

# “Operational experiences of process bath analysis in a wire rod pickling plant”

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## Content of Presentation

- › Overview of DEW wire rod pickling plants for carbon steel and stainless steel
- › Online concentration monitoring at HCl-Fe wire rod dip tank pickling plant
- › Online concentration monitoring at HF-HNO<sub>3</sub> mixed acid wire rod dip tank pickling plant (project MACO Pilot)
- › Summary and outlook

# Wire rod production at DEW Hagen



# Wire rod production at DEW Hagen



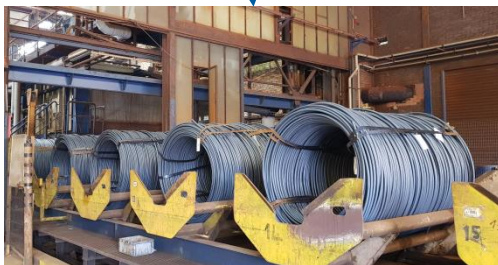
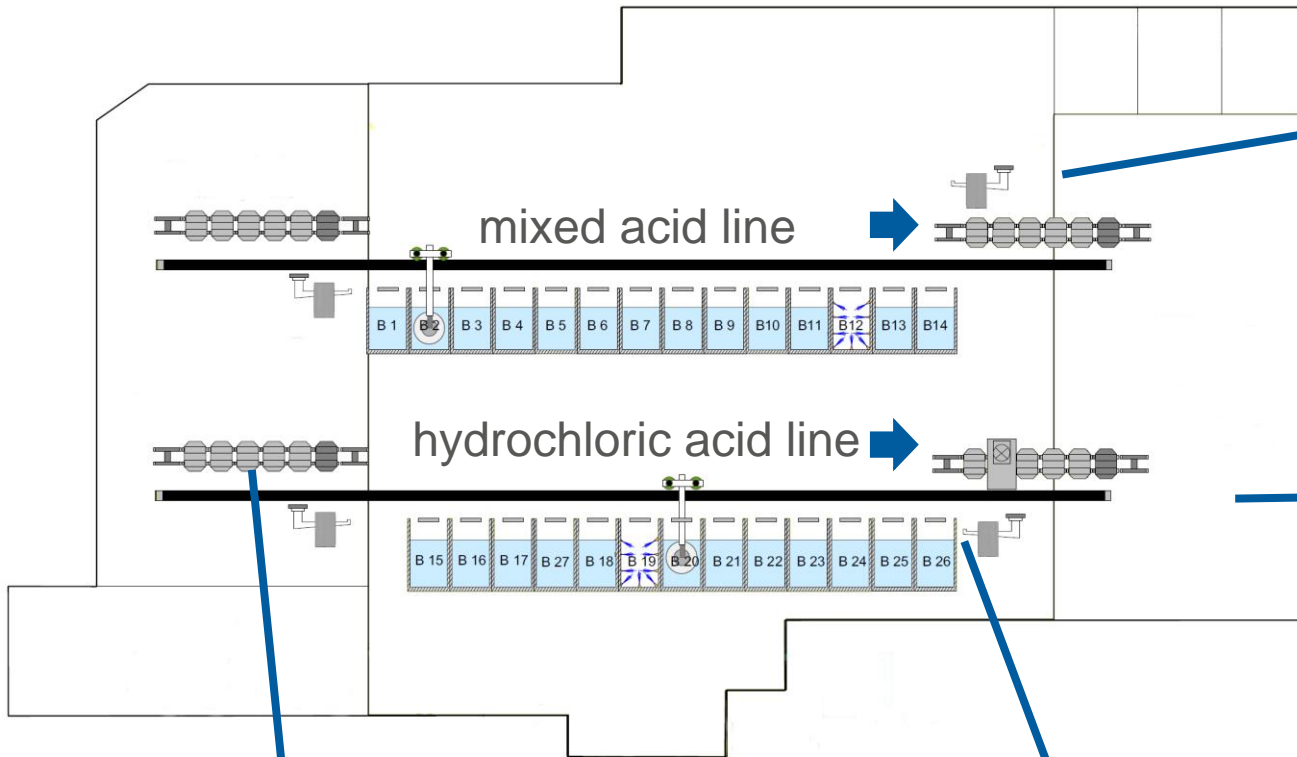
## Production Site Hagen:

