INOVATIVE CHEMICAL TECHNOLOGIES LLP

SPECIALISTS IN DISTILLATION TECHNOLOGIES

DISTILLATION SYSTEMS

TROUBLESHOOTING AND UPGRADATION

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Distillation:

- Most widely used separation process in the chemical industry !
- Accounts for 40% of equipment cost and 60% of operating costs of a chemical plant.

Yet, many distillation systems do not perform to full capacity or efficiency!

This presentation discusses troubleshooting and upgradation of distillation systems.

Troubleshooting, Upgradation and Debottlenecking

Procedures for studying and modifying existing plants to improve their performance.

Troubleshooting

Plant is not performing properly. Errors could have originated during design, manufacturing or operation.

Upgradation

Plant is performing, but the product recovery, product purity, energy consumption, plant capacity or operational flexibility can be improved.

Debottlenecking

Plant is performing, but is not giving full capacity. Some equipment are undersized and causing ‘bottlenecks’ to capacity.

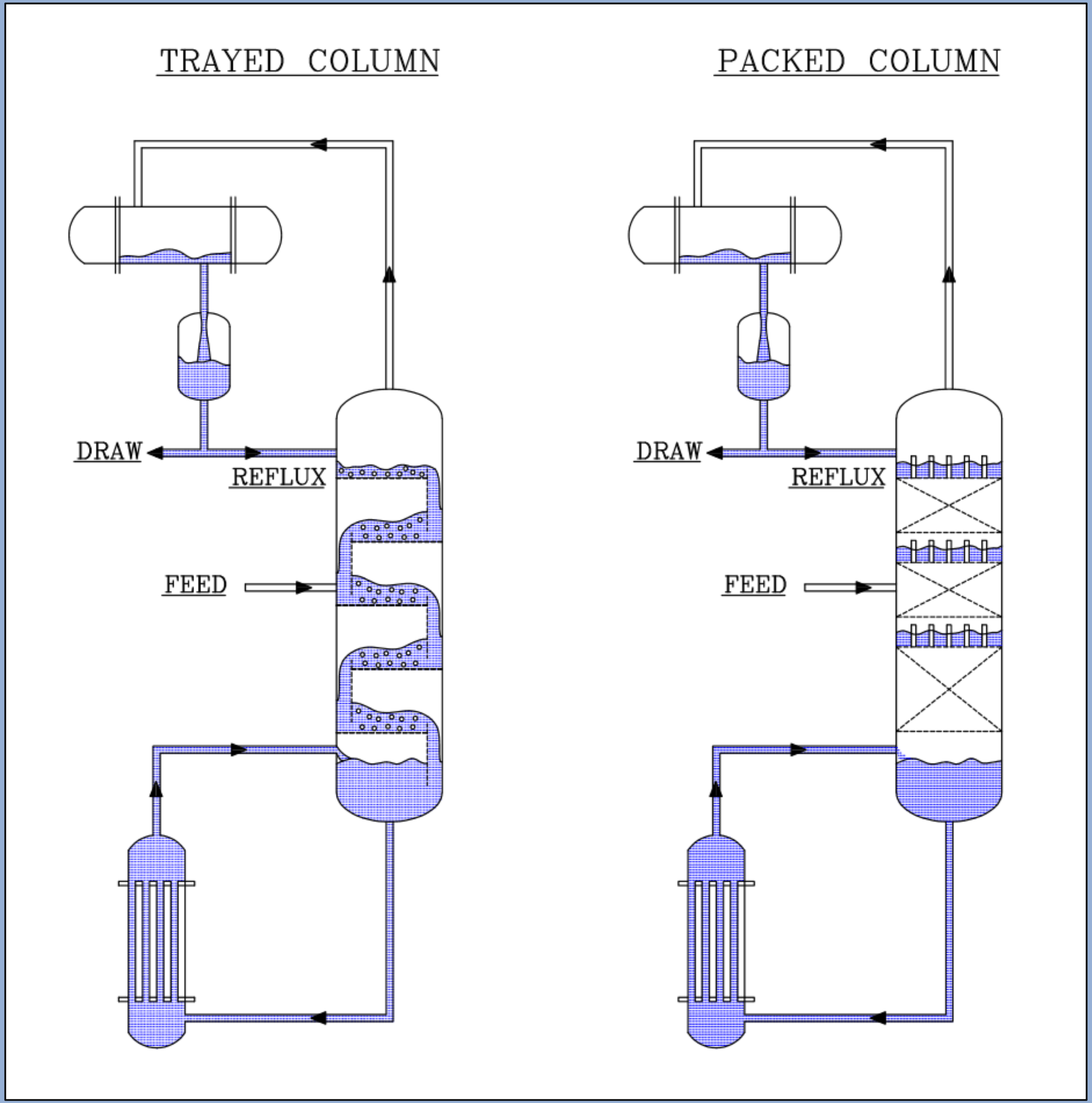
Troubleshooting Errors

- Ignoring components in feed
- Incorrect design algorithm
- Mistakes in detailing of trays and internals
- Problems with layout and erection
- Piping errors
- Excess holdup
- Issues with heat exchangers
- High column pressure drop.
- Operational issues like fouling.
- Changing properties of operating fluids.

Upgradation Techniques

- Convert batch to continuous operation (or vice versa): Viz batch plants are flexible but less efficient than continuous plants.
- Use appropriate contact devices, such as trays and packings. Viz, trays are resistant to fouling, but have higher pressure drops.
- Install robust instrumentation and control systems.
- Replace, repair or modify underperforming equipment.
- Use heat recovery systems, such as multi-pressure distillation and mechanical vapour recompression (MVR)
- Re-evaluate operating process and parameters.

