Operational experience from a 300 kg/h post-combustion capture test plant

using Monoethanolamine

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EnBW

Energie
braucht Impulse

Brief portrait
EnBW Energie Baden-Württemberg AG

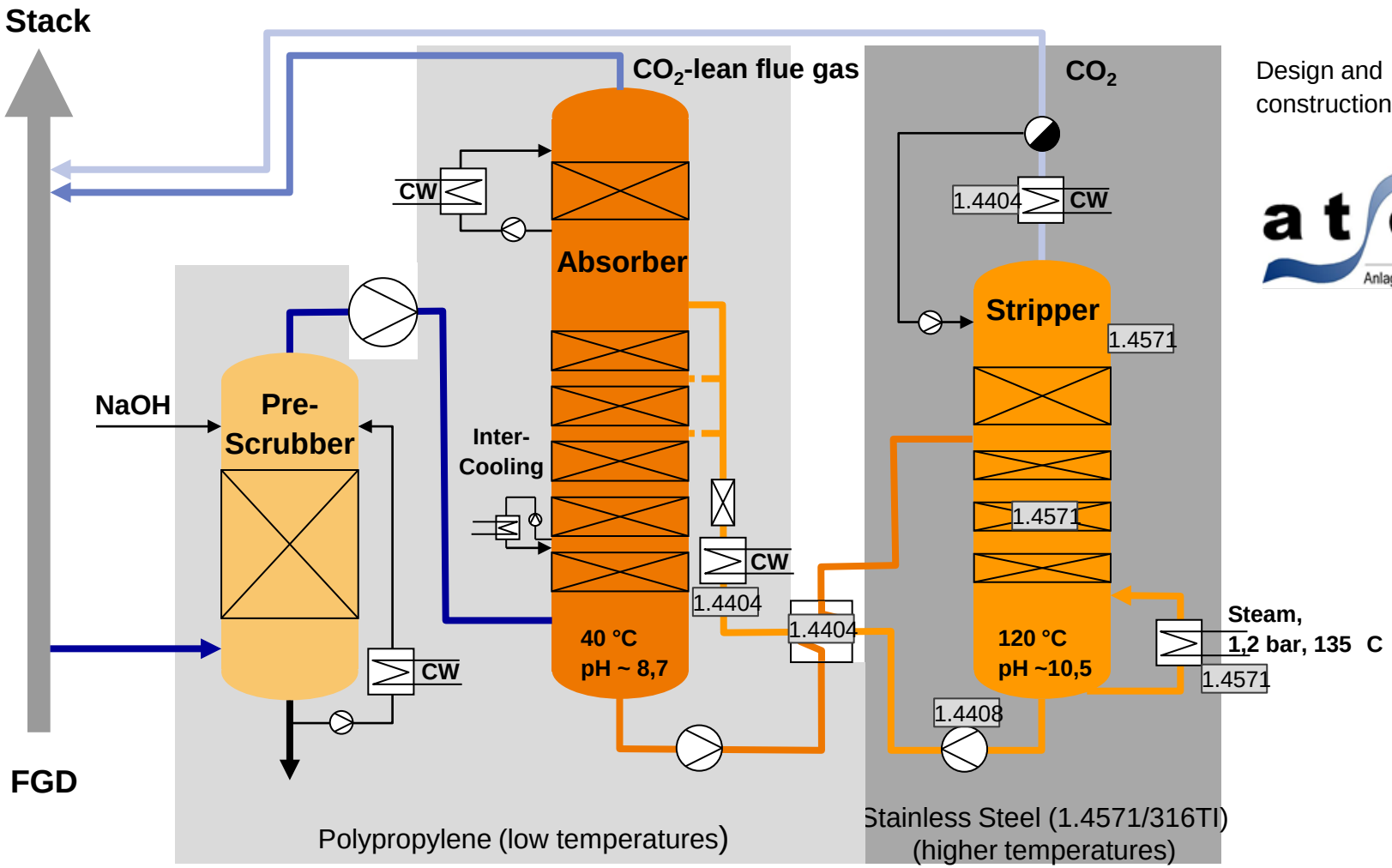
- One of the largest energy companies in Germany and Europe
- Business segments:
  electricity generation and trading, electricity grid and sales, gas, energy and environmental services
- Annual revenue 2012: in excess of € 19 billion
- Customers: some 5,5 million
- Employees: some 20,000

Place and characteristics of the Heilbronn test plant

- **Heilbronn Combined Heat and Power Plant**
  - Electric output (site): 1.020 MW
  - District heating: 300 MW
  - Electric output Unit 7: ~ 815 MW
  - Commissioning of Unit 7: 1985
  - Flue gas treatment: DeNOx, ESP, wet FGD, Sewage sludge cofiring (1998)

- **Amine scrubbing test plant**
  - Flue gas volume flow: ca. 1.500 Nm^3/h (f)
    ~ 0,05 % of total flue gas volume flow of Unit 7
  - CO_2 capture rate: ~ 90 %
  - Captured CO_2 quantity: ~ 300 kg CO_2/h
  - Design solvent: 30 wt.% Monoethanolamine (MEA), (20 – 50 w%)
  - Space requirement: ~ 10 x 15 x 5 m, height: 40 m (absorber), 25 m (stripper)
  - Commissioning: March 2011

