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VACUUM DISTILLATION APPARATUS

Original Filed July 31, 1939

FIG. 1

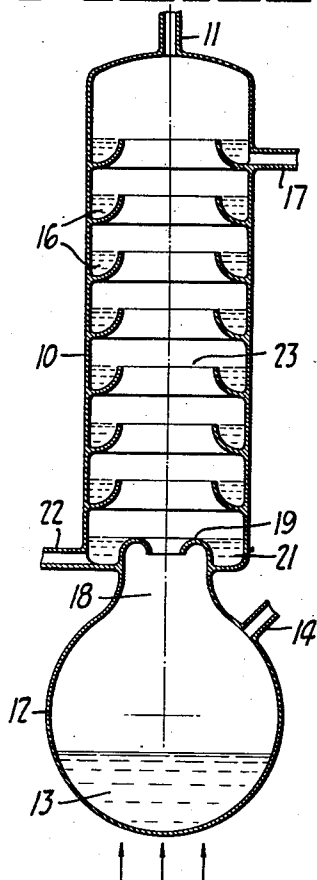


FIG. 2

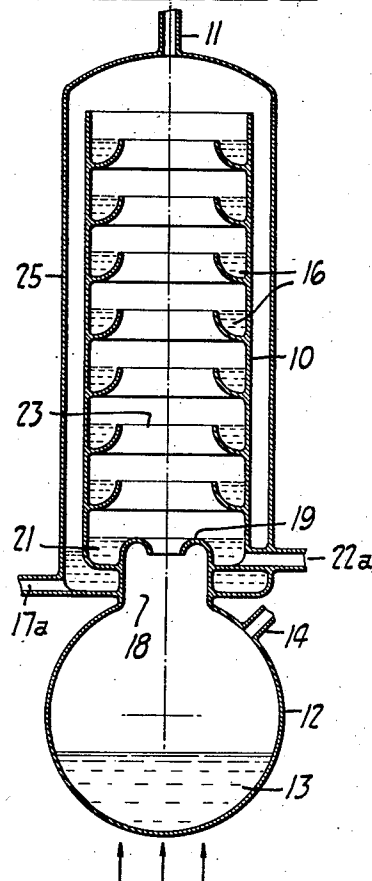
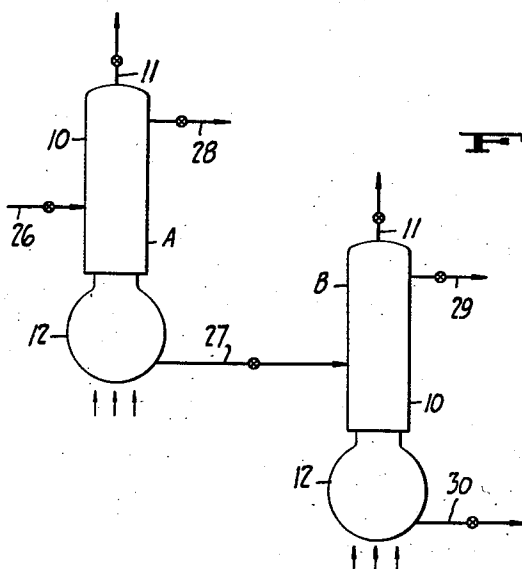


FIG. 3



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VACUUM DISTILLATION APPARATUS

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Original application July 31, 1939, Serial No. 287,525. Divided and this application September 15, 1941, Serial No. 410,855

2 Claims. (Cl. 202—205)

This invention relates generally to high vacuum distillation apparatus such as is useful for the fractional distillation of a wide variety of materials. By high vacuum, I have reference to vacuums of the order of 10–5 mm. of mercury or higher.

It is an object of the invention to provide an apparatus of the above character which will be relatively simple in construction and operation, and which will make possible the separation of fractions having a high degree of purity.

Another object of the invention is to make possible progressive fractionation and reflux action in connection with distillation at high vacuums.

