Batch distillation

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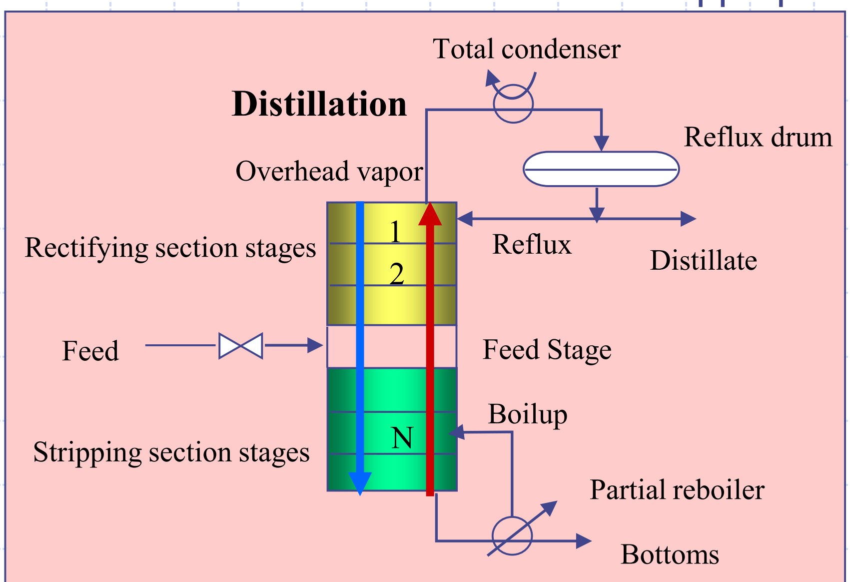
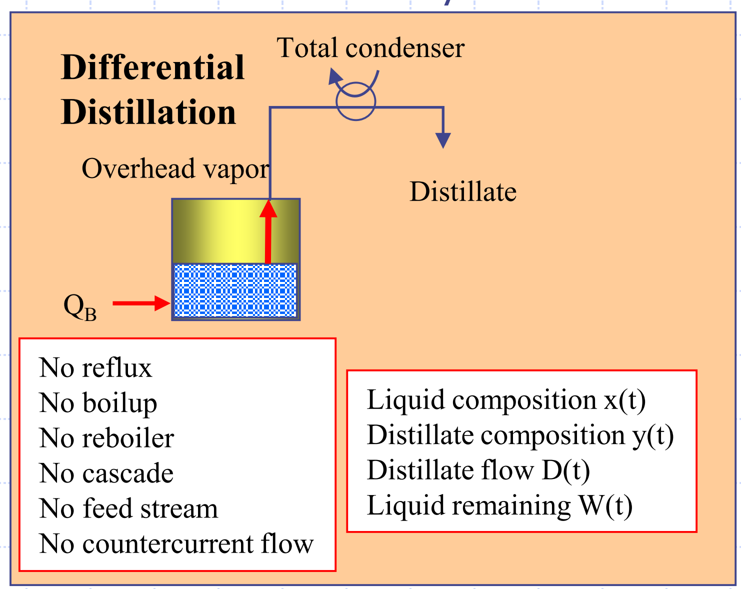
Agenda

- Batch distillation principles
- Multistage batch distillation
- Binary distillation: Rayleigh equation
- Batch distillation at total reflux
- Semi batch distillation
  - Constant x_D operation
  - Constant reflux operation
- Multicomponent batch distillation

Batch Distillation

- A feed mixture of a given composition is placed in a single stage separator and heated to boiling.
  - The vapor is collected and condensed to a distillate.
  - The composition of the remaining liquid and the distillate are functions of time.
- There may be several reasons for running a batch process this:
  - Small capacity doesn’t warrant continuous operation
  - Separation is to be done only occasionally
  - Separation is preparative to produce a new product
  - Upstream operations are batchwise or feedstocks vary with time or from batch to batch
  - Feed materials are not appropriate for a continuous flow system.





The **Differential Distillation** operation requires a much **simpler** apparatus, but is **complicated** because the process is now a function of time.

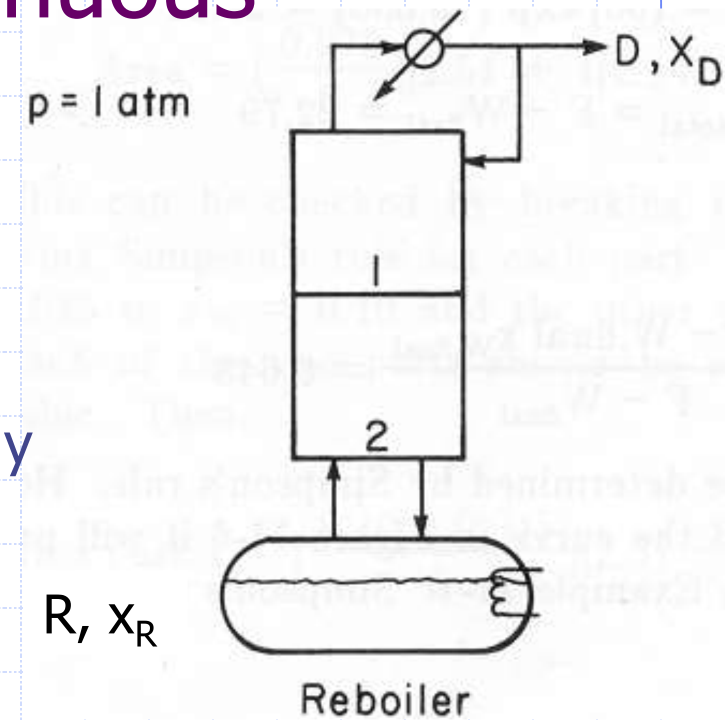
Batch Distillation

- Batch distillation uses
  - Relatively small amounts of product and charge.
  - Non-continuous operation (batch).
  - Different distillations are to be done using the same equipment.
- Batch distillation peculiarities
  - Unit may be a single pot or multi-staged.
  - There is no continuous feed – the pot is charged with liquid and then drained at the end of the run.
  - Distillate (usually the desired product) may be withdrawn continuously or collected in an accumulator.
  - Batch installations used in ‘campaigns’
- Industries interested
  - Pharmaceuticals
  - Fine chemistry and specialty chemistry
  - Food
  - cosmetics

Batch Distillation

- One is more interested in the amounts of bottoms and distillate collected rather than the rates.
  - Design of the column is not important
  - Same column may be used for different separations
  - It is important rating a batch distillation equipment
- The amount and compositions in the pot (bottoms) change as the more volatile component(s) decrease(s) with time and the less volatile component(s) increase(s) with time.
- Because the bottoms amount and concentrations change with time, the distillate amount, D, and concentration, x_D, in general, change with time.
- Operating a batch distillation may be done in 3 different ways:
  - Constant reflux operation and variable distillate composition (semi-batch)
  - Constant distillate composition and variable reflux (semi-batch)
  - Total reflux operation (fully batch)
- Each operating mode gives different time concentration profiles in the column

Multistage Batch vs. Continuous



- The batch system can be operated with a constant L/D, which means that x_D will change with time, or it can be operated with a constant x_D, which means that the L/D must be continuously changed.
- For a constant L/D, the distillate concentration fed to the accumulator decreases with time.
- The composition of the desired component in the distillate is at a maximum at the beginning of the batch run and decreases with time as it is distilled from the bottoms pot.
- The concentration of the more volatile component in the accumulator also decreases with time – the trade off is a lower concentration with more distillate accumulated.

