

CH industrial technologies

Distillation, desalting

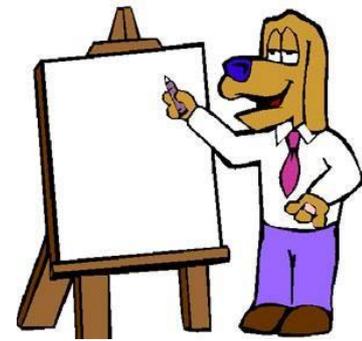
Klára Kubovicsné Stocz

Oktober 2023



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Agenda



Distillation

Operation parameters_effect

Crude distillation

Analytical method(feed, product)

CDU units/operation parameters

Desalting

Internals at distillation columns

Distillation

Traditional, simple distillation

- column with 2 products:
feed, top/bottom product
condenser/reboiler

What influences the optimal operating parameters ?

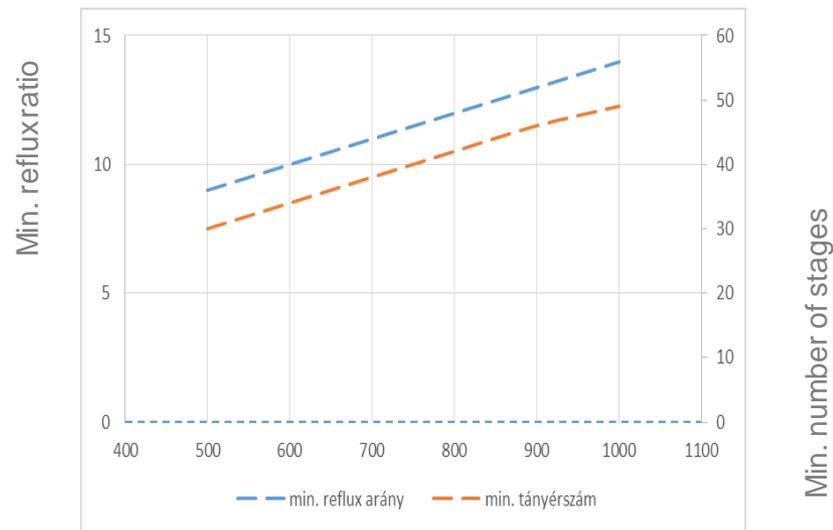
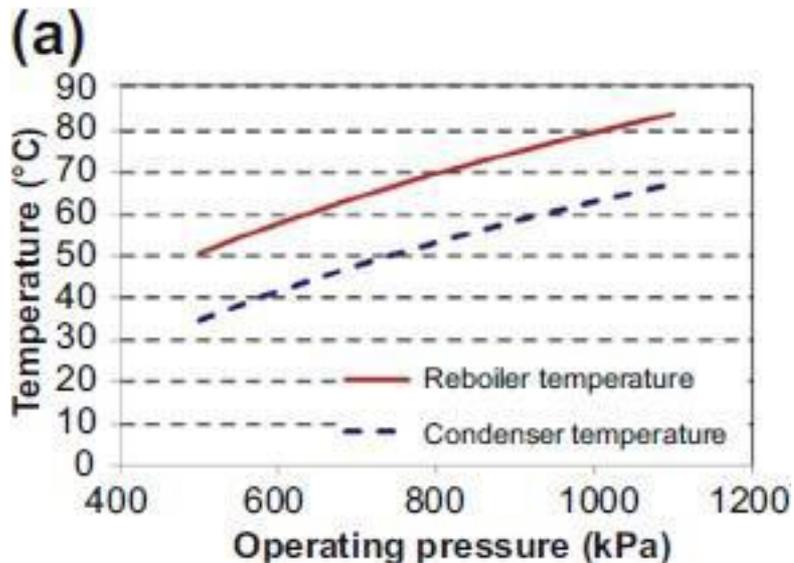
(planning/degrees of freedom)

- operation pressure/pressure drop
- Feed inlet temperature/pressure
- Theoretical tray number/ tray efficiency
- Place/position of feed
- Type of condenser
- Type of reboiler

(the quantity and quality of the feed and sharpness of the separation are defined)

These parameters have significant effect on the heating and cooling energy demand of the separation.

Operation pressure of distillation column



distillation operating pressure affects the temperatures of heating and cooling. The bubble point and dew point temperatures of a mixture of a given composition depend strongly on pressure.



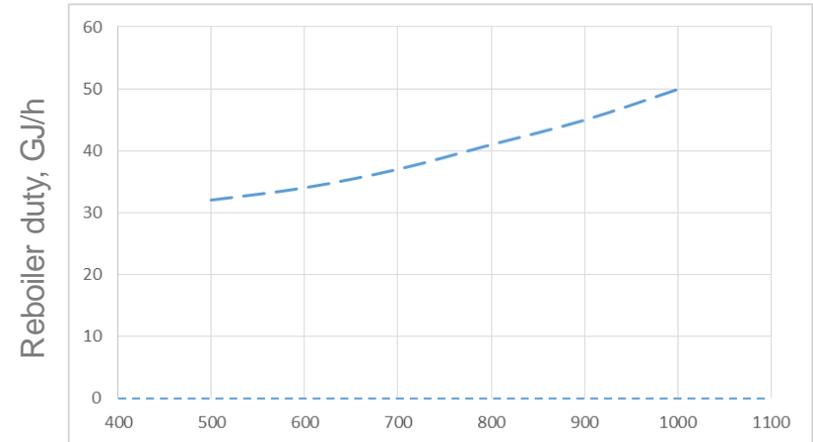
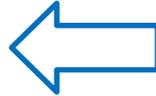
pressure
Minimum reflux ratio and minimum number of stages both increase with pressure.
 With a higher operating pressure, the capital cost of the column will increase.



Temperature increase with increased pressure.
 the components in the mixture typically become more similar in volatility and their separation becomes more difficult,
 the distillation column will need more reflux and more theoretical stages to compensate it

Column operating pressure

The increase in reflux resulting from the pressure increase is likely to increase the reboiler and condenser duties



pressure, kPa

The logical conclusion is that it is **best to operate at atmospheric pressure unless there are good reasons not to do.**

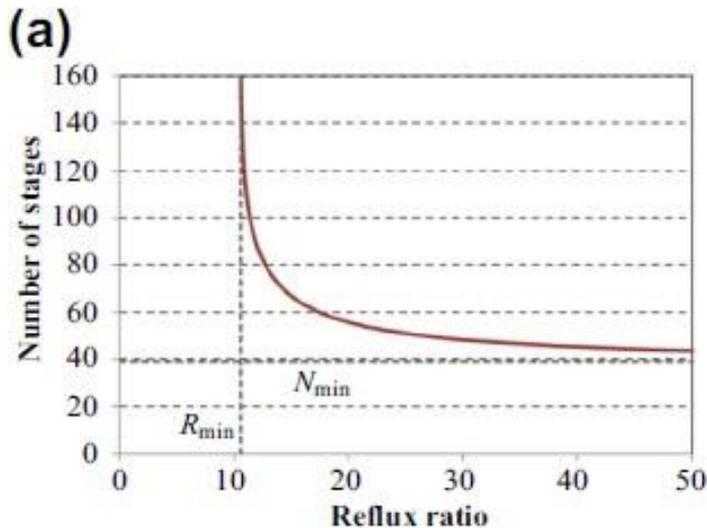
In particular, if:

1. increasing the operating pressure allows to avoid refrigeration (cooling) or help the use of refrigeration at more moderate conditions;
2. operating under a vacuum avoids degradation of thermally sensitive materials because of the lower temperatures in the column;
3. changing the pressure (up or down) creates an opportunity for heat recovery within the wider process
4. the cost of higher pressure feed (to meet downstream specifications) outweighs the benefits of increased operating pressure.

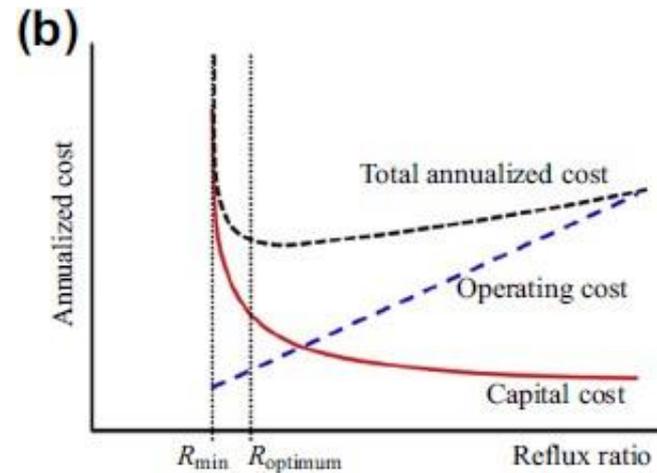
Number of theoretical stages

The number of theoretical stages required to carry out a specified separation in a simple distillation column.

A key design decision is to select the number of stages in the column.



For different numbers of stages, different reflux ratios will be required, corresponding to different reboiler and condenser duties.



There is a **trade-off between operating costs and capital investment** - increasing the number of stages would increase the height of the column and therefore its cost, but would decrease reflux requirements and hence reduce duties and operating costs, as well as costs of heat transfer equipment.

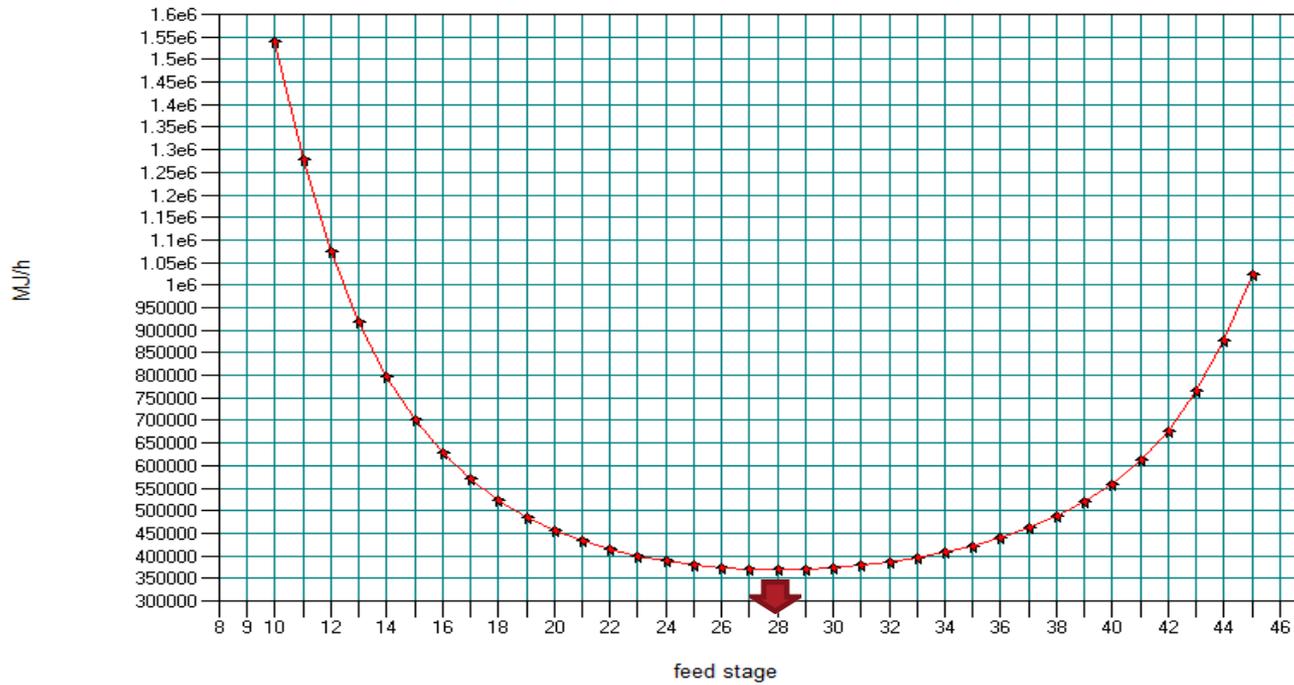
Feed stage location

If the feed composition or temperature are very different to those on the feed stage, the mixing of the feed with the material within the column disrupts the composition profile in the column. These “mixing effects” are thermodynamically inefficient and cause the heating and cooling duties of the column to increase.

On the other hand, if the composition and temperature of the feed and the feed stage are similar, then the feed almost not influences the mass transfer taking place on the feed stage, which is more energy efficient.

