DISTILLATION UNDER VACUUM WITH LIQUID PROTECTIVE SEAL

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This invention relates to separation under vacuum of substances of different vapor pressures and more particularly refers to a liquid protective seal for use of vacuum distillation equipment.

In the distillation of substances having low vapor pressures, it is usually advantageous, particularly in the case of heat sensitive materials, to conduct the distillation under a partial vacuum. As is well known in the art, in plant operations, such partial vacuum is best created through the agency of high velocity steam jets.

A disadvantage of such distillation apparatus using steam jets to generate vacuum is that in the event of a sudden shut-down or failure of the steam-jets, condensed water from the steam jets and water from the intercondensers rushes rapidly into the evacuated distillation apparatus, thereby causing contamination of the contents of said apparatus with water. Water contamination is particularly undesirable when the material being rectified in the apparatus is sensitive to moisture. Such is the case, for example, in the distillation of organic isocyanates, acid halides, and anhydrides and the like. A different form of contamination in vacuum distillation operations although less frequent sometimes results when for example due to sudden increase in the volatility of the steam-jets, such as may occur after a momentary failure, rapid boiling of the contents of the reboiler may occur, such that some hot distillate may be carried over, past the receivers, into the steam jets. Interaction of distillate with the steam-jets is undesirable and hazardous where the distillate is known to react with water exothermically, for example in the distillation of tolylene diisocyanates. Such interaction of the distillate with the steam jets is equally undesirable where the distillate is known to react with water to form solid particles, for in such instances extremely hazardous conditions may develop due to plugging up of the steam paths.

Another object of our invention is to provide apparatus and method which will overcome these disadvantages. Another object is to effectively isolate the evacuation system, namely the steam-jets, from the remainder of the distillation apparatus, such that, in the event of a sudden failure of the steam-jets, contamination of the various parts of the distillation apparatus with condensed steam does not occur and also that in the event of a sudden over-loading of the receivers, interaction of the hot distillate with the steam-jets is avoided.

Our invention is best illustrated by reference to the accompanying view diagram which represents a vacuum distillation apparatus comprising our novel liquid protective seal.

Referring to the view diagram a typical distillation plant layout may comprise a fractionation column 1, a reboiler, which is not shown, a condenser 3, a liquid separator 6, a receiver 18, steam jet conduit 10 leading to steam jets, not shown, and means for connecting these various components so as to render the apparatus operable. Our liquid seal comprises the combination of chamber 8, tube 11, reservoir 12, overflow tank 23 and means for connecting these various components to each other and to a typical industrial distillation plant, as illustrated in the drawing.

In the actual operation of the distillation plant represented in the drawing, vapors of the substance to be purified by distillation pass from column 1 into condenser 3 through line 2. From condenser 3 distillate is withdrawn through lines 5 and 7 into receiver 18. Uncondensed vapors generally carried entrained droplets from the top of condenser 3 pass through line 4 into a liquid separator 6 desirably equipped with a mist separator 25 such as a screen or spaced brickwork, where the vapor velocity is reduced and any additional condensate collected in liquid separator 6, passes down through lines 19 and 7 into receiver 18 from the bottom of which distillate is discharged through line 14 and valve 26. Non-condensables from the top of liquid separator 6 pass through line 20 into chamber 8 desirably at a point remote from vapor conduit 10 and preferably at one side of interposing baffle 9. Reservoir 12 contains a volume of an inert liquid 21 which is immiscible with and whose density is less than water and desirably less than the substance being distilled. The volume of liquid 21 is such that when a partial vacuum is generated in 8 through the operation of steam-jets 10 and the liquid 21 rises in tube 11 to countervance the vacuum that is created, the bottom of tube 11 will remain completely submerged in liquid 21. Overflow line 13 is desirably situated so that the level of liquid 21 in reservoir 12 and in line 11 will be at the same height as line 13 when steam jets 10 are not in operation.

To illustrate the operation of our liquid protective seal, when the steam jets 10 are turned on, liquid 21 rises in tube 11 from an original level equal to the level of line 13 until the column of liquid in tube 11 counteracts the vacuum applied by steam jets 10. Minor fluctuations in the vacuum generated by steam jets 10 will be reflected in minor variations in the height of the column of liquid in tube 11. In the event of a sudden failure of steam jets 10, any water entering chamber 8 will be deflected by baffle 9 away from line 20 and the receiver, down through the column of liquid 21 present in tube 11 and into the bottom of reservoir 12, provided that, as already mentioned, liquid 21 is immiscible with and its density is less than that of water. Any water is accumulated in reservoir 12 it will overflow through line 13 into overflow tank 23, open to the atmosphere through line 27 from which it can be withdrawn through valve 22. Alternately, the water can be withdrawn from the bottom of reservoir 12, thru line 28 and valve 24. Thus, receiver 18, liquid separator 6 and other components of the distillation plant preceding or connecting receiver 18 and liquid separator 6 are protected against water contamination in the event of a sudden failure of steam jets 10.
Conversely, when overloading of condenser 3 and liquid separator 6 forces hot distillate of the product being purified, through line 20, the distillate will be deflected by baffle 9 through the column of liquid 21 present in tube 11 into the bottom of reservoir 12 from which it can be subsequently withdrawn thru line 13 and ultimately valve 22 or alternatively through valve 24.

It has already been specified that for the successful operation of the liquid protective seal of our invention, liquid 21 is one which is inert, immiscible with and of lower density than water. Another requirement is that the liquid have a very low vapor pressure such that contamination of the apparatus by vapors of the liquid in reservoir 12 is avoided and such that loss of the liquid through evaporation, at the distillation conditions employed, is negligible. Any liquid material inert to water, immiscible with water, of lower density than water and having a low vapor pressure, i.e., a boiling point of at least 200° C., preferably above 275° C. may be employed. Hydrocarbons such as alkyl benzenes, e.g. dodecylbenzene, mineral oils, and diphenyl have been found satisfactory.

From the foregoing description it will be evident to those skilled in the art that our novel liquid protective seal can be used with a wide variety of distillation equipment for the distillation of a large number of different substances. Our invention is intended to include all such variations and modifications as will be apparent to those skilled in the art and is not restricted to the typical distillation plant described herein.

We claim:

1. A vacuum distillation apparatus comprising a still and condenser means adapted in normal operation to condense substantially all desired condensables from the distillation and discharge therefrom substantially only non-condensables, a steam jet for producing a vacuum in said still and a liquid seal device separating the condenser means and steam jet, said liquid seal device comprising:

(a) a chamber connected to the steam jet and condenser means.
(b) a reservoir below said chamber containing an inert liquid of lower density than water and immiscible therewith.
(c) a tube communicating with said reservoir and chamber and terminating below the surface of the liquid in said reservoir so that in vacuum operation liquid can rise in said tube according to the applied vacuum and a baffle in said chamber adapted to deflect liquid impinging thereon downwardly and into said reservoir.

2. Apparatus as claimed in claim 1 having an overflow tank communicating with said reservoir for the discharge of water collecting in the reservoir to discharge in said overflow tank.

3. Apparatus as claimed in claim 1 wherein said inert liquid in said reservoir is a hydrocarbon having a boiling point above 200° C.

4. Apparatus as claimed in claim 3 wherein said inert liquid is dodecylbenzene.

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