Multistage Batch Distillation

- We have only one operating line since there is no feed.
- We can plot this operating line on a McCabe-Thiele plot along with our equilibrium curve.
- We can step down the operating line from x_D to x_R to determine the number of stages.
- Note that the bottoms concentration, x_R, keeps changing with time as the liquid is boiled off.
- Also note that x_D changes with time for a constant reflux ratio, L/V or L/D.
- We need to relate x_D and x_R with time…

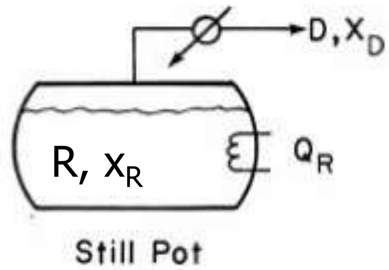
Binary batch distillation: Rayleigh equation

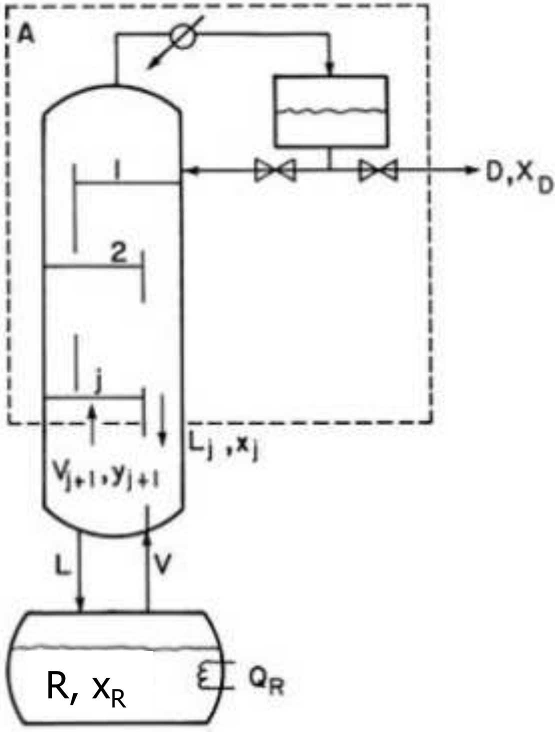
- Mass balance around the entire system for the entire operation time:

  $F = R_{final} + D_{total}$

  $F z_F = R_{final} x_{R,final} + D_{total} x_{D,avg}$

- F, z_F and the desired value of x (one) are specified
  - An additional equation is required to solve for 3 unknowns (Rayleigh equation)
  - Assumption: column and accumulator hold-up is negligible → -out=accumulation in reboiler → $-x_D dR = -d(Rx_R)$
  - rearranging: $-x_D dR = -R\, d(x_R) - x_R d(R)$
  - Finally: $$\ln\frac{R}{F} = \int_{z_F}^{x_R} \frac{dx_R}{x_D - x_R}$$
- Rayleigh equation
  - is valid for simple and multistage batch distillation.
  - We must relate x_D to x_R and do the appropriate integration





Rayleigh Equation Notes

- Note that x_R will change with time starting at z_F ($t = 0$) and ending at $x_{R,final}$ (t = final).
  - However, x_D also changes over time (note that it is not the same as the $x_{D,average}$).
  - Thus, in order to integrate the right-hand side of Eq. (11-6), we need a relationship between x_D and x_R → $x_D = f(x_R)$
- If we have a column
  - with an N number of stages,
  - and we are operating at constant L/D,
  - we can use the McCabe-Thiele analysis to step off the stages from x_D and determine the resulting x_R to give us an $x_D = f(x_R)$.
  - We just assume different x_D's , step-off, and determine the resulting x_R's.
- Simple binary batch distillation
  - Rayleigh equation can be integrated analytically

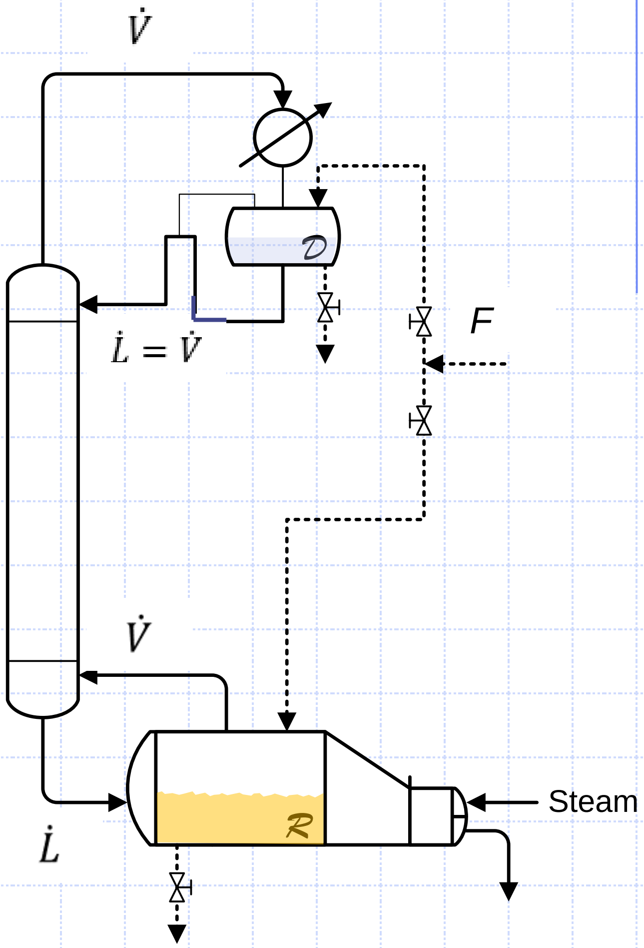
$$\ln \frac{R}{F} = \int_{z_F}^{x_R} \frac{dx}{x - f(x)}$$

Total Reflux

- Note that a multistage batch still, operated such that all of the distillate is returned to the top of the column, is essentially the same as a multi-stage distillation column operated under **total reflux.**
- For a binary separation, given a column containing an N number of equilibrium stages, one can measure x_D at the top of the column and x_B at the bottom of the column and perform a McCabe-Thiele analysis to determine the theoretical N_{min}.

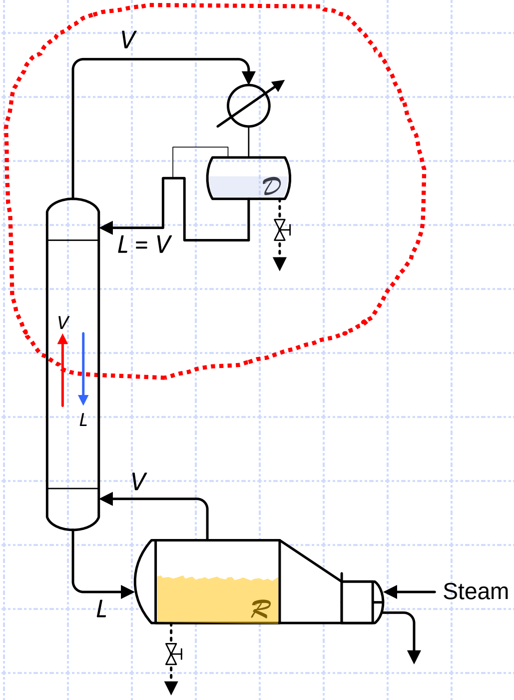
Multistage Fully Batch operation (total reflux)

- Feed F is divided between the boiler and the top accumulator
- After start up (stages flooding) the column is operated at total reflux: L=V at all times → D and R are constant
  - the light component will concentrate at the top and the heavy at the bottom
  - Operation is stopped when specification for both product is reached.
- Note:
  - Final compositions x_D and x_R depend on how the feed is divided
  - if $D < D_{spec}$, bottom will be polluted by light components
  - If $D > D_{spec}$, top will be polluted by heavy components



$$\frac{D}{F} = \frac{z_F - x_{R,spec}}{x_{D,spec} - x_{R,spec}} \quad \frac{R}{F} = \frac{x_{D,spec} - z_F}{x_{D,spec} - x_{R,spec}}$$

Multistage Fully Batch operation (total reflux)



- Tank compositions varies continuously during operation
- At each time the global material balance is:

$$F = D + R \qquad F z_F = D x_D + R x_R$$

- Balance of the upper part of the column:

$$\frac{dDx_D}{dt} = \dot{V} y_{j+1} - \dot{L} x_j \xrightarrow{D = cost, \dot{L} = \dot{V}} \boxed{y = x + \frac{D}{\dot{V}} \frac{dx_D}{dt}}$$

- Assuming negligible hold-up in the column with respect to the thanks:

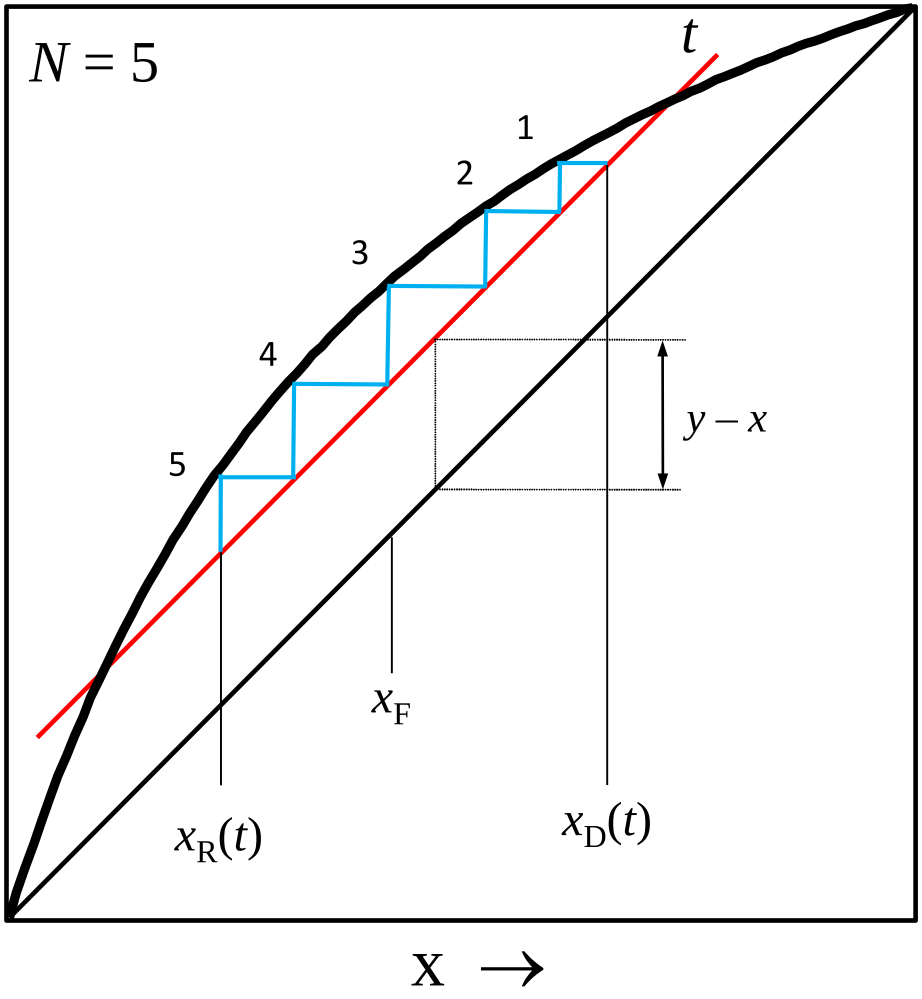
$$D \frac{\mathrm{d} x_D}{\mathrm{d} t} = -R \frac{\mathrm{d} x_R}{\mathrm{d} t}$$

Multistage Fully Batch operation (total reflux)

- At each time, the OL has a slope = 1 and its distance from y=x line is (y-x)
- Fix an $x_D(t)$ initial guess
- Step down the column and find the value $x_R(t)$ compatible with the initial guess
  - The two compositions are correct if they satisfy the global balance too

$$D(x_D - z_F) = R(z_F - x_R)$$

- The OL moves in time getting closer to the diagonal

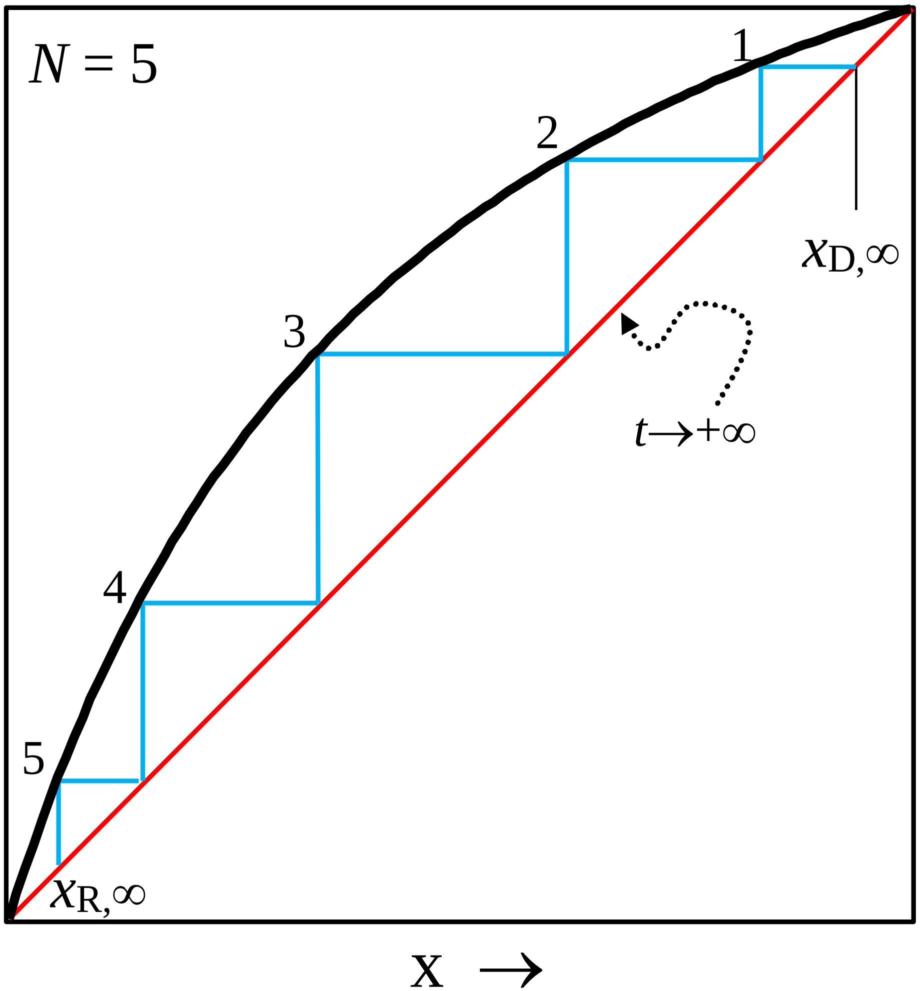


Multistage Fully Batch operation (total reflux)

- After a long time (infinite time) the OL overlaps to the diagonal
- Compositions will have to staisfy the global balance

$$D(x_{D,\infty} - x_F) = R(x_F - x_{R,\infty})$$

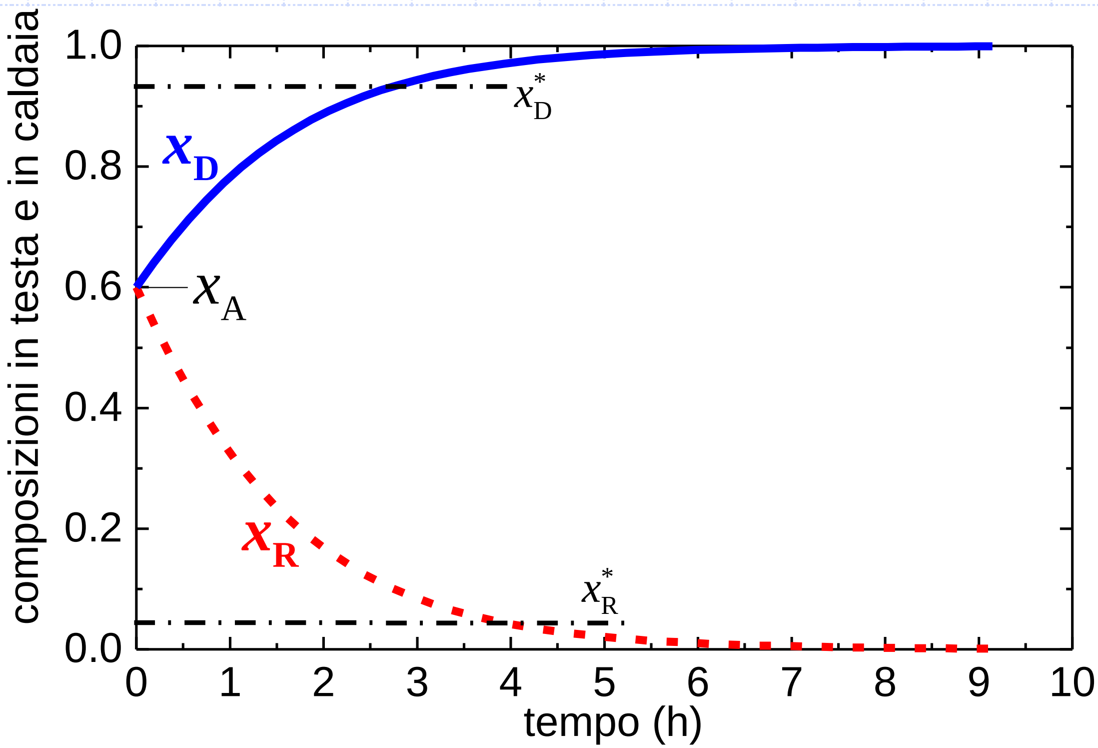
- Desired purity $x_{D,spec}$ and $x_{R,spec}$ will always be less than the maximum ones,
  - reachable at stationary regime only (i.e. at $t \rightarrow \infty$)