SOME CASE STUDIES



Case 1: Holdup

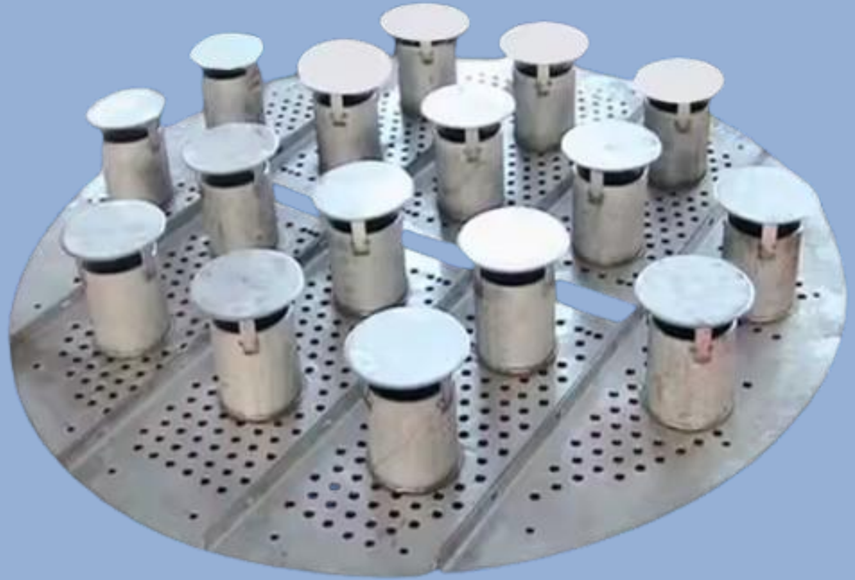
Holdup: Liquid remaining within the system during operation

Case-1

A large diameter packed column, with six distributors, was being used to separate close-boiling isomers. The column took very long to stabilize and did not give product of required quality.

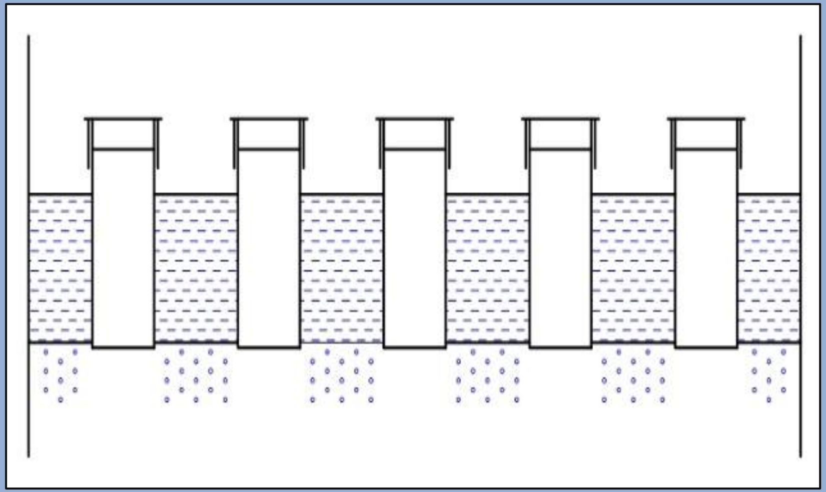
Diagnosis

Diameter of liquid distribution holes was too small. Therefore, liquid level (hence, the holdup) on each distributor increased. Total holdup became so high that almost all the low-boiler accumulated in the holdup itself, without coming out as distillate.

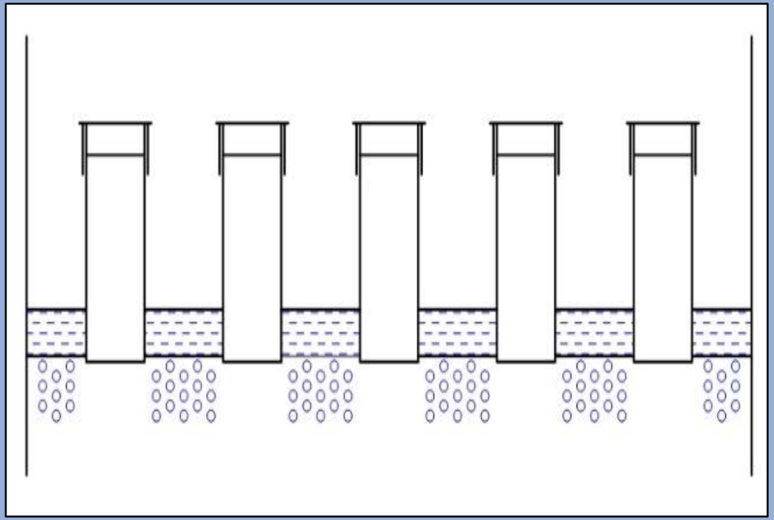


Solution Diameter of distributor holes was increased.

Smaller holes → lower discharge rate → higher liquid level → higher holdup



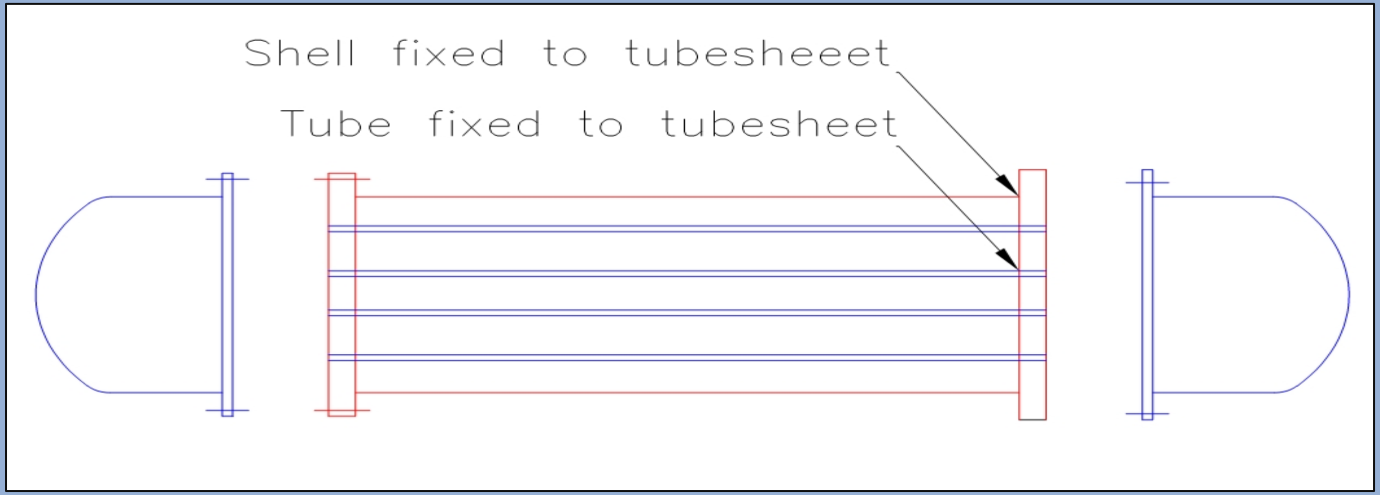
Larger holes → higher discharge rate → lower liquid level → lower holdup