- › 400 Employees
- › Output per year around 100.000 t
  - › ca. 59 % Construction Steel
  - › ca. 37 % Heat and Acid Resistant Steel
  - › ca. 4 % Tool Steel

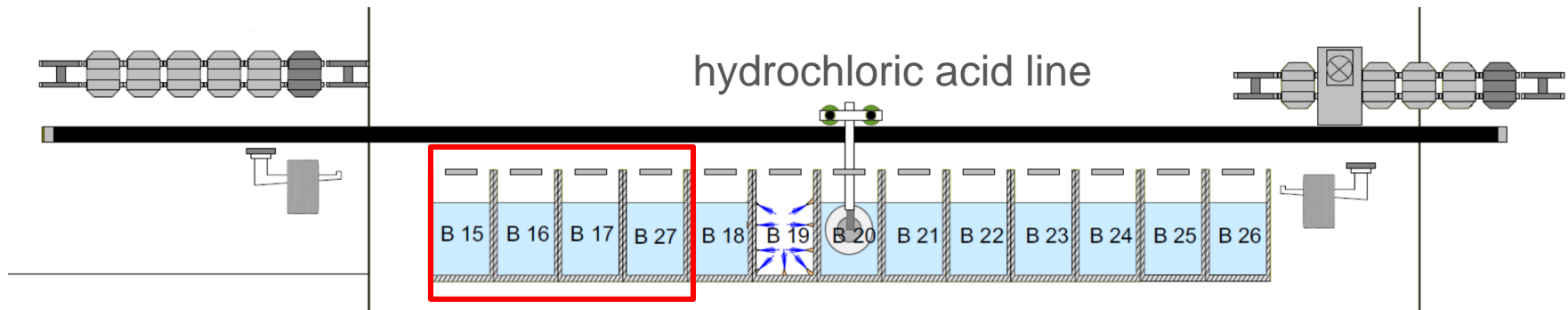
## Main products:

- › Wire rod steel : Ø 5,5 bis 30 mm
- › Bright Steel
  - › Long Products : Ø 2 bis 26 mm, length from 250 bis 6200 mm
  - › Wire rod: Ø 4 bis 22 mm