Multistage Fully Batch operation (total reflux)

Typical concentration profiles

- If, for a fixed z_F D and F are correctly chosen and the column has a sufficiently high number of stages,
- it is possible to gat both products at high purity



Multistage Fully Batch operation (total reflux)

- The heat required for a given separation is:

$$Q_r = \lambda \int_0^{t_F} \dot{V} d\,t = \lambda D \int_{z_A}^{x_{D,spec}} \frac{d x_D}{y - x}$$

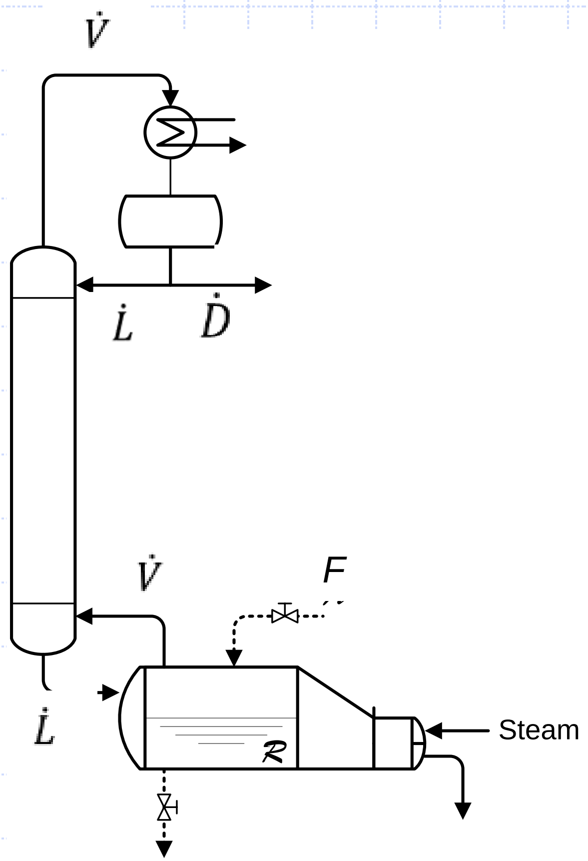
- And the time t_F to reach the product specifications is:

$$t_F = \frac{Q_r}{\lambda_{st} W_{st}}$$

- The value of the integral does not depend on the vapor flow rate.
  - Q_R is a function of the required separation ($x_{D,spec} - x_{R,spec}$),
  - Vapor consumption is independent on how the separation is operated
  - This means that no control is necessary: it should only be run at total reflux.

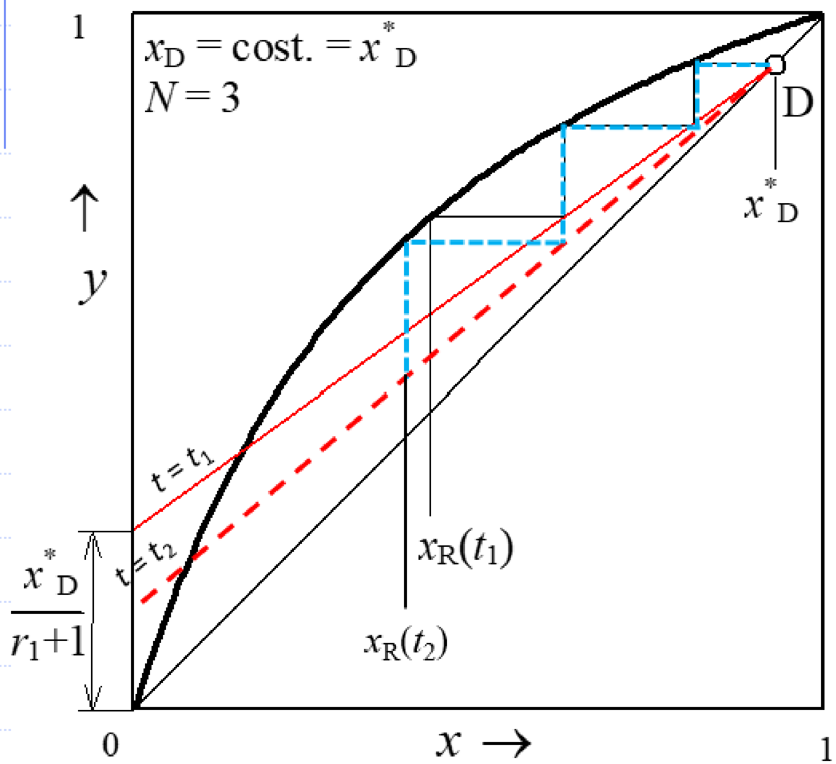
Semi-batch distillation process

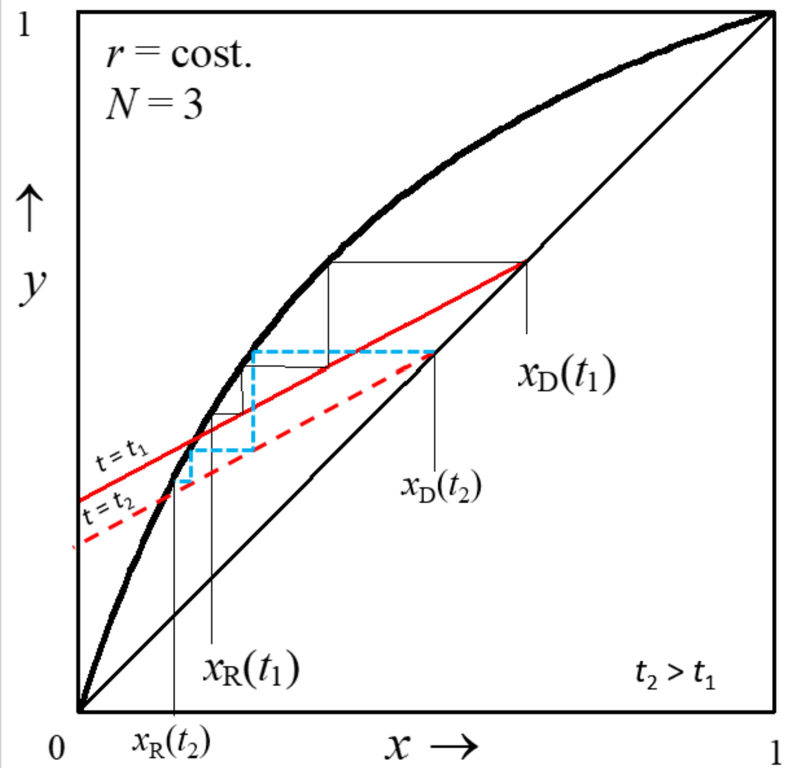
- Start up phase
  - Capacity of condenser accumulation tank is reduced, since distillate is continuously removed
  - Reboiler should be large enough
  - Mixture is fed to the reboiler only
  - Operation is started at total reflux, up to the column is in stable conditions from the fluid-dynamic view point (verify with stable pressure drops)
  - Afterwards it is possible to operate::
  - a) variable reflux, keeping constant distillate composition x_D;
  - b) constant reflux (i.e. constant flow rate of distillate), with variable distillate composition