Process design of amine scrubbing test plant



Design and construction by

Amine scrubbing test plant

Absorber top

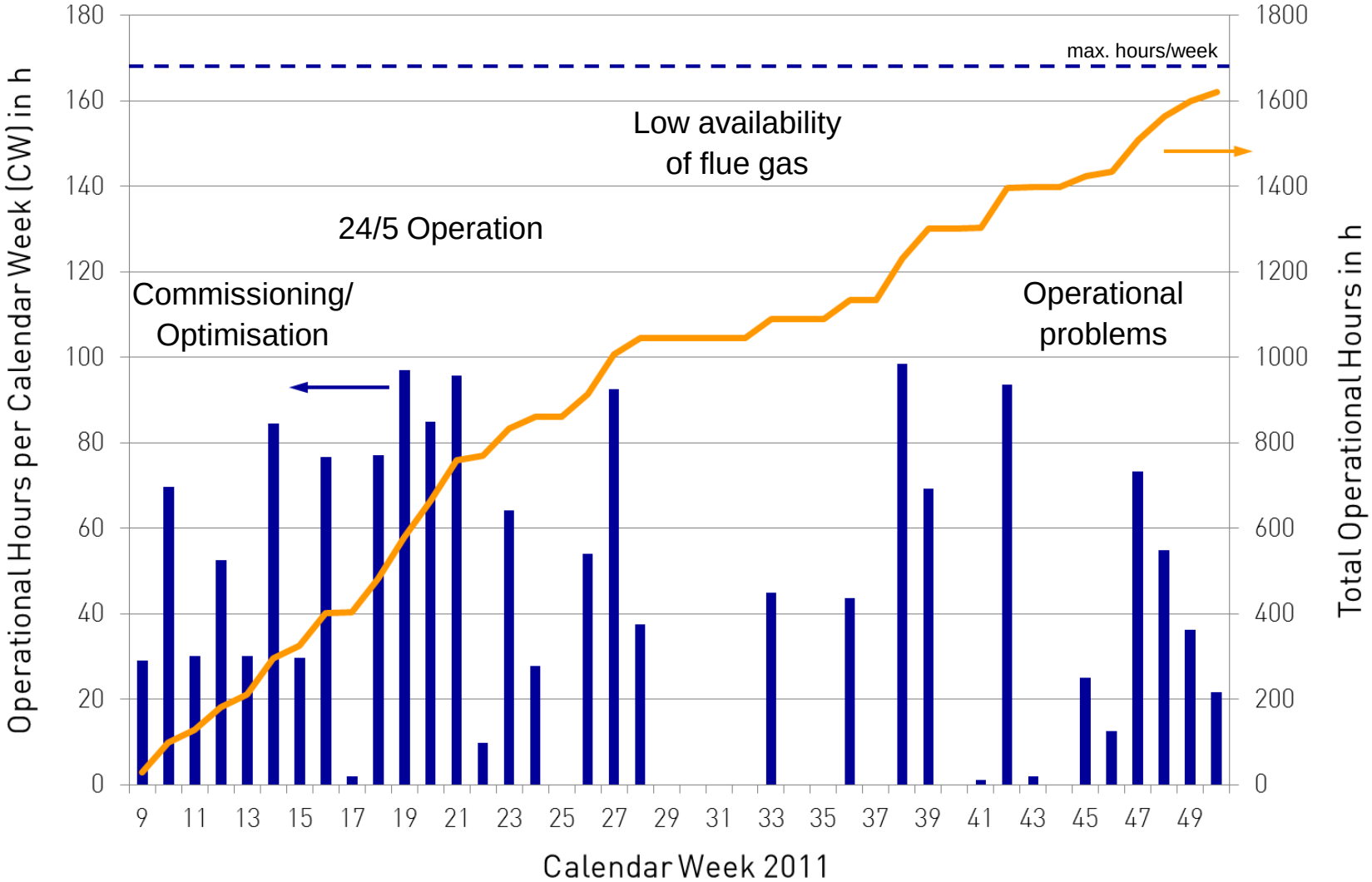
Absorber water wash

Absorber inter-

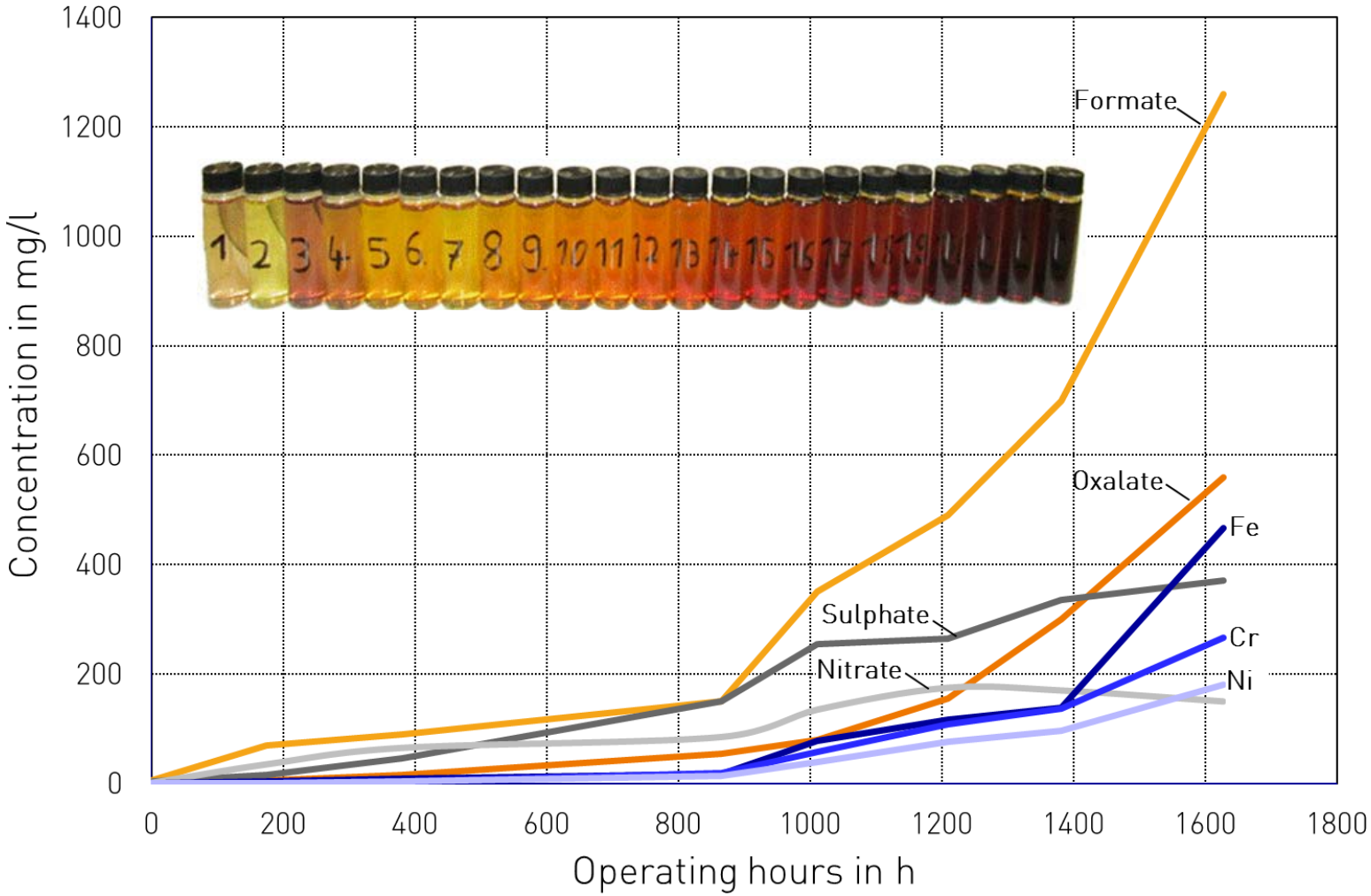
Stripper top - overhead condenser

