Sept. 25, 1956 A. G. NERHEIM 2,764,534

HEAD FOR SPINNING BAND FRACTIONATOR

Filed May 11, 1955

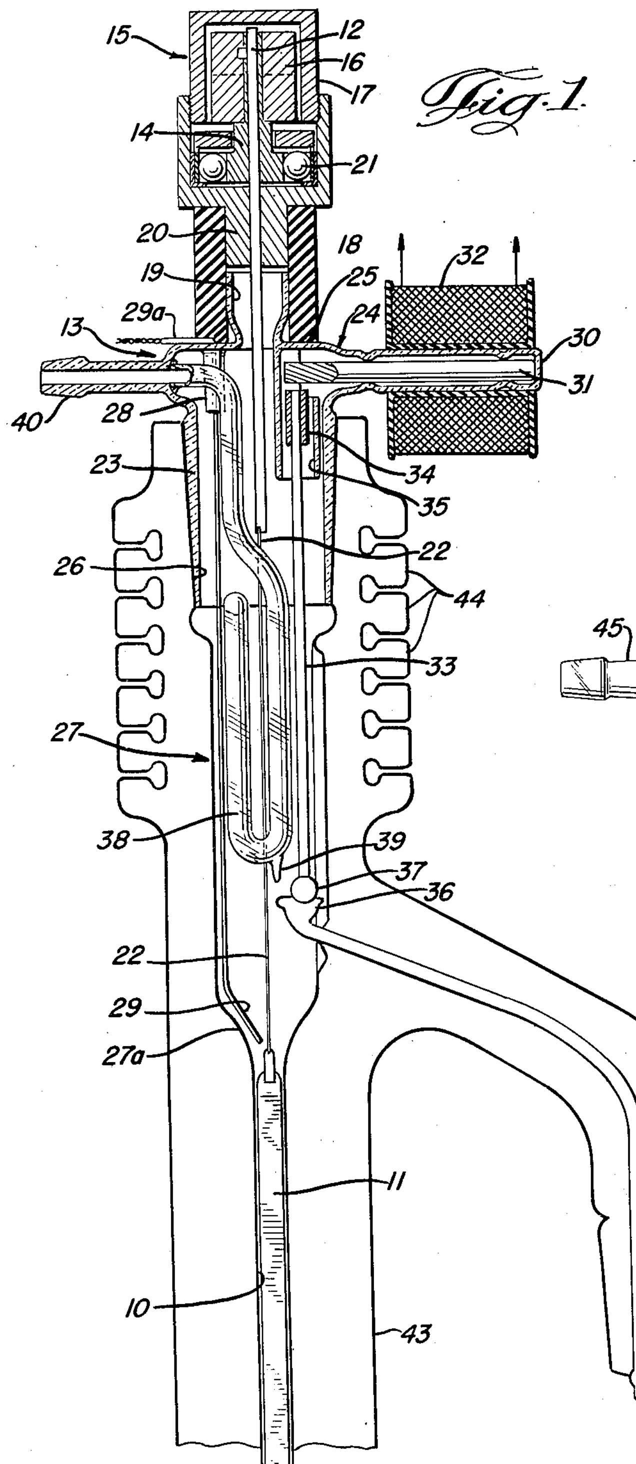
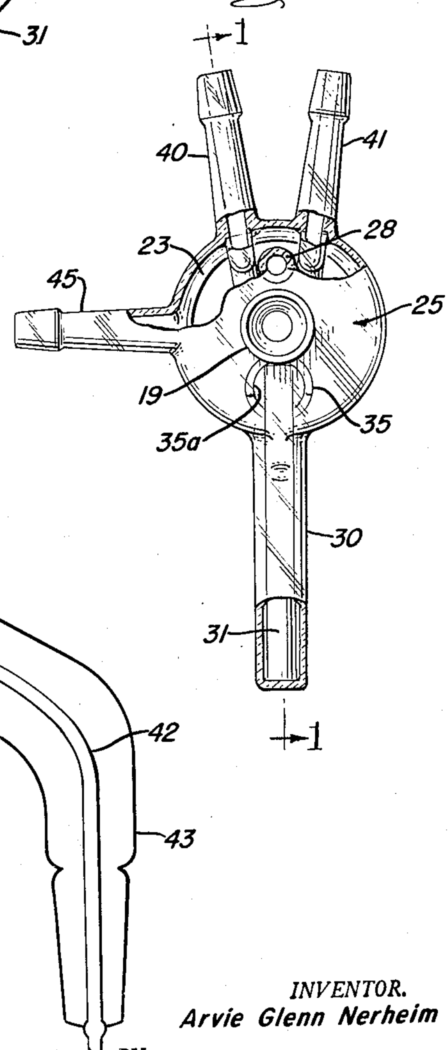


