

Plant Design and Economics
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Lecture No -40
Introduction to Sequencing of Ordinary Distillation Columns

Welcome to lecture 40 of plant design and economics. Now, you have seen in a binary distillation column that a mixture of say components A and B can be separated into A and B with very high degree of purity provided of course their relative volatility supports this. Now when you have multi component mixtures, let us say we have 3 components A B and C. Well it was possible to separate the binary mixture into pure fractions of A and B, almost pure fractions of A and B, in a single ordinary distillation column.

It is no more possible to separate a mixture of 3 components using a single ordinary distillation column. Note that we are not talking about say divided column where a single distillation column may be enough to take out pure A B and C. Using ordinary distillation column to separate a 3 component mixture will require at least 2 distillation columns. Now, we have now a choice in the order in which the products are separated. So do I separate A from B and C in the first distillation column or do I separate C from E and B in the first distillation column.

So these leads to distillation column sequencing. So in today's lecture we will start with an introduction to the sequencing of ordinary distillation column.

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Distillation for Multi-component Mixture

Multi-component mixtures are often separated into more than two products in a sequence of two-product separators.

For nearly ideal feeds (hydrocarbon mixtures, alcohols) the most economical sequence will often include only ordinary distillation columns provided that the following conditions hold:

1. The relative volatility between the two selected key components for the separation in each column is greater than 1.05.
2. The reboiler duty is not excessive. Example: If light key component is water, it has a very high heat of vaporization.

NPTEL

Multi component mixtures are often separated into more than 2 products in a sequence of 2 product separators, such as 2 product distillation columns. For nearly ideal feeds such as hydrocarbon mixtures or alcohols the most economical sequence will often include only ordinary distillation columns provided certain conditions are met. So let us look at what are those conditions?

The relative volatility between the 2 selected key components for the separation in each column is greater than 1.05. Note that when the relative volatility is below 1.05, the separation through distillation column generally becomes impractical. The reboiler duty is not excessive. An example where the reboiler heat can be excessive is when the light key component is water. Note that it has very high heat of vaporization.

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Distillation for Multi-component Mixture

3. The tower pressure does not cause the mixture to approach its critical temperature.
4. The overhead vapour can be at least partially condensed at the column pressure to provide reflux without excessive refrigeration requirements.
5. The bottoms temperature at the tower pressure is not so high that chemical decomposition occurs.
6. Azeotropes do not prevent the desired separation.
7. Column pressure drop is tolerable, particularly if operation is under vacuum.

NPTEL

Dr. Manoj Kumar

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The tower pressure does not cause the mixture to approach its critical temperature. The overhead vapour can be at least partially condensed at the column pressure to provide reflux without excessive refrigeration requirement. Note that the refrigeration is expensive. So our column pressure should be such that the vapour that is coming out from the top of the distillation column can at least be partially condensed so that we get the reflux flow.

The bottoms temperature at the tower pressure is not so high that chemical decomposition occurs. Azeotropes do not prevent the desired separation. Column pressure drop is tolerable particularly if the operation is under vacuum. So when these conditions are met the separation through a sequence of ordinary discussion column becomes economic.

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Distillation Sequencing Using Simple Columns

Consider: A homogeneous multi-component fluid mixture needs to be separated into a number of products, rather than just two products.

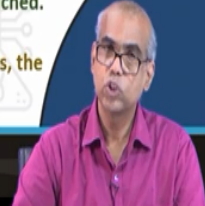
First consider a sequence of ordinary distillation columns, where a single feed is sent to each column and the products from each column are: the distillate and the bottoms.

If a mixture contains N components and we want to separate it into pure components, then $(N - 1)$ columns are required. Because each component can be removed in order of volatility until the final binary pair is reached.

There is a choice of order in which the products are separated that is, the choice of *distillation sequence*.



Dr. K. Raghav



Consider a homogeneous multi component fluid mixture that needs to be separated into a number of products rather than just 2 products. First, we consider a sequence of ordinary distillation columns where a single feed is sent to each column and the products from each column are just 2; one distillate stream and the bottom stream. So we are considering a sequence of ordinary distillation columns where a single feed is sent to each column in the sequence and the products from each column are just 2, number of products are just 2; the distillate and the bottoms.