Inter-cooling and lean cooling HTX

Test plant operation in 2011

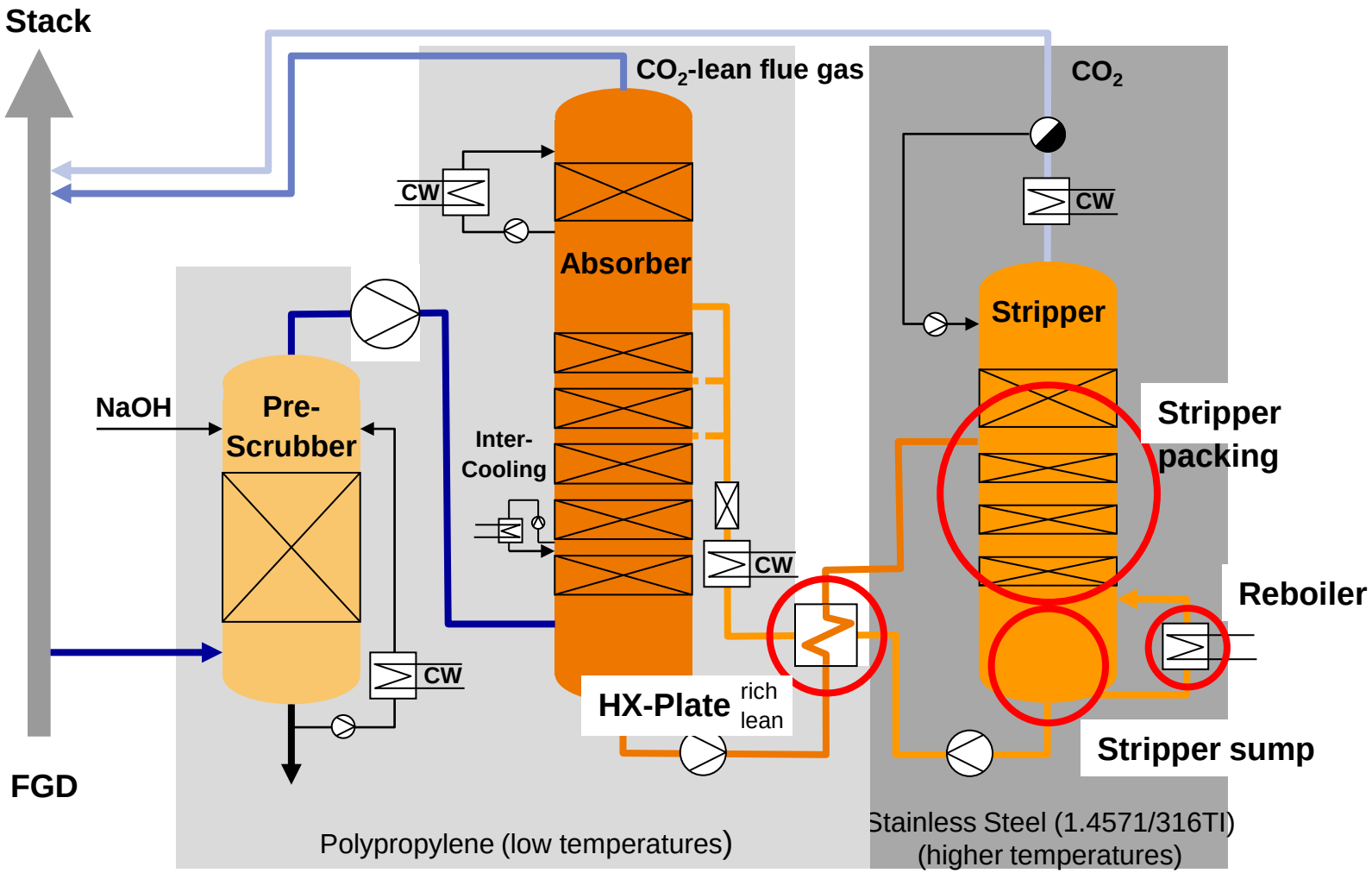


Solvent concentration of heat stable salts and metals

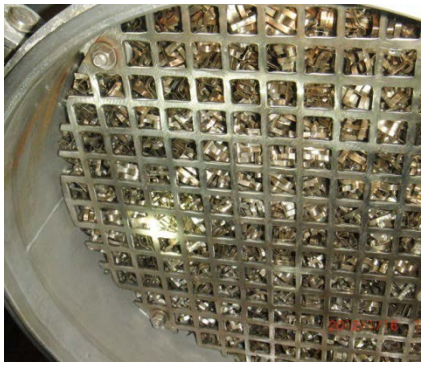


Rieder, A., Unterberger, S., GHGT-11, 2012