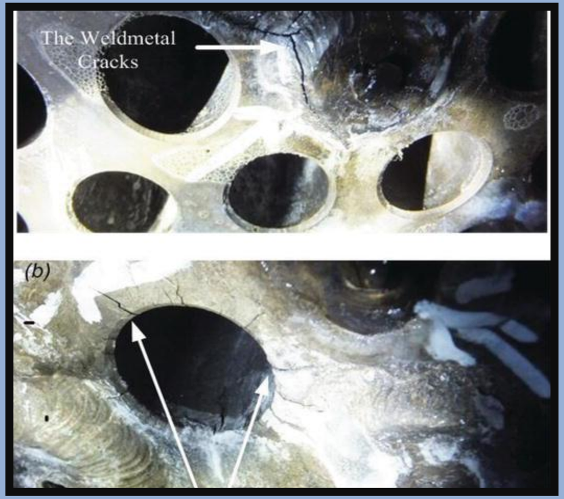


Case-2 Tube to tubesheet joints in a shell-and-tube condenser were getting damaged.

Diagnosis Differential thermal expansion was creating stresses that damaged the joints.

Solution The fixed tubesheet condenser was replaced by U-tube bundle condenser.

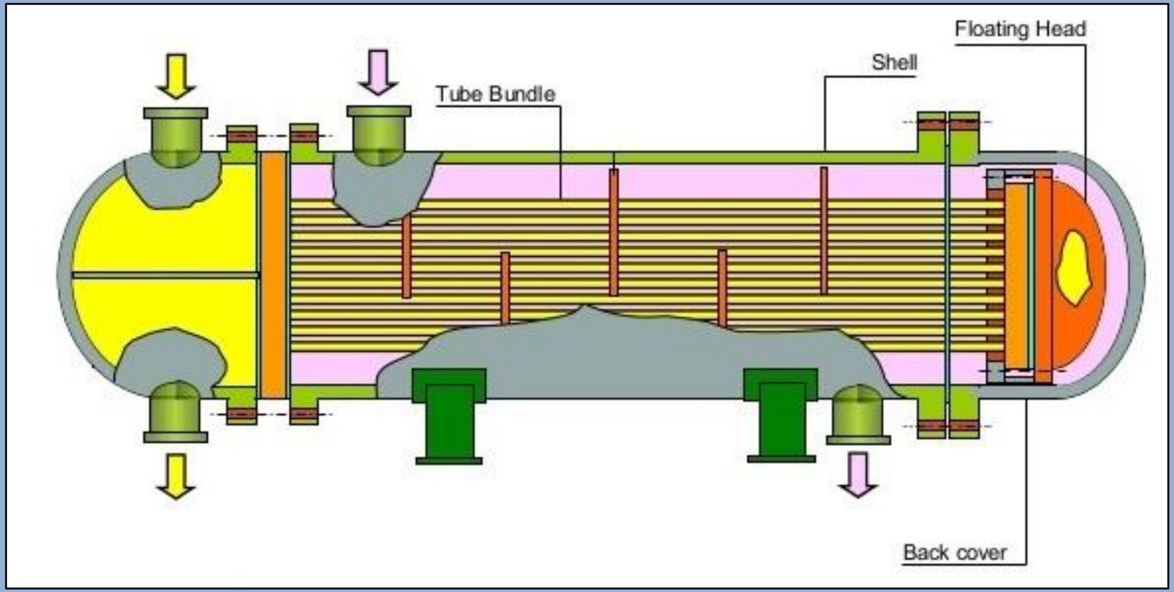




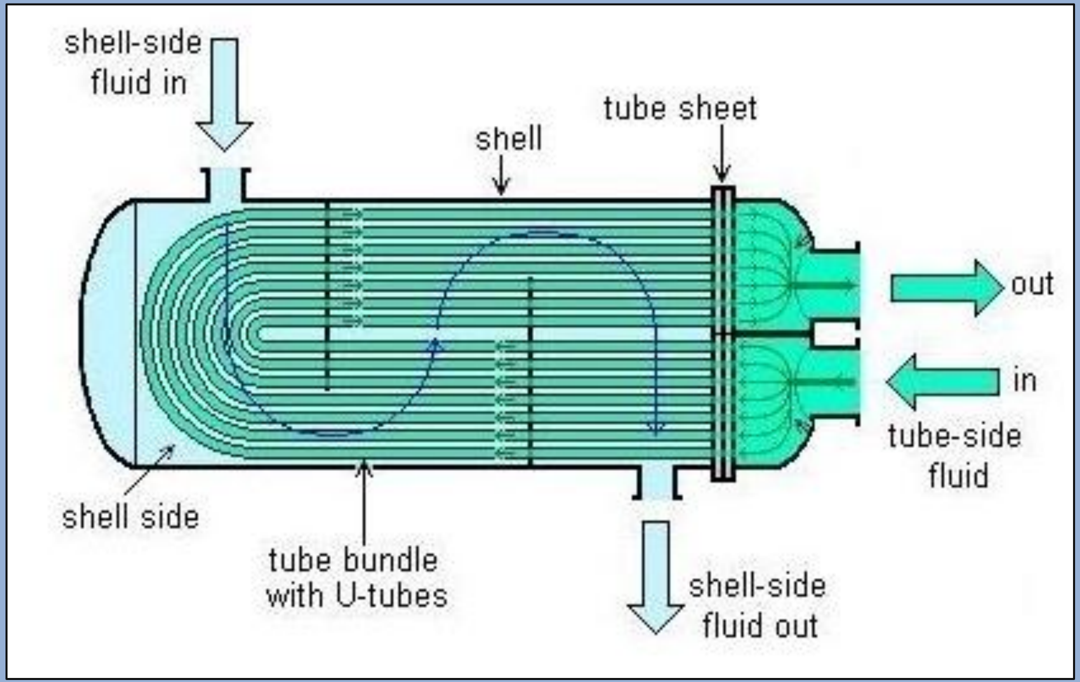
- Shell and tubes are both fixed to the two tube sheets.
- Shell is hotter so it expands more. Tubes are cooler, and expand less.
- Difference in the expansions of shell and tubes create thermal stress.