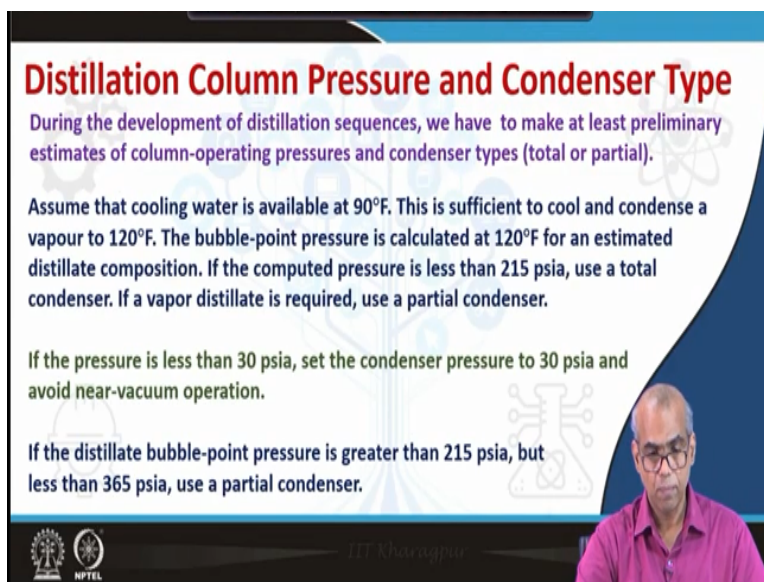
If a mixture contains n components and we want to separate it into pure components then n minus 1 columns are required. For 3 components, we have seen that we need 2 distillation columns, a sequence of 2 distillation columns. In the first column, let us say I can separate A and in the second column I can separate B and C. You can also separate C in the first column and A and B in the second column.

So when a mixture contains n components and you want to separate it into pure components, then n minus 1 columns are required. This is because each component can be removed in order of volatility. Let us say the volatility decreases as we move from A to B to C, then I separate may be A in the first column and then B and C in the second column like that. So each component can be removed in order of volatility.

And then when final pair is reached that can be separated in the last column. For example, when

I separate A B C, component A is separated in the first column. But now the final pair is reached B and C. So they can be separated in one column only. So 1 plus 1, 2 columns are required. So for n component mixtures, I need n minus 1 columns to separate them into pure components. Now when you have several such components, then we have a choice of order in which the products are separated, that is the choice of the distillation sequence.

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Distillation Column Pressure and Condenser Type

During the development of distillation sequences, we have to make at least preliminary estimates of column-operating pressures and condenser types (total or partial).

Assume that cooling water is available at 90°F. This is sufficient to cool and condense a vapour to 120°F. The bubble-point pressure is calculated at 120°F for an estimated distillate composition. If the computed pressure is less than 215 psia, use a total condenser. If a vapor distillate is required, use a partial condenser.

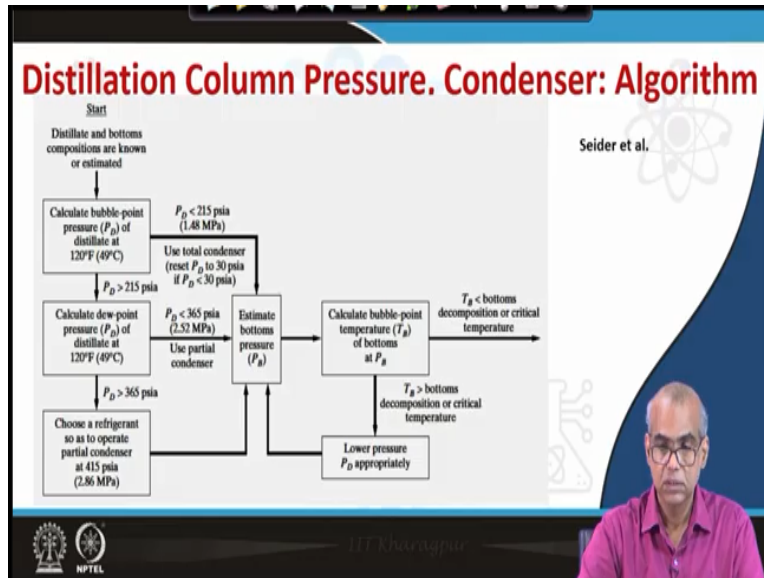
If the pressure is less than 30 psia, set the condenser pressure to 30 psia and avoid near-vacuum operation.