Observations points



Observations after 1.600 h of test plant operation

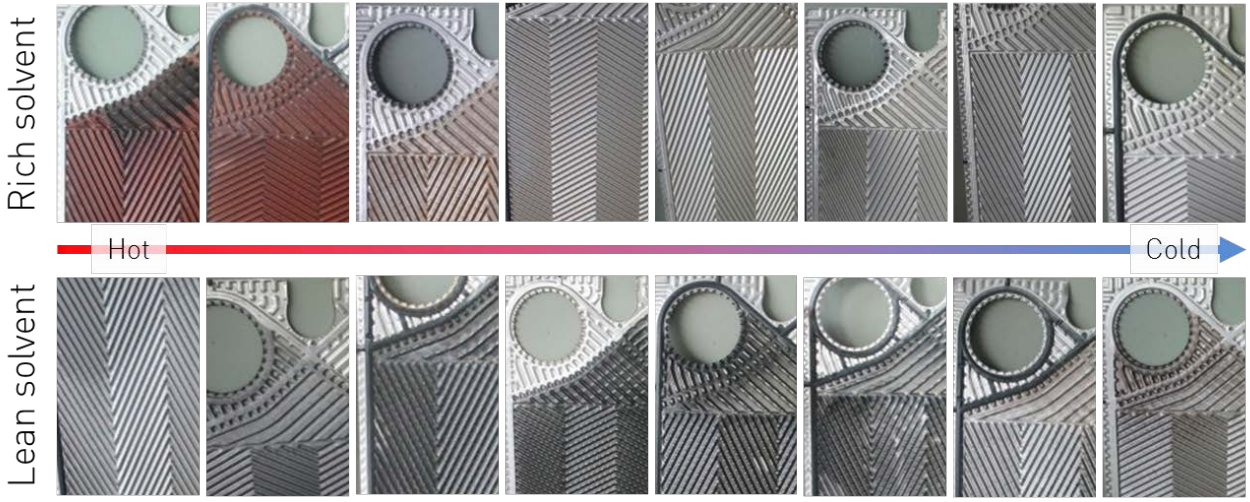


Stripper packing above rich inlet

Stripper packing below rich inlet

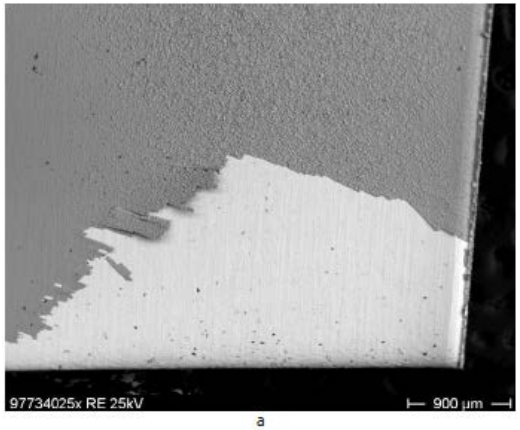
Sludge formation in inter-cooling storage

