially from the axis of the column, for a purpose to be described presently.

A heating or resistance wire has a series of turns 20 carried by the heating jacket, including the post 18. The wire is wound closely around the jacket 11 in a spiral, the ends of the wire being connected to terminals 21 mounted on the post 18 near the top and the bottom. The retaining post is provided with means for holding the turns 20 in vertically spaced relation, such means as illustrated being transverse detents 22 on the outer face of the post forming transverse grooves 18a for receiving the turns. It will be apparent that the tension in the wire turns serves to hold the post in its slot 11a. More particularly, when the post is inserted in the slot and the heating wire wound around the jacket 11, with the turns in the grooves 18a, the tendency of the wire turns to force the edges of the slot together is exactly counterbalanced by the tendency to force the post into the slot and spread the edges. It is for this reason that the cuts forming the interengaging edges of the post and the slot 11a are radial, since the post then acts, insofar as balancing these forces is concerned, as if it were an integral part of the glass jacket, whereby breakage of the glass is avoided. A further advantage is that if the width of the slot 11a is not exactly as planned, the tapered cross-section of the post makes it possible to compensate by seating it farther into or out of the slot. To compensate for variations in the width of the slot from top to bottom, it may be desirable to use two or more sections of post arranged end to end in the slot. Each section will then seat itself at the appropriate depth, thereby giving sufficient support along the glass jacket to prevent collapse.

In regard to the radial cuts forming the vertical slot 11a in the heating jacket, it is well known that a glass tube as usually made is non-uniform in thickness, diameter and cross-sectional shape, and is not exactly straight. However, if the radial cuts are made to intersect an arbitrary straight axis approximating a true axis, and the subtended angle between the slot edges is the same as the extended angle between the tapered edges of the retaining post, the post will seat in the slot along its entire length even though the glass tube is misshapen.

In its outer face, the post 18 has a vertical groove 18b which is of sufficient width to receive adjustable contacts 24 for energizing the heating wire. As shown in Fig 5, there is a pair of such contacts, each of which may be a standard alligator clip (Fig. 1). The groove 18b is sufficiently deep to allow the jaws of the clip to grasp one of the wire turns 20, without interference from the post.

The retaining post 18 is also provided with holes, opening into the groove 18b, for receiving one or more temperature indicating devices. As shown, I employ three such devices 25, one for each of the heating sections 20a, 20b and 20c into which the heating wire is divided by the two adjustable contacts 24 (Fig. 5). Each indicating device 25 may conveniently be in the form of a thermocouple assembly comprising a pair of ceramic insulators 27, 27a projecting from holes in the

post outwardly through spaces between the wire turns 20. The insulator 27 projects inwardly into the annular space 19 where it terminates near the distilling column 10, and it contains a "Constantan" wire 28 connected at its inner end to a coil spring 29 at a junction 30, as by brazing or soldering or in some other manner whereby the temper of the wires is retained. The spring 29, made of iron wire, is coiled around the insulator 27, and its outer end is connected to a "Constantan" wire 28a extending through the insulator 27a. With this construction, the spring 29 urges the junction 30 against the column 10, so that by connecting a galvanometer 31 (Figs. 4 and 5) across the wires 28 and 28a outside the apparatus, the temperature differential across the space 19 can be easily determined. Also, the spring 29 enables the junction 30 to be retracted from the column, either by sliding of the wire 28 in its insulator 27 or by sliding of insulator 27 in the post, which facilitates replacement of the distilling column 10; and the junction 30 will automatically position itself against the column regardless of its diameter.

If desired, the upper insulator 27 may contain a second wire 28b of iron located in a separate passage in the insulator and connected at its inner end to the junction 30, as shown in Fig. 4. In this way, by connecting a milli-voltmeter 32 (Fig. 4) across the wires 28 and 28b, the actual temperature of the column can be determined where it is engaged by the junction 30.

Referring now to the wiring diagram, Fig. 5, the terminals 21 of the heating wire 20 are connected to conductors 34 and 35, respectively, which in turn are connected through a variable transformer (or rheostat) 36 to a current source 37. The upper and lower contacts 24 are connected through leads 24a and 24b to the conductors 34 and 35, respectively, and are adjustable vertically along the heating jacket, such adjustment being permitted either by the flexibility of the leads 24a and 24b or by sliding of the leads along the conductors 34 and 35. Thus, the contacts divide the heating wire into upper, intermediate, and lower heating sections 20a, 20b and 20c, respectively, the three sections being connected in parallel across the current source. This arrangement permits quick and easy adjustment not only of the total amount of heat from wire 20 (by adjusting of the transformer or rheostat 36) but also of the distribution of the heat along the column within the jacket 11.