If the distillate bubble-point pressure is greater than 215 psia, but less than 365 psia, use a partial condenser.

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So in today's lecture we will see some introductory concepts of sequence of distillation columns using ordinary distillation columns. Now before the development of distillation sequence or during the development of distillation sequence we have to make at least some preliminary estimates of column operating pressure as well as condenser types, whether we require a total condenser or whether we require a partial condenser.

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Now you can use an algorithm provided by Seider to determine the condenser types. This algorithm, basically states as follows: assume that cooling water is available at 90 degree Fahrenheit. If the cooling water is available and 90 degree Fahrenheit, this is sufficient to cool and condense a vapour to 120 degree Fahrenheit. The bubble point pressure is calculated at 120 degree Fahrenheit for an estimated distillation composition.

If the computed pressure is less than 215 PSI absolute, it is recommended that you use a total condenser. However, if a vapour distillate is required in that case we use a partial condenser. So you first assume that cooling water is available at 90 degree Fahrenheit which is sufficient to cool and condense a vapour to 120 degree Fahrenheit. You estimate a distillate compositions and the bubble point pressure is calculated at 120 degree Fahrenheit.

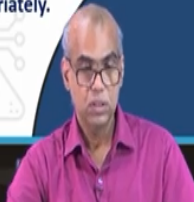
If the computed pressure is less than 215 psi absolute, use a total condenser. If a vapour displace is required use a partial condenser. If the pressure is less than 30 psia, set the condenser pressure to 30 psia and avoid near vacuum operation. If the distillate bubble point pressure is greater than 215 psia, but less than 365 psia, use a partial condenser. So if the distillate bubble point pressure is between 215 to 365 psi absolute, a partial condenser is recommended.

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Distillation Column Pressure and Condenser Type

If the distillate bubble-point pressure is greater than 365 psia, determine the dew-point pressure for the distillate as a vapour. If the pressure is greater than 365 psia, operate the condenser at 415 psia with a suitable refrigerant in place of cooling water.

For the selected condenser pressure, add 10 psia to estimate the bottoms pressure (for nonvacuum duty, acceptable column pressure drops are in the range 0.05–0.15 psi/tray), and compute the bubble-point temperature for an estimated bottoms composition. If that temperature exceeds the decomposition or critical temperature of the bottoms, reduce the condenser pressure appropriately.



If the distillate bubble point pressure is greater than 365 psi absolute, determine the dew point pressure for the distillate as a vapour. If the pressure is greater than 365 psi absolute, operate the condenser at 415 psi absolute with the suitable refrigerant in place of cooling water. For the selected condenser pressure add 10 psi absolute to estimate the bottoms pressure. And compute the bubble point temperature for an estimated bottoms composition.

If that temperature exceeds the decomposition or critical temperature of the bottoms, reduce the condenser pressure appropriately. So this were the statement from this algorithm. You can make use of this algorithm to decide what type of condenser will be used for the distillation column.

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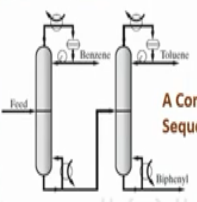
Sequences of Ordinary Distillation Columns

Simple columns employ:


- One feed split into two products
- Key components adjacent in volatility, or any components that exist in small quantities between the keys will become impurities in the products
- A reboiler and a condenser

Consider separation of:

	Benzene	Toluene	Biphenyl
Normal Boiling Point (°C)	80.1	110.8	254.9



Another Sequence: Take Biphenyl as Bottoms in Column-1



Now when I say that, let us first consider the sequence of ordinary distillation column. What I mean by ordinary distillation columns are as follows. So these ordinary distillation columns or simple columns employ 1 feed split into 2 products. Key components adjacent in volatility so that sub separation is possible or any components that exist in small quantities between the keys will become impurities in the products.