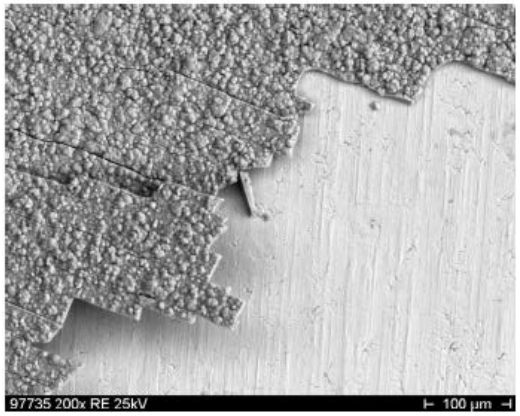
Solids formation at stripper pressure retention valve



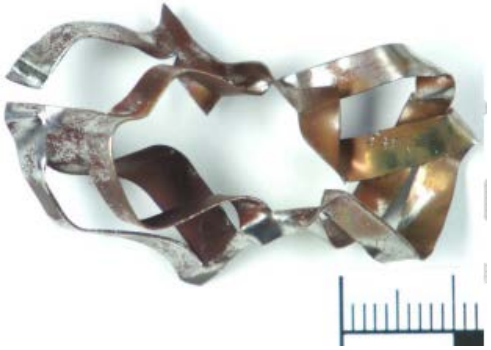
Lean-rich solvent cross heat exchanger inspection

SEM examination of the packing material grade 316 Ti



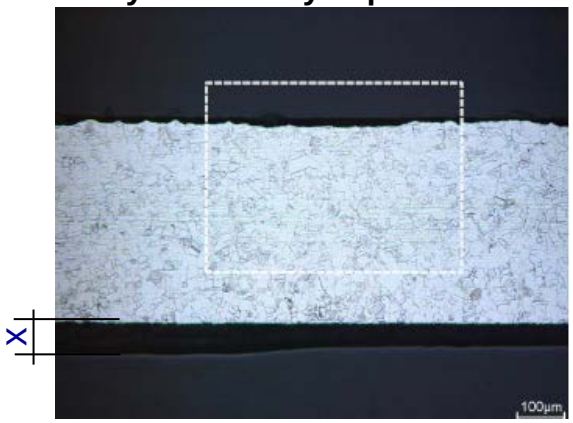


Surface without significant corrosion under the film

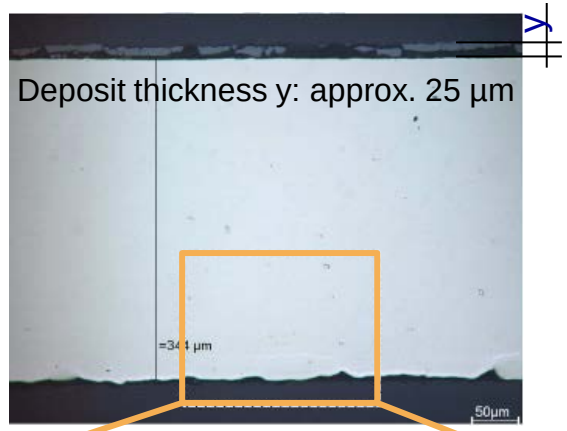


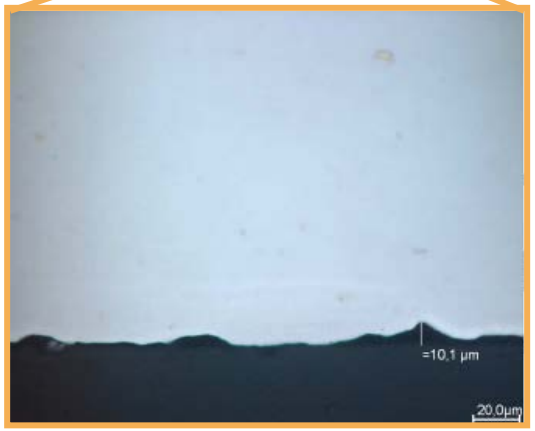
Packing material thickness: 0,25 mm

Partly covered by deposits



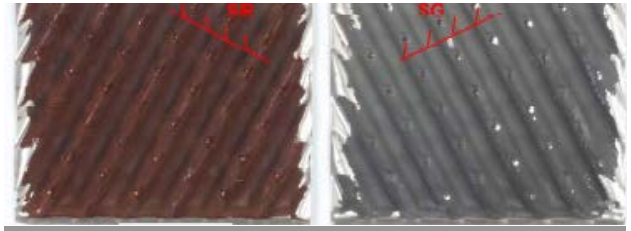
Deposit thickness x: approx. 50 µm



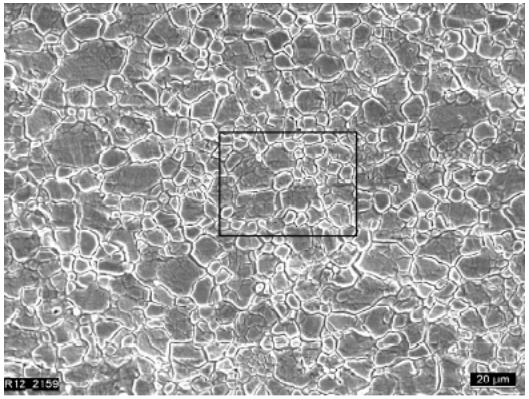


2 examples of pitting corrosion in the microsection approx. 10 µm
at the non covered surface