# Wire rod pickling plants



# Online-measuring technique for automated concentration supervision at wired rod HCl-pickling plant



## Operation HCL Pickling

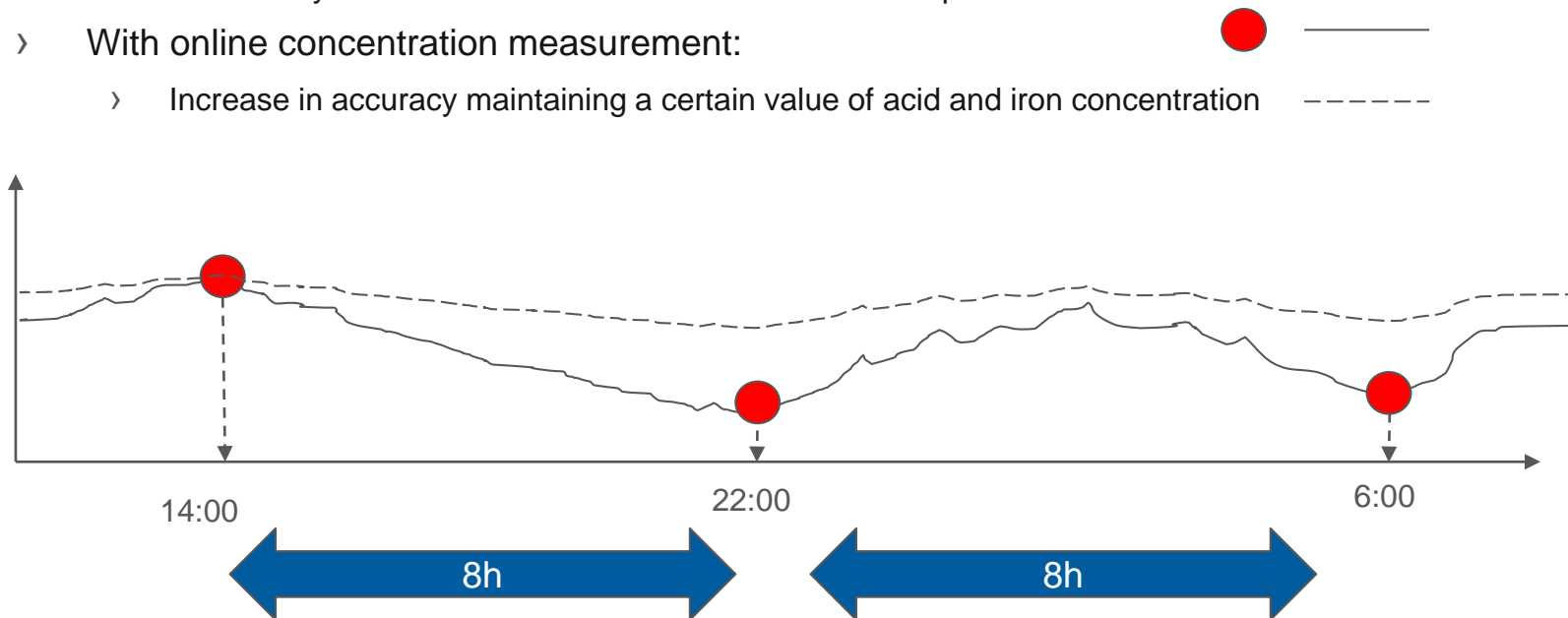
- › Pickling of Construction Steel, Bearing Steel
- › 4 HCL pickling baths, management of
  - › HCL concentration in g/l
  - › Fe concentration in g/l
- › Both values need to be maintained inside certain tolerances

# Online-measuring technique for automated concentration supervision at wired rod HCl-pickling plant



## Online- Measuring – Advantages

- › Offline – measurement – samples every 8h
- › Concentration management auto dosing depending on Amount of pickled material
- › With offline concentration measurement:
  - › Uncertainty in acid and iron concentration between samples for 8h
- › With online concentration measurement:
  - › Increase in accuracy maintaining a certain value of acid and iron concentration

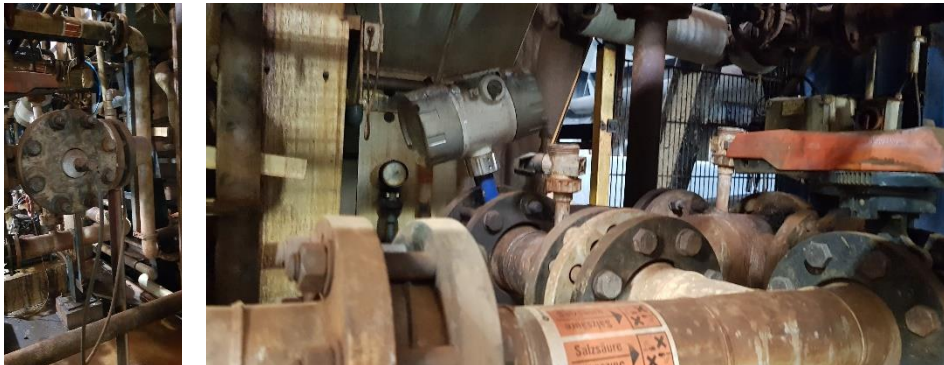


# Online-measuring technique for automated concentration supervision at wired rod HCl-pickling plant



## Operation of Online- Measuring

- › Two sensors per measurement unit
  - › Ultrasonic and conductivity sensor
- › 2 Units for 4 HCL Pickling Baths
- › Positioned inside the heating Loop
- › Continuous Measurement and Visualization of
  - › HCL Concentration in g/l
  - › Fe Concentration in g/l