Multistage Semi-Batch Distillation

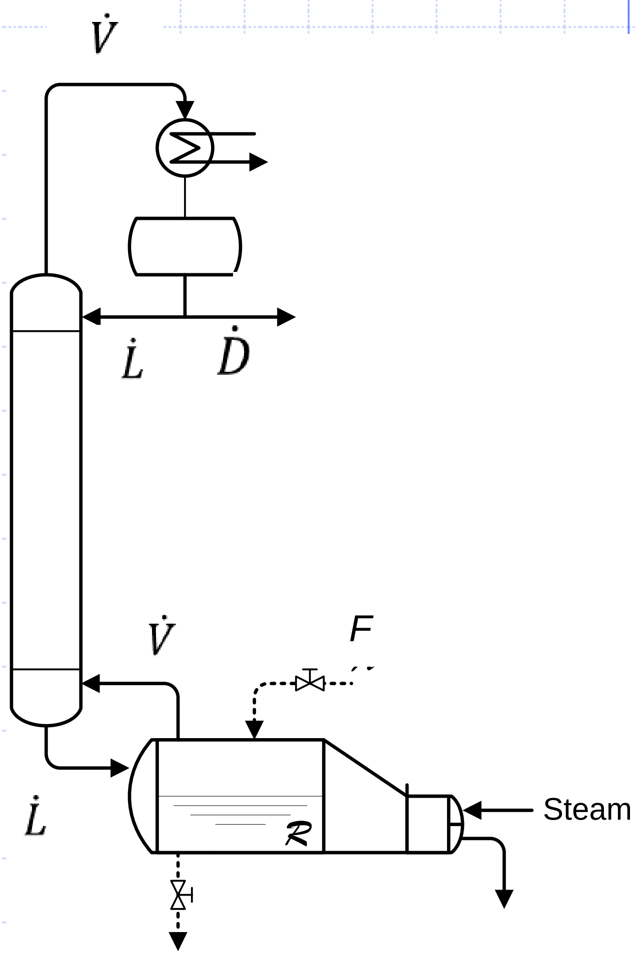
- Multistage semi batch operation may be operated in two different conditions:
- Constant x_D Constant L/D





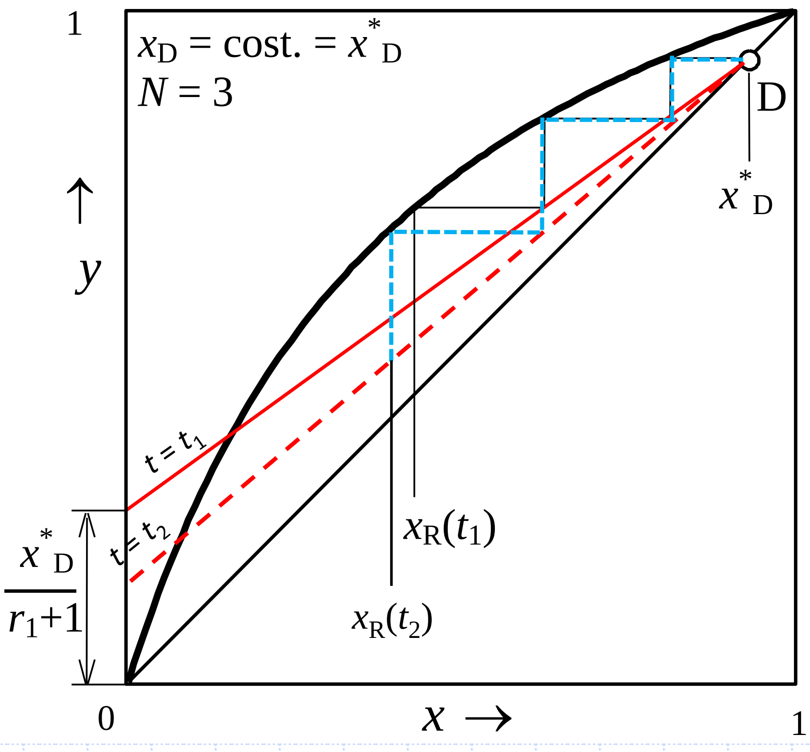
Semi-batch distillation process

- Assumptions
  - Adiabatic column
  - Hold-up negligible in the column and in the condenser
  - Hold-up in reboiler only
  - Constant column efficiency during operation (time)
  - Column at steady state at each time (quasi-stationary hypothesis)
- In these conditions
  - The column is a simple enrichment section only column
  - With variable composition feed
  - With constant feed flow rate



Semi-batch distillation process

- After startup, reflux flow rate L is varied so that $x_D = x_{D,spec}$
  - At any time t_1, with fixed r, it is possible to find the composition in the reboiler at that time t_1
  - Since light component is output from the top, its concentration in the reboiler goes down
  - To keep x_D at the desired value, it is necessary to push the separation and therefore raise the value of r



Constant x_D operation

- The column is operated at constant x_D
  - First value for r is such to fit exactly N stages between x_D and x_F
  - Last value for r is such to fit exactly N stages between x_D ande x_R
- Energy balance and steam consumption
  - Reflux ratio (instantaneous)

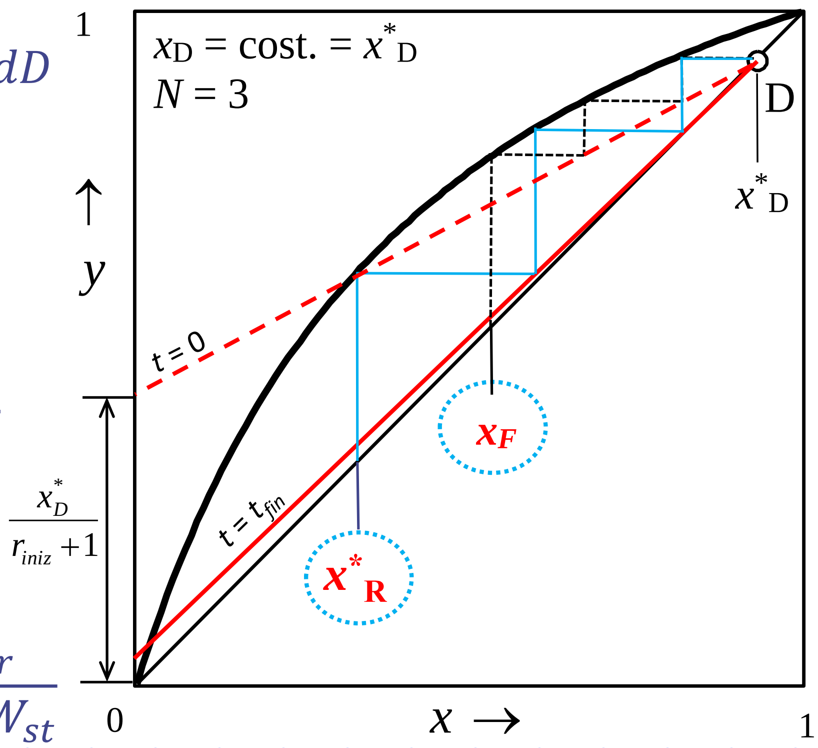
$$r = \frac{\dot{L}dt}{dD}$$

$$dQ_r = \lambda(r+1)dD \longrightarrow Q_r = \lambda \int_0^{D,spec} (r+1)dD$$

  - It is necessary to know the function r(D). Starting from Rayleigh eq.:

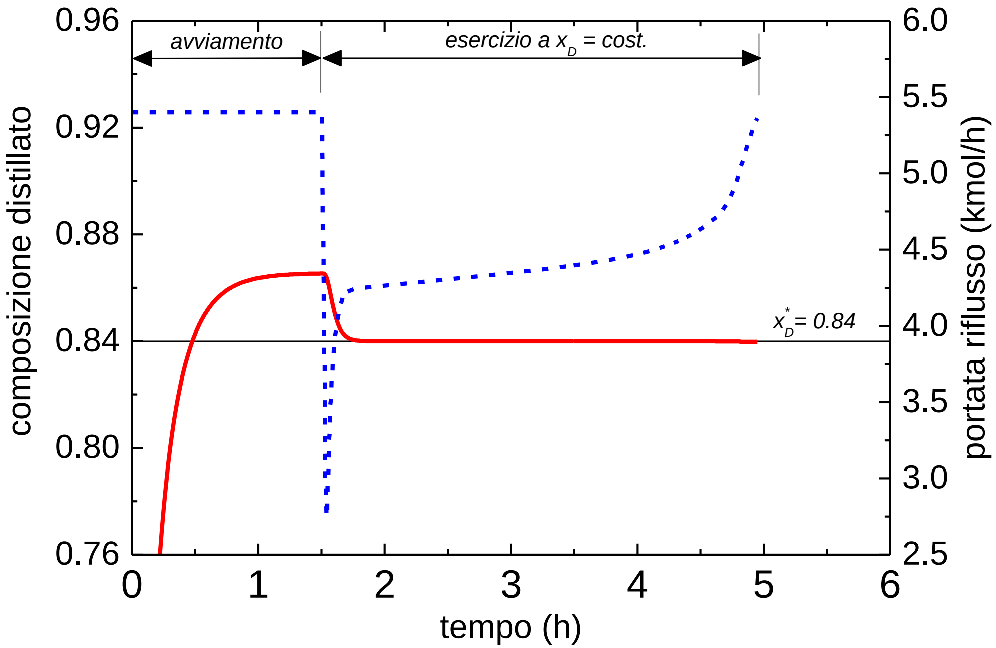
$$\ln\frac{R}{F} = \int_{z_F}^{x_R} \frac{dx_R}{x_D - x_R} \xrightarrow{x_D = \text{cost}} R = F\frac{x_D - x_F}{x_D - x_R}$$