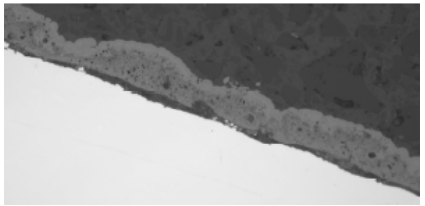
Metallurgic examination of cross heat exchanger plates grade 316 L



Rich solvent side



Surface: Visible grain boundaries on the cleaned material surface

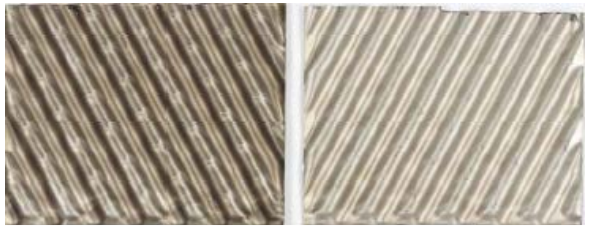


Cross section: Slight corrosion under deposit

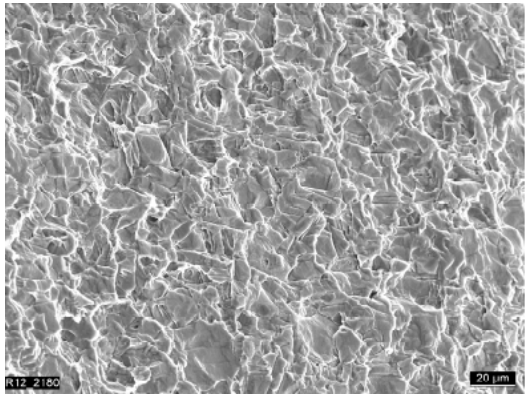
Rich

Lean

Lean-rich cross heat exchanger plate (hot section)



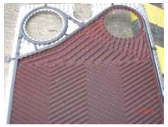
Lean solvent side



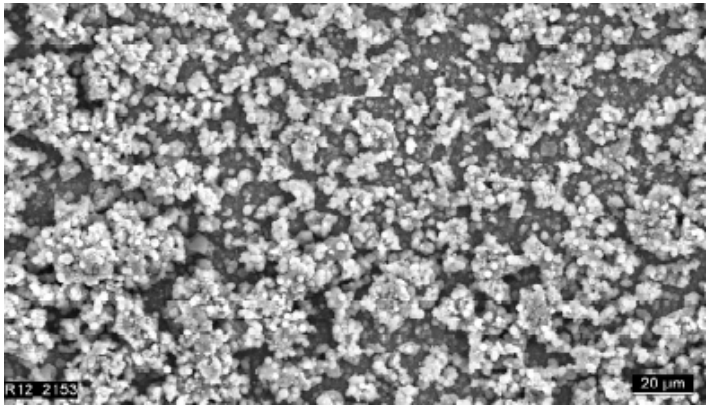
Rough surface without visible grain boundaries

Cross section: Pitting corrosion approx. 10µm

Deposit and sludge examination



Rich solvent side (hot)
Red to brown area

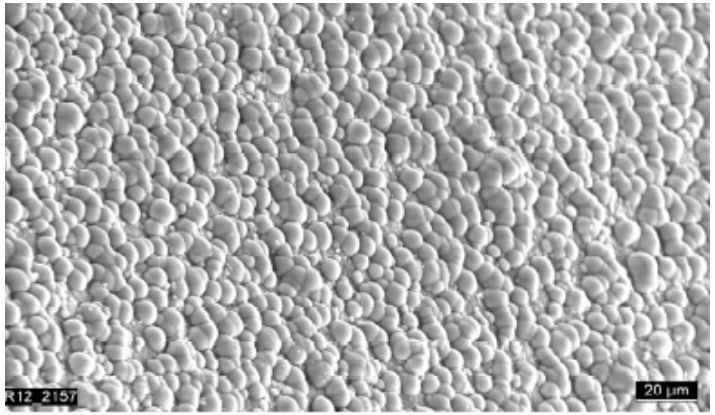


Analytical methods:
- EDS (Energy dispersive spectrometry)
- XRF (x-ray flourescence)

Results of the EDS analysis:

Analyse 1 auf Belag 25kV							
O	Al	Si	Cr	Fe	Ni	Mo	Summe
10,2	0,7	0,6	7,0	81,0	0,3	0,3	100

Rich solvent side (hot)
Grey area

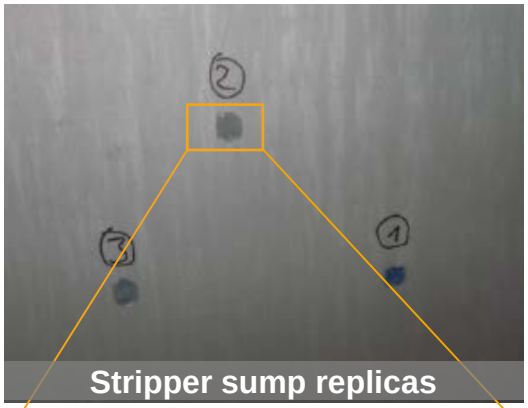


Results of the EDS analysis:

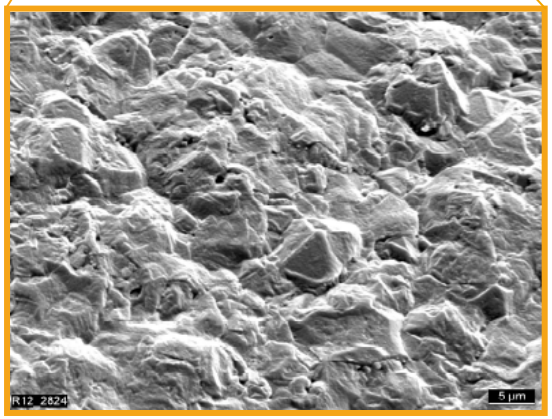
Analyse 11 auf Belag 25kV						
O	Si	Cr	Fe	Ni	Mo	
19,6	0,6	4,0	75,7	0	0,1	100

- With XRF, ferrous and chromium oxides, as well as ferrous carbonates could be confirmed
- The stripper sludge does not contain **nickel and molybdenum compounds**
  => These elements must stay in solution/do not precipitate for the entire temperature/ pH conditions of the absorption/desorption cycle

Microstructure of stripper bottom /endoscopy of reboiler grade 316 Ti



Stripper sump replicas



Microstructure of stripper wall shows same characteristic surface as lean side of cross heat exchanger

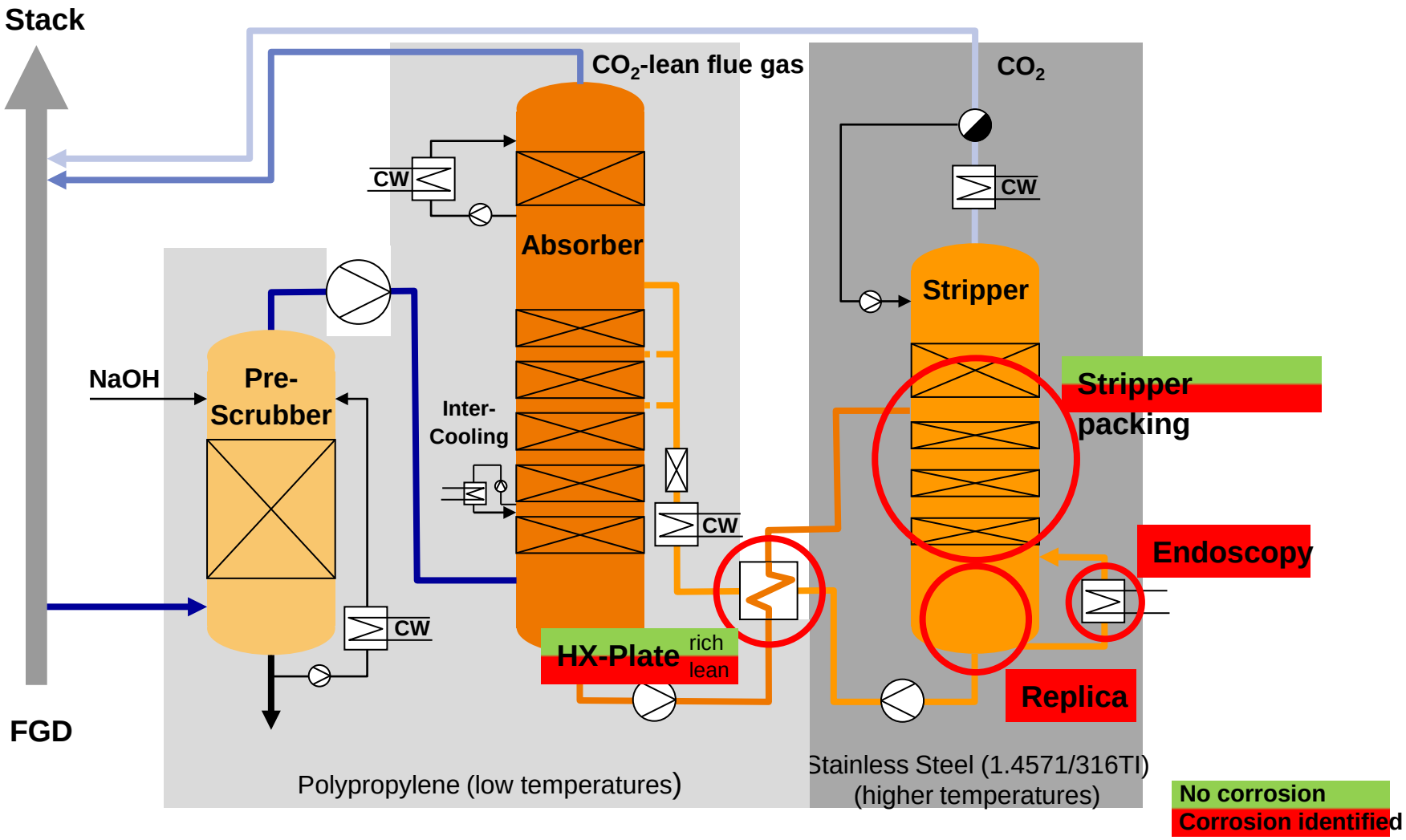
Tube weld seams (reboiler top)

Drum weld seam (reboiler top)