Also these simple columns or ordinary distillation columns has a reboiler and a condenser. Now consider the separation of a 3 component mixture, the simplest multi component mixture is a 3 component mixture. And let us consider a mixture of benzene, toluene and biphenyl. The normal boiling points are listed as 80.1 degree Celsius for benzene, 110.8 degree Celsius as toluene and 254.9 degree Celsius for biphenyl.

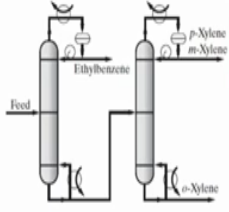
You can see that the normal boiling points of benzene, toluene and biphenyl are widely separated. So it is possible to separate them into pure fractions or almost pure fractions using ordinary distillation columns or sequence of distillation column. So 1 common sequence is shown. So if you look at the normal boiling points for these components, you see that benzene is most volatile, then comes toluene and biphenyl is the least volatile.

So one common sequence may be in the first distillation column you separate the most volatile component benzene as top product. So the bottoms which is a mixture of toluene and biphenyl goes to column number 2 where they are separated into toluene as top product and biphenyl as bottom product. So this is a common sequence. We can also think of another sequence where we can take the least volatile the biphenyl as a bottom product in column 1.

Then the mixture of benzene and toluene from the top goes to column number 2 where this is separated into benzene and toluene. Benzene will come as top product of column 2 and toluene will come as bottom product of column 2. So this is another sequence. Both the sequence are possible.


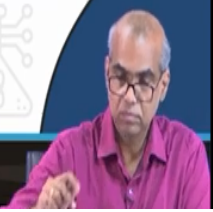
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Nearly Pure Products: Not Always Possible



The para and meta isomers are not separated because the normal boiling points of these two compounds differ by even $< 1\text{ }^{\circ}\text{C}$, making separation by distillation impractical.

	Ethylbenzene	o-xylene	m-xylene	p-xylene
Normal Boiling Point ($^{\circ}\text{C}$)	136	144	139	138.3

Now in the previous case benzene, toluene and biphenyl we could separate them into pure fractions using ordinary distillation column. But it is may not be possible to obtain pure products using ordinary distillation column always. Consider the case of separation of ethylbenzene, ortho-xylene, meta-xylene and para-xylene. Note that the boiling border, the boiling points between meta-xylene and para-xylene are very, very low, less than 1 degree Celsius.

Among these ethylbenzene is most volatile. So if I consider a sequence as follows: ethylbenzene is taken out as the top product in the first column. And then the bottoms goes to column 2 where the mixture of ortho-xylene and meta-xylene, sorry the mixture of meta-xylene and para-xylene which are very close boiling comes as top product and ortho-xylene which has higher boiling point compared to meta-xylene or para-xylene come as bottom product.

Now further separation of para-xylene and meta-xylene is in distillation column or ordinary distillation column is not practical because of very, very low difference in the boiling points between these 2 components.

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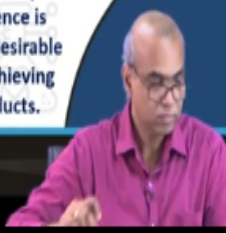
Direct/Indirect Sequences

(a)

(b)

Direct Sequence: Here all final products but one are distillates. It is widely used in industry because distillate final products are more free of impurities such as objectionable high-boiling compounds and solids.

Indirect Sequence: Here all products except one are bottoms products. This sequence is generally considered to be the least desirable sequence because of difficulties in achieving purity specifications for bottoms products.



Now, we will define two terms indirect sequence and direct sequence. So these are linked to the order in which the products are separated. For example, in a mixture with 3 components, as you have seen we can separate component A in the first column and B and C in the second column or we can also separate C in the first column and A and B in the bottom, A and B in the second column.