Methods of handling thermal stress-1

Floating Head Heat Exchanger



U-tube bundle Heat Exchanger



Methods of handling thermal stress-2

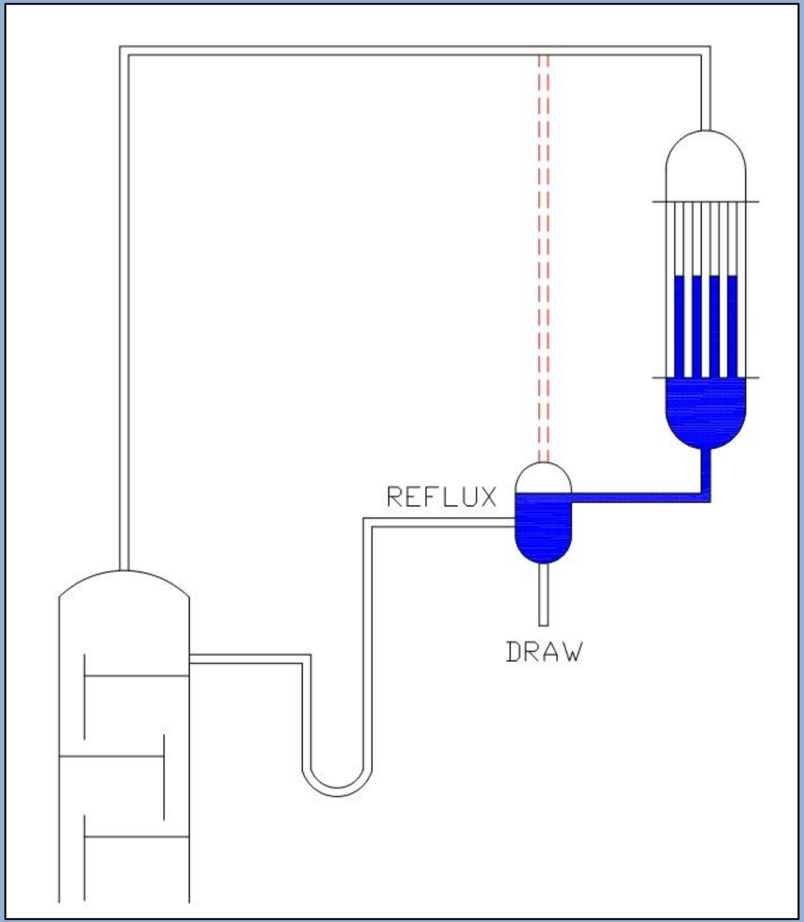
Heat Exchanger with Expansion Joint

Expansion Joint Detail

Case-3 Reflux to a column was flowing in sudden spurts. Distillate purity was not acceptable, even though separation was technically very easy.

Diagnosis

- The condenser was not vented. When vapour condensed, a partial vacuum was generated in the condenser. This did not allow condensate to flow to column. Instead, it backed up into the reflux drum as well as condenser. After it reached a certain level, hydrostatic head overcame the pressure differential, and reflux flowed to the column.
- Because reflux was not smooth it was not possible to obtain required distillate concentration.

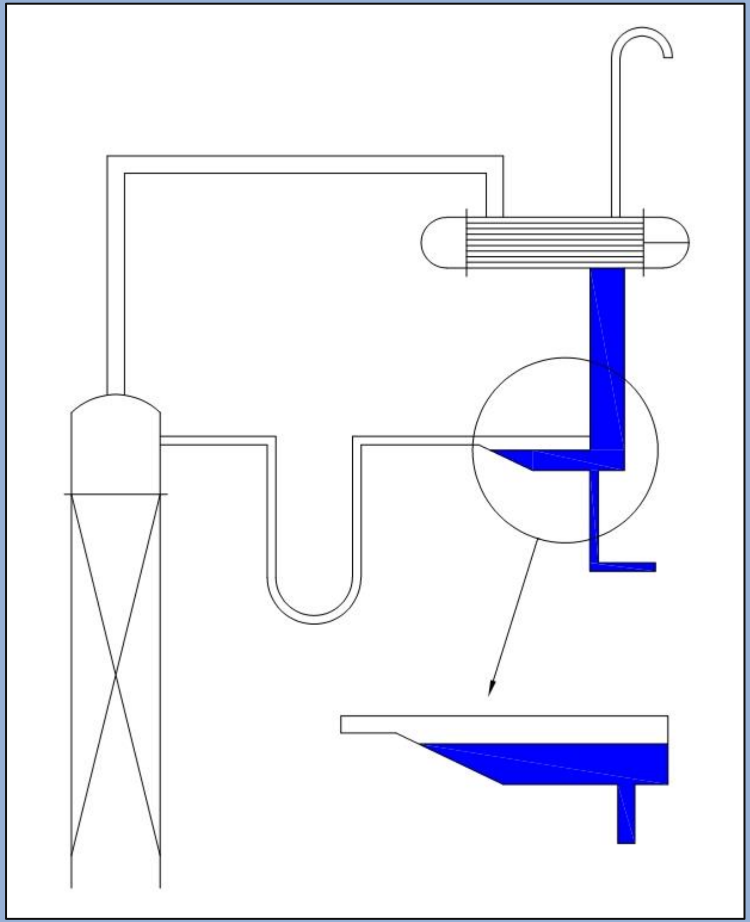


Solution Pressure equalisation line was provided to the reflux pot.