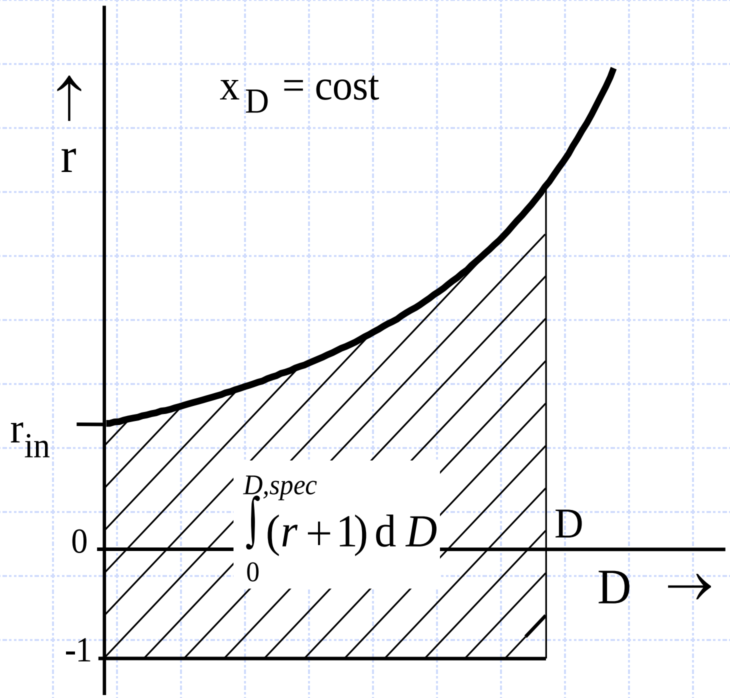
  - $r(x_R)$ is transformed in r(R), and consequently one gets r(D).
  - The time of batch process is: $t_F = \dfrac{Q_r}{\lambda_{st} W_{st}}$



Constant x_D operation

- At the end of the operation, big steam consumption is required for small quantities of distillate
- It is possible to decide a maximum value of D
  - Beyond which the operation is not economical any more
  - And column operation is very difficult since the raise of r is not linear in time





Constant reflux operation

- After start up the reflux flow rate L is kept constant and therefore also D is constant:

$$Q_r = \lambda(r+1)D$$

- r is determined by a trial and error procedure.
  - Material balances must be satisfied in the form:

$$R = F\frac{x_{D,spec} - z_F}{z_F - x_{R,spec}}$$

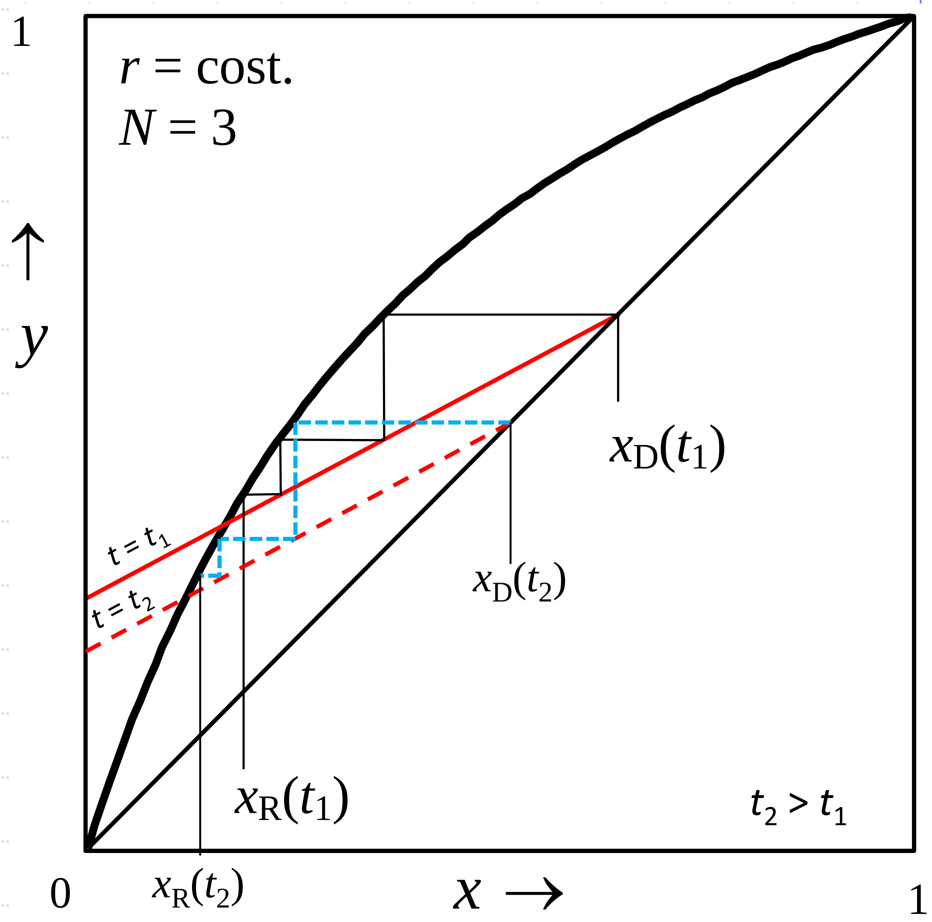
  - And Rayleigh equation is:

$$\ln\frac{R}{F} = \int_{z_F}^{x_R} \frac{dx_R}{x_D - x_R}$$

Integration requires to know the function $x_D(x_R)$, to be obtained through a McCabe-Thiele construction.

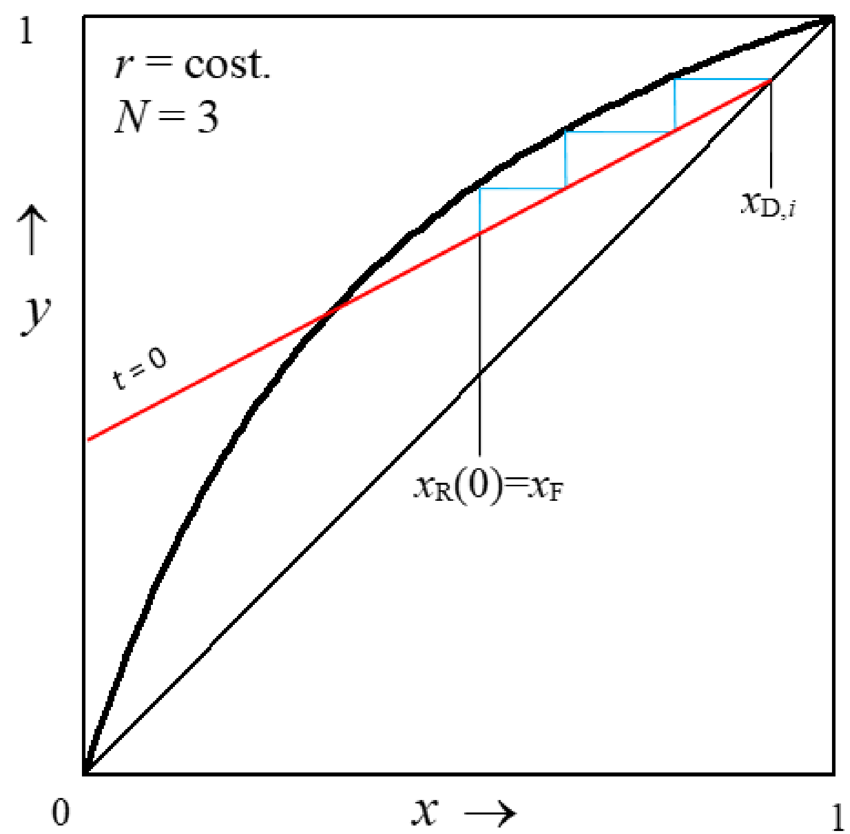
Constant reflux operation

- An arbitrary value of r is chosen
  - between the instantaneous composition of the distillate and the residue
  - so that the exact number of stages fits in.
- Light component is extracted in the distillate, → the concertation of the light component goes down in the reboiler (x_R falls in tempo)
- Since N is fixed →if x_R goes down in time, x_D goes down too
- The OL moves downwards keeping the same slope
- Final composition of the distillate will be the average of the compositions in time