Now, the direct sequence is defined as the sequence where all the final products but one are taken as distillates. So in a direct sequence all the final products except one will be taken out as distillates. So note here, my products are A, B and C, only C is bottom product, both A and B are top product. So this is an example of direct sequence. So in case of direct sequence all final products except one are distillates.

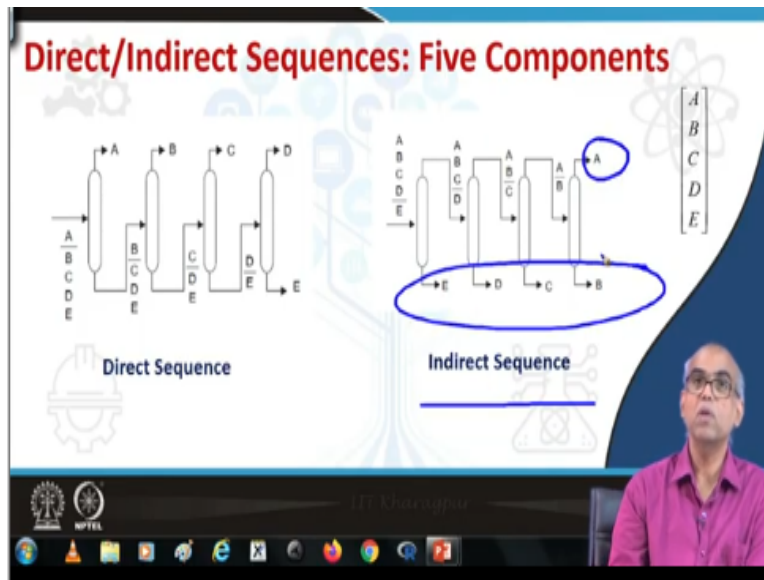
It is widely used in industry because distillate final products are more free of impurities such as objectionable high boiling components and solids. This is in comparison to bottom products. So distillate final products are always preferred in industry because they are relatively free of impurities when you compare to bottom final products. Now indirect sequence are defined as the sequence where all products except one are bottom products.

Note here where you take out C as bottom in the first, A and B in the second column. So only one is top product, both B and C products are bottom products. So this is an example of indirect

sequence. So in case of indirect sequence all the products, except one are bottom products. This sequence is generally considered to be the list desirable sequence because of difficulties in achieving purity specifications for bottom products.

So an indirect sequence is generally considered to be less desirable because of difficulties in achieving purity specifications of bottom products.

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So let us another example of direct sequence using 5 components A,B,C,D,E. So you have 5 components and let us say that components are arranged according to the relative volatility. So this is the, A is most volatile and E is the least volatile. So in case of direct sequence, all the products A,B,C,D except E are top products, only E is bottom product. Note, in the first column we make a separation here, so A is taken out.

In the second we make a separation here, so B is taken out and so on and so forth. Similarly, look at the indirect sequence where except A all are bottom products. So, this is an example of indirect sequence.

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Number of Sequences of Ordinary Distillation Columns

	Benzene (A)	Toluene (B)	Biphenyl (C)
Normal Boiling Point (°C)	80.1	110.8	254.9

Here, can we separate the mixture into toluene and a multi-component product of benzene and biphenyl? A
B

The separation of toluene from benzene and biphenyl by ordinary distillation in the first column is impossible because toluene is intermediate in volatility.

With ordinary distillation, first separate benzene, toluene, and biphenyl and then blend the benzene and biphenyl.

ONLY TWO SEQUENCES ARE POSSIBLE

Separation of A, B, and C

We have seen, in case of Benzene, toluene and biphenyl mixtures that they can be separated into benzene, toluene and biphenyl using 2 ordinary distillation columns and 2 different sequences are possible. So in the first case, the benzene is separated in the first column and toluene and biphenyl are separated in the second column. In the second sequence, the biphenyl is separated in the first column and benzene and toluene are separated in the second column.

Now, can we separate the mixture into toluene and a multi-component product of benzene and biphenyl? So is it possible for me using these ordinary distillation columns to separate a mixture of biphenyl, toluene and benzene into toluene and benzene plus biphenyl? It will not be possible. The separation of toluene from benzene and biphenyl by ordinary distillation column in the first column is not possible because toluene has intermediate volatility.