Case-4 A distillation column was working satisfactorily under normal design conditions. But, when capacity was reduced to 30% of design, reflux stopped and distillate purity became unacceptable.

Diagnosis

- Nozzle diameters of condenser and column reflux did not match, and an eccentric reducer had been used. The draw line was tapped from lower part of pipe, while the upper part continued to reflux.
- When flows were turned down, condensate flowed down the lower (draw) pipe, while upper (reflux) side became empty, which affected distillate purity.



Solution Size of column reflux, condenser outlet and connecting piping were made equal and eccentric reducer eliminated.

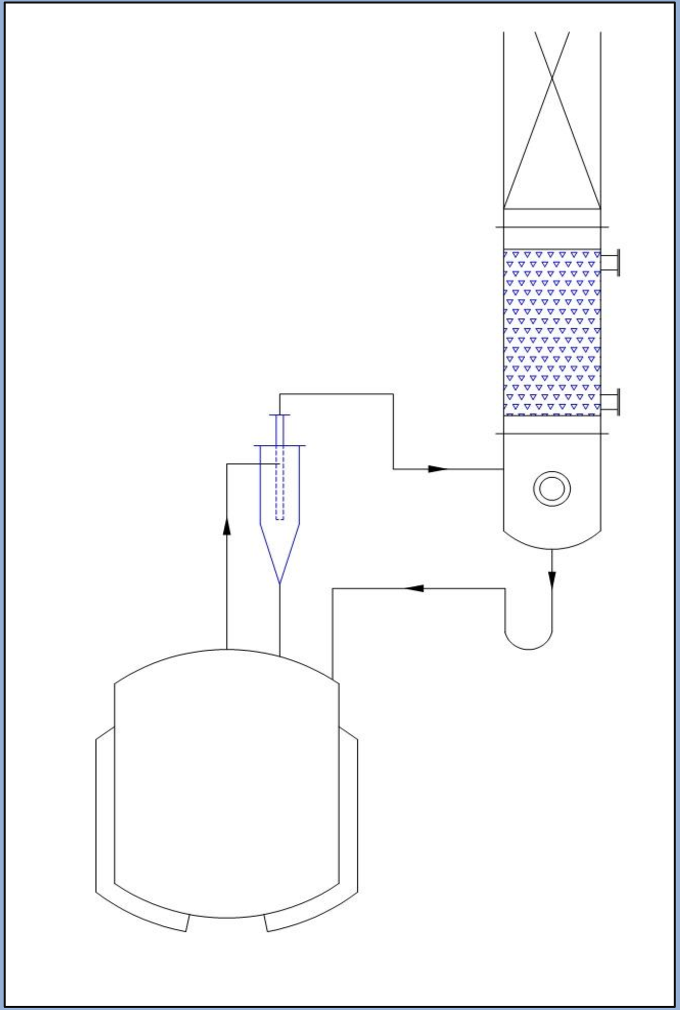
Case-5 In a batch distillation, bottom section of the column was getting corroded frequently. Removing and replacing damaged structured packings was a tedious process.

Diagnosis

Vapour from still was entraining liquid droplets to the column, which contained some inorganic acids.

Solution

- The vapour was taken to a cyclone separator that removed much of the entrained liquid.
- Furthermore, a small section of random packings was introduced at column bottom. Damaged random packings could now be removed and replaced in-situ easily.

