

Reactor Boiler and Auxiliaries - Course 133

THE DISTILLATION UPGRADING PROCESS

Water distillation for D₂O upgrading is based on the difference in boiling points of D₂O and H₂O. Generally, the magnitude of this temperature difference is a measure of the difficulty of separating the two components, and in this case the difference (1.4°C) is small so that the difficulty of separation is appreciable.

Mechanism of Operation

A D₂O-H₂O mixture contains D₂O of boiling point 101.4°C and H₂O of boiling point 100°C at atmospheric pressure. If sufficient heat is added to such a mixture, the H₂O-component will boil first, and any vapor leaving the mixture will contain a slightly increased proportion of H₂O. The mixture left behind will then contain a slightly increased proportion of D₂O. In this case, the mixture left behind will ultimately become "reactor grade" D₂O (and will remain at the bottom of the column) and the mixture leaving as vapor will ultimately become waste product (and will be drawn off at the top of the column).

Several stages connected in series and assembled in a vertical cylinder form a cascade which is known as a distillation column. Heating of the D₂O-H₂O mixture takes place at the bottom of the column in a reboiler. Vapor rises through the column, is condensed at the top of the column, and descends through the column as liquid while other vapor rises and comes in contact with it. The descending liquid, already slightly depleted in D₂O, tends to pick up higher boiling point component (D₂O) from the vapour, while at the same time giving up lower boiling point component (H₂O) to the rising vapor. The internals of the column are designed to promote thorough and efficient mixing of the liquid and vapor phases.

The fluid obtained by condensing the overhead vapor (called distillate) is depleted in D₂O content from that of the original mixture. This distillate may be either partially or wholly drawn off the top of the column or it may be allowed to descend the column by gravity, to contact the vapor rising up the column. The portion descending is called the reflux (Fig. 1). When all the distillate is allowed to descend, the column is operating on total reflux.

The distillate produced during D_2O upgrading using a distillation column contains proportionately more light water than the original D_2O-H_2O mixture. Unless the D_2O content is 2% by weight or less, it is uneconomical to discard it; rather, it is cheaper to store the distillate and upgrade it at some later time. During operation of the column, the portion of the distillate not recycled as reflux is "tapped" or "drawn" off, and although of lower grade D_2O than the column feed, must be saved for later re-processing.

Since γ increases as the pressure at which distillation is done is reduced, a vacuum distillation unit is used for upgrading heavy water. The vacuum, usually about 0.25 atmos, is limited due to the large vapor volumes that would result at pressures much below this point.

Types of Columns Used

The columns presently in use for D_2O upgrading are of two types, classified according to the internals of the column:

1. Sieve-plate columns.
2. Packed columns.

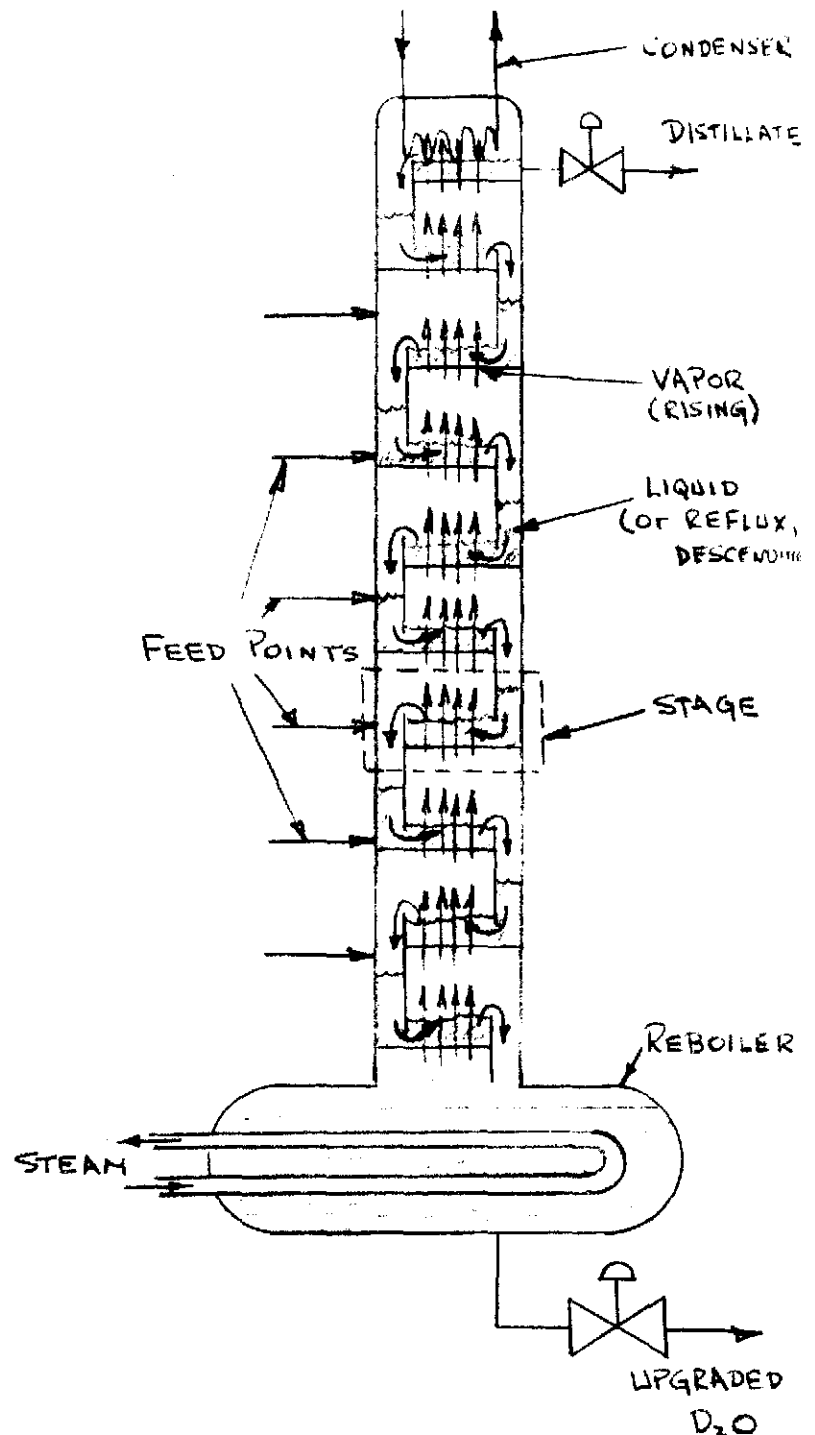


Fig. 1

Sieve-plate Columns

The internals of a sieve-plate column consist of a series of horizontal perforated plates through which rising vapor can pass, together with a weir arrangement to allow the liquid to descend in the column (Fig. 2). Repeated contacting of liquid and vapor is thus promoted as the vapor rises and the liquid descends in the column. The vapor leaving a given plate is slightly more enriched in H_2O ; the liquid, slightly more enriched in D_2O . The column at NRU is a sieve-plate type, designed and built by Canadian Badger Ltd.

Generally speaking, it is now usually possible to obtain more efficient vapor-liquid mixing with a packed column, resulting in increased separating ability for a given size of column. Sieve-plate columns on the other hand have a comparatively low first cost. However, to accommodate the upgrading needs of present-day nuclear installations, a sieve-plate column becomes impractical due to the large number of stages and hence size of column required.

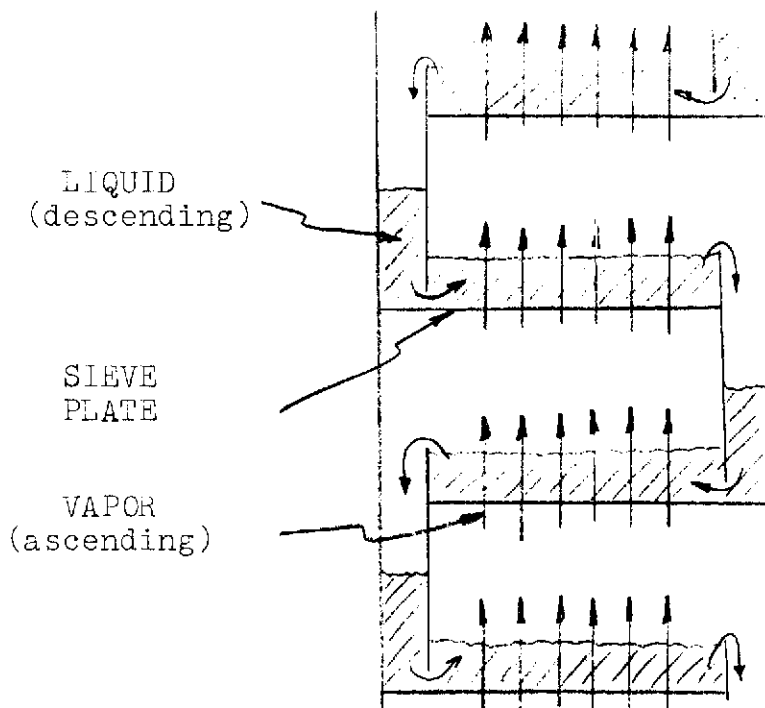
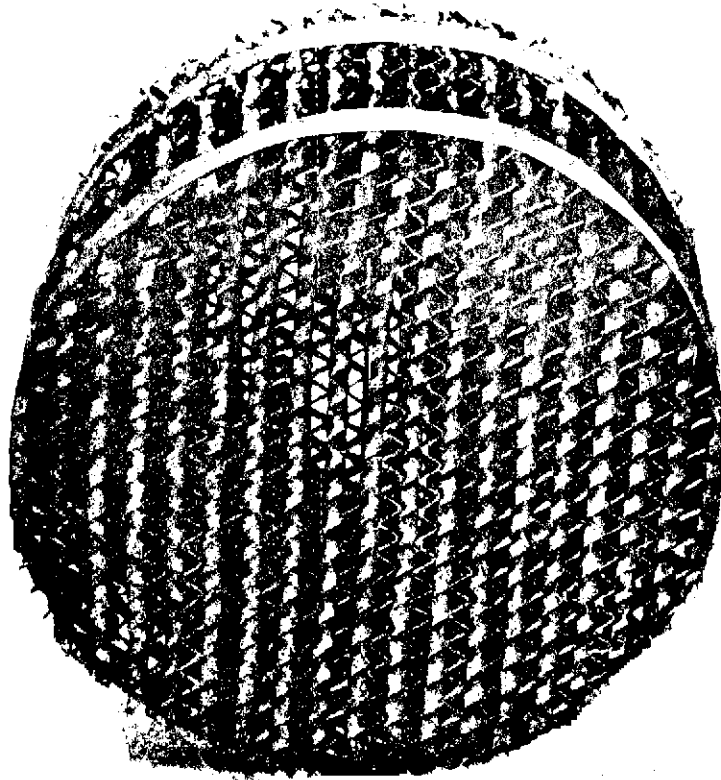