Fig. 1.

Fig. 2.



INVENTOR.
Arvie Glenn Nerheim

BY Everett A. Johnson
Attorney

United States Patent Office

2,764,534

Patented Sept. 25, 1956

2,764,534

HEAD FOR SPINNING BAND FRACTIONATOR

Arvie Glenn Nerheim, Calumet City, Ill., assignor to Standard Oil Company, Chicago, Ill., a corporation of Indiana

Application May 11, 1955, Serial No. 507,630

7 Claims. (Cl. 202—153)

This invention relates to an improved laboratory miniature fractionator of the type known as a spinning band column. More particularly, the invention relates to a column head construction for such a miniature fractionator.

The need for fractional distillation of small quantities of organic liquids has increased with the trend in research for small scale experiments. Miniature distillation columns have been designed which give separations of 15 to 50 ml. samples equal to those obtainable with large samples on macro high-efficiency laboratory columns. However, effective miniature distillation requires more than just scaling down a large column. On smaller scale, control of throughput and reflux ratio must be more precise. Losses of sample must be avoided and entrainment of vapors during vacuum operation must be eliminated. Contamination of fractions must be minimized; a single drop of distillate can represent 10% contamination in a succeeding fraction if it is held on the walls of the receiver system.

Miniature fractionation equipment has been designed to meet these requirements, three types of which are in common use. They include the so-called spinning band, "Hyper-Cal," and concentric tube fractionating columns. These three types of columns make it possible to fractionate most organic liquids and all have heights equivalent to a theoretical plate (H. E. T. P.) of about 1 cm. Each of the various types, however, has advantages and disadvantages when compared to the others. The spinning band type has low holdup and low pressure drop and is especially useful at reduced pressures. The Hyper-Cal, which contains a fixed heli-grid packing, is effective in separations of larger samples at atmospheric pressure. The all glass concentric tube column has a low H. E. T. P. but requires a high reflux ratio.

I prefer to use a spinning band column and my invention is particularly adapted for use on columns of that type.

Such a unit comprises in general a still pot for holding the charge, a fractionating column or tube, a condenser system, a liquid product receiver, and a rotatable element disposed within the fractionating tube.

Accurately controllable heating means are provided for supplying heat to the still pot, the still pot and heating element ordinarily being enclosed within a vacuum flask which is removably seated at its upper end in contact with a second vacuum flask which substantially encloses the fractionating column and the take-off line.

Column throughput in a spinning band column is conventionally controlled by controlling the heaters. However, it has been found that for a given speed of rotation of the spinning band the separation efficiency increases with decreasing throughput. Further, it has been found that column efficiency may be increased by increasing the rotation speed of the spinning band. However, if vibration occurs in spinning columns, the efficiency is lowered. Heretofore, vibration was likely to occur at

speeds above about 2200 R. P. M. and could occur at even lower speeds if the band and column were not properly aligned.

It is, therefore, an object of my invention to provide a spinning band column which may be operated at high throughputs without loss in efficiency. It is a further object of the invention to provide a head for a spinning band column and a band shaft mounting adapted to obtain vibration-free band rotation up to 6000 R. P. M. An additional object is to provide a vibration-free column which has a high efficiency while permitting a doubling of the throughput. Still another object of my invention is to provide an apparatus which permits efficient operation at high throughput by maintaining high efficiency at high R. P. M. It is an additional object of the invention to provide a miniature distillation apparatus which is capable of a wide range of operating conditions adapted to meet the needs of individual distillations. These and other objects of the invention will become apparent as the description thereof proceeds.