Location of the feed stage

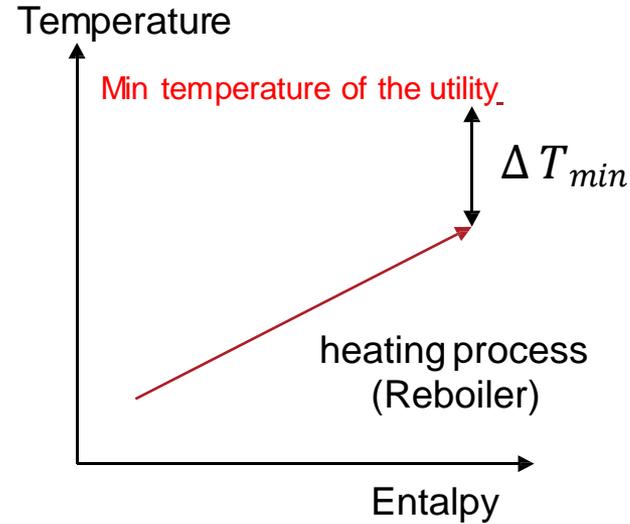
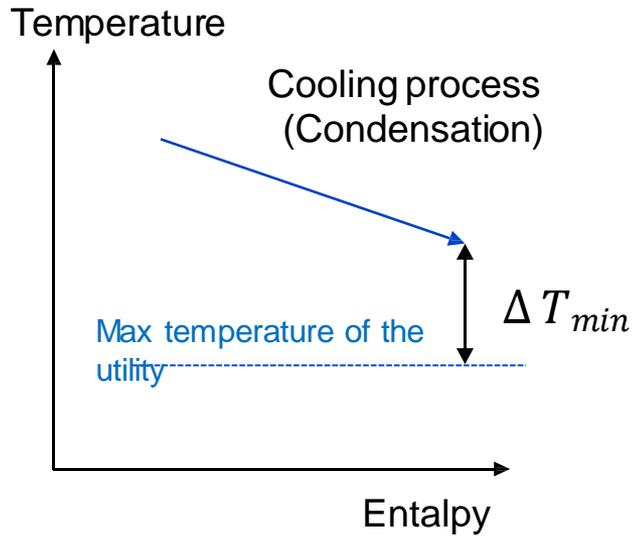
feed tray



★ reb duty

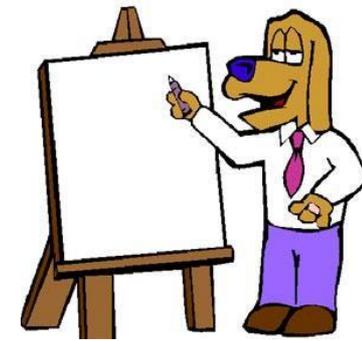
If we know the number of stages, the optimal feed stage can be defined.
Minimum duty has to define at defined product quality

Heating- and cooling medium(Utility) temperature



The temperature demand of the utility stream is determined by the level of the condenser and reboiling temperature.

Agenda



Distillation

Operation parameters_effect

Crude distillation

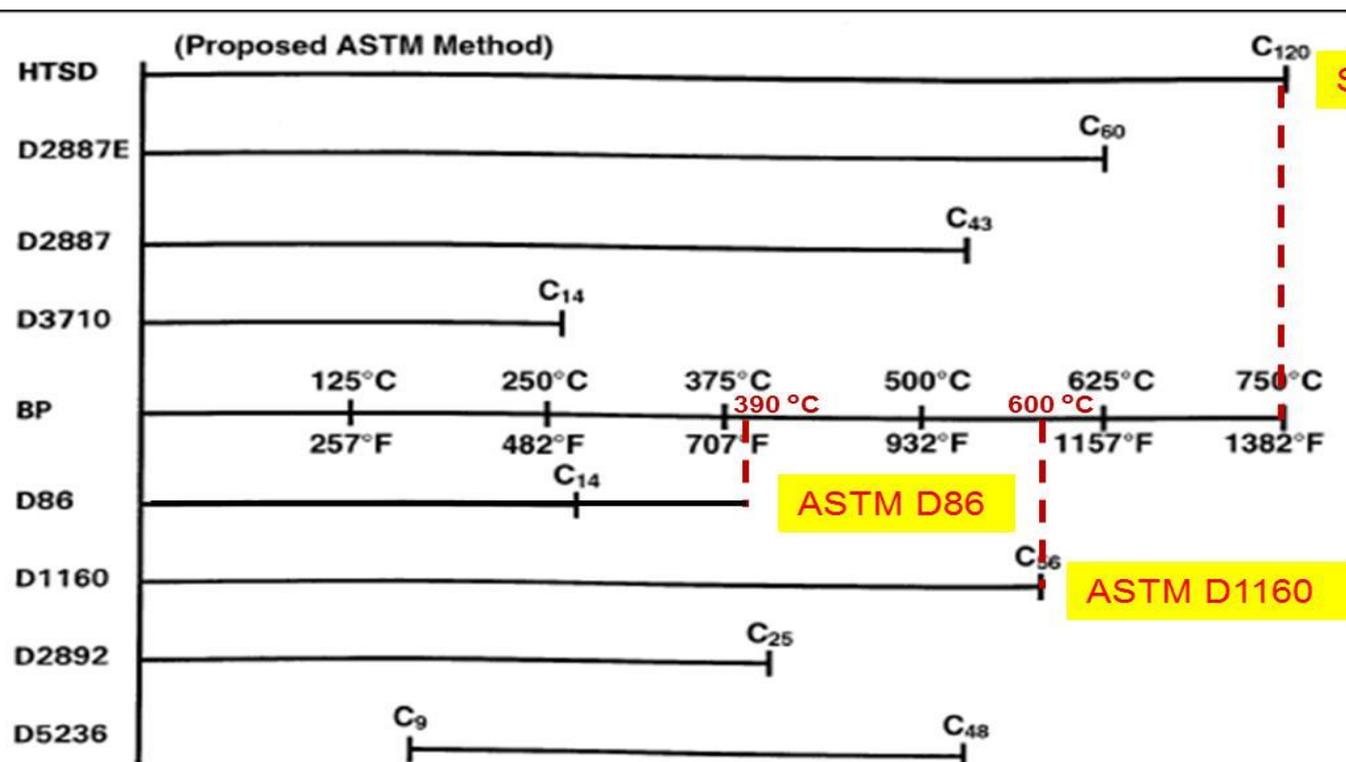
Analytical method(feed, product)

CDU units/operation parameters

Desalting

Internals at distillation columns

Analitical methods vs. Boiling point



SIMDIS HT 750

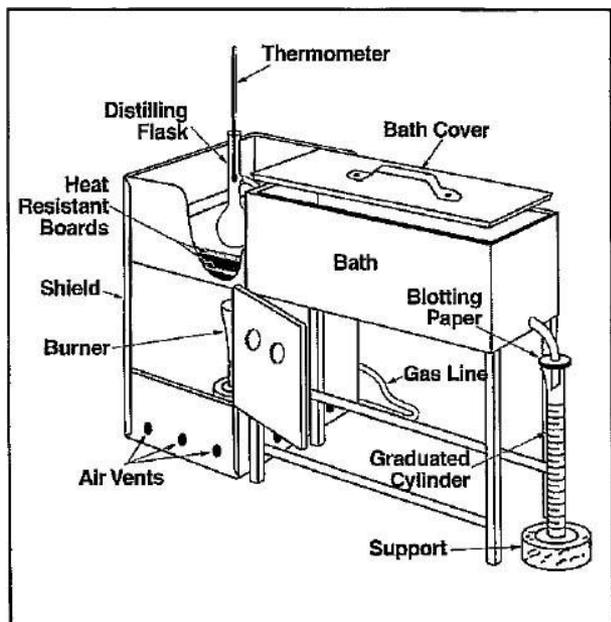
ASTM D86

ASTM D1160

ASTM and SIMDIS methods application range
(boiling point vs n-paraffin carbon number)

Engler desztilláció (ASTM D86)

- Kezdő forráspont:** az első csepp megjelenésekor leolvasott gőztéri hőmérséklet, normál légnyomásra korrigálva, °C
- Végő forráspont:** az a hőmérséklet, amelynél még szedünk párlatot és a hőmérséklet nem csökken, °C
- Átdestillált mennyiség:** a végő hőmérsékletnek megfelelő desztillátum mennyisége, ml
- Maradék:** a lombikban maradt anyag mennyisége, ml
- Veszteség:** (bemért – átdestillált – maradék) anyag mennyiség, ml

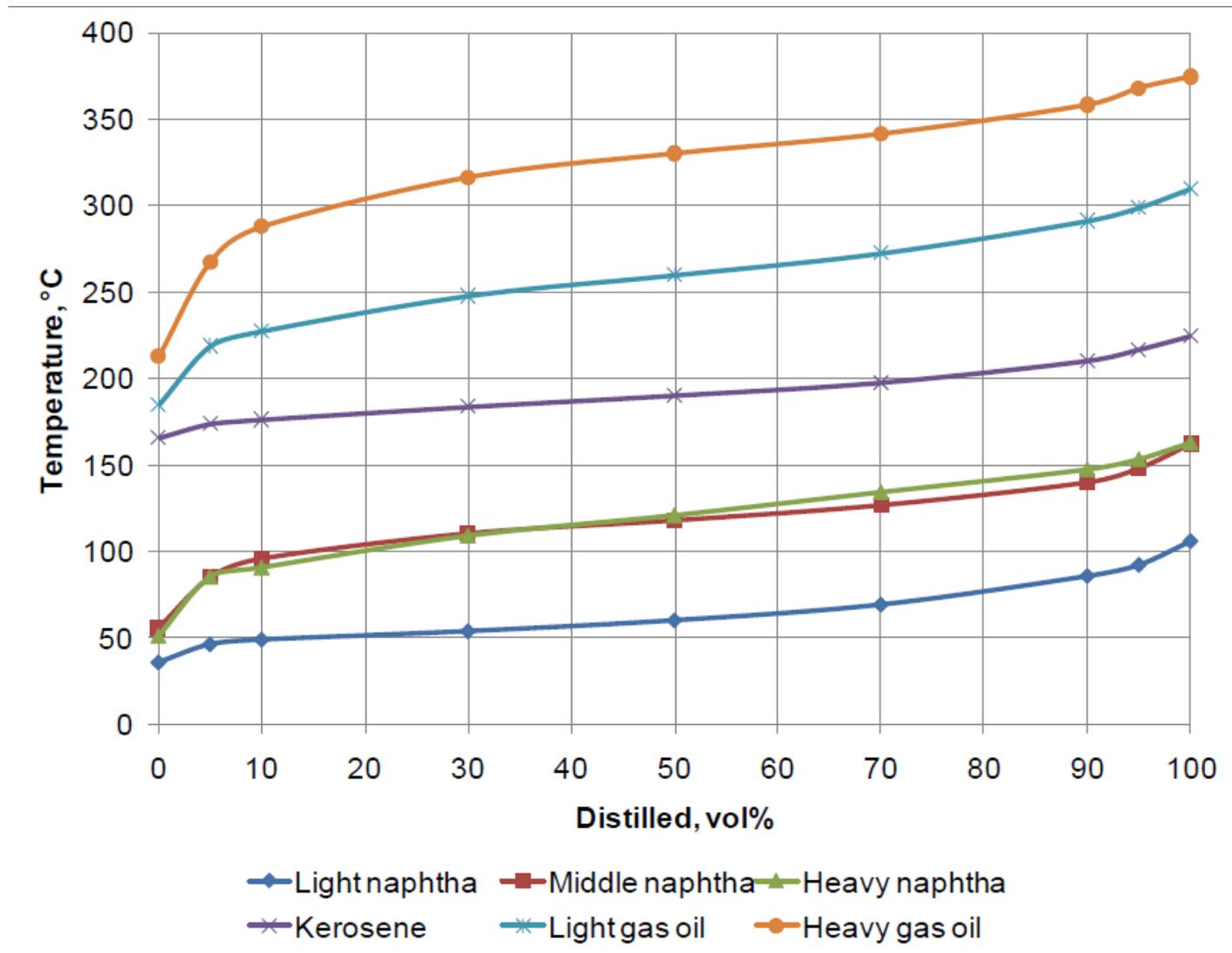


Térfogat alapú mérés

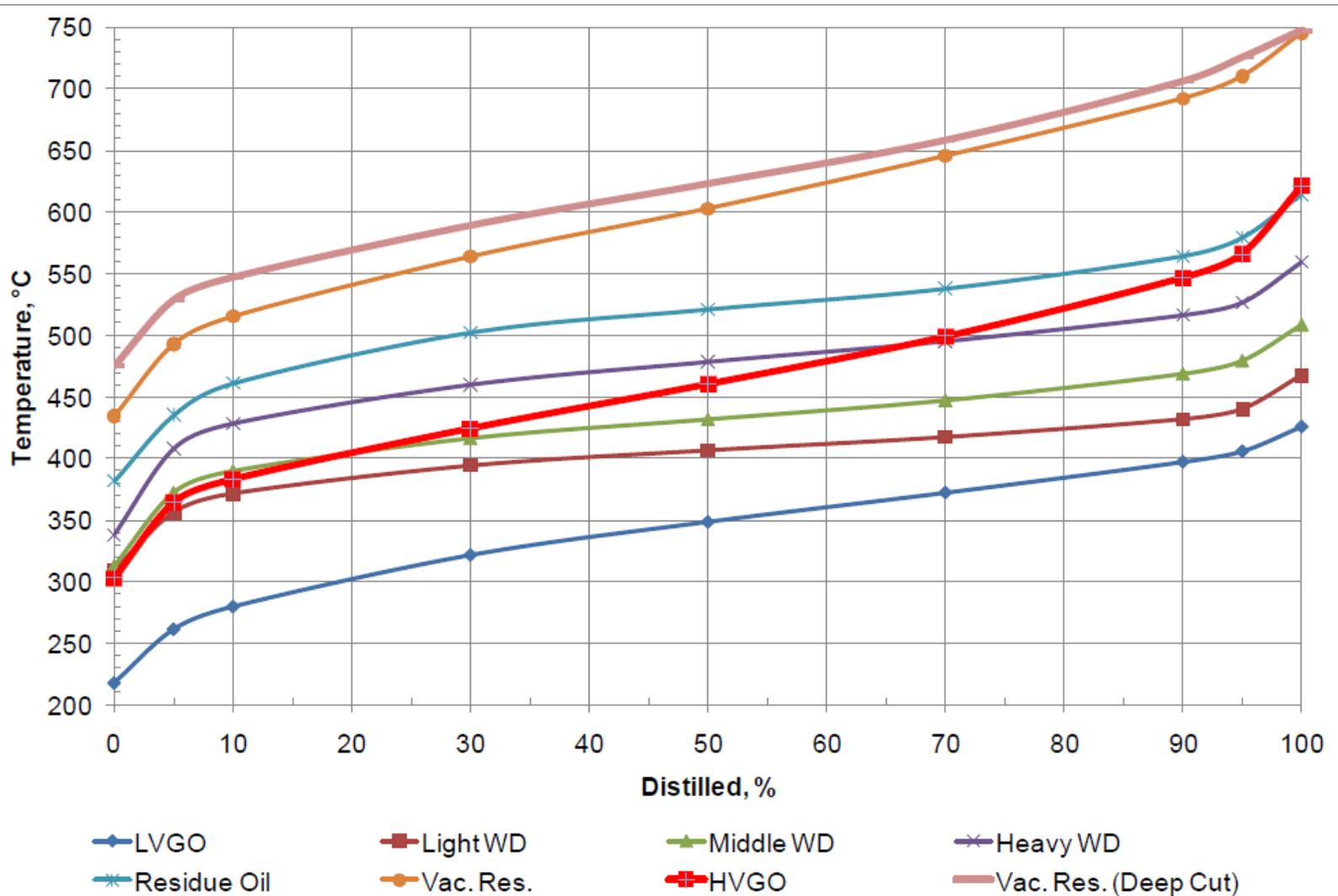
Elpárolgott mennyiséget mérik hőmérséklet függvényében