Fig. 2

Packed Columns

The internals of a packed column are not arranged in a series-connected cascade of stages like those of a sieve-plate column; rather the internals consist of a packing, usually in the form of a metallic gauze throughout the length of the column. An equivalent number of stages is then calculated, based on the separating ability of the column. Fig. 3 shows the type of gauze pellet used in the Douglas Point packed column. Provision must be made along the column for the addition of feed, and uniform spreading of the feed across the column at the particular elevation(s) chosen.

Depending on the particular application, the equivalent of between 150 and 500 stages are needed for an H_2O - D_2O separation. By comparison, columns employed in chemical plants seldom contain



Sulzer Packing

This "pellet" is a few centimetres in diameter. These gauze "pellets" are packed into thin tubes, which run the length of the column, with provision for the addition of feed. The tubes are assembled into a cluster of about the same inside diameter as the column. Each Douglas Point column is about 25 centimetres in diameter.

Fig. 3

more than 100 stages. Working at reduced pressures proves advantageous as with the sieve-plate column, since the single stage η increases with falling pressure.

The following comparison serves to emphasize further advantages of the packed column:

	<u>Packed Column</u>	<u>Plate Column</u>
Stages/m	10 - 60	1 - 10
Pressure loss (mm Hg/stage)	0.2 - 0.5	2 - 10
Hold-up (ℓ/m^2 of stage)	3 - 8	10 - 50
Starting-up time (days)	3 - 4	7 - 8

In a packed column, the gauze "pellets" are arranged in long thin-walled tubes as described under Fig. 3. An important feature of most packed columns is that they usually consist of two or more towers, connected in parallel to the reboiler at the bottom only; each tower has its own condenser at the top. The reason for doing this is to prevent the individual towers from "competing" with one another, and can best be appreciated by considering the column during operation on total reflux (ie, when no product is being withdrawn). In this instance, the quantity of vapor ascending through any cross section is equal to the quantity of liquid descending. This applies to towers in parallel at the bottom only, even though the quantities may differ slightly from one tube to another, since phase reversal takes place at the top of the tube. However, if the columns are connected in parallel at the top also, the separation is quite adversely affected, since a net flow up one tower and down another can easily result, reducing the reflux ratio. Optimum operation for an H_2O - D_2O separation will result at very high reflux ratios.

A simplified flow diagram of a typical upgrading unit showing the main components which would be used on-site at a nuclear station is shown in Fig. 4. Most of the auxiliary equipment is labelled in the diagram; note the use of separate condensers for each tower, connected in parallel at the reboiler end only. Packing in the towers is indicated by double cross-hatching. Top and bottom product purities are measured by a density controller, operating on the principle of density difference between H_2O and D_2O .

H_2O - D_2O distillation columns are now being built having a large diameter with as many as 40 - 50 towers connected to a single reboiler, each with its own condenser; the over-all diameter of the towers assembled into a single column may measure as much as 2 - 3 metres. The design and separating ability of a given installation will depend on the specified duties of the unit.