Briefly, I attain the objects of my invention by providing a head for a miniature distillation column which has a flat top wall and a minimal vertical height. Thus, the length of the band shaft for the spinning band passing through the head is considerably shortened. To eliminate a main source of vacuum leaks, the conventional external needle guide sleeve is eliminated. According to my invention, the guide sleeve is within the head and the needle shaft is provided with a paramagnetic section at its upper end. The needle valve may be lifted from its seat by an electromagnetic bar sealed within the head. The thermocouple sleeve is turned wholly within the head and the thermocouple leads are flush with the top wall.

The enclosed or introverted guide and thermocouple sleeves permit substantial reduction in the height of the head and consequently the length of the band shaft. It has been found that such reduction in band shaft length permits the use of high speeds of rotation without vibration and hence has been found to increase the fractionating efficiency of the column.

Further details and advantages of my invention will become apparent as the description thereof proceeds with reference to the accompanying drawings wherein:

Figure 1 is a vertical section taken along the line **1—1** of Figure 2 showing the improved head construction and related column components and spinning band drive means; and

Figure 2 is a plan view with some parts removed.

Referring to the drawings, a column **10**, comprising a relatively long thin glass tube, contains the spinning band **11** having a drive shaft **12** which extends through the head **13** and is secured to the drive pin **14** within the magnetic coupling **15**. This coupling **15** includes a pair of opposed magnets (only one of which is shown), each being of generally cylindrical configuration and arranged axially of each other and of the shaft **12**. The magnet not shown is secured, for example, by a chuck means (not shown) so that it may be driven, and the other magnet **16** is within a vacuum-tight and non-magnetic housing **17** which is fixed to the head **13** by means of a flexible connector **18** engaging the stem **19** on the head **13** and the stem portion **20** of the housing **17**.

The elongated drive pin **14** disposed within the separable housing **17** is arranged to be rotated by the enclosed magnet **16**. An anti-friction bearing means **21** rotatably supports the drive pin **14** within the housing **17**. The drive shaft **12**, fixed to drive pin **14**, extends longitudinally from the lower end of the housing **17**, through the stem **19**, the head **13** and engages the band shaft **22** which in turn supports the rotatable element or band

11. A magnetic coupling and housing, such as that illustrated, is described and claimed in co-pending application Serial No. 309,034, filed September 11, 1952, and entitled "Seal and Magnetic Drive for Spinning Band Column."

The rotatable element 11 is slightly smaller in its greatest lateral dimension than the internal diameter of the fractionating tube 10. Rotation of the close-fitting band 11 produces a "wetted wall" effect within the fractionating tube 10 with the result that there is intimate contact between the up-flowing vapors within the column and the down-flowing reflux on the wall thereof.

The head 13 includes a tapered body 23, a pancaked portion or cap 24 having a flat top wall 25, the tapered body 23 being arranged in a vacuum-tight seal with the seat 26 of the column 10. The cap 24 of the head 13 includes laterally-extending condenser inlet 40 and outlet 41, each passing through the side wall thereof and being connected to opposite ends of the looped condenser 38 which depends below the hollow body portion 23 into the condenser section 27 merging with the top of the column 10. A drip tip 39 is provided on each of the separate legs of the condenser 38. A vacuum connection 45, also laterally extending from the side wall of cap 24, permits vacuum operation of the column 10 and completes the construction of head 13.

The thermocouple 29 is encased within a small tubing 29*a* which passes through the thermocouple well 28 and is disposed laterally adjacent the upper flat wall 25 so as to lie flush therewith beneath the lower end of the flexible connector 18. To give correct readings, the thermocouple 29 is experimentally located within the condenser section 27 while refluxing a pure compound.

A solenoid coil 32 is fixed about the sealed arm 30 which contains the soft iron core 31. Immediately below the exposed inner end of the core 31 within the head 24 is the upper end of the needle 33 having its upper paramagnetic end 34 within the enclosed needle guide 35 and its tip 37 on the needle seat 36. The needle tip 37 is thus raised from the seat 36 by the action of the electromagnet 31 on the paramagnetic top 34 of the needle 33 when the solenoid coil 32 is energized.