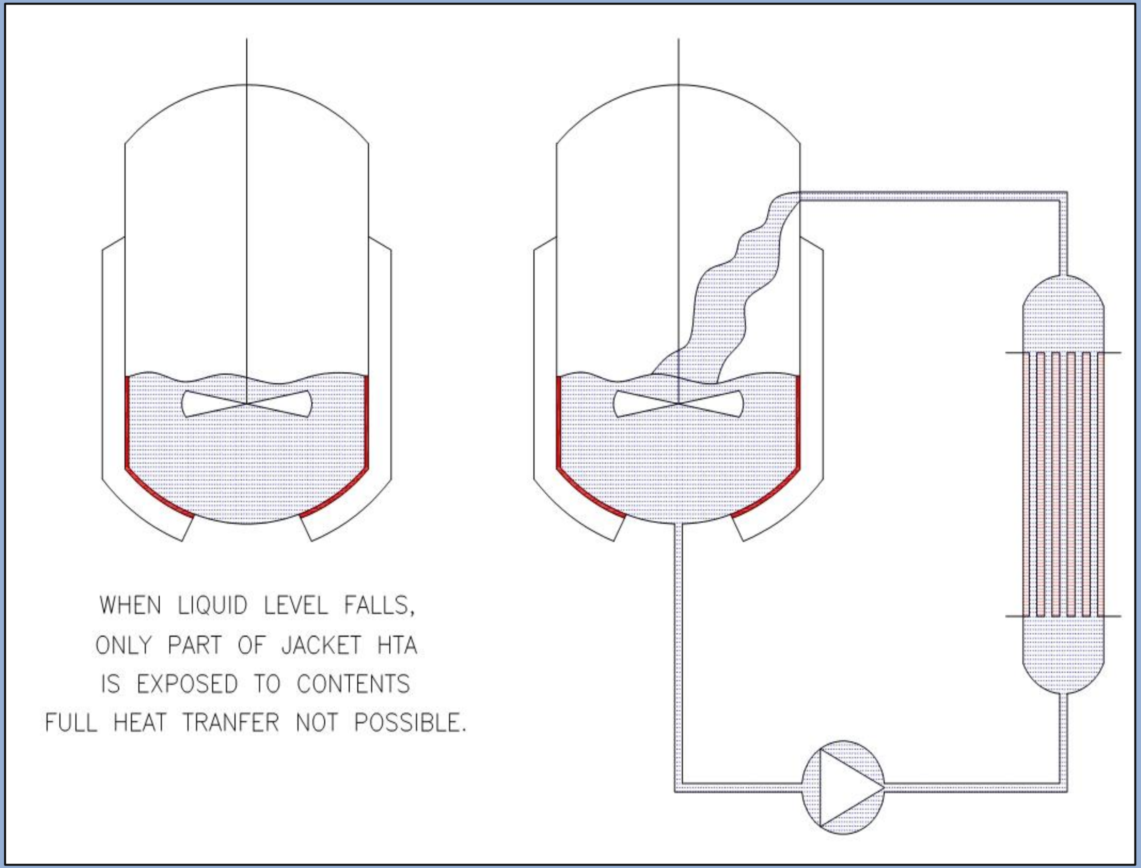


Case-6 In a batch distillation, vaporisation rate decreased as cycle progressed, resulting in sub-optimal performance.

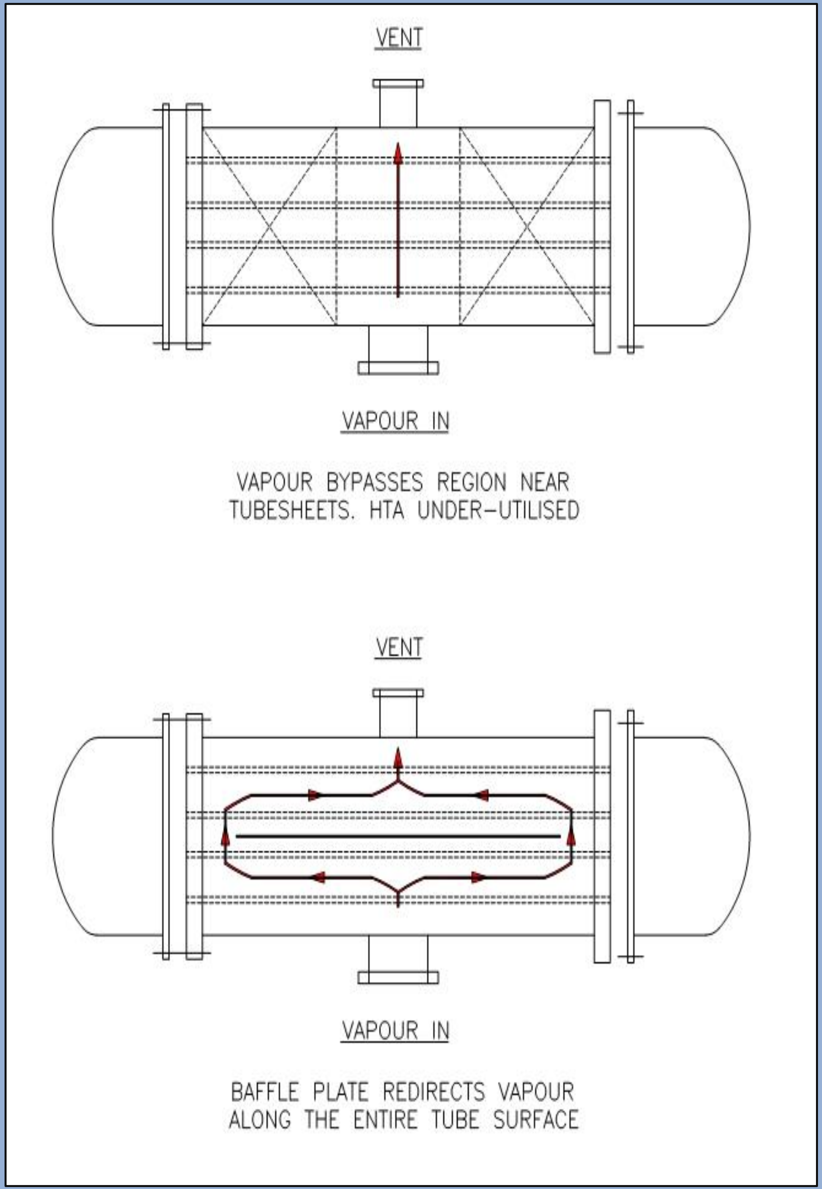
Diagnosis

As level of liquid inside the distillation still decreased, less of jacket area was exposed to heat transfer, resulting in lowered vaporisation rates.

Solution An external forced circulation reboiler was provided. The entire area of the reboiler could now be utilised - even when still level had dropped.



Case-7 A column-mounted condenser had adequate heat transfer area, but was not performing as per design.