Additional objects of the invention will appear from the following description in which the preferred embodiment of the invention has been set forth in detail in conjunction with the accompanying drawing.

This application is a division of my copending application Serial Number 287,525, filed July 31, 1939, Patent No. 2,266,053 entitled "Vacuum distillation method."

Referring to the drawing:

Figure 1 is a side elevational view in cross section, illustrating diagrammatically apparatus incorporating the present invention;

Fig. 2 is a side elevational view, in cross-section, showing another embodiment of the invention.

Fig. 3 is a side elevational view, illustrating a plurality of units constructed somewhat as illustrated in Fig. 1, and connected together to form a continuously operating series.

Referring first to Figure 1 of the drawing, I have illustrated apparatus comprising the fractionating column 10, having a connection 11 to a vacuum pump or other form of evacuating equipment capable of maintaining a high vacuum. Communicating with the lower end of the fractionating column there is a vessel 12 adapted to contain a charge of material 13, and which under normal conditions receives heat from a suitable source during the distillation cycle. An inlet pipe 14 is indicated for introducing material into the vessel 12.

The fractionating column 10 includes the series of fractionating cups 16. These cups can be in the form of rings as illustrated, and they are formed so that when filled with a condensed fraction, the fraction can overflow the inner lip into the next lower cup. The uppermost fractionating cup 16 is shown provided with a pipe connection 17, whereby the lightest fraction collected in this cup can be withdrawn.

The throat or passage 18 which connects the vessel 12 with the interior of the fractionating column 10, is preferably provided with means to prevent a washing up of any of the mother liquor into the fractionating column. This means is preferably in the form of a downturned lip 19, surrounding the passage 18. The space surrounding the region of lip 19 can form an additional fractionating cup 21, as illustrated. A tube 22 can be provided to draw off the liquid accumulating in cup 21, which in a typical instance represents material from which light ends have been removed, and which does not contain waxes, color components, and miscellaneous contaminants that are caused to remain in the vessel 12. Above the throat 18 the fractionating column 10 is provided with a substantially unobstructed passage 23, for free movement of molecules of the vaporized material leaving the charge of material undergoing treatment.

Use of the apparatus described above can be described as follows: A charge of the material undergoing treatment, which can be termed a mother liquid, is introduced into the vessel 12. The vacuum pump connected with tube 11 is placed in operation, whereby the interior of the apparatus is subjected to a high vacuum of the order of say 10–5 mm. of mercury. The charge 13 is maintained at a suitable temperature consistent with the character of the fraction or fractions to be removed by applying heat to the same. Assuming maintenance of proper vacuum and temperature conditions, and also assuming that the exterior of the fractionating column 10 is suitably heat insulated, as by means of heat insulating material or an evacuated space, various fractions are vaporized from the charge 13. The vacuum is such that the mean free path of movement of the molecules is at least as great as the distance between the lower fractionating cups and the surface of the mother liquid. Because of the slightly different temperature and pressure conditions existing at the various levels represented by the different fractionating cups, fractions are condensed and collected in the fractionating cups, and eventually all of the cups become filled with condensed fractions.

The distillation cycle is now in normal operation with the lower fractionating cups continuously receiving molecules of the vaporized material from the space 23. Some re-vaporization occurs continuously from the lower condensed fractions, and this re-vaporized material is condensed in upper fractionating cups. At the same time some overflow occurs from each cup and

the overflow liquid passes into the next lower fractionating cup. This progressive fractionating and reflux action makes for utmost purity of the separated fractions, particularly with respect to the lightest fraction removed from the uppermost fractionating cup.

The lip 19 serves the useful purpose of preventing contamination of the condensed fractions with mother liquid. Without this lip there is a tendency for the mother liquid to be washed upwardly to intermingle with the collected fractions. However, with this lip such mother liquid is returned back to the charge 13.