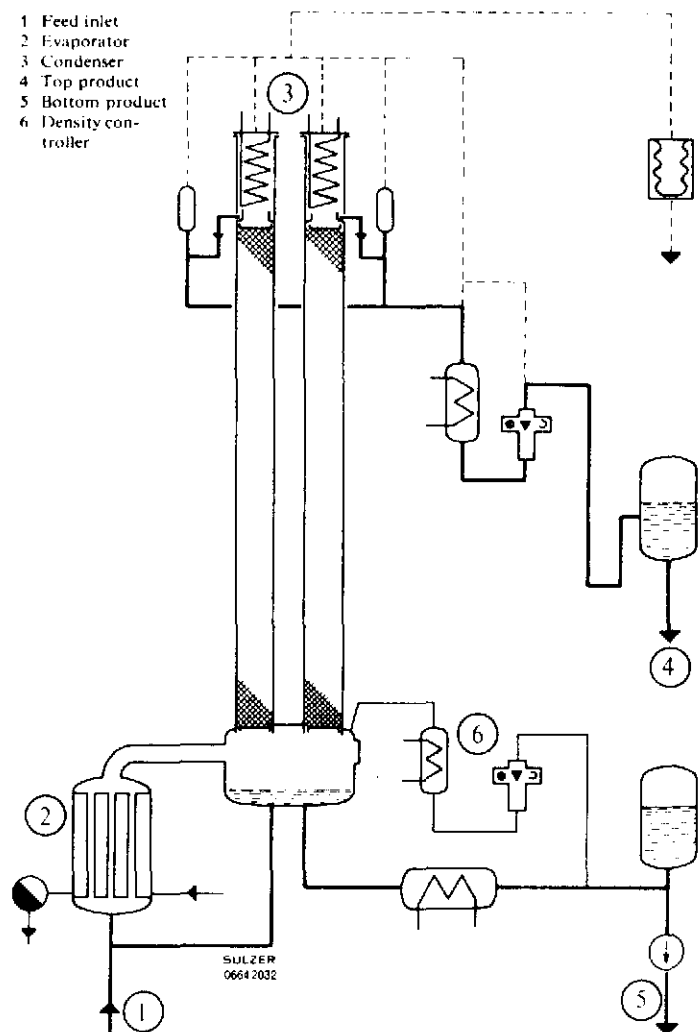


Fig. 4

Method of Operation

There are two ways in which a heavy water upgrading distillation unit may be used. While a particular column may be designed primarily for one mode of operation it is desirable, for upgrading work, that it be flexible enough to work in both ways. The first mode of operation is called "stripping". This technique removes or strips light water from the incoming feed without appreciably raising the D₂O concentration of the feed. In this type of operation the column vapor flow or boiling rate is essentially the same as the feed rate. The second method of operation is called "enriching". In this case downgraded heavy water is fed to the distillation column at a much lower rate than the column vapor flow or boil-up rate. The heavy water flows downward with the column reflux while the light water is carried off in the vapor rising up the column. The heavy water thus becomes enriched or upgraded as it flows to the bottom of the column.

In both cases the high grade product is removed from the bottom of the column while the light water is removed in a portion of the condensed overhead vapor or distillate. In the stripping operation the product is reactor grade heavy water while in the enriching operation the product may either be reactor or may be a lower grade depending on D₂O content of the feed and on the characteristics of the column. In both cases, of course, the product flow rate is the feed flow rate less the quantity of water that is collected as distillate.

The distillation column must operate continuously in order to achieve the best possible performance. The best separation of components, in this case light and heavy water, is only obtained if the column operates "at equilibrium". This is a steady state condition where the values of D₂O concentrations, from the bottom of the column through the many stages to the overhead vapor, remain steady. A period of 2 to 8 days is required for a heavy water distillation column to reach equilibrium conditions after it has been started up. For the start-up period, the column is operated on total reflux; once equilibrium has been achieved, feed to the column is started. Most packed columns require "flooding" before operation at total reflux commences, to allow proper wetting of the gauze.

In order to increase the flexibility of the distillation column for upgrading work, facilities can be provided to permit the column to work on what may be called a "semi-batch" operation. This uses fairly large batches of feed of a wide range of D₂O concentrations and requires the column to come to a new equilibrium condition for each batch, although the operation is continuous while working on a batch. The column may be operated either as in stripping or as in enriching. The increased flexibility is, of course, obtained at the expense of some capacity since time is required to bring the column to equilibrium for each batch of feed.

ASSIGNMENT

1. Explain how an H_2O - D_2O separation takes place in a distillation column. Is it an easy separation? Why?
2. What is meant by total reflux and when is this mode of operation used in practice?
3. How are distillation columns classified and what types are currently in use for D_2O upgrading? Which is generally better suited to D_2O upgrading and why?
4. How does an "enriching" operation differ from a "stripping" operation?

R. Popple