Chemistry - PI 24

FRACTIONAL DISTILLATION

OBJECTIVES:

1. Given Figure 1 from the text; use it to verbally explain to the satisfaction of a colleague (or the course manager if requested) the effect of heating a mixture of D_2O/H_2O liquid to its boiling point and of cooling a mixture of D_2O/H_2O vapour to its condensation point.

2. Verbally describe how the process of fractional distillation produces reactor grade D_2O in a BHWP finishing unit or a station upgrader. (Choice of above to coincide with trainee's deployment). The description should satisfy a colleague (or the course manager if required).

When water leaves the enriching portion of the Girdler-Sulphide process, it is between 10 and 30% D_2O. (This concentration is varied to obtain the optimum match between the enriching and finishing units.) The finishing units at BHWP use fractional distillation in towers using **sieve trays** to achieve intimate mixing of the rising vapours and descending **reflux.**

Distillation is the separation of the constituents of a liquid mixture by partial vapourization of the mixture and separate recovery of vapour and residue. The more volatile constituents of the original mixture are obtained in increased concentration in the vapour, the less volatile in greater concentration in the liquid residue. Completeness of separation depends upon certain properties of the components involved and upon arrangement of the distillation process.

Fractional Distillation is a distillation in which a vapour is continuously and countercurrently contacted with a condensed portion of the vapour. This process secures a greater enrichment of the vapour in the more volatile components than could be secured with a single distillation operation using the same amount of heat. The condensate returned to accomplish this object is termed reflux.

The generally used devices in which vapours from a still on their way to a condenser can flow countercurrently to a portion of the condensate returned as reflux are called rectifying columns or towers. Rectifying columns are most commonly fed at or near the center of the column, in which case the section above the feed is known as the rectifying section, while the part below the feed is the stripping section.

Vapour-Liquid Relationships

For a two component system, eg, heavy water/light water, one is able to develop a diagram, see Figure 1 which represents boiling point versus liquid composition. The diagram also shows compositions of "first vapours" at the point of boiling.

The points to remember as you study this diagram are:

1. For any mixture of D_2O and H_2O, the boiling point is below the weighted average of the two boiling points due to the presence of the more volatile H_2O.

2. The "first vapours" are richer in H_2O the more volatile component (but still contain significant quantities of D_2O).

3. The liquid becomes richer in D_2O due to the loss of H_2O. Eventually the "pot" contains reactor grade D_2O.

Figure 1 represents the boiling point and equilibrium composition relationships, at constant pressure, of all mixtures of liquid A (boiling point t_a) and liquid B (boiling point t_b).

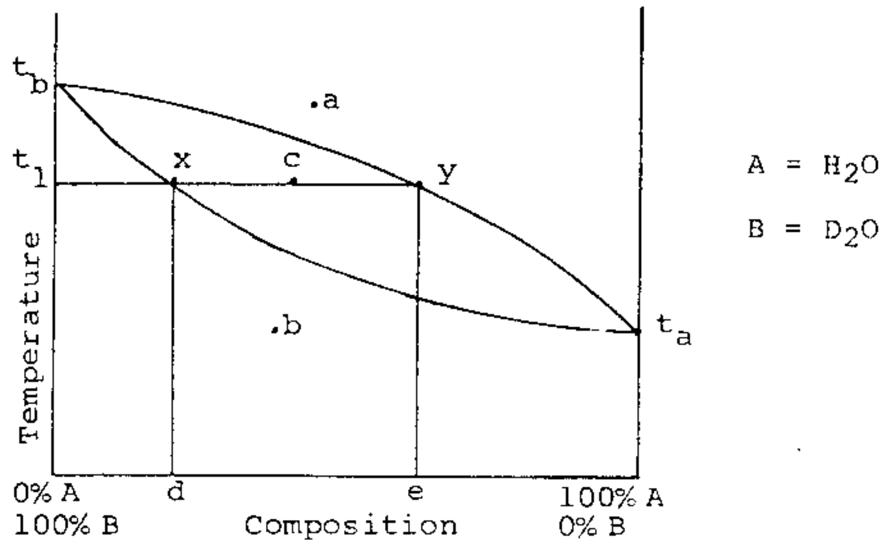

Figure 1

Boiling Point Diagram

Any point (such as point x on the lower curve) has for its abscissa, the composition of liquid that will just begin to boil at temperature t_1, giving vapour of composition(e).

Any point (such as point y on the upper curve) has for its abscissa, the composition of vapour (e) that will just begin to condense at the temperature given by its temperature (t_1) and will give liquid of composition (d).

Any two points on the same horizontal line (such as x and y) represent the composition of liquid and vapour in equilibrium with each other at the temperature given by the horizontal line through them. For all points above the top curve, such as point (a), the mixture is entirely vapour. For all points below the bottom curve, such as point (b), the mixture is completely liquified. For points between the two curves, such as point (c), the system consists partly of liquid and partly of vapour.

Suppose that a liquid mixture of composition (d) is heated slowly. It will begin to boil at t_1. The first vapour produced will have a definite composition, represented on the diagram by (e). As soon as an appreciable amount of vapour has been formed, the composition of the liquid will no longer correspond to (d), since the vapour is richer in the more volatile component than the liquid from which it was evolved and hence the point (x) tends to move toward (t_b) as the liquid is enriched in D_2O and the boiling point of the mixture tends to move towards the boiling point of pure D_2O.

The boiling point diagram must, in general, be determined experimentally. It will vary with the total pressure. Changes in pressure, though, merely move the whole diagram up or down but do not greatly affect the relation between the vapour and liquid curves. The experimental determination of such curves will not be described here.

In the process of reflux, steam rises from the stillpot, encounters the packing, condenses, then descends countercurrently through the rising vapours (this descending liquid itself is often called reflux). Since the rising vapours are slightly warmer than the falling drops, heat exchange occurs, causing each drop slowly but continuously to re-volatilize (flash off). The "2nd generation" steam is slightly richer in the lower boiling liquid. By the same token, the drops as they descend, become richer in the higher boiling liquid. Thus H_2O tends toward the upper portion of the tower, and D_2O to the lower. The tower has the effect of performing an almost infinite number of micro-distillations. It is important to realize that the separation is gradual rather than sharp, and has nothing to do with difference in density of the waters.

The packing in our station upgrading and BHWP Finishing Unit towers is either sieve trays (stainless steel baffle plates with holes in them), or a kind of gauze made of bronze.

FURTHER READING:

COURSE 438: Heavy Water Production and Management

LESSONS: 438.30-1
438.30-4

PRACTICE EXERCISES:

1. Explain Figure 1 in the text to a colleague in terms of:

   (a) The effect on a mixture of D_2O/H_2O liquid that is heated just to its boiling point.

   (b) The effect on a mixture of D_2O/H_2O vapour that is cooled to its first condensation.

2. To a different colleague explain:

   (a) How a Sulzer Upgrader uses fractional distillation to produce reactor grade heavy water.

   OR (b) How a Lummus sieve tray tower at BHWP "finishes" the feed from a Girdler-Sulphide process to produce reactor grade heavy water.

   Your choice of (a) or (b) should reflect your deployment.

P. Dodgson
P. Morovan