Note that the normal boiling point of toluene is in between that of benzene and biphenyl. So with ordinary distillation if we want to have toluene and a mixture of biphenyl plus benzene, what we have to do is, using any of the sequence we have to separate benzene, toluene and biphenyl and then can blend benzene and biphenyl if that is so required. So what we conclude from 3 component mixtures that only 2 sequences are possible.

In the first sequence, we separate A in the first column and B and C in the second column. In the another sequence, we separate C in the first column and B and C, sorry A and B in the second

column. So we have mixture A, B and C either we make a split here in the first column or we make a split here in the first column. So only two sequences are possible.

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So separation of 2 component feed, only 2 sequences are possible.

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What, when you have 4 component mixture? So we have 4 component mixture A, B and C, D. Here we have 5 different sequences. Note one direct sequence, one indirect sequence and then another three mix sequence. So in the direct sequence, I take out A, B, C as top products in the three columns and D as bottom product. Note that I have 4 components, so the distillation column that will be required, number of distillation column that will be required is 3, $4-1$ is 3.

Sequence that are possible is 5. So 2 sequences as direct sequence and indirect sequence, another 3 as mix sequence. So these all sequences are obtained by taking the separation points at different locations in the list of relative volatility or the list of the components A, B, C, D arrange in the order of volatility.

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Number of Sequences of Ordinary Distillation Columns

The diagram illustrates the possible sequences for a four-component mixture (A, B, C, D) in order of decreasing volatility. The root node is 'ABCD'. It branches into two main categories: 'Direct Sequence' and 'Indirect Sequence'.
 - Under 'Direct Sequence', there are two branches: 'BCD' leading to 'CD' and 'BCD' leading to 'BC'.
 - Under 'Indirect Sequence', there are two branches: 'ABC' leading to 'BC' and 'ABC' leading to 'AB'.
 The total number of sequences shown is 5.

Now, this can also be graphically representative, pictorially represented as follows.

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Number of Sequences of Ordinary Distillation Columns

What should be the number of different sequences of ordinary distillation columns (N_s) to produce P number of products from a multi-component mixture?

For the first separator in the sequence, $(P - 1)$ separation points are possible. **Example:** If the desired products are A, B, C, D, and E in order of decreasing volatility, then the possible separation points are $5 - 1 = 4$. A-B, B-C, C-D, and D-E.

Now let j be the number of final products that must be developed from the distillate of the first column. For example, if the separation point in the first column is C-D, then $j = 3$ (e.g. A, B, C).

Then $(P - j)$ equals the number of final products that must be developed from the bottoms of the first column.

A
B
C
D
E

Now, how do I compute the number of different sequences of ordinary distillation columns that is possible to produce P number of products from a multi component mixture? So I have been

given a multi component mixtures with P components, what should be the number of different sequences of ordinary distillation column to produce P number of products? We have seen for 3 components, there are 2 sequences. For 4 components, there are 5 sequences.

So in general when there are P number of products P component mixtures, what should be the number of sequences? For the first separator in the sequence, P-1 separation points are possible. For example, if I have 5 components A, B, C, D, E, then the possible separation points are 1, 2, 3 and 4, that means the number of components minus 1. So AB, BC, CD and D. So in the first distillation column, it is possible to have P-1 separation points.

Now, let J be the number of final products that must be developed from the distillate of the first column. So what you say is that J be the number of final products that must be obtained from the distillate of the first column. So for example, if the separation point in the example is between C and D, then J is 3, A, B and C. So, what should be the number of final products that must be developed from the bottoms? It should be P-J.

Total is P, if J needs to be developed from distillate of the first column, then P-J should be the number of final products that must be developed from the bottoms of the first column.

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Number of Sequences of Ordinary Distillation Columns

If N_i is the number of different sequences for i final products, then for a given separation point in the first column, the number of sequences is $N_i N_{P-j}$

But in the first separator, $(P - 1)$ different separation points are possible.