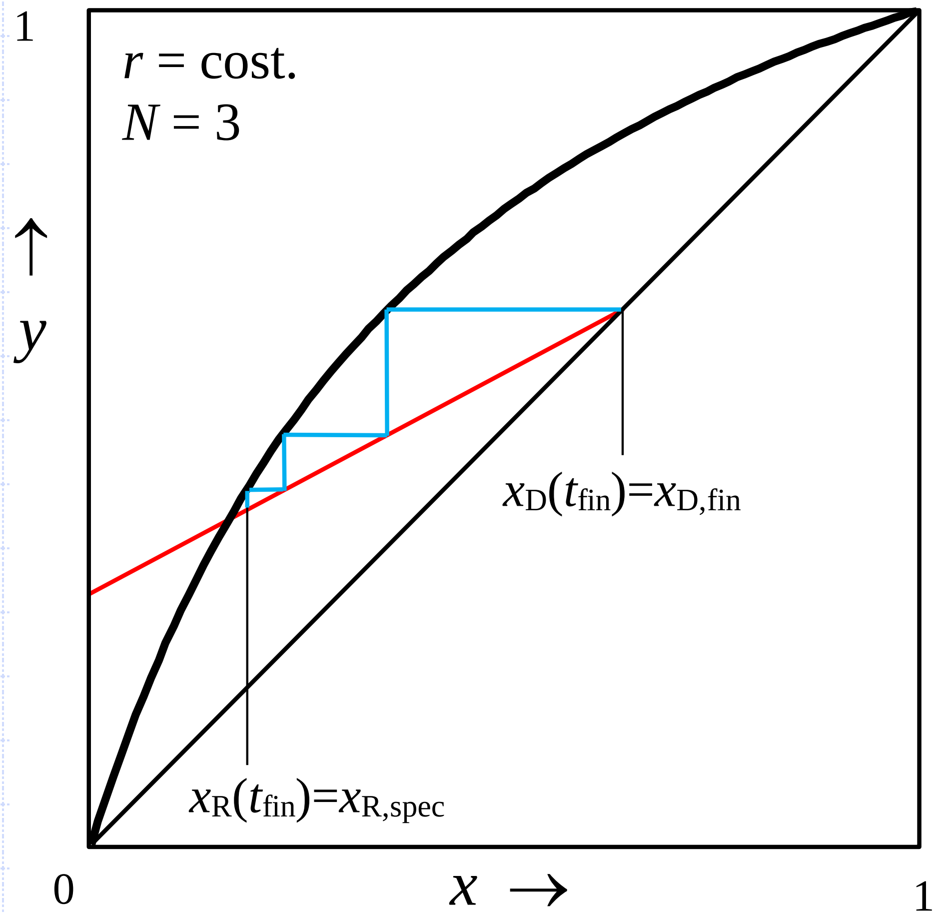
Constant reflux operation

- Initial distillate composition $x_{D,i}$ allows to get to the x_F composition of the feed to the reboiler with exactly N = 3 stages



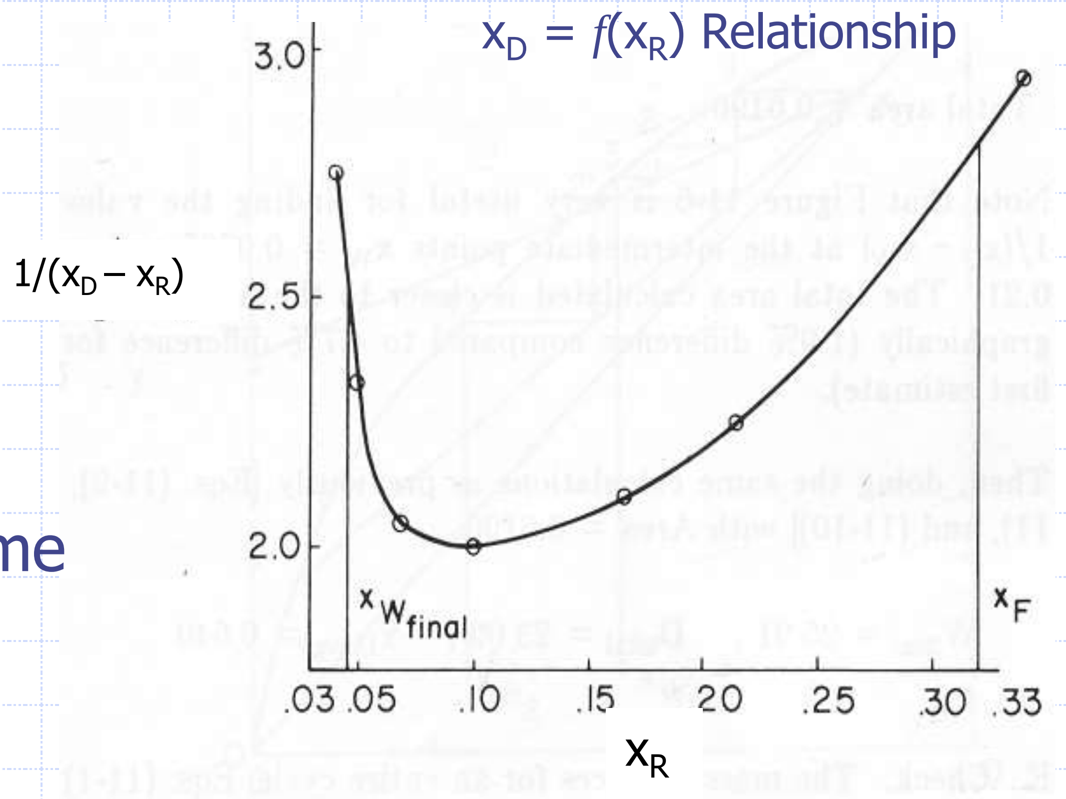
Constant reflux operation

- Construction stops when bottom composition is equal to the specification ($x_{R,spec}$)



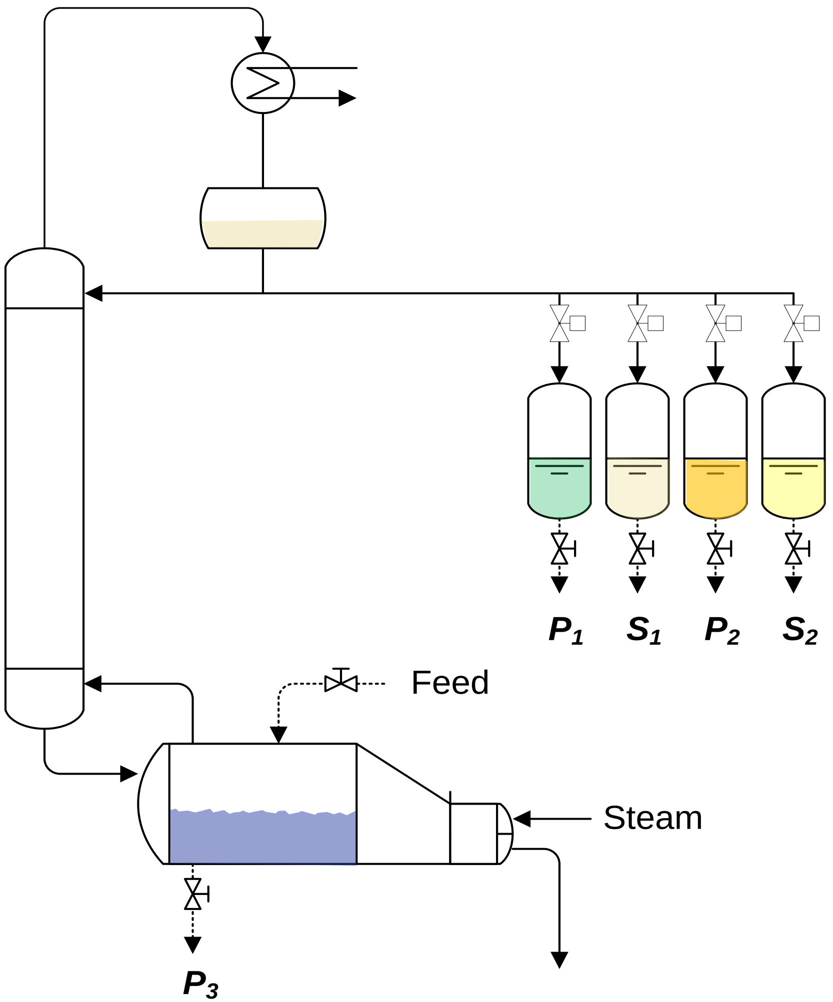
Constant reflux operation

- For a given L/D and number of column stages, assume x_D's, perform a McCabe-Thiele Analysis at each x_D stepping down to determine the corresponding x_R's.
- Plot $1/(x_D - x_R)$ vs. x_R.
- Graphically integrate
  - or do a polynomial curve fit between $x_R = z_F$ and $x_R = x_{R,final}$ and integrate.
  - Use Polymath or Excel for integration
- The correct value of r satisfies at the same time both Rayleigh equation and material balances