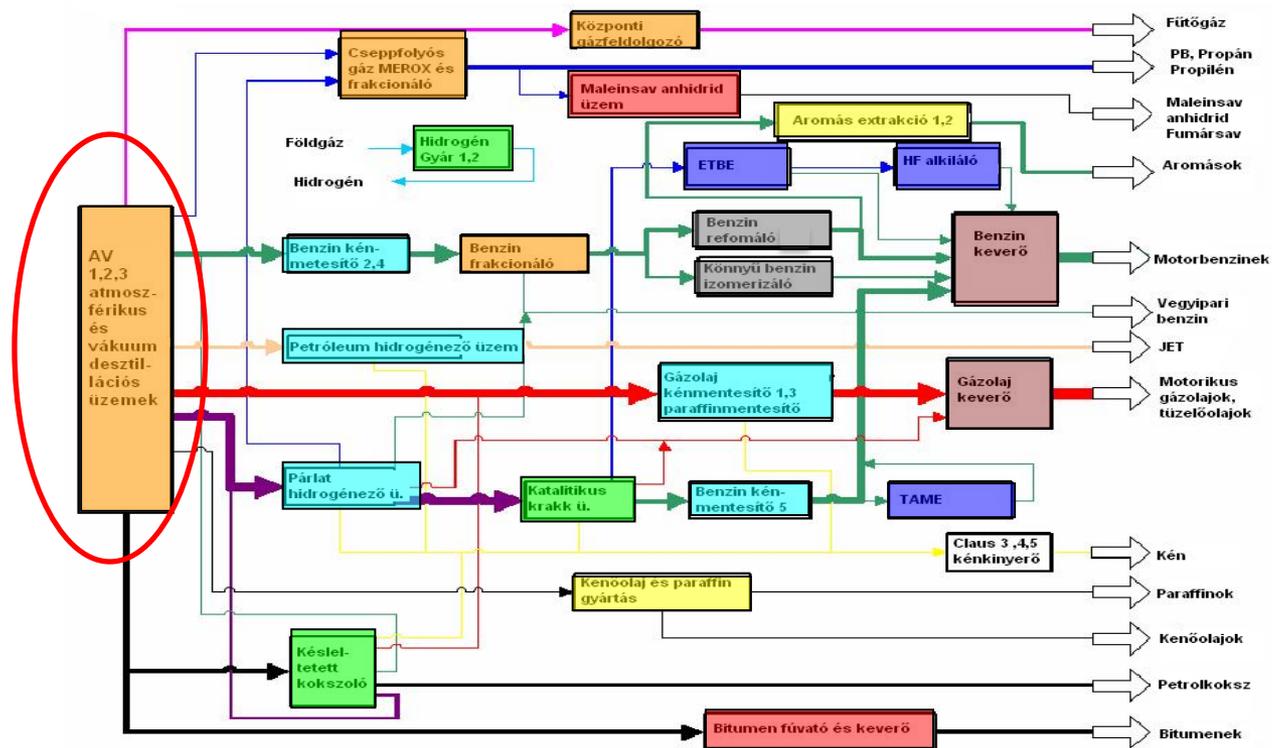


Atmoszferical product distillation curves, ASTM D86



Vacuum product distillation curves, SIMDIS HT-750





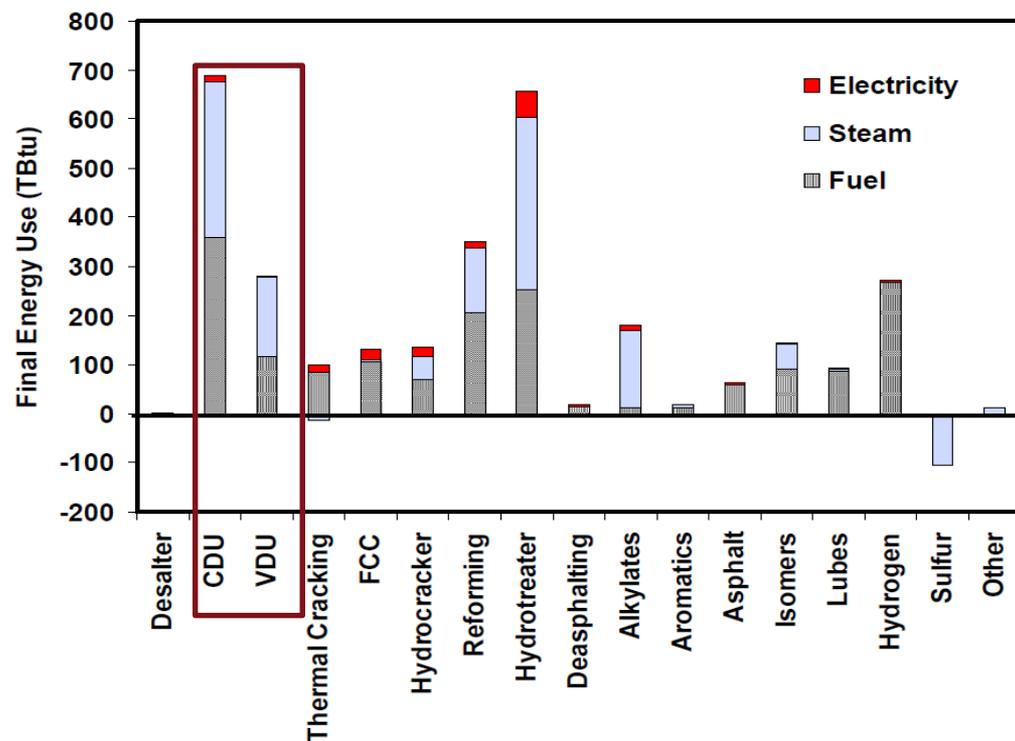
- | | | | |
|---|---|--|--|
| Desztilláció | Konverzió | Oxidáció | Addíció |
| Kéntelenítés | Oldószeres finomítás | Oktánszám növelés | Keverés |

*DUF: Danube refinery

Forrás: MOL

Energy demand of distillation

- ▶ Distillation technology is one of the largest consumers of energy in the oil refining process
- ▶ The design and operation of an efficient equipment requires the presence of relevant physico-chemical knowledge



Difference between Crude distillation units

Configuration

- Integrated or separated atm and vacuum units
- Number of Distillation columns
- Number of Internal circulation reflux (PA)

Operation

- Type of processed crude
- Product yields
- Temperature(preheating-line outlet temp., furnace (inlet/outlet), Drawing temperture)
- pressure
- Steam is used to strip or not (wet vs. dry)

Energy efficiency

- Heatintegration
- Separation efficiency
- Status of furnace and/or heatexchangers

Crude distillation at Danube Refinery



CDU1: - Lube-
production feed

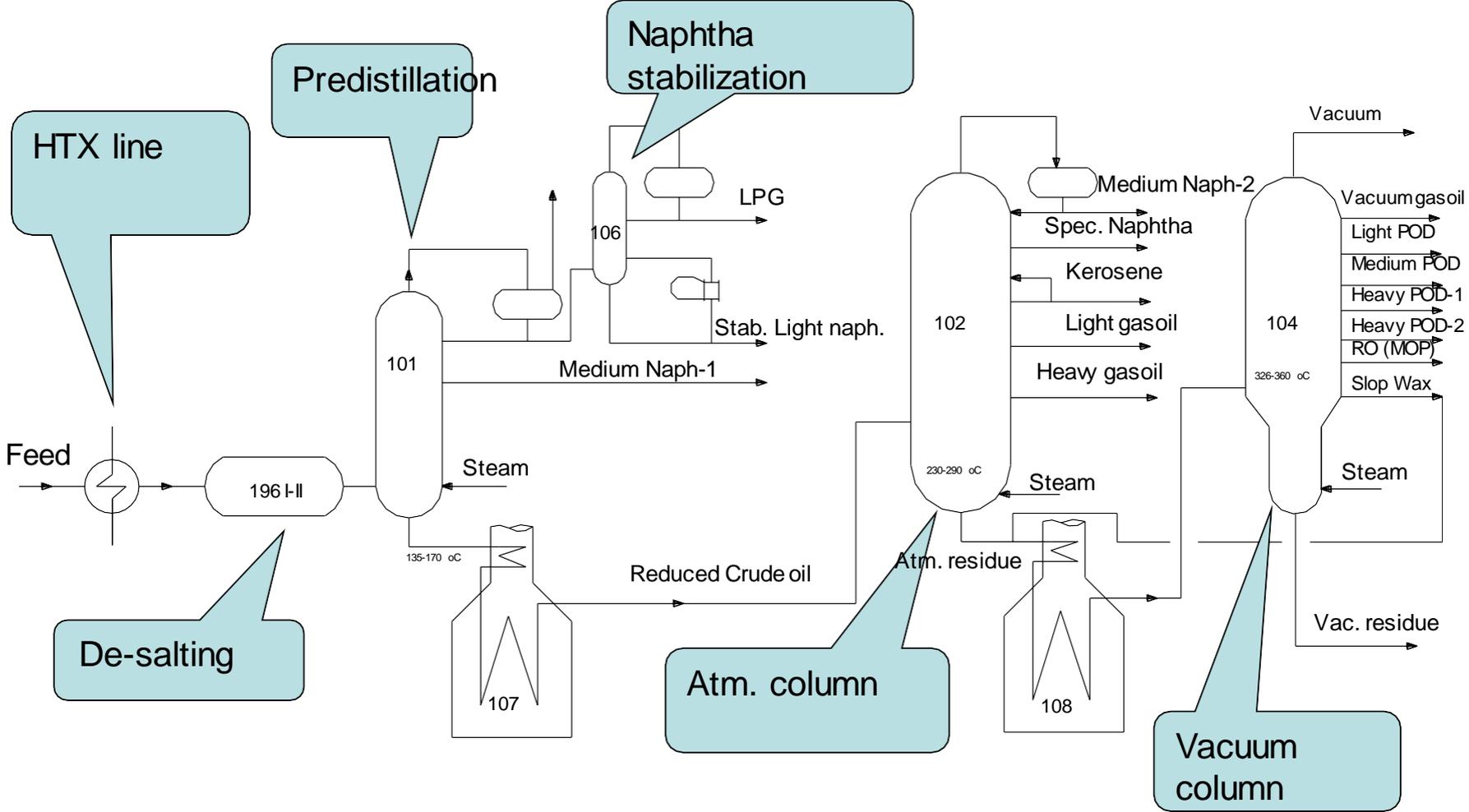


CDU2: - motorfuel and lube
production feed



CDU3: - motorfuel
production feed

Main part of CDU units



PROCESS STEPS – CRUDE PROCESSING

The crude oil distillation generally involves the *following process steps*:

- crude pre-heating
- desalting
- pre-flash of the unstabilized light naphtha
- atmospheric distillation
- vacuum distillation
- naphtha stabilizer



Main part of CDU unit

Crude pre-heating



Two main part:

- before desalter:

till 120-140 °C is heated the crude, 2 or 4 parallel line

- After desalter:

To 170-180 °C is heated the crude

Heating medium:

products and pumparounds

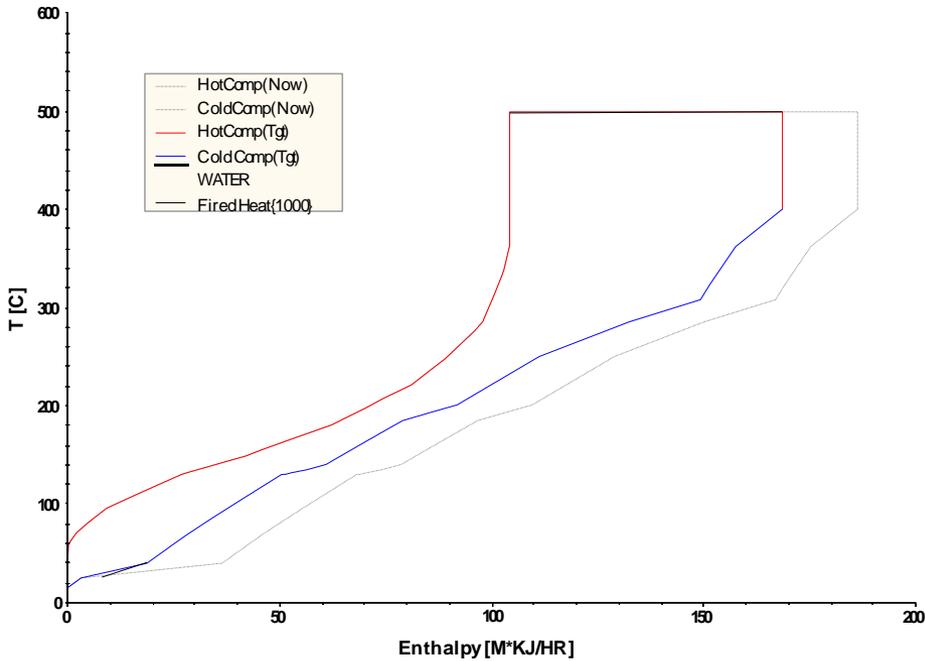


Crude pre-heating

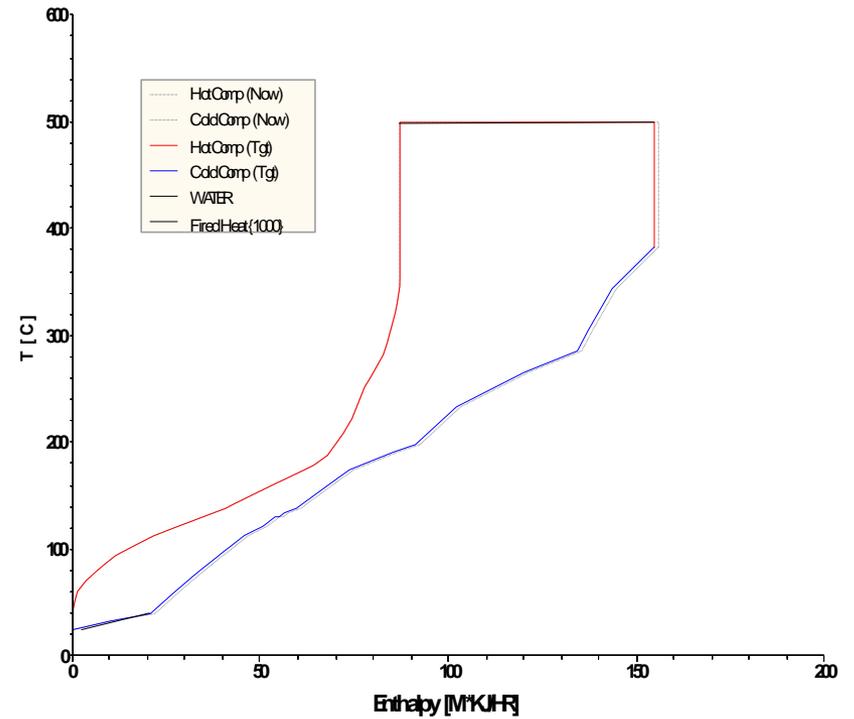


Heatintegration, pinch analysis – kompozit curve

% ToSave e[\$]=21,8%, QNow_H =82,1, QNow_C =28,5



%ToSave e[\$]=17%, QNow_H=68,8, QNow_C=19,7



Desalter



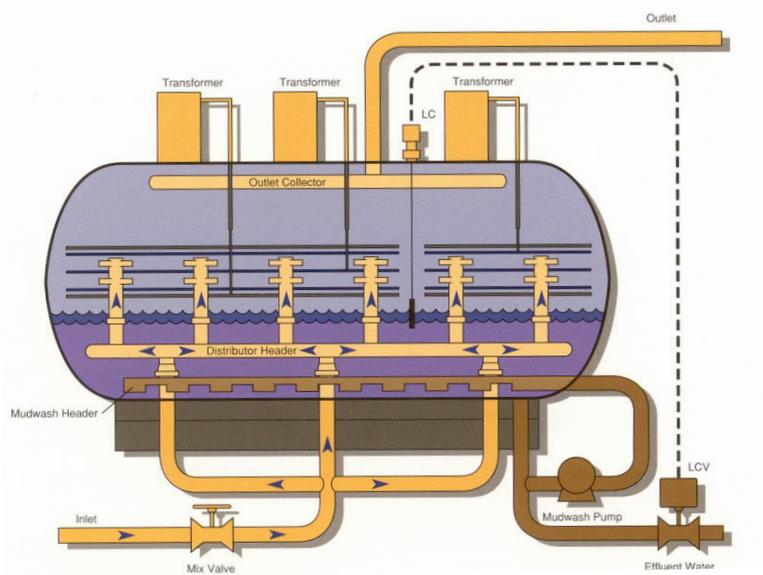
De-salter

Function:

Crude salt- and water removal

The desalting is a key preparation step for crude oil separation and refining process

Why?



- Fouling in Furnaces or HTXs
- Corrosion at top product lines, condensers, pipes (CaCl_2 , MgCl_2 , NH_3 ,)
- Fouling, plugging in vac furnace (Na)
- Shorter operation period in VB unit
- Catalyst poison (Na)
- Energy demand (water)

Effluent water treatment

- oil /grease contamination
- solid contamination

Desalter

Contamination	effect
water	Capacity decreasing Energy demand encresing
salt	Corrosion deposit/plugging Catalyst poison
solid	plugging erosion Rag Layer Stability
Surface Active Agents	Rag Layer Stability Poor Control Chemical Costs

Desalter

- **Salt content of crude oils consist of mainly chlorides:**

: NaCl 70-80 wt %

MgCl₂ 20-10 wt %

CaCl₂ 10 wt %

• Small amounts of sulfates and carbonates

Salts are either in the form of **crystals** or **ionized** in the **water** present in the crude

Crystals can be eliminated by washing with water because they are ionized and hydrated → increase water solubility (temperature !)



Generally accepted rule: the chloride content of the overhead condenser water should not exceed 10 ppm, otherwise serious corrosion may occur.

Desalter



Solid contamination	
<u>Classification</u>	<u>Composition/type</u>
Basic Sediments (20-200 Microns) <i>alap</i>	1) Derived from producing/mining <i>sand, mud(silt), clay</i>
Filterable Solids <20 Microns <i>Szűrhető</i>	2) Corrosion agent <i>Fe sulfate, FeOx</i> 3) Scales <i>Sulfates, carbonates</i>

Desalter

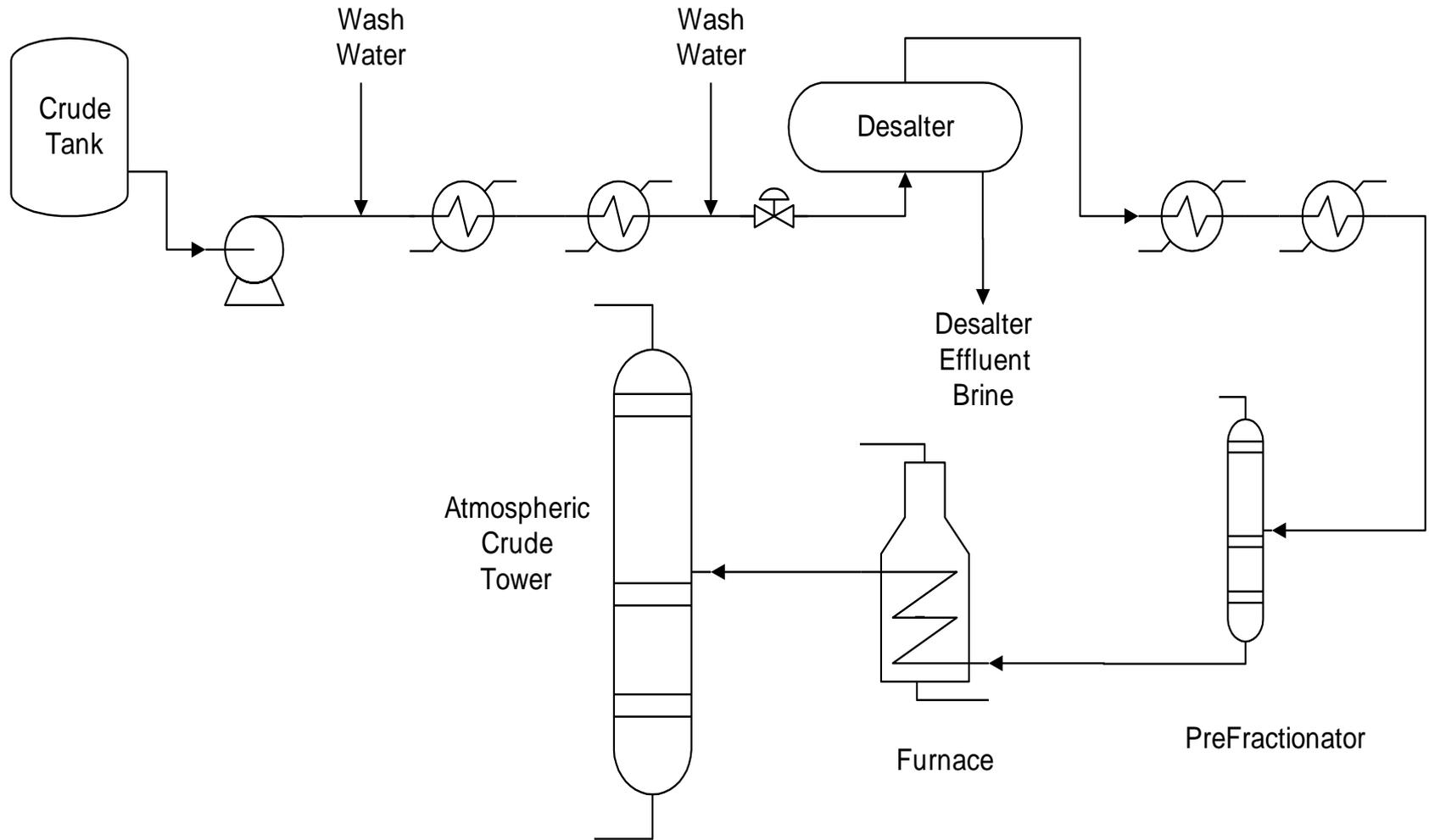
What type of impurities can be removed from crude in desalter:

- water soluble salts: approx. 90-99%
- solid contamination: approx 50% (depends on crude and contamination)

What type of contamination NOT removed from crude in desalter:

- organic metal salt
- naphthenic acid

Desalter



Desalter

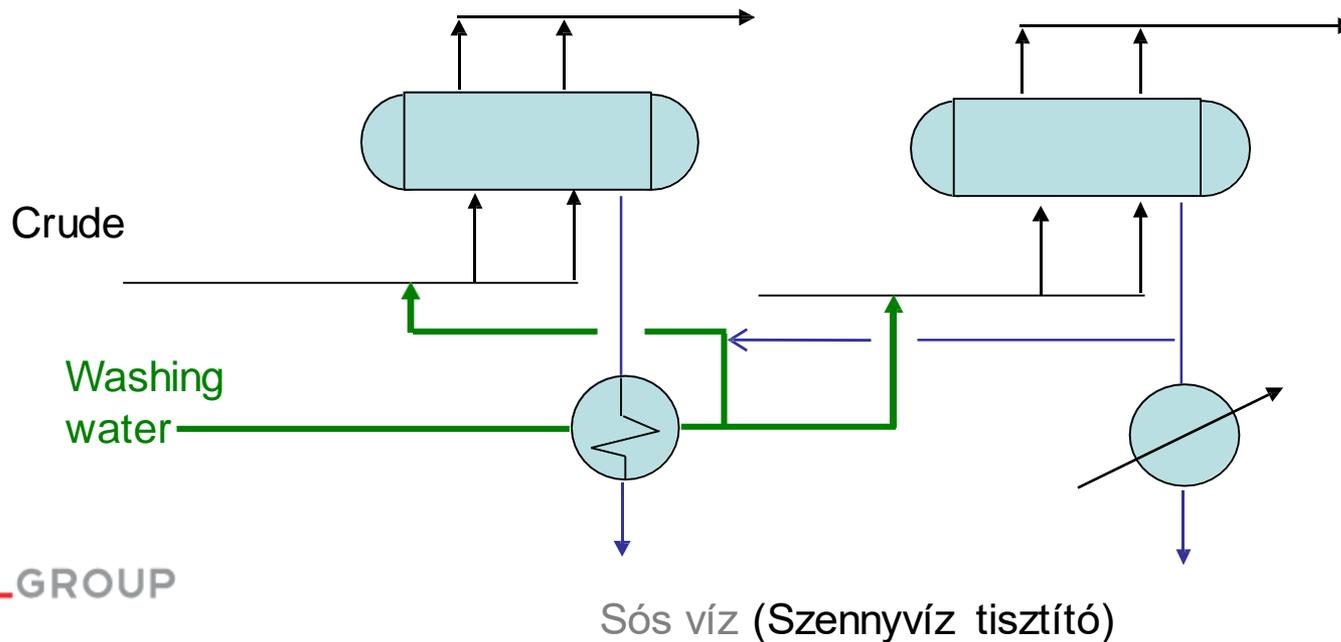


Two steps de-salter operates in DR CDU units

Average salt content of crude: 20-40 ppm

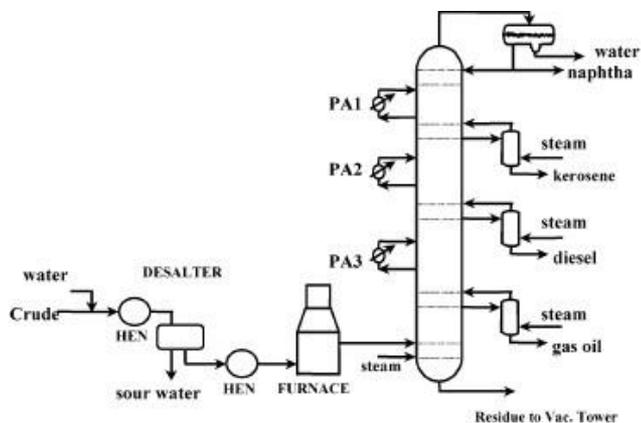
Aim: <4 ppm

Less water request

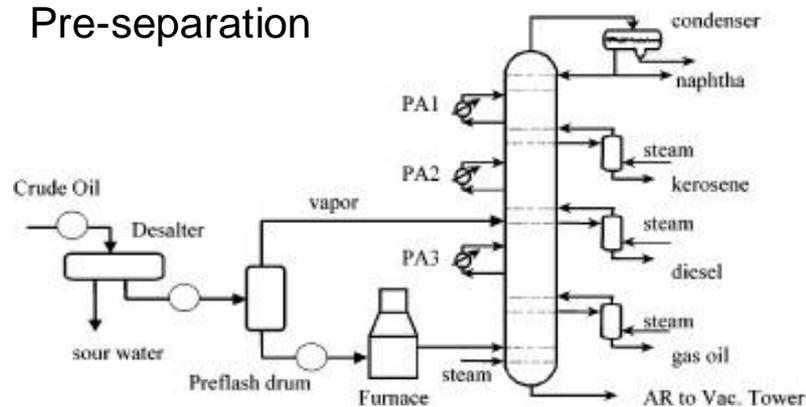


Crude distillation

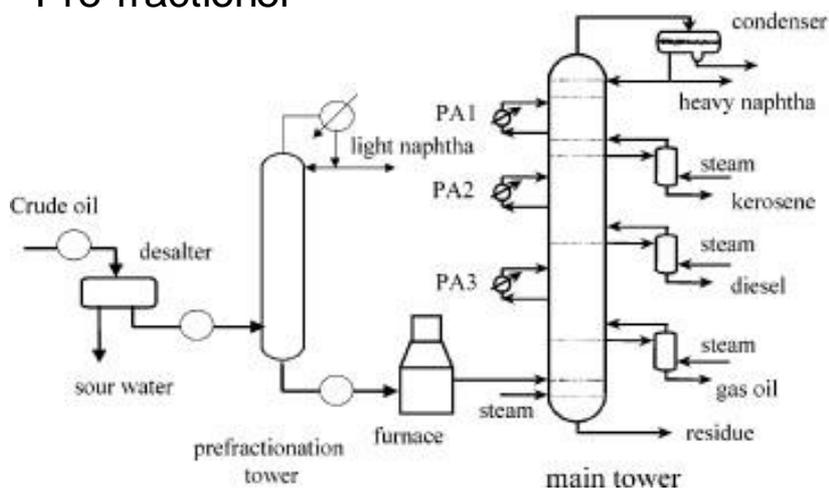
conventional



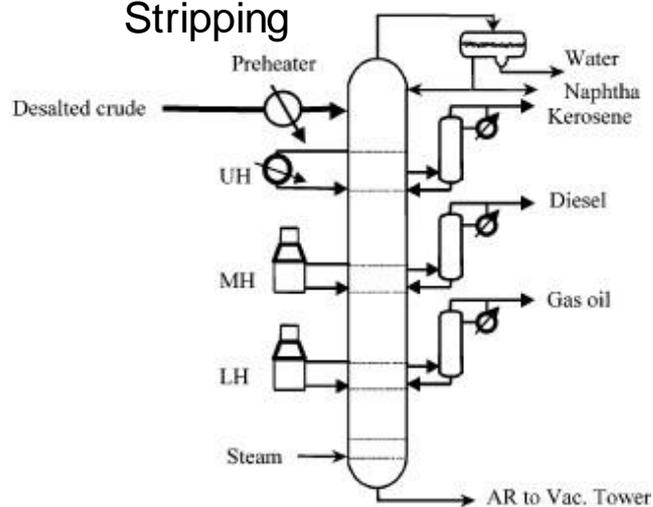
Pre-separation



Pre-fractioner



Stripping



What is the function of pre-flash column?

Pre-flash column is one of the major part of a Crude Distillation Unit.

As the name suggests, its job is to flash, that is to vaporize the lighter (volatile) portion of the crude oil before it enters the atmospheric heater. The basic principle of this vaporization is the sudden decrease of pressure from around 4 bar to 1 bar (in our case). Which makes a large chunk of the volatile portion of the crude to get vaporized and is directed to the main distillation column bypassing the furnace.

The remaining heavy portion is heated in the furnace and finally introduced in the main distillation column.

The advantage of having the pre-flash drum is to reduce the load in the furnace resulting in saving of Fuels.



PRE-FLASH DRUM VS. PRE-FLASH COLUMN

Pre-flash drum reduces downstream exchanger network and heater flow rates, but the atmospheric column and its overhead system needs sufficient capacity to process the vapours.

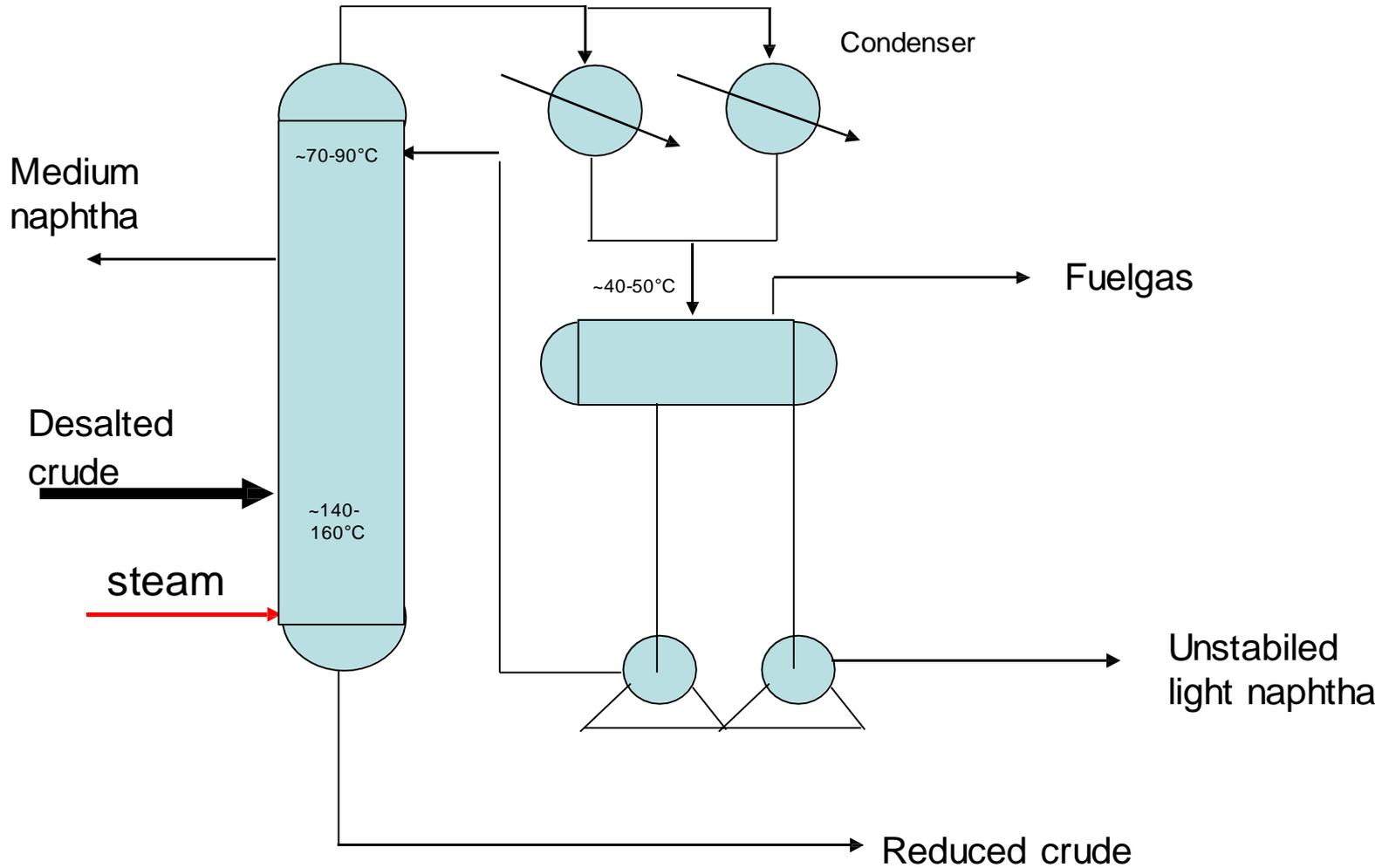
Pre-flash column reduces the vapour flow rate through the atmospheric column. If it has its own overhead system, it also reduces atmospheric column condenser drum and overhead pump loads.

Drum or column sizing depends largely on flashed crude liquid rate and its composition. If sharp separation is needed between the naphtha and kerosene the pre-flash column is the right choice.