Thus, the number of different sequences for P products is the following sum:

$$N_s = \sum_{j=1}^{P-1} N_j N_{P-j} = \frac{[2(P-1)]!}{P!(P-1)!}$$

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So if N_i is the number of different sequences of i final products, then for a given separation

point in the first column, the number of sequences should be N^j multiplied by N^{P-j} . But in the first sequence or what but in the first distillation column in the sequence, there are $P-1$ different separation point. So the number of different sequences for P products will be given by this sum. So you have to take the sum $N^j N^{P-j}$ for all values of j equal to 1 to $P-1$ because $P-1$ separation points are possible in the first distillation column. This can also be written using the factors as shown.

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Number of Sequences of Ordinary Distillation Columns

$$N_s = \sum_{j=1}^{P-1} N^j N^{P-j} = \frac{[2(P-1)]!}{P!(P-1)!}$$

Number of Products, P	Number of Separators in the Sequence	Number of Different Sequences, N_s
2	1	1
3	2	3
4	3	14
5	4	42
6	5	132
7	6	429
8	7	1,430
9	8	4,862
10	9	14,300

Number of Possible Sequences for Separation by Ordinary Distillation

Now this equation tells me how many different sequences are possible for given number of products? So we have for 3, say number of distillation column will be 2 and this sequences possible are 2, we have seen that. For 4 also, we have seen that we need 3 ordinary distillation columns and 5 sequences are possible. Note that the number of sequences that are possible rapidly increases as we increase the number of products.

For example, with 7 number of products, with 7 components, I need $7-1=6$ ordinary distillation column, but the possible number of sequences becomes 132.

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Practical Constraints Restricting Options

1. Safety considerations might dictate that a particularly hazardous component be removed from the sequence as early as possible to minimize the inventory of that material.
2. Reactive and heat-sensitive components must be removed early to avoid problems of product degradation.
3. Corrosion problems often dictate that a particularly corrosive component be removed early to minimize the use of expensive materials of construction.
4. If thermal decomposition in the reboilers contaminates the product, then this dictates that finished products cannot be taken from the bottoms of columns.

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Now, although that those equation gives me many, many number of possible sequences particularly when I increase the number of components, there are practical constraints which restricts these options. What are those practical constants? Let us briefly discuss. Safety constraints. Safety constants might dictate that a particularly hazardous component be removed from the sequence as early as possible to minimize the inventory of that material.

Because you do not want that hazardous component to be in the inventory, so you want to minimize the inventory of this hazardous material and then you need to remove this component as early as possible. Reactive and heat sensitive components must be removed early to avoid problems of product degradation. Corrosive, corrosion problems often dictate that a particularly corrosive component be removed early to minimize the use of expensive material of construction.

If thermal decomposition in the reboiler contaminates the product, then this dictates that finished products cannot be taken from the bottom of the distillation column.



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Practical Constraints Restricting Options

Some compounds tend to polymerize when distilled unless chemicals are added to inhibit polymerization. These polymerization inhibitors tend to be non-volatile, ending up in the column bottoms. If this is the case, it normally prevents finished products being taken from column bottoms.

There might be components in the feed to a distillation sequence that are difficult to condense. Total condensation of these components might require low-temperature condensation using refrigeration and/or high operating pressures. Such condensation increases operating costs significantly.

Under these circumstances, the light components are normally removed from the top of the first column to minimize the use of refrigeration and high pressures in the sequence.



Some compounds tend to polymerize when distilled unless chemicals are added to inhibit polymerization. These polymerization inhibitors tend to be non-volatile ending up in the bottoms of the column. If this is the case it normally prevents finished products being taken from the column bottoms. There might be components in the feed to a distillation sequence that are difficult to condense.

Total condensation of this components might require low temperature condensation using refrigeration and/or high operating pressures. Such condensation using low temperature, that means using refrigeration or high pressures increases the operating cost significantly. So under these conditions the light components are normally removed from the top of the column to minimize the use of refrigeration and high pressure in the sequence.

So these practical considerations must be made and these practical constraints restricts the practical options that are available to the designer for possible number of sequences of ordinary distillation column for a multi component separation. With this we stop our discussion here and we will continue with synthesis of separation systems in the next module where we will discuss more about the sequences of distillation column.