For example, by moving the lower contact 24 downwardly so that it engages a lower turn of wire 20, the resistance of the lower section 20c is decreased as well as the vertical extent of this section. Since the heat output of this section is E^2/R and E is fixed by the variable transformer or rheostat 36, then the heat output is inversely proportional to R. Thus, decreasing R by decreasing the number of turns in section 20c increases the heat output in section 20c. Conversely, the resistance and vertical extent of the intermediate section 20b is increased, but its heat output is decreased. By raising the lower contact 24, these changes in the output and vertical extent of the respective sections 20b and 20c are reversed. Similar adjustment of the vertical position of the upper contact 24 causes similar changes in the output and vertical extent of the respective heating sections 20a and 20b. By raising or lowering the contacts 24 equal amounts, the top and bottom heating sections 20a and 20c can be adjusted to vary their respective heat outputs and vertical extents, without affecting the intermediate section 20b except to change its vertical position. Thus, the distribution of the heat output can readily be varied by small steps over a wide range, which is a highly advantageous feature in apparatus of this type, as is well known in the art.

The contacts 24 may, of course, be of different form than that shown in Fig. 1. For example, they may be sliding contacts, as shown at 38 in Fig. 6, in which case the vertical groove 13b in the post may be reduced in depth or eliminated. As shown in Fig. 6, each contact 38 is carried by a slide 39 mounted in the vertical slot 12a in the draft jacket. By sliding the contacts up and down on their respective slides 39, the distribution of the heat may be varied, as previously described. The contact should be long enough to make contact with any wire turn before breaking contact with an adjacent turn, thus avoiding sparking.

The location of the thermocouples 26 may be varied according to the adjustments of the heating sections 20a, 20b and 20c, or as otherwise desired, by inserting them in selected holes spaced along the retaining post. Usually, it is desirable to position a thermocouple at the central part of each heating section. By use of the differential thermocouples, it is a relatively simple matter to determine the proper adjustment of the contacts 24 (38) for the necessary heat output from each heating section in order to obtain the desired temperature conditions along the column, as indicated by the thermocouples. Determination of the temperature differentials at the several thermocouple locations can be facilitated by use of a selector switch 40 (Fig. 5) having an arm 41 engageable selectively with contacts 42a, 42b and 42c connected to one side of the upper, intermediate and lower thermocouples 26, respectively. The other side of each thermocouple is connected to a common wire 43 leading to one side of the galvanometer 31, the other side of which is connected to the arm 41. Thus, engagement of arm 41 with any of the contacts 42a, 42b, 42c connects the corresponding thermocouple across the galvanometer, the reading of which indicates the temperature differential.

It will be apparent that in the new apparatus, the turns of the heating wire are readily accessible from the side of the apparatus along the entire height of the heating jacket, and because of this feature I am able to provide for the aforementioned adjustment of the heat distribution in a simple and effective manner. Also, by virtue of this feature, the wire turns may be arranged to form a plurality of separate heating elements which can be eneergized and deenergized automatically under control of respective thermostats extending into the space 19, thereby maintaining the desired temperature condition. The vertical slot in the outer or draft jacket 12 need be only of a width sufficient to accommodate the contacts 24 or the slides 39, so that the usual functioning of the draft jacket is not impaired. The column 10 can be operated in the manner which is customary in distilling apparatus (or in a chemical reactor), the adjustable contacts and the thermocouples, however, enabling easy regulation of the temperature conditions.

The end caps 13 and 14 may, if desired, be provided with annular grooves 15 on both faces, so that the heating and draft jackets can be stacked to accommodate columns of any length. The column 10 may be supported by a bulge in its upper or lower portion overlying the upper or

lower collar 16. It will be understood that the apparatus is susceptible of other modifications within the scope of the invention.

I claim:

1. In an apparatus of the character described, having a column, a heating jacket surrounding the column and spaced therefrom, and a draft jacket surrounding the heating jacket and spaced therefrom, the improvement which comprises a heating wire having a series of turns carried by the heating jacket and spaced vertically from each other, the draft jacket having a vertical slot through which the turns are accessible, an adjustable contact extending through the slot and engaging one of the wire turns, the contact dividing the wire into upper and lower heating sections and being adjustable vertically in the slot to engage different turns of the wire and thereby vary the resistances and the vertical extents of the respective sections, a current source, and electrical connections, including the adjustable contact, for connecting said sections in parallel to the current source.

2. The improvement as defined in claim 1, in which the heating jacket has a recess opposite the draft jacket slot and over which the wire extends, the recess being adapted to receive part of said contact.

3. The improvement as defined in claim 1, in which the heating jacket has an opening opposite the draft jacket slot and located between adjacent turns of the wire, and a temperature indicating device extending through the slot and the opening into the space between the heating jacket and the column.

4. The improvement as defined in claim 1, in which the heating jacket has a vertical slot opposite the draft jacket slot, and a retaining post disposed in the heating jacket slot and engaging the wire turns to hold them in vertically spaced relation.