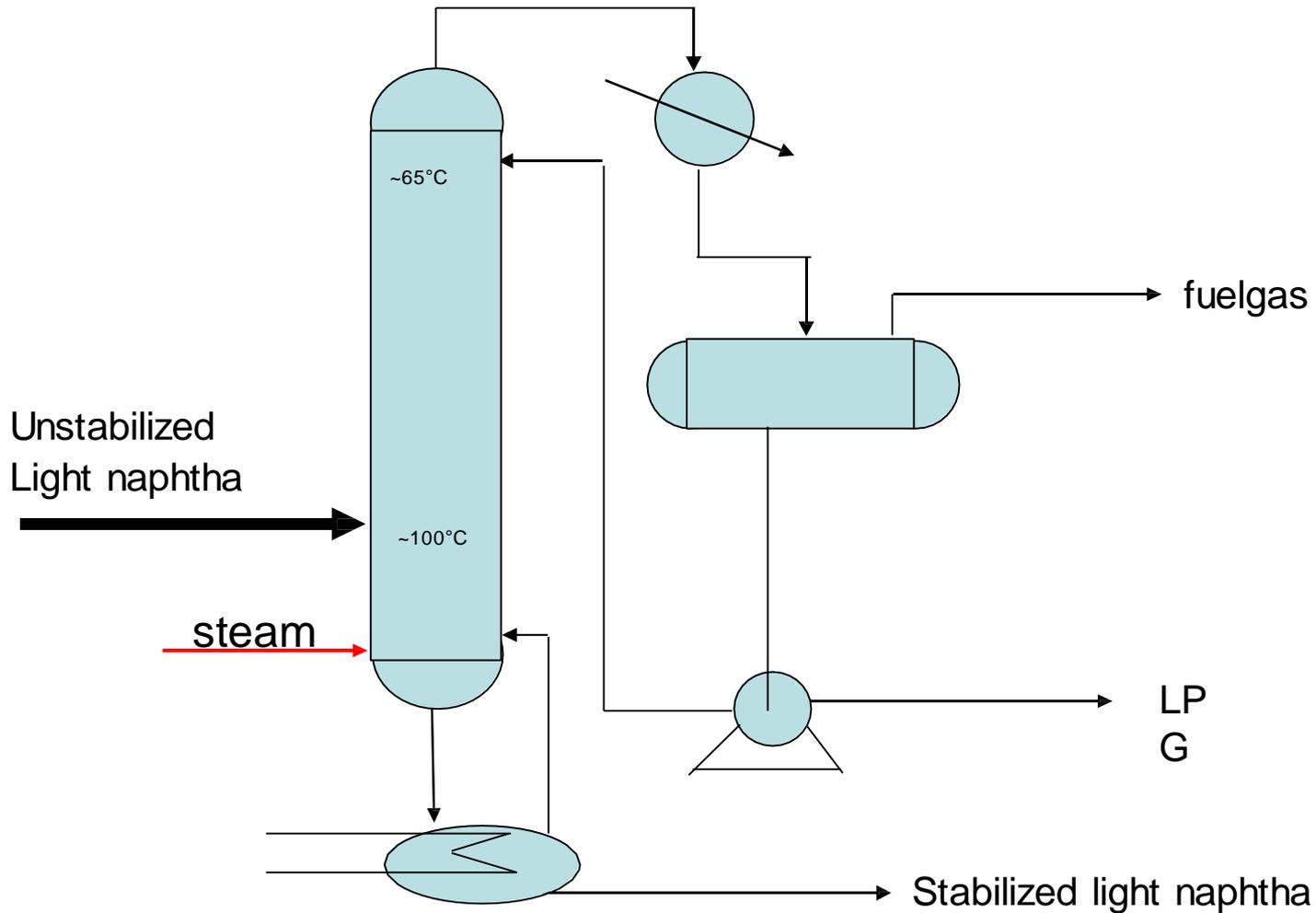
Pre-distillation

Aim: remove light components from crude



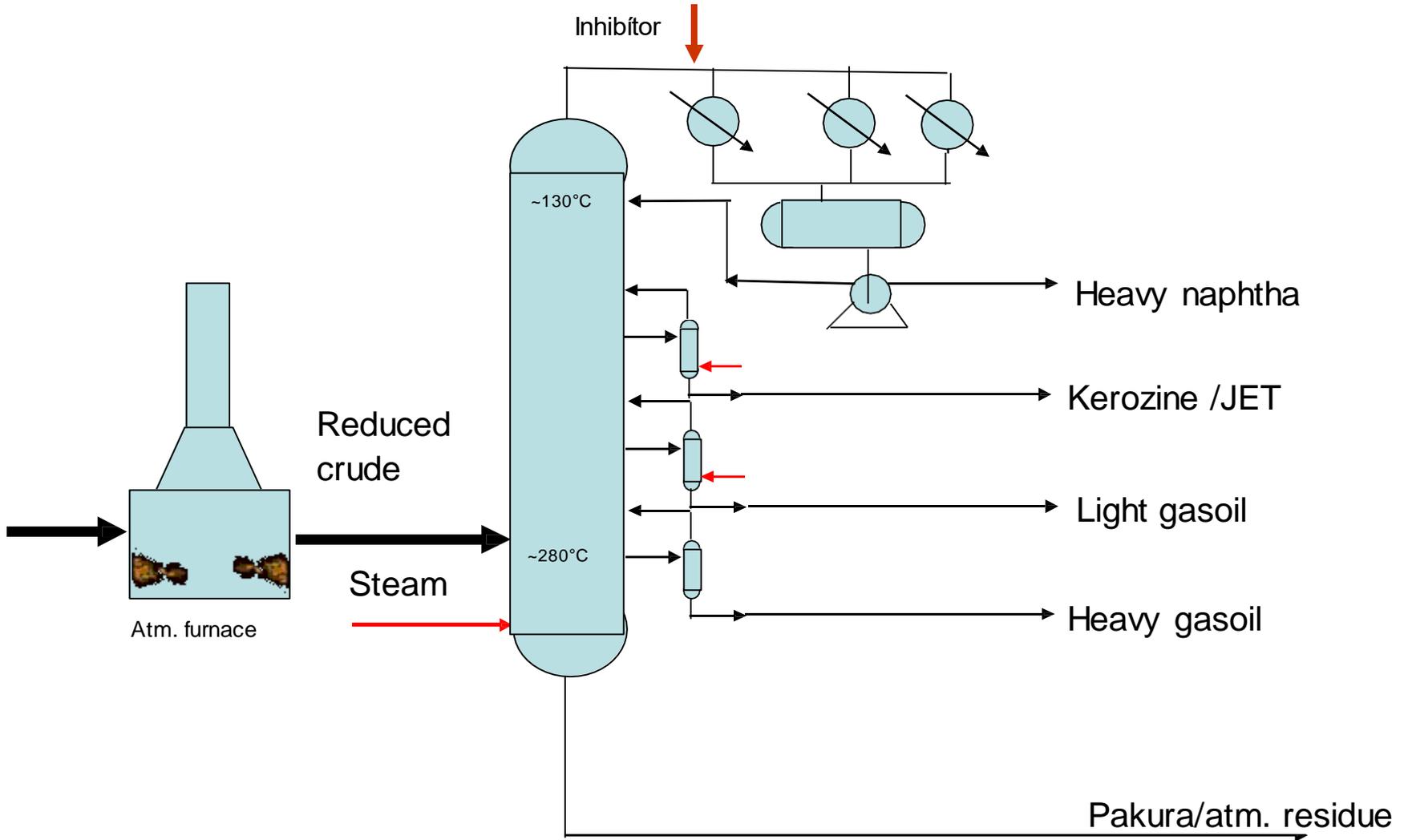
Light naphtha stabilizer

Aim: stabilization of Light naphtha



Atmospheric column

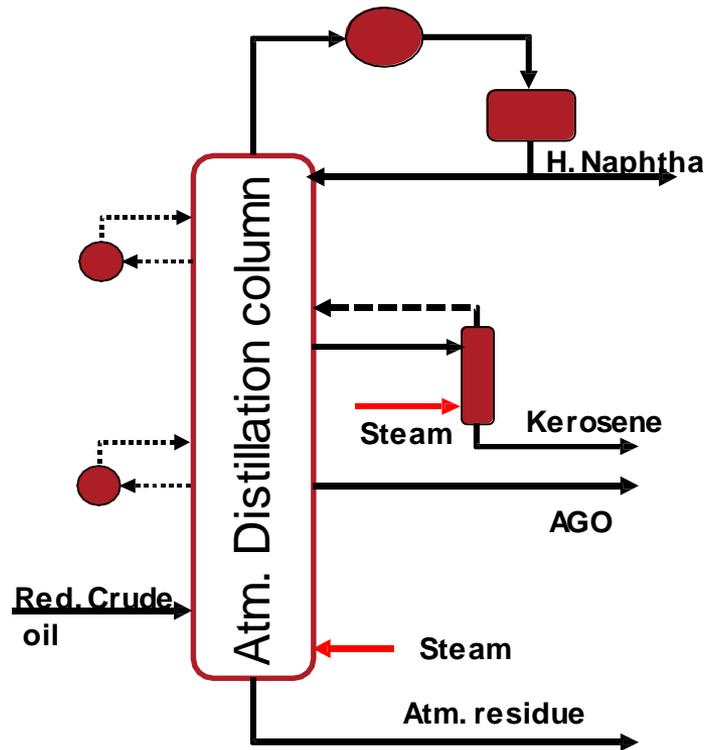
Aim: cuttigg reduced crude into different fraction at atmosferic pressure



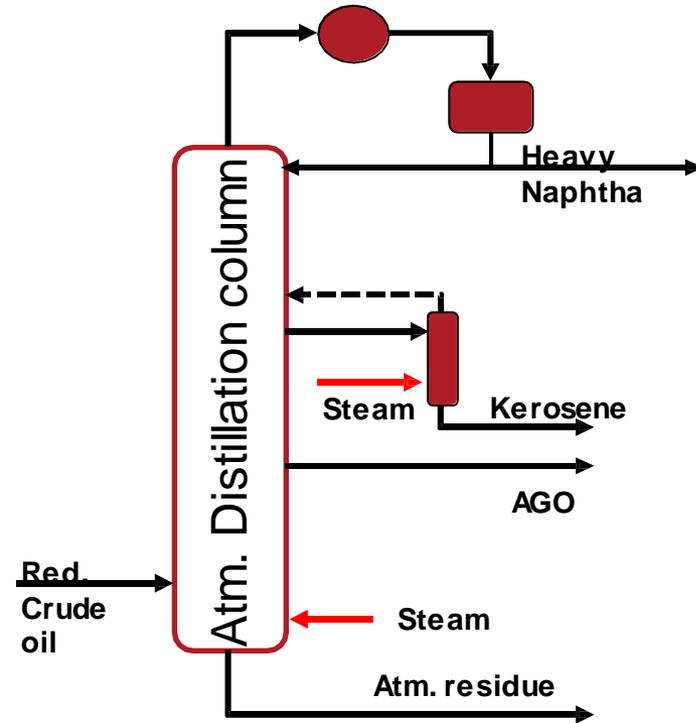
Typical trays number – atm. column

Fraction	Number of trays
naphtha / JET	8 - 9
JET / LGO	9 - 11
LGO / HGO	5 - 9
HGO / feed	8 - 11
feed / bottom	4 - 9
Side stripper	4 - 10

ATMOSPHERIC COLUMN W/- AND W/O PA EXAMPLE



A case



B case

Column configuration

	CASE A	CASE B
Red. crude flowrate, kg/h	65 500	65 500
Red. crude temperature, °C	325	325
Red. crude pressure, bar	3,15	3,15
Feed tray number	14	14
Condenser temperature, °C	40	40
Tray Number	16	16
Condenser temperature, °C	40	40
Kerosene, kg/h	18 000	18 000
Amount of stripper steam, kmol/h	20	20
Steam temperature	170	170
Steam pressure	8	8
AGO	18 800	18 800
Top PA	Yes	No
Flowrate, m3/h	20	
Delta temperature, °C	-50	
Bottom PA	Yes	No
Flowrate, m3/h	50	
Delta temperature, °C	-110	
Flowrate of Bottom stripper steam, kmol/h	60	60