Drum weld seam (Reboiler top)

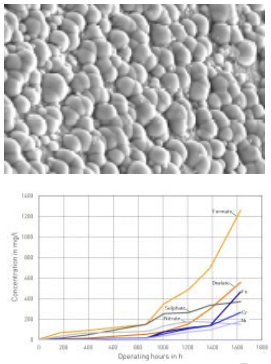
Reboiler endoscopy shows rough surfaces, discoloration and corroded weld seams

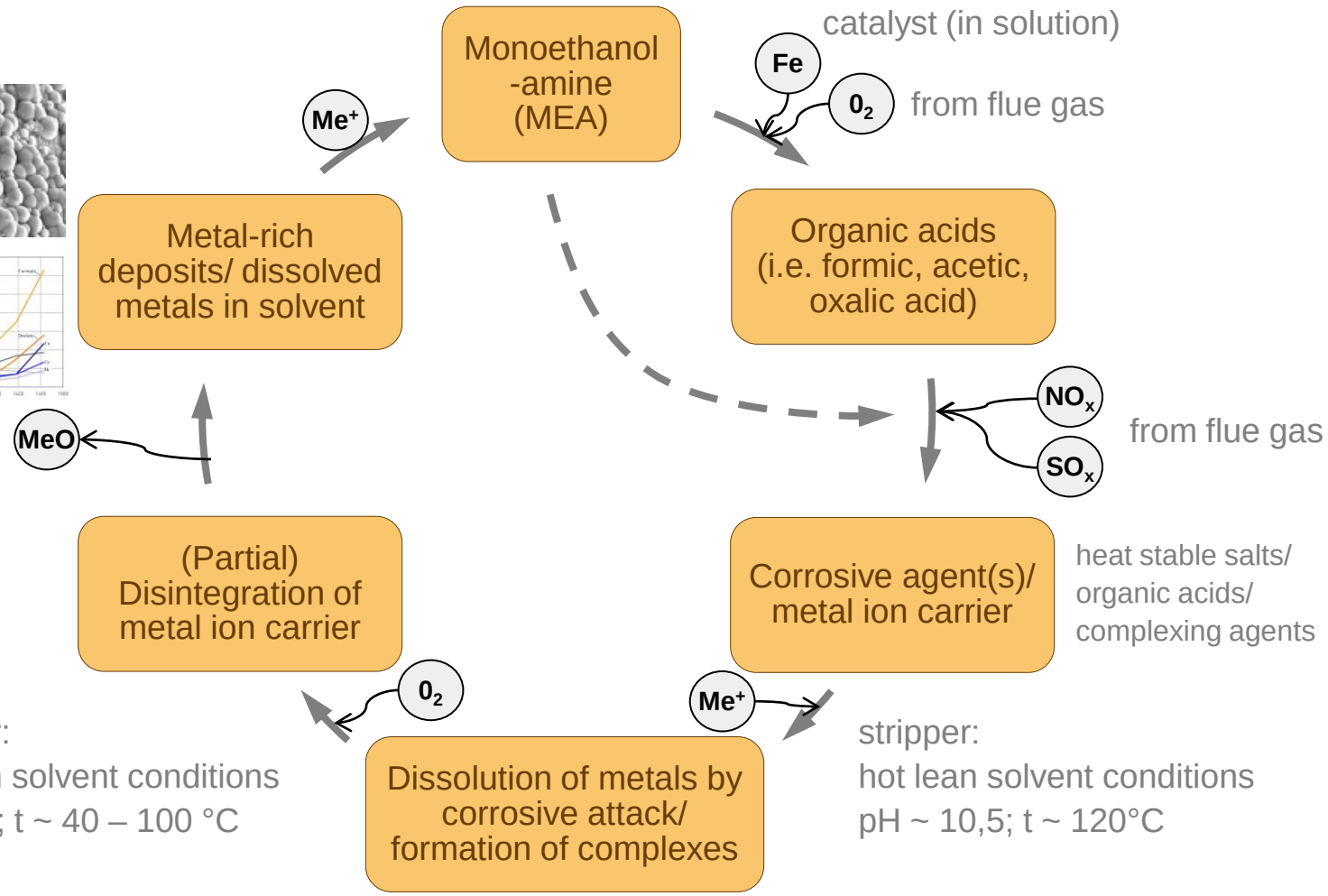
Overview results from previous investigations



Hypothesis: Degradation product-based corrosion cycle

Metal-complex induced corrosion generated by MEA degradation products





Conclusions

- Results are plant- and solvent-specific – and still not complete
  - First test campaign
  - Confirmation required!
  - Comparability questionable, due to 'used' material surfaces compared to 'new' conditions?
- Corrosion effects clearly identified in the hot lean solvent area
  - 'Critical' corrosion rate from a technical point of view?
  - To be resolved by a corrosion allowance during equipment design?
  - Use of alternative/higher-grade materials necessary? Suitable materials?
  - Effect of self-accelerating solvent degradation on corrosion?
  - Beneficial effect of reclaiming/solvent management to be investigated
- Reaction/corrosion mechanism proposed
- Further investigations on materials and components for:
  - Further characterisation of corrosion mechanism
  - Determination of corrosion rate
  - Corrosion testing of selected materials

Thank you for your attention!

Questions?