5. In an apparatus of the character described, having a column, a heating jacket surrounding the column and spaced therefrom, and a plurality of turns of heating wire carried by the jacket for heating the space between the jacket and the column, the improvement which comprises an insulating retaining post disposed within said turns and holding the turns in spaced relation on the jacket, the post having a plurality of holes each aligned with a lateral opening in the jacket for inserting a temperature indicating device through the post and jacket into the space between the jacket and the column.

6. The improvement as defined in claim 5, in which the heating jacket is slotted vertically to form said lateral opening, the post being fitted into the vertical slot to give mechanical support to the jacket.

7. In an apparatus of the character described, having a column, a heating jacket surrounding the column and spaced therefrom, and a heating wire carried by the jacket for heating the space between the jacket and the column, the improvement which comprises a retaining post of insulating material forming part of the jacket wall, the jacket having a vertical slot in which the post is fitted and providing access to the interior of the jacket, the heating wire including a plurality of turns extending at least partly around the exterior of the jacket, means on the post for holding said turns in vertically spaced relation, a contact for energizing the wire and adjustable along the post to engage different turns of the wire, and a draft jacket surrounding the heating jacket and spaced therefrom, the draft jacket having a vertical slot opposite said first slot and through which the adjustable contact extends.

8. The improvement as defined in claim 7, in which the wire turns extend across the outer face of the post to hold the post in the slot.

9. The improvement as defined in claim 7, in which the post and the slot have interengaging edges, each edge lying in a vertical plane extending radially from the column.

10. In an apparatus of the character described, having a column, a heating jacket surrounding the column and spaced therefrom, and a heating wire carried by the jacket for heating the space between the jacket and the column, the improvement which comprises a retaining post of insulating material forming part of the jacket wall, the jacket having a vertical slot in which the post is seated, the heating wire including a plurality of turns extending at least partly around the exterior of the jacket, means on the post for holding said turns in vertically spaced relation, and a contact for energizing the wire and adjustable along the post to engage different turns of the wire, the post and the slot having interengaging edges, each edge lying in a vertical plane extending radially from the column, the wire turns extending under tension across the outer face of the post to hold the post in the slot, whereby the tendency of the wire to force the slot edges together is counterbalanced by the tendency of the wire to force the post into the slot and spread the slot edges.

11. In an apparatus of the character described, having a column, a heating jacket surrounding the column and spaced therefrom, and a heating wire carried by the jacket for heating the space between the jacket and the column, the improvement which comprises a retaining post of insulating material forming part of the jacket wall, the jacket having a vertical slot in which the post is seated, the heating wire including a plurality of turns extending at least partly around the exterior of the jacket, means on the post for holding said turns in vertically spaced relation, and a contact for energizing the wire and adjustable along the post to engage different turns of the wire, the post having a vertical groove in its outer face, the turns extending across the groove, said contact including a releasable clamping device extending into the groove.

12. In an apparatus of the character described, having a column, a heating jacket surrounding the column and spaced therefrom, and a heating wire carried by the jacket for heating the space between the jacket and the column, the improvement which comprises a retaining post of insulating material forming part of the jacket wall, the jacket having a vertical slot in which the post is seated, the heating wire including a plurality of turns extending at least partly around the exterior of the jacket, means on the post for holding said turns in vertically spaced relation, a contact for energizing the wire and adjustable along the post to engage different turns of the wire, and a draft jacket surrounding the heating jacket and spaced therefrom, the draft jacket having a vertical slot opposite said first slot and through which the adjustable contact extends, and a slide mounted in the draft jacket slot for supporting the contact.

13. In an apparatus of the character described,

having a column, a heating jacket surrounding the column and spaced therefrom, and a heating wire carried by the jacket for heating the space between the jacket and the column, the improvement which comprises a retaining post of insulating material forming part of the jacket wall, the jacket having a vertical slot in which the post is seated, the heating wire including a plurality of turns extending at least partly around the exterior of the jacket, means on the post for holding said turns in vertical spaced relation, a contact for energizing the wire and adjustable along the post to engage different turns of the wire, and a plurality of temperature indicating devices extending through the post into said space for indicating the column temperature and also the temperature difference across the space.

14. In combination with a column for fractionating, and the like, and a heating jacket surrounding the column and providing an intervening space, a device for determining temperature conditions in said space and comprising an insulating member disposed in an opening in the jacket, a thermocouple including a pair of wires extending through the insulating member and interconnected in said space, a second insulating member disposed in an opening in the jacket, a differential thermocouple including a third wire extending through the second member into said space, a spring wire connected between said last wire and the interconnection of the first wires

and acting to urge said interconnection against the column, a voltmeter connected across the first wires to indicate the column temperature, and a galvanometer connected between said third wire and one of the first wires for indicating the temperature differential across said space.

15. The improvement as defined in claim 7, comprising also a temperature indicating device extending through an opening in the post into said space.

16. The improvement as defined in claim 7, comprising also a temperature indicating device extending through an opening in the post into said space, and a spring connected to said device for urging the same against the column.

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