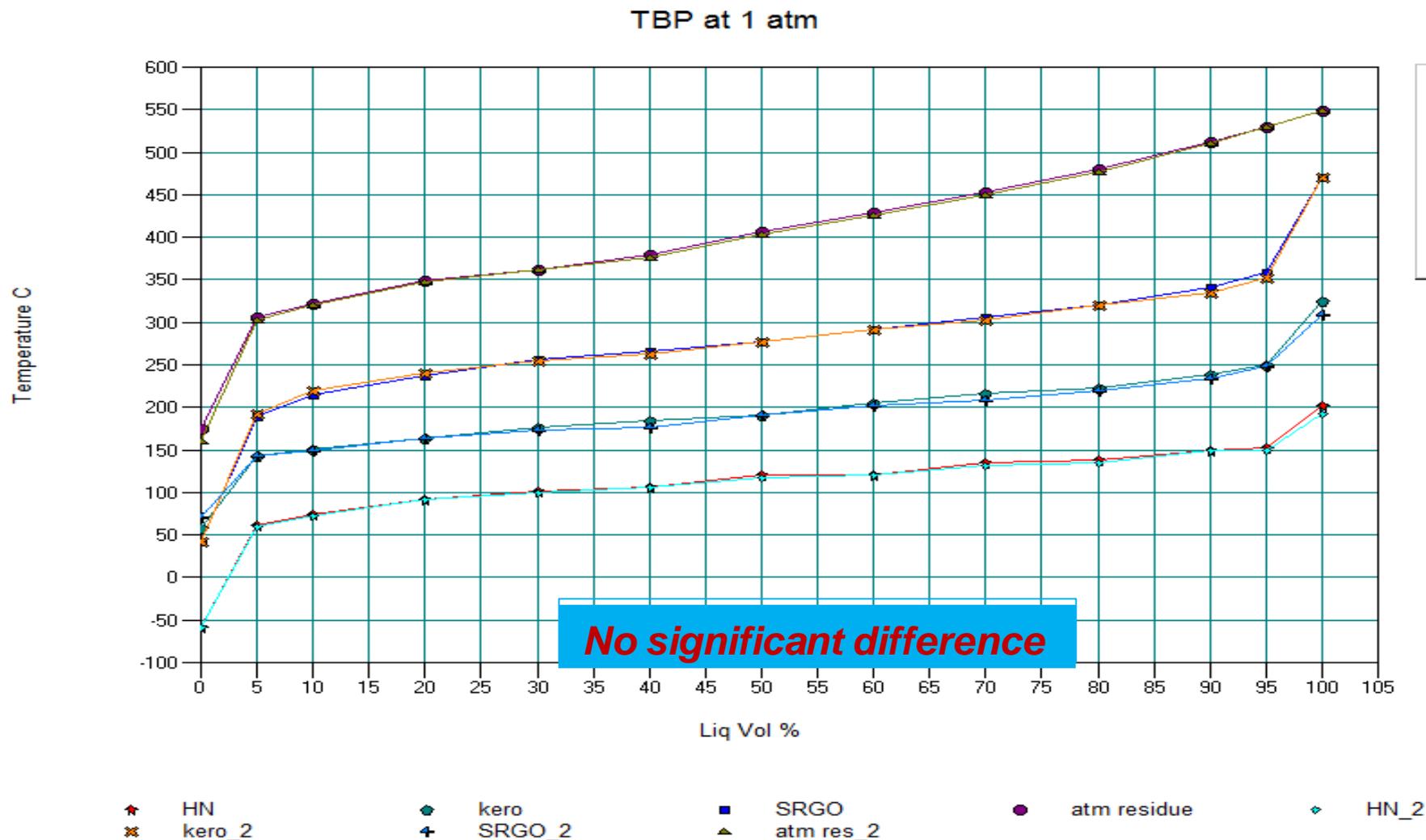
Inlet feed parameters are same

Side product parameters are same

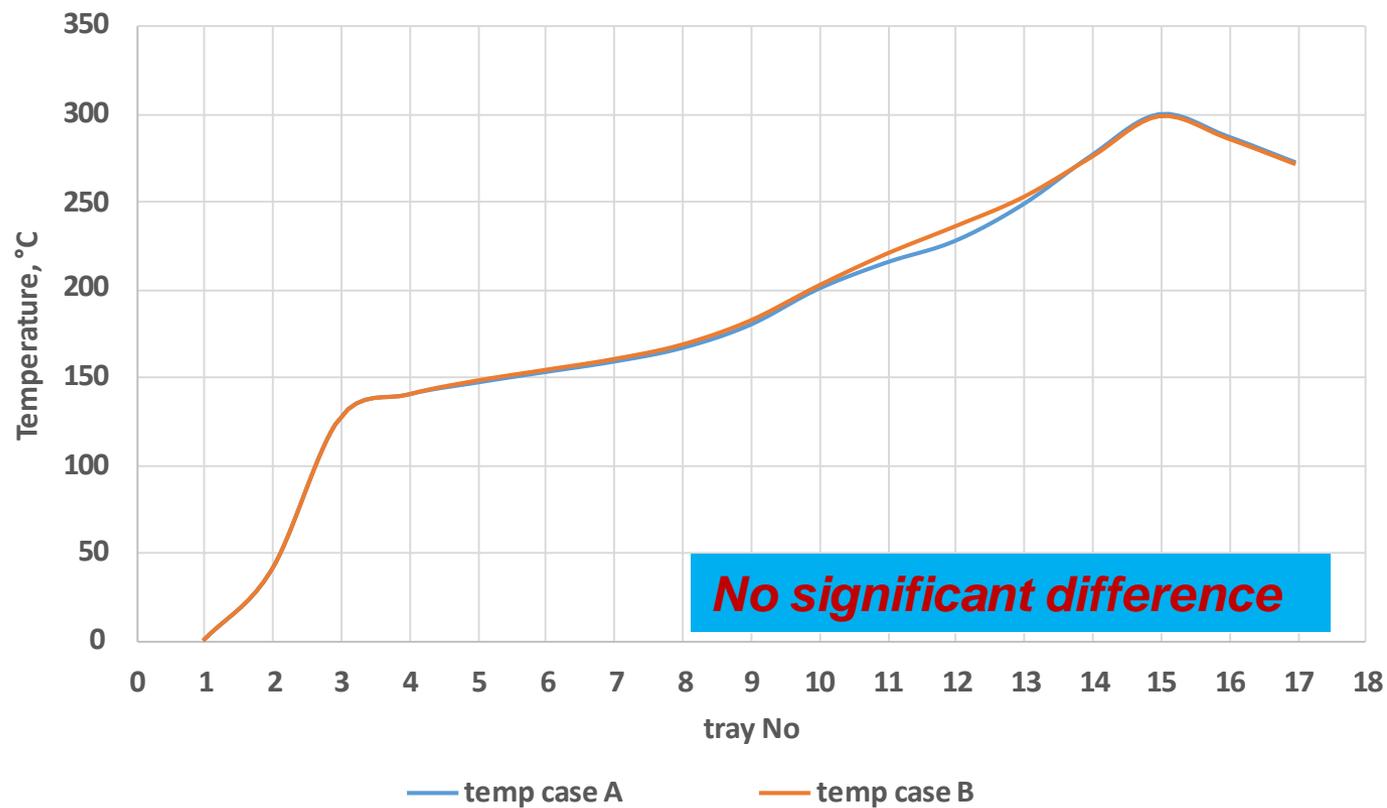


Bottom steam is same

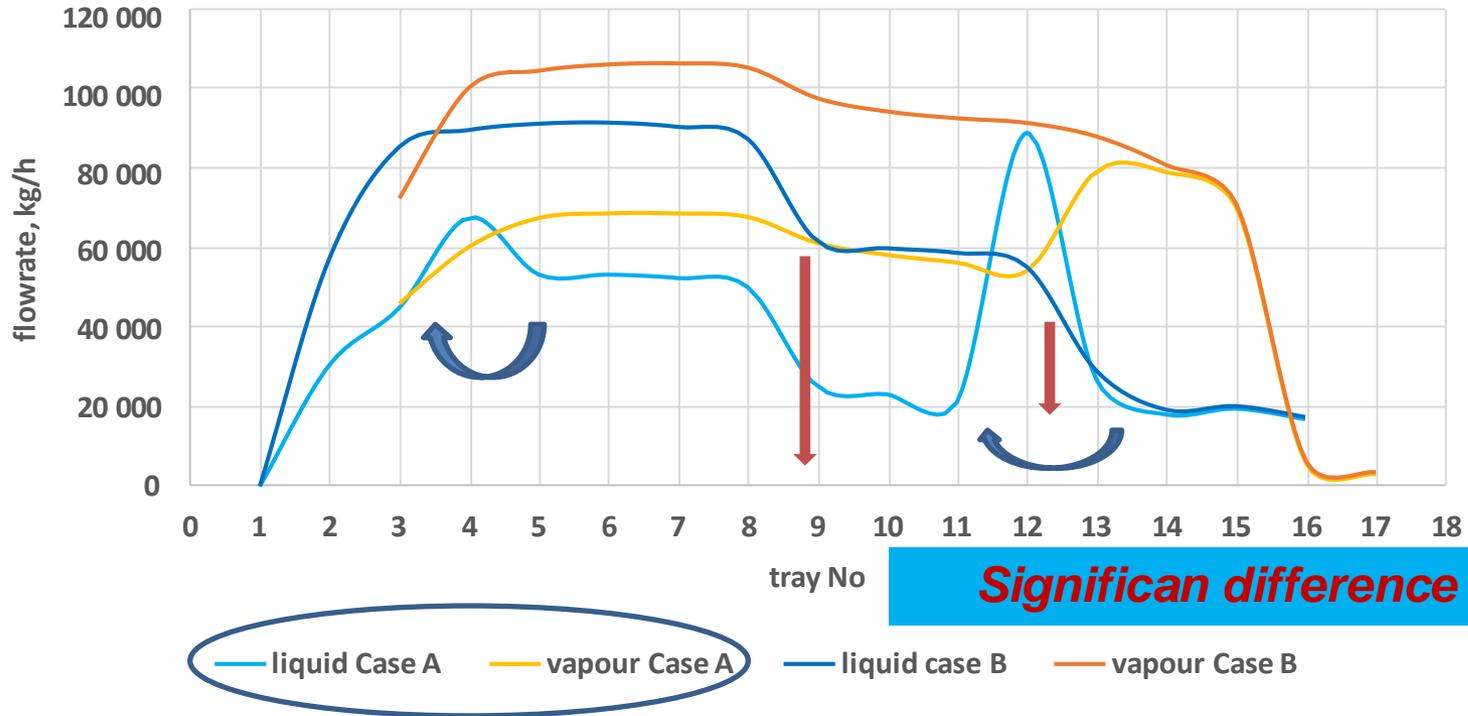
COMPARISON OF THE TBP CURVES OF PRODUCTS



COLUMN TEMPERATURE PROFILE



Column vapour-liquid loading



Heat management

	CASE A (PA)			CASE B		
	Removed heat, MJ/h	Temp in, °C	Temp out, °C	Removed heat, MJ/h	Temp in, °C	Temp out, °C
Condenser	26 020/ 65%	127	40	39 230	127	40
Top PA	1 840/5 %	147	97			
Bottom PA	11 850/30%	248	138			
Total	39 710			39 230		

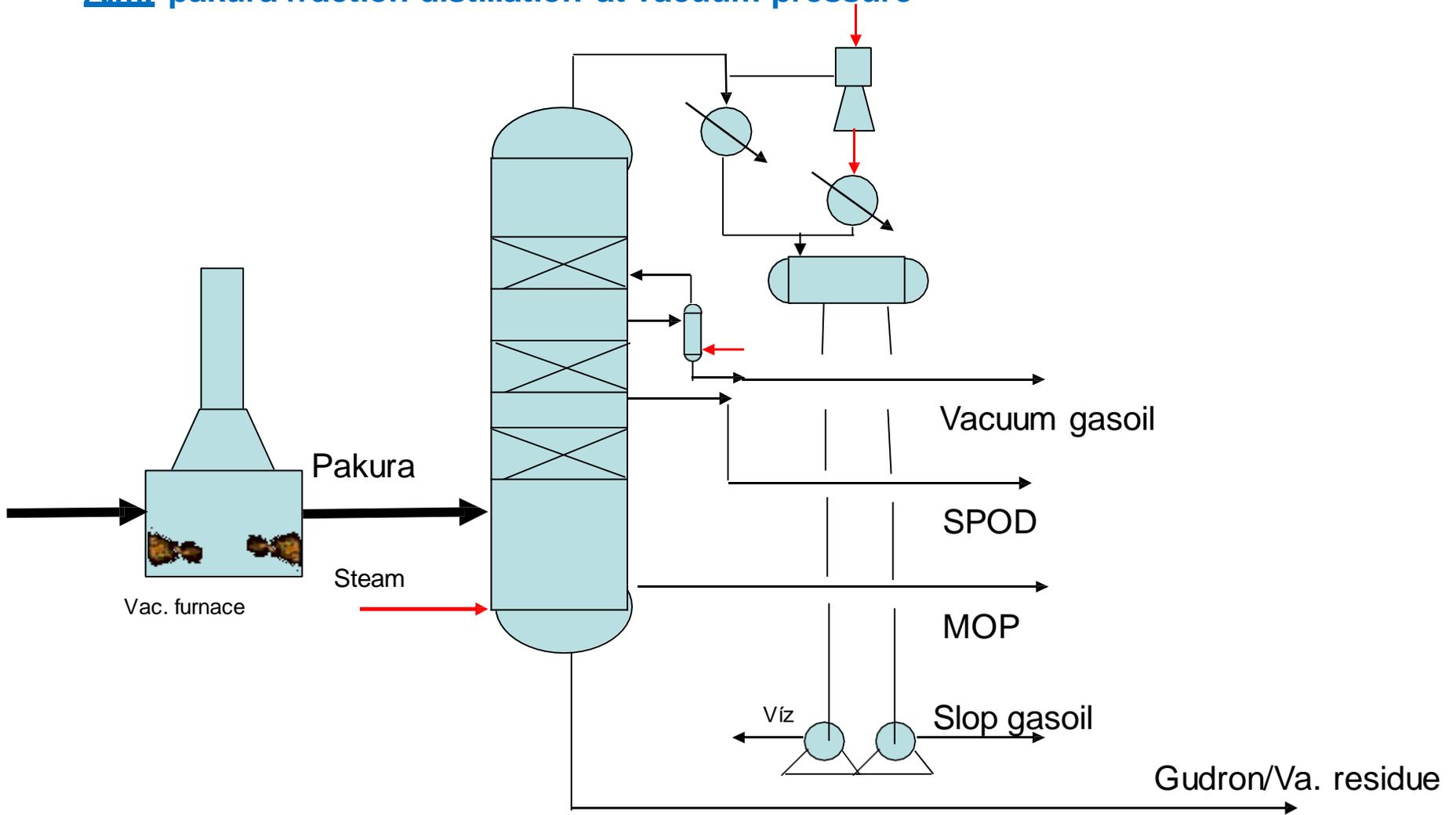
Heat recovery at higher and more temperature level !!

REFLUX ratio:

Case A: 2,2
Case B: 4,2

Vacuum column

Aim: pakura fraction distillation at vacuum pressure



Operation parameters

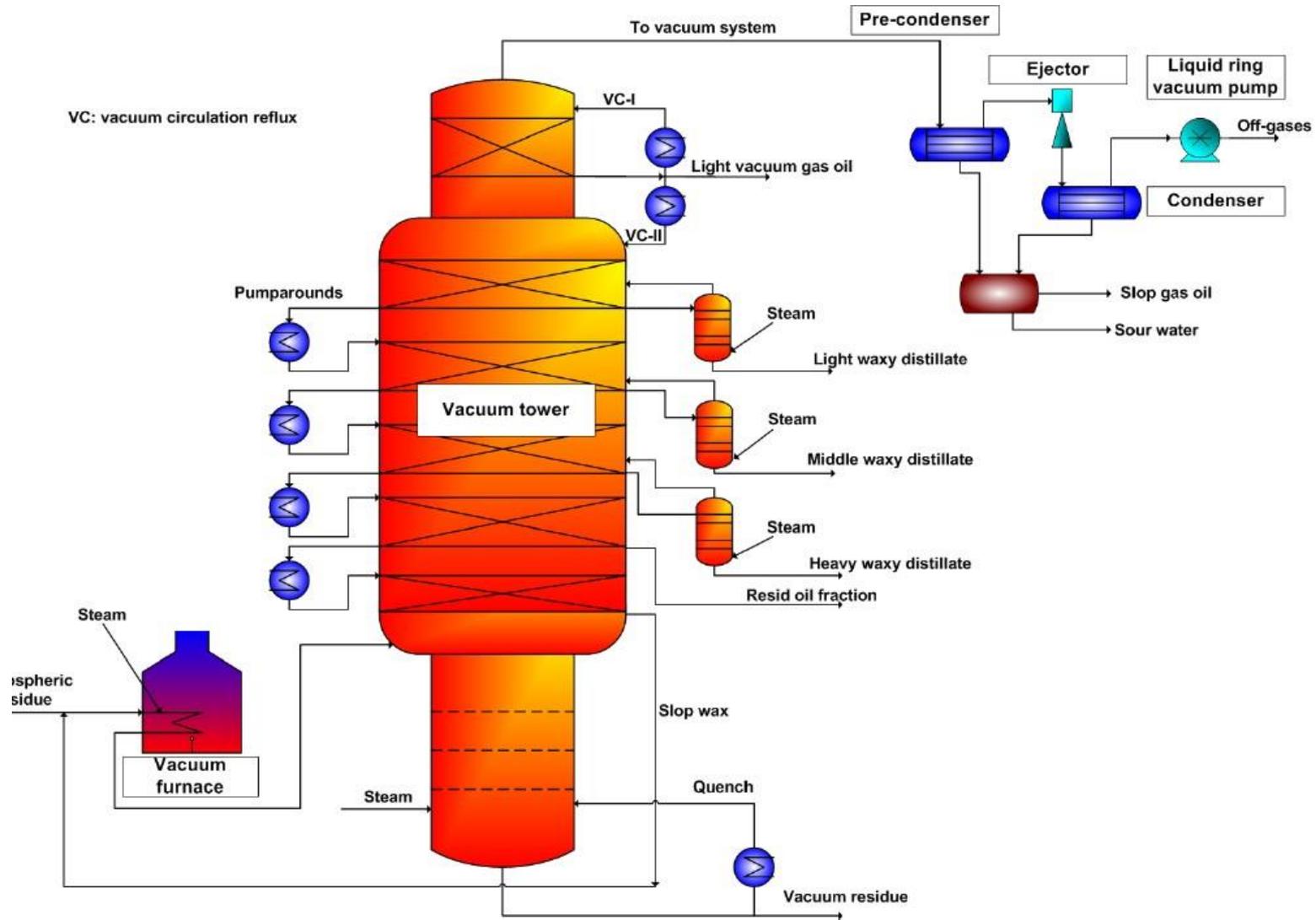
Vacuum furnace:

- Outlet temperature: 385 - 415 °C
- Outlet pressure: 0.35 - 0.50 bar

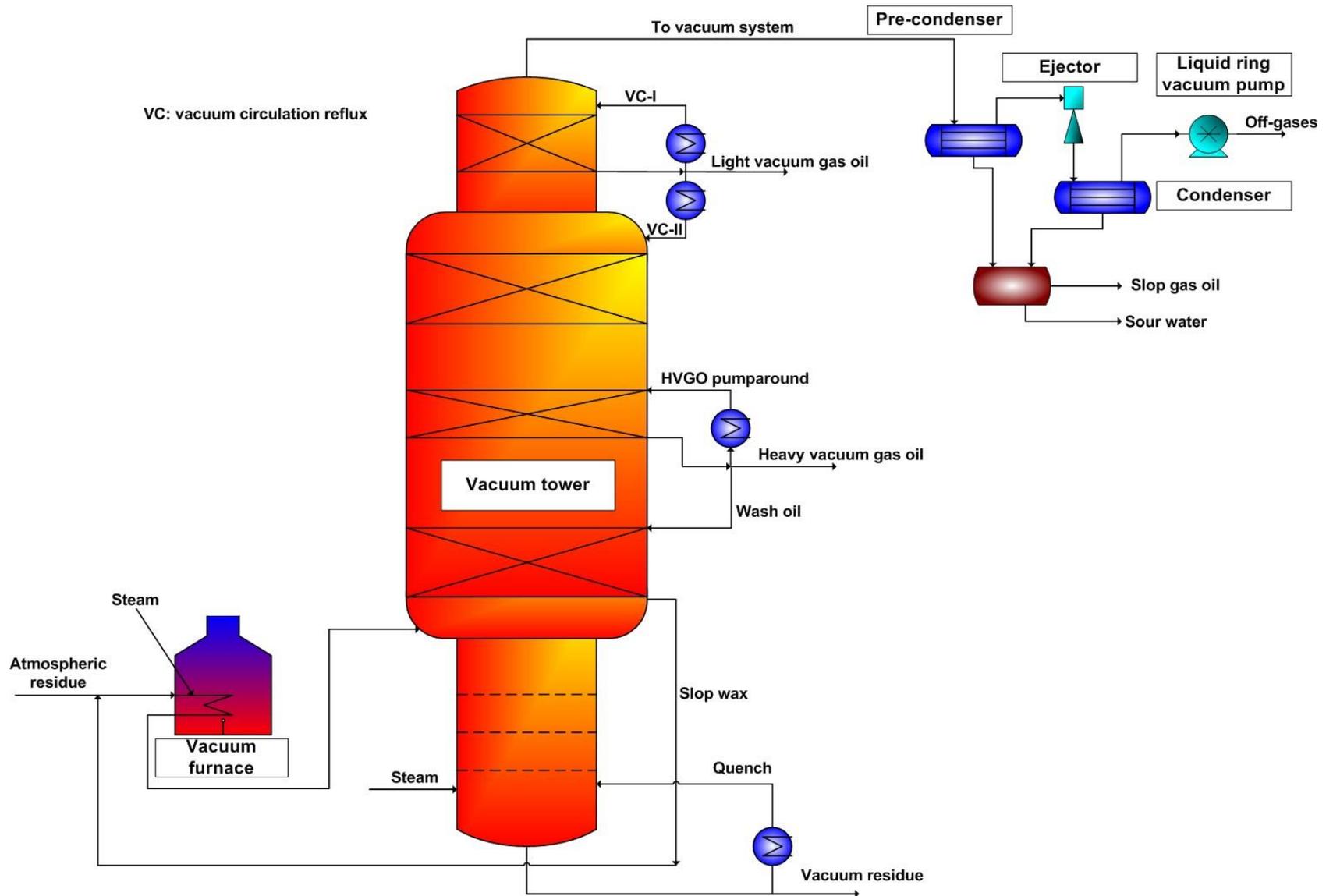
Vacuum column

- Top temperature: 70 - 80°C
- Top pressure: 40 - 80 mbar
- flash zone temperature : 375 - 398 °C
- flash zone pressure: 60 - 170 mbar
- bottom temperature: 320 - 340 °C

Vacuum distillation– I. Lube production



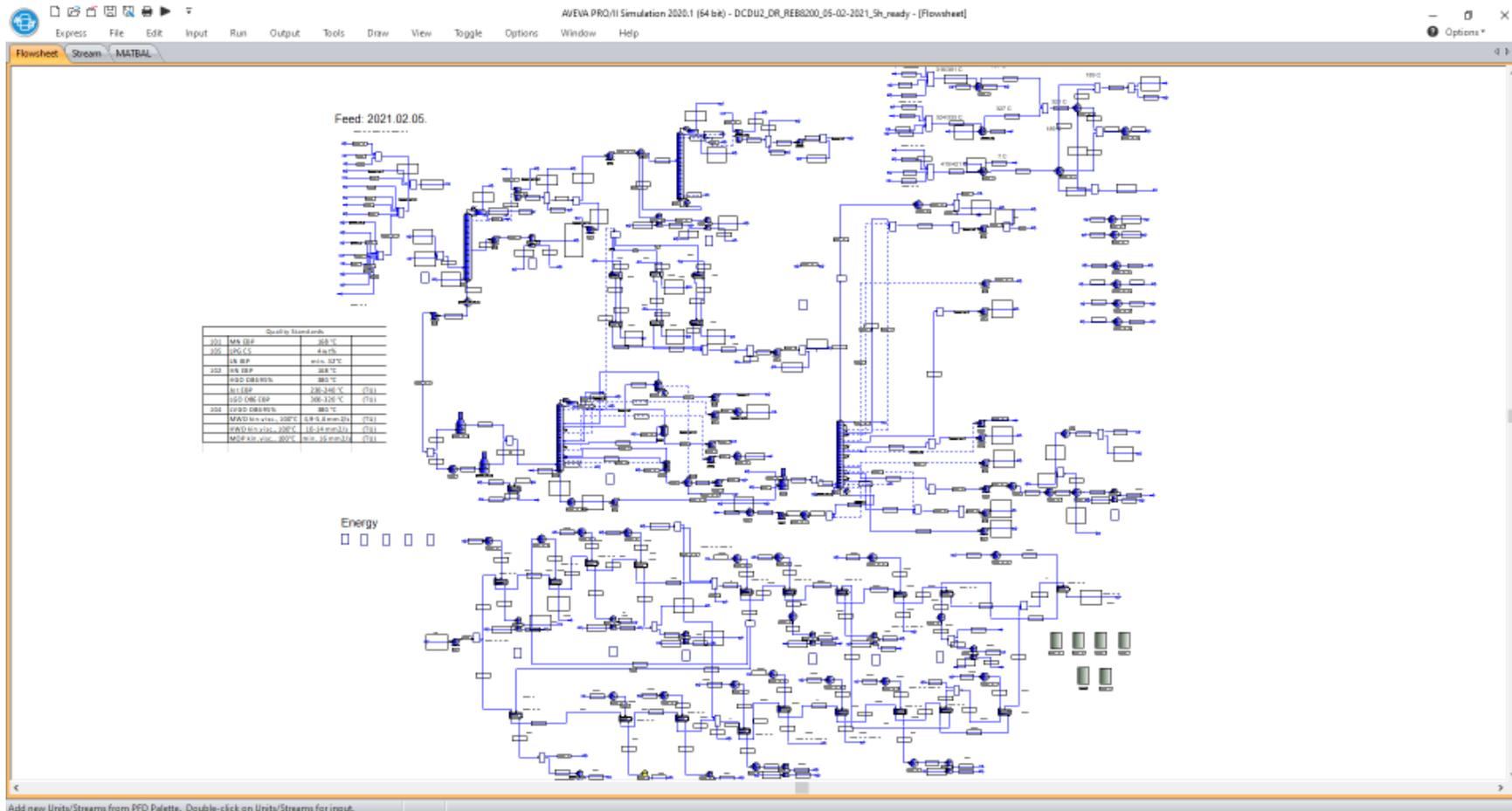
Vacuum distillation – II. motorfuel production



Deep- cut operation

- ▶ Deep - cut mode aims to increase HVGO yield to reduce vacuum residue(gudron) yield.
- ▶ Deep - cut mode, when the cutting point is higher than 565 °C.(1050 F) 95 % point of HVGO and 5 % of residue
- ▶ Operation parameters:
 - ▶ Low top pressure
 - ▶ Low pressure drop
 - ▶ High outlet furnace temperature (>410 °C)
 - ▶ Provide an adequate amount of washing liquid on the sink

CDU simulation model



Internals

[Back](#)

Internals

Trays

Packing

Valve-tray

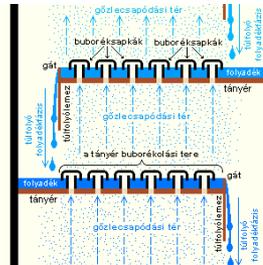
With bubble cup

Sieve tray

Random

Structured

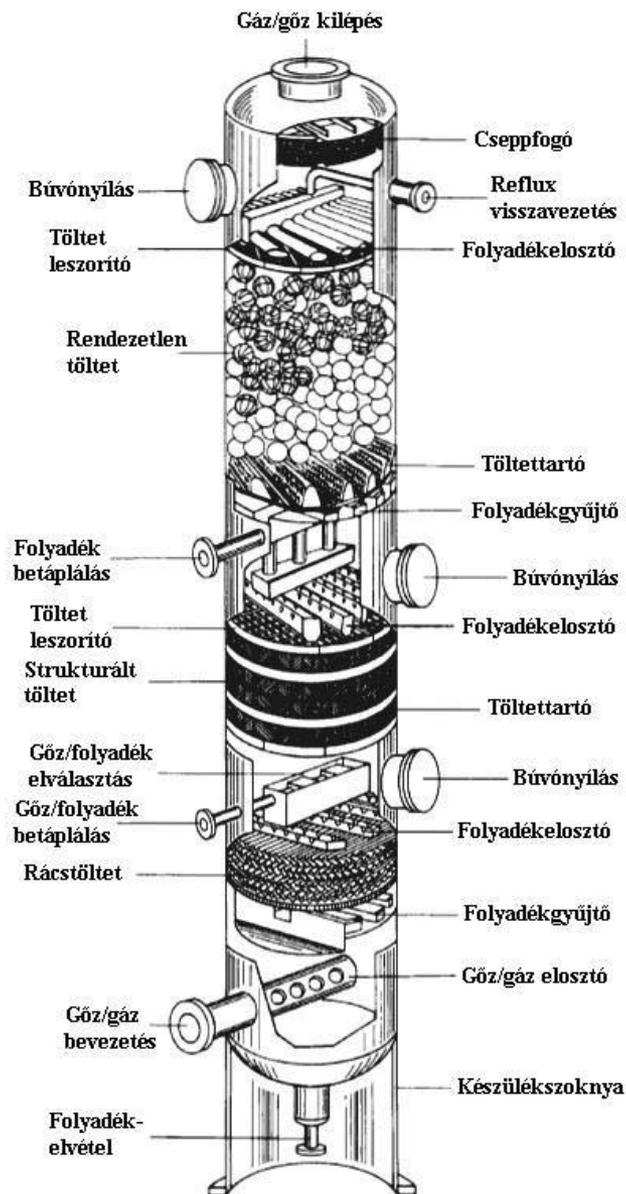
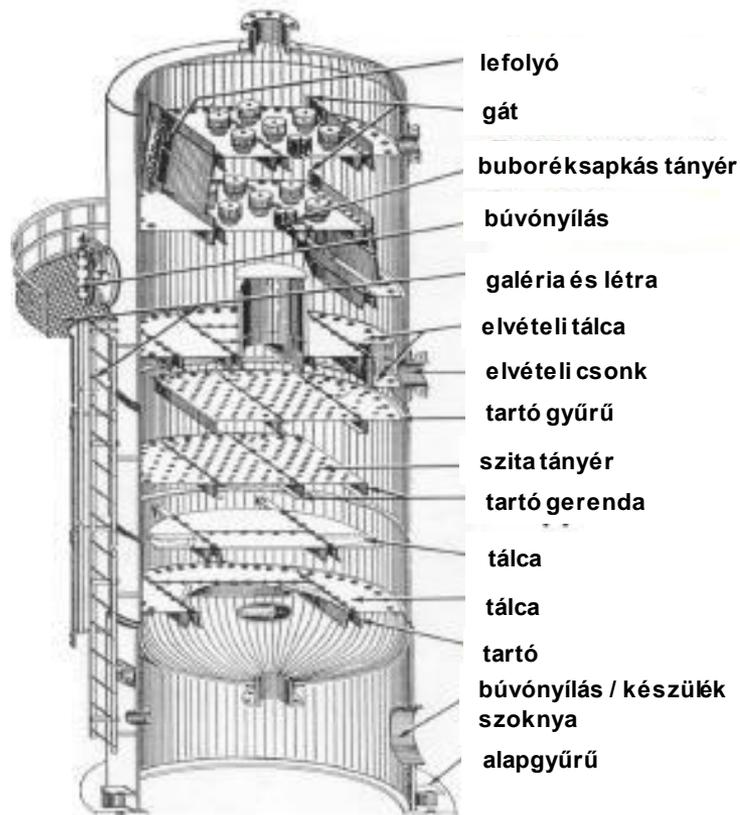
Grid



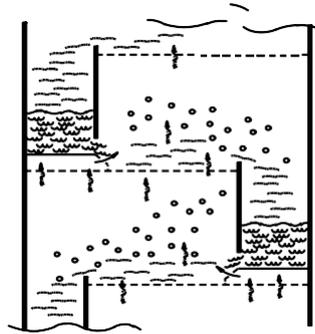
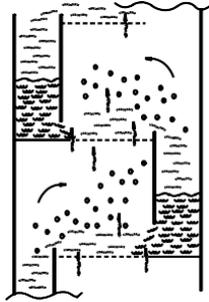
Main part of distillation column

Packing column

Plate/tray column



Conventional and High-efficiency trays structure Comparison



► Advantage /compare to high efficiency trays structure:

- Higher capacity: 30%
- Lower pressure drop: 20%
- Same or better material transfer capacity
- Smooth fluid flow
- Smooth vapour flow
- Better resistance to deposition of contaminants



ULTRA-FRAC® trays



SUPERFRAC® trays



VGPlus Trays



Köszönöm a figyelmet !