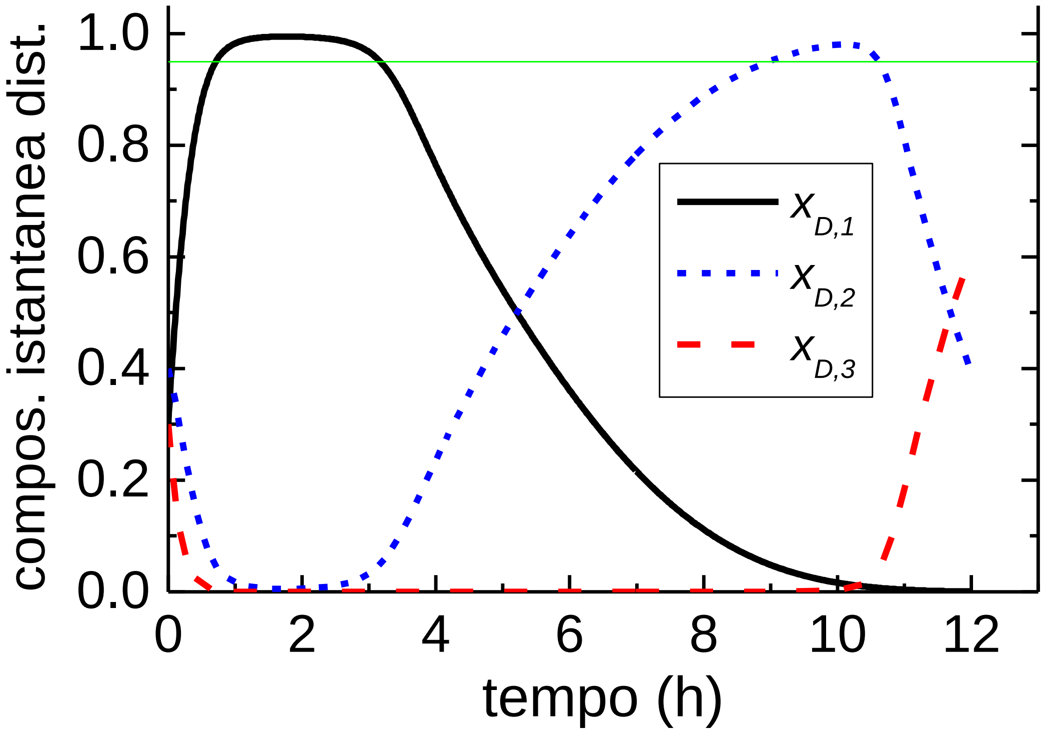
Multicomponent batch distillation

- It is possible to treat multicomponent mixtures (N_c = 3 in the example)
  - Different product cuts are obtained (P_1, P_2 e P_3)
  - wastes (slop cuts) are obtained (S_1 and sometimes S_2)
  - Heavier product is always obtained in the reboiler at the end of the batch.
- Tanks are needed
  - One for each cut;
  - slop cuts could be mixed and treated again in the same column
- One column is sufficient for obtaining all requested N_c cuts
  - A continuous operation would have required N_c −1 columns



Multicomponent batch distillation

- Typical composition profiles



Column Overall Efficiency

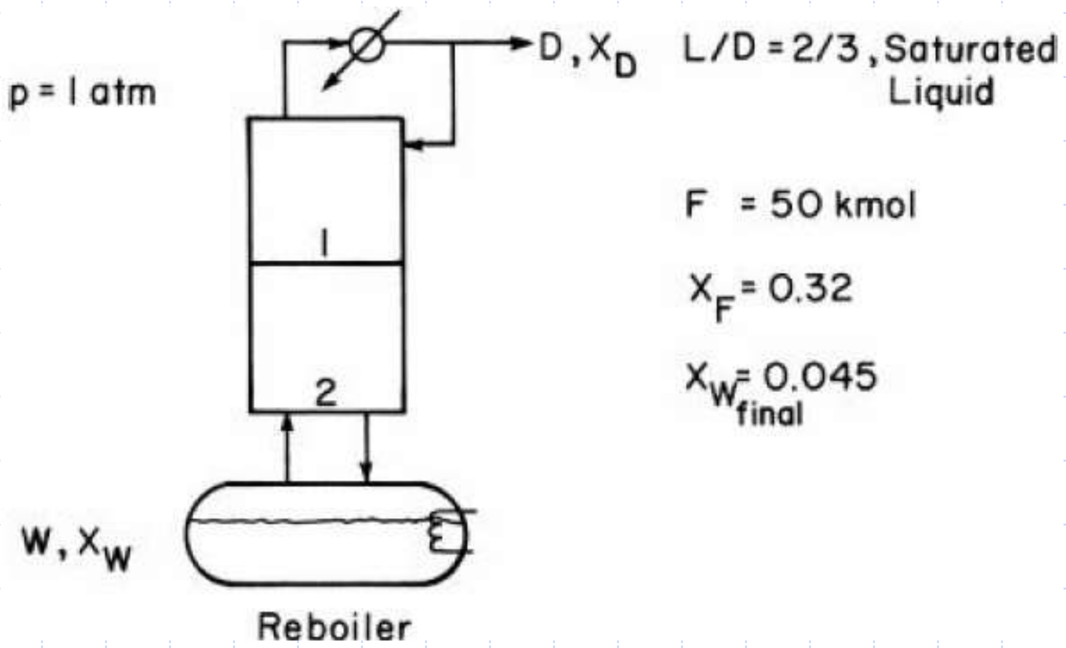
- For a column containing an N_{actual} number of stages, the overall efficiency can be determined from

$$E_o = N_{equil} / N_{actual}$$

where N_{equil} is the theoretical N obtained from the McCabe Thiele analysis.

Example: ethanol – water batch distillation

- We wish to batch distill 50 kmol of a 32 mol% ethanol, 68 mol% water feed. The system has a still pot plus two equilibrium stages and a total condenser. Reflux is returned as a saturated liquid, and we use L/D = 2/3. We desire a final still pot composition of 4.5 mol% ethanol.
  Find the average distillate composition, the final charge in the still pot, and the amount of distillate collected.
  Pressure is 1 atm.



Example: ethanol – water batch distillation

- Solution
  - The McCabe-Thiele diagram for several arbitrary values of x_D is shown in Figure.
  - The top operating line is
  - $y = \frac{L}{V}x + \left(1 - \frac{L}{V}\right) x_D$
  - Where L/V = (2/3)/(5/3) = 2/5
  - The corresponding x_W and x_D values are used to calculate $x_D - x_W$ and then $1/(x_D - x_W)$ for each x_W value.
  - These values are plotted in Figure. The area under the curve from $x_F = 0.32$ to $x_{W,final} = 0.045$ is 0.608 by graphical integration.
  - From Eq.: $W_{final} = F\exp^{-Area} = (50)\exp(-0.608) = 27.21$
  - From Eq.: $D_{total} = F - W_{final} = 22.79$
  - From Eq.:

$$x_{D,avg} = \frac{Fx_F - W_{final}\, x_{W,final}}{F - W_{final}} = 0.648$$

  - The area can also be found by Simpson's rule.