A condenser 38 comprises a loop of tubing which doubles back on itself with a pair of drip tips 39 at the lower extremities of the loop which discharges on the edge of the needle seat 36. When the needle tip 37 is raised from the seat 36, the condensate is removed from the column by take-off line 42 into a receiver (not shown).

By regulating the flow of cooling fluid into the total condenser 38, the evolved vapors within the fractionating tube 10 may be condensed and returned as reflux within the fractionating column 10 when the take-off line 42 is closed by the needle valve.

Condensate is prevented from leaking past the needle seat 36 by careful alignment of the needle 33 and by keeping the tip 37 clean. The head 13 should be adjusted so that the needle 33 is vertical and moves freely within the introverted guide 35 depending from the upper flat wall 25 of the cap portion 24. A port 35*a* in the upper end of the guide 35 is in alignment with the arm 30 and admits one end of the electromagnetic core 31 into the upper end of the guide 35. It will be understood that the paramagnetic end 34 of the needle 33 is disposed immediately below the protruding end of the electromagnet 31 and is lifted thereby when the coil 32 is energized.

The bottom portion 27*a* of the condenser section 27 is tapered as shown and merges with the upper end of column 10 whereby reflux from the total condenser 38 is uniformly distributed on the wall of the fractionating column 10.

To minimize radiation heat losses from column 10, the vacuum jacket 43 may be insulated up to the condenser section 27 to assure adiabatic operation of the still. The upper end of the vacuum jacket 43 is provided in the

region of the condenser section with expansion bellows 44.

The enclosed or introverted needle guide 35 and the thermocouple sleeve 28 are both turned into the pancaked cap 24 of the head 13 thereby substantially reducing the total height of the head and consequently the length of the band shaft 22. It has been found that such reduction in band shaft length permits the use of higher speeds of rotation without vibration and hence has been found to permit increasing the throughput of the column at a desired fractionating efficiency and to increase the efficiency at a given throughput.

Conventionally, spinning band columns of the type to which my improvement relates employs a rotatable element comprising a metallic band. It is contemplated, however, that the metallic band 11 may be replaced by a non-metallic or plastic band or a metallic band coated with such a plastic. A preferred material is Teflon or Kel–F (tetra-fluoroethylene resin), polyethylene, nylon, polyurethanes or the like.

The configuration of such plastic or plastic-coated band is significant. A preferred form is a rod-like element having a cross-section of a square with concave sides, the edges of such rod-like element having inclined uniform serrations which are uniform but non-aligned with respect to serrations on adjacent edges of the band.

One way in which such a band can be fabricated is to thread a rod of square cross-section and then cut away a symmetrically curved portion of the faces of the rod located between the four "threaded" corner edges. It is also contemplated that such a band may have from 2 to 6 or more "threaded" edges.

In any event, the threaded edges are mounted in close running fit with the interior of the column wall and, depending upon the direction of rotation, can be used to affect the flow of reflux along the column wall and thereby control the efficiency of the vapor-liquid contact within the column. Advantages of this type of spinning band include a low holdup of liquid and low pressure drop as well as giving low H. E. T. P. values which are from ½ to ⅔ lower than the H. E. T. P.'s for spinning band columns employing conventional metallic rotatable elements.

Although the invention has been described with reference to a preferred embodiment thereof, it is to be understood that this is by way of illustration only. Accordingly, it is contemplated that modifications and variations can be made in the apparatus by those skilled in the art in light of the preceding description and without departing from the spirit of the invention.

What I claim is:

1. A take-off head for a spinning band fractionating column including an exteriorly tapered hollow body portion, a superposed hollow cap portion integral with said body portion, a substantially parallel top wall on said cap, hollow connector stem means extending axially of said wall, an introverted thermocouple sleeve extending inwardly from said upper wall and offset with respect to the axis of said connector stem means, a laterally extending tubular chamber communicating with said cap portion and closed at its outer end, said chamber being elongated and adapted to be surrounded by a solenoid coil, electromagnetic core means within said tubular chamber and extending within said head, a needle guide means within said head and extending inwardly from said top wall into said tapered body portion, said guide means being axially offset with respect to the axis of said connector stem means and of said body portion and having its upper end in alignment with said laterally extending chamber, a lateral port in said guide at the upper end thereof in alignment with said tubular chamber, said port accommodating the inner end of said core means, said guide means being adapted to receive the upper end of a needle valve means having a paramagnetic portion at the upper end thereof, a condenser means