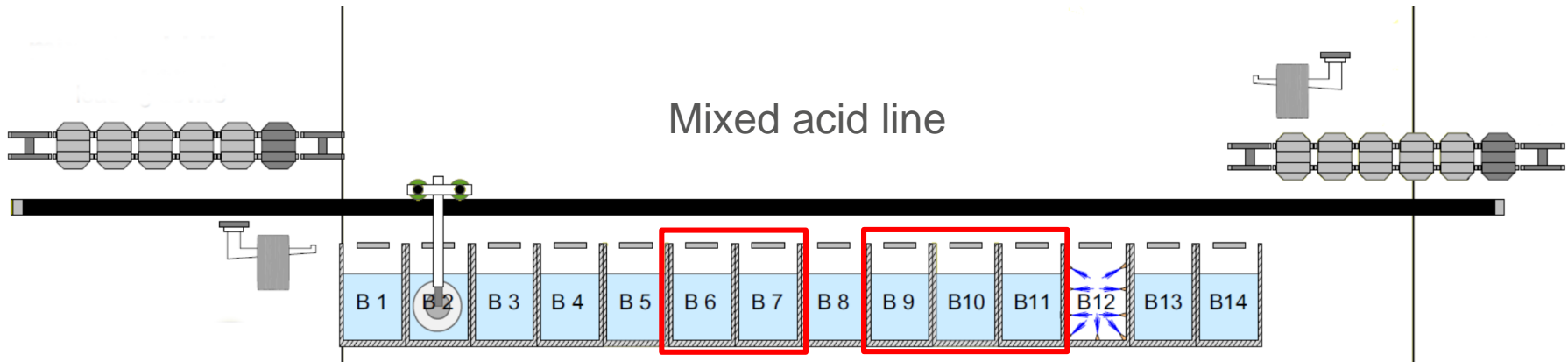


## Maintenance of Online - Measuring

- › Rinsing with NaOH once per month for 24h
- › Every 6 month visual inspection and recalibration
- › Operation since 2006 (13 years)



# Concentration monitoring and control at stainless steel wire rod mixed acid pickling (Project MACO Pilot)



## Operation Mixed Acid Pickling

- › Pickling of Heat and Acid Resistant Steel
- › 5 mixed acid pickling baths, management of
  - › HF concentration in g/l
  - › HNO<sub>3</sub> concentration in g/l
  - › Fe concentration in g/l
- › All three values need to be maintained inside certain tolerances

# Concentration monitoring and control at stainless steel wire rod mixed acid pickling (Project MACO Pilot)

## Initial situation at mixed acid line

- › The bath concentration supervision by at-line analysis is time consuming (manual sample drawing, analysis time about 30 minutes per tank sample) and thus the sample frequency limited to about 8 h.
- › Some steel grades can cause high temperature increase  $\gg 40\text{ °C}$  in the pickling tanks during the complex chemical reactions of the HF-HNO<sub>3</sub>-acid mixture
- › The adjustment of the acid concentration within the set-point ranges and the control of the tank temperature is very important for safe process control