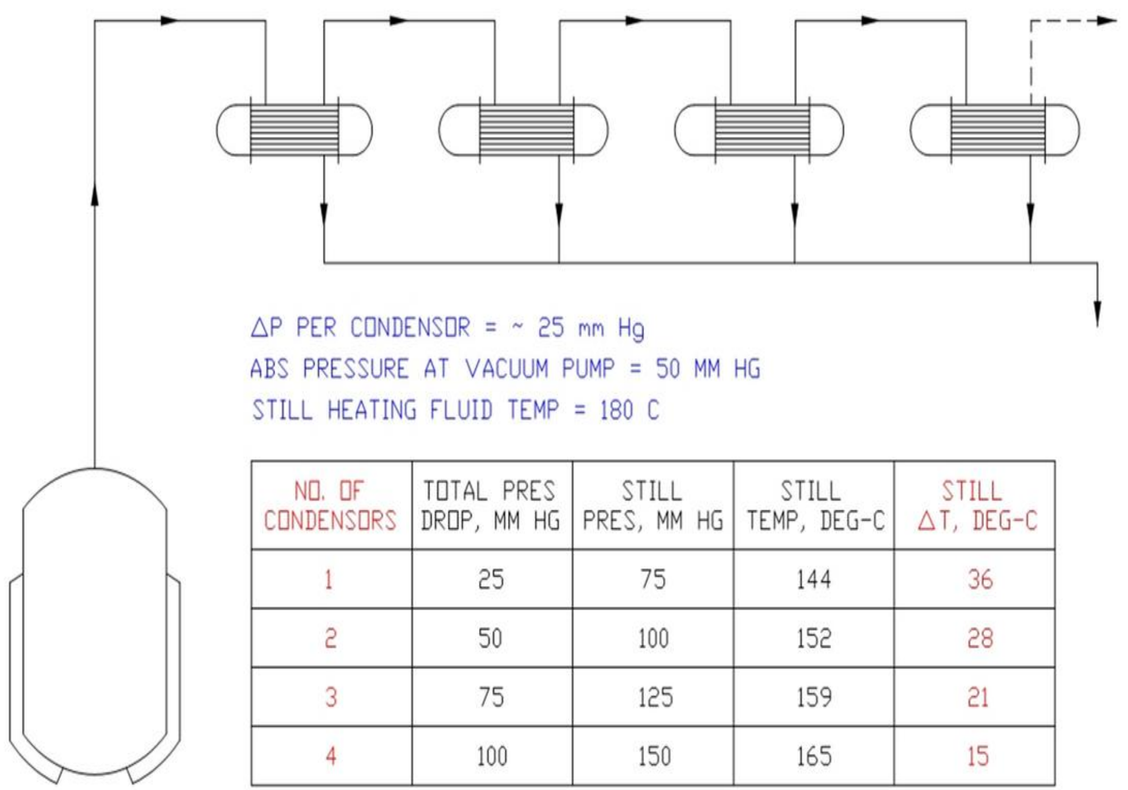


Diagnosis

Condenser vent nozzle was located right above its vapour inlet nozzle. Vapour was bypassing most of the heat transfer area, and going directly to vent.

Solution The condenser was replaced by another one which had a horizontal baffle. This redirected vapour so that it flowed over the entire heat transfer area.

Case-8



NO. OF CONDENSORS	TOTAL PRES DROP, MM HG	STILL PRES, MM HG	STILL TEMP, DEG-C	STILL ΔT, DEG-C
1	25	75	144	36
2	50	100	152	28
3	75	125	159	21
4	100	150	165	15

Problem Statement :

A vacuum distillation system was not giving an adequate rate. When condensers were added in series to increase total heat transfer area, recovery rates actually decreased.

Diagnosis:

Adding more condensers in series increased overall pressure drop. This increased still pressure and temperature, and thus, reduced ΔT and vaporization.

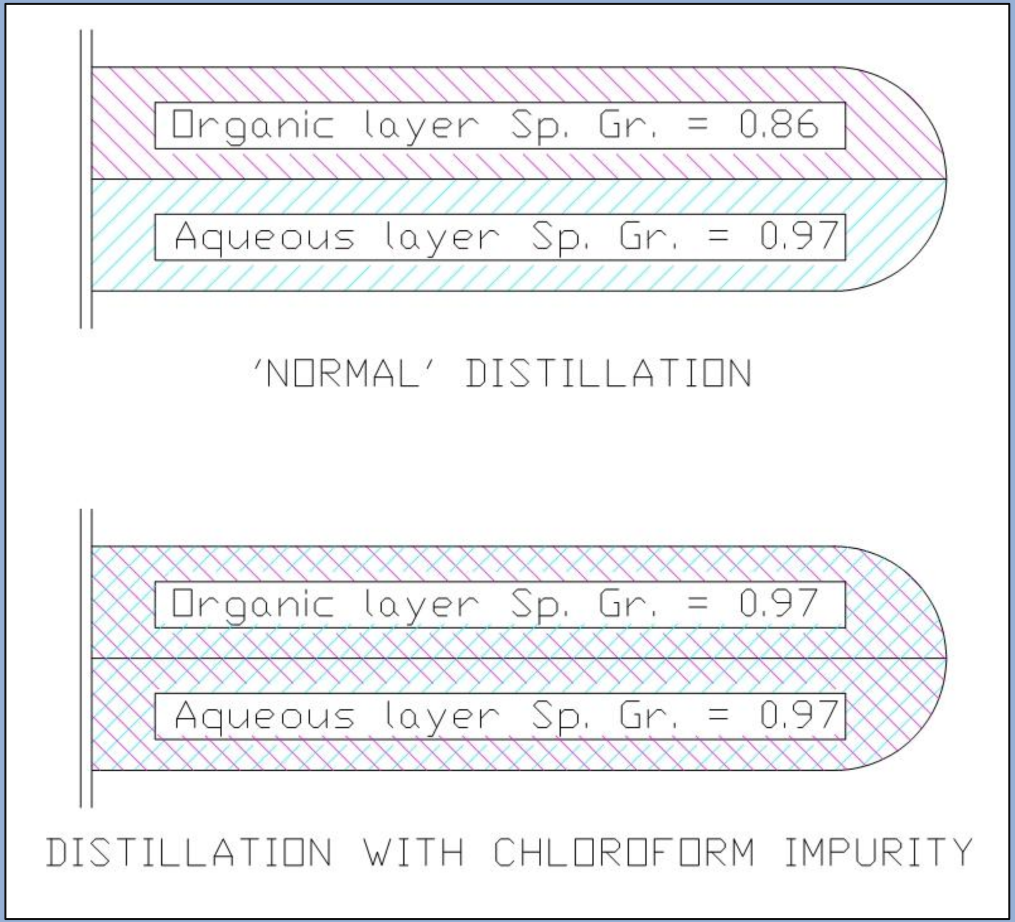
Solution:

The condensers were replaced by a single condenser with large heat transfer area, which had much lower overall pressure drop.