The degree of vacuum maintained and the temperature of the charge undergoing treatment is, of course, to be selected in accordance with the material undergoing treatment and the fractions to be removed. In most instances it will be found desirable to maintain such a vacuum that the mean free path of movement of the molecules is of the order of the distance between the surface of the liquid undergoing treatment, and an intermediate point about midway between the ends of the fractionating column.

Figure 3 illustrates an arrangement for providing an evacuated heat insulating jacket about the fractionating column. Thus in this instance the walls of the column are surrounded by a sealed jacket 25 which is in direct communication with the upper end of the column. Pipe 11 connects to the jacket and the lightest fraction can be withdrawn from pipe 17a, instead of from the top cup of the column. Also liquid can be withdrawn from the cup 21 through pipe 22a, as for example where one desires a liquid from which the light ends have been removed, and which does not contain waxes, color components and miscellaneous contaminants remaining in 12.

Figure 2 represents a plurality of units similar to that shown in Figure 1, connected in series. Pipe 26 in this instance represents introduction of the material undergoing treatment into an intermediate one of the fractionating rings of the first unit A. The lower vessel 12 of the unit A is shown connected to the drain pipe 27, which in turn leads to an intermediate one of the fractionating cups of the second unit B. Pipes 28 and 29 are shown for removing the light fractions from the uppermost cups of units A and B. Material from the lower end of unit B can be drained through the pipe 30.

In operating the apparatus of Figure 2, the material undergoing treatment can be continuously bled into unit A through pipe 26, and material can be continuously drained from vessel 12 of unit A, into the unit B, through pipe 27. The material in the lower vessel 12 of unit B can be maintained at a slightly higher temperature than the temperature of material in the vessel 12 of unit A. It will be apparent that with such continuous operation the collected material in the receiver 12 of unit A will maintain constant characteristics, thus making possible con-

tinual or continuous removal of a light fraction through pipe 28. The same is also true of the material in the vessel 12 of unit B, assuming of course that the volume of material in this receiver is maintained constant by continual drainage through pipe 30.

With reference to Figure 2, one of the units illustrated can be operated continuously in place of batch operation as described for Figures 1 and 3. For such operation it is desirable to introduce the liquid into an intermediate fractionating cup as indicated by lines 26 or 27 and to continually drain liquid from the lower vessel 12 to maintain a substantially constant liquid level.

The apparatus described above is capable of producing fractions of extreme purity. It can be used in connection with the distillation treatment or refining of a wide variety of products, including various mineral oils or petroleum products, and vegetable or animal oils. It can also be used to advantage to remove fractions such as vitamins or vitamin-like substances, from materials such as vegetable or animal oils.

I claim:

1. In high vacuum molecular distillation apparatus of the character described, a vertically disposed fractionating column of the reflux type, said column including a plurality of vertically spaced collecting cups arranged whereby a condensed fraction in one cup may reflux into the cup below the same, said cups surrounding a substantially unobstructed column like vertical path for free movement of vaporized molecules, means for introducing molecules of the vaporized material into the fractionating column, a jacket surrounding the column and having its upper end communicating with the upper end of said space above the uppermost cup of the column, and means for subjecting the fractionating column and said jacket to a high vacuum, the lower portion of said jacket serving to collect a fraction from a region above the uppermost cup of the column.

2. In high vacuum molecular distillation apparatus, a chamber serving to retain mother liquor undergoing distillation, a vertically disposed fractionating column comprising a plurality of vertically spaced cups arranged one above the other and each arranged for refluxing collected material into the next lower cup, said cups being formed to surround and freely communicate with a substantially unobstructed column like space of substantial cross sectional area extending upwardly from the surface of liquid undergoing treatment, means for subjecting the interior of the fractionating column to a relatively high vacuum, and a depending lip surrounding said space at a level intermediate the lowermost fractionating cup and the surface of the liquid, to prevent entrainment of mother liquor in the lowermost fractionating cup.

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