supported by said cap portion and extending through and below said body portion, and condenser inlet and outlet means extending laterally from the side wall of said cap portion, said condenser means comprising a pair of looped legs offset with respect to the axis of said connector stem means and said body portion whereby a band shaft for driving a spinning band may pass through the apparatus without interference by the thermocouple sleeve, the needle valve guide, or the condenser means.

2. A take-off head for a laboratory fractionating column including an exteriorly tapered hollow body portion and a superposed hollow cap portion, a substantially planar upper wall on said cap, tubular connector stem means extending axially of said cap portion, an introverted thermocouple sleeve extending inwardly from the upper wall, a laterally extending tubular chamber communicating with said cap portion and closed at its outer end, electromagnetic core means within said tubular chamber and extending within said head, a needle guide within said head and extending inwardly into said tapered body portion from said planar wall, a lateral port in said guide at the upper end thereof in alignment with said tubular chamber, said port accommodating an end of said core means, a needle valve means extending within said tapered body portion and having an upper end disposed adjacent the inner end of said core within said introverted needle guide, and paramagnetic means on the upper end of said valve means.

3. The apparatus of claim 2 which includes a flexible connector means surrounding said tubular stem means, the end of said flexible connector being flush with the planar top wall of said cap portion.

4. The apparatus of claim 2 which includes a tubular condenser means extending longitudinally through said cap portion and below said body portion, a loop in said condenser doubling back on itself, said condenser being offset with respect to the longitudinal axis of said tubular stem means and said body portion.

5. In a spinning band column including a still pot, a fractionating column connected thereto, rotatable means disposed within said column, a condenser, and take-off head means superposing said column, the improvement which comprises a take-off head including an exteriorly tapered hollow body portion and a top chamber portion, a substantially planar upper wall on said chamber portion, an exterior hollow stem extending axially of said wall and adapted to receive said rotatable means, a thermocouple sleeve extending inwardly of the said upper

wall, a laterally extending enclosed tubular arm fixed to said head portion, a soft iron core within said tubular arm extending within said head, said arm being adapted to receive a solenoid coil encircling a major portion of said core, a needle valve guide sleeve within said head and extending inwardly into said tapered body portion from said wall, a port in said sleeve at the upper end thereof in alignment with said tubular arm and accommodating an end of said core, said needle valve means extending within said tapered body portion and having an upper end disposed subjacent the end of said core protruding within said needle sleeve, paramagnetic means on the upper end of said valve means, and a high pressure hose connection surrounding said hollow stem, said high pressure hose abutting the upper flat wall of said cap portion.

6. In a spinning band column including a still pot, a fractionating column connected thereto, rotatable means disposed within said column, a condenser, and take-off head means superposing said column, the improved apparatus which comprises a take-off head including an exteriorly tapered hollow body portion and a head chamber portion, a substantially planar upper wall on said chamber portion, an exterior hollow stem extending axially upward of said wall and adapted to receive said rotatable means, a thermocouple sleeve extending inwardly of the said upper wall, a laterally extending enclosed tubular arm fixed to said head portion, a soft iron core within said tubular arm extending within said head, said arm being adapted to enter a solenoid coil about a major portion of said core, a needle valve guide sleeve within said head and extending inwardly into said tapered body portion from said wall, and a port in said sleeve at the upper end thereof in alignment with said tubular arm and accommodating an end of said core.

7. The apparatus of claim 6 which includes a needle valve means extending within said tapered body portion and having an upper end disposed subjacent the end of said core protruding within said needle sleeve, paramagnetic means on the upper end of said valve means, and a high pressure hose connection surrounding said hollow stem, said high pressure hose abutting the upper flat wall of said cap portion.

References Cited in the file of this patent

UNITED STATES PATENTS

2,573,807	Piros	Nov. 6, 1951
2,608,528	Piros	Aug. 26, 1952