Case-9 During azeotropic distillation of isopropanol with cyclohexane entrainer, water layer did not form in the phase separator, hence water could not be removed.

Diagnosis

- The feed contained small amounts of chloroform, which has a low boiling point and is hydrophobic. So, it rose to column top and accumulated in the organic layer.
- Since density of chloroform is high, overall density of the organic layer increased until it became almost equal to the water layer.
- Consequently, the two layers could not separate, and remained intermixed.

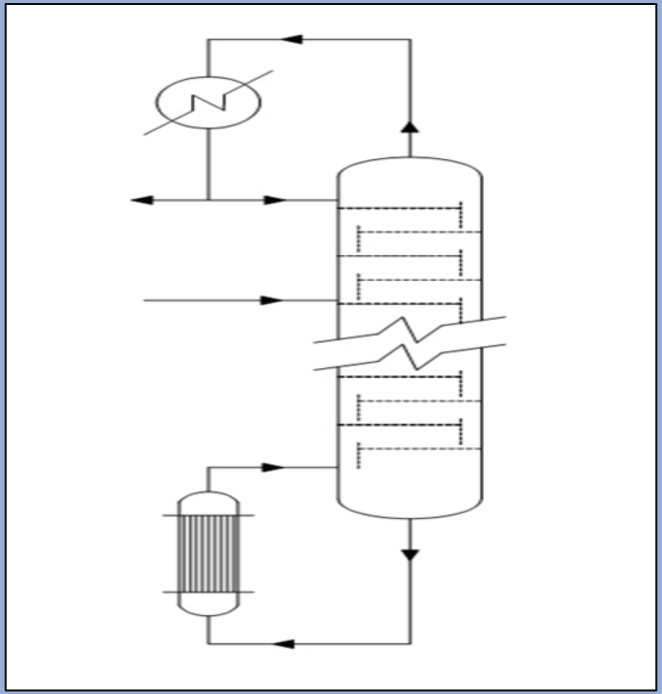


Solution The feed was pre-distilled to remove chloroform. Then, chloroform-free feed was distilled with cyclohexane entrainer to remove water.

Case-10 A bubble-cap tray column was being used for vacuum distillation of isomers. Vaporisation was inadequate and separation could not be achieved.

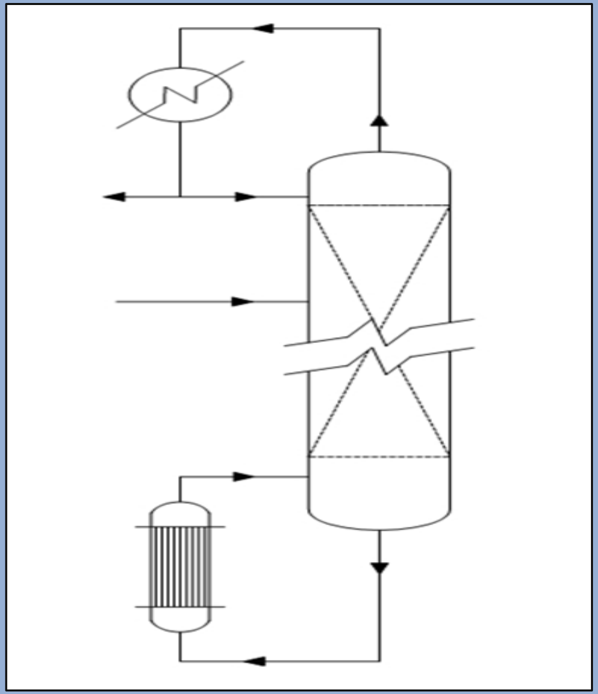
Diagnosis Bubble-cap tray column had high pressure drop. This resulted in a high bottom pressure, high bottom temperature, and thus lower temperature difference with the reboiler. Consequently, vaporisation was reduced.

Solution The tray column was replaced with a structured packing column. Column bottom pressure and temperature decreased, which permitted proper vaporisation.



TRAYED COLUMN

- No. of trays: 40
- Pressure drop per tray: 3 mm Hg
- Total pressure drop: 120 mm Hg
- Column top pressure: 50 mm Hg
- Column bottom pressure: 170 mm Hg
- Column bottom temperature: 155 deg-C



PACKED COLUMN

- Packed height: 8 mts
- Pressure drop per meter: 1 mm Hg
- Total pressure drop: 8 mm Hg
- Column top pressure: 50 mm Hg
- Column bottom pressure: 58 mm Hg
- Column bottom temperature: 122 deg-C

MISCELLANEOUS CASES	
CASE	DIAGNOSIS / SOLUTION
In a Dimethyl Sulfoxide (DMSO)-Water batch distillation, vent condenser was getting choked at the end of batch cycle.	Vent condenser was on chilled water, whose temperature was less than the melting point of DMSO (19 C). This caused it to solidify. Vent condenser was changed to cooling water with higher temperature.
There was an explosion while distilling a THF (Tetrahydrofuran)-water mixture, which had been stored for some time.	THF had formed peroxides during storage - which exploded. The mixture was treated with ferrous sulphate to remove peroxides,

MISCELLANEOUS CASES	
CASE	DIAGNOSIS / SOLUTION
A continuous column was flooding, starting at a point just above the feed.	Column was designed for feed at boiling point, but actual feed was superheated. It flashed on entering the column, increasing the vapour load.
Gas chromatograph indicated an impurity in the distillate which was not present in feed.	Another impurity in feed (acetic acid) was reacting with the distilled solvent (ethanol) to produce ethyl acetate – and that showed up in the distillate.

MISCELLANEOUS CASES

CASE	DIAGNOSIS / SOLUTION
In a DMF-water batch distillation, AF-120 gaskets were provided. The gaskets at column top were getting damaged, but those at column bottom nozzles were not damaged.	The feed had small amounts of toluene, which accumulated at column top and damaged top gaskets. But, toluene concentration at bottom remained low, and bottom gaskets were not damaged. The AF-120 gaskets were replaced with PTFE gaskets.

THANK YOU