## Solution

- › Additional installation of an high-rate mixed acid analysis system

# Concentration monitoring and control at stainless steel wire rod mixed acid pickling (Project MACO Pilot)

## Special Conditions inside mixed acid line

- › Problem: Very high solid matter accumulation in pickling acid  $>> 10$  g/L



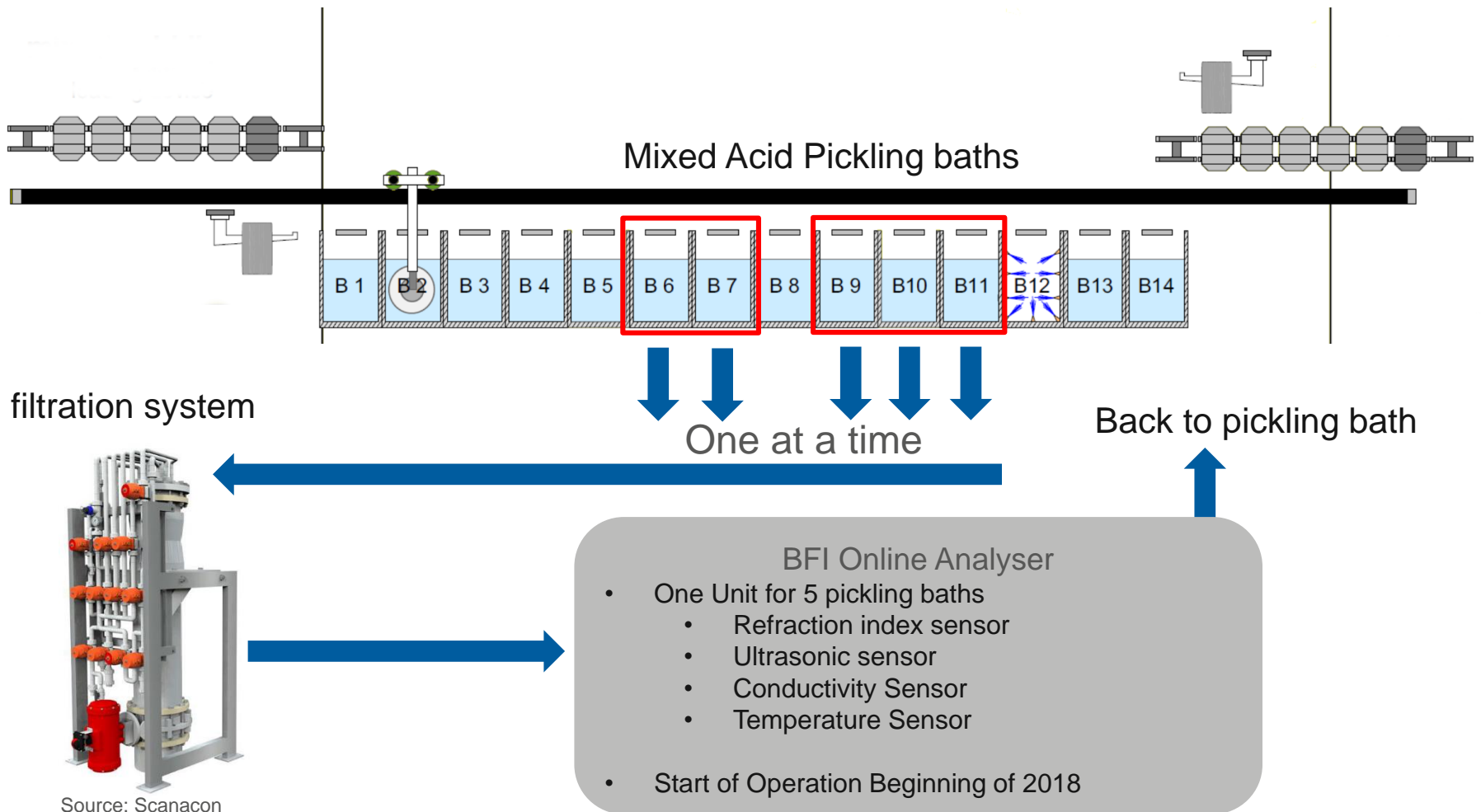
Solid matter inside  
Pickling bath

- › Refraction index sensor functionality of online analysis system limited to 9 g/L

## Solution

- › Solution: online analyser sample stream pre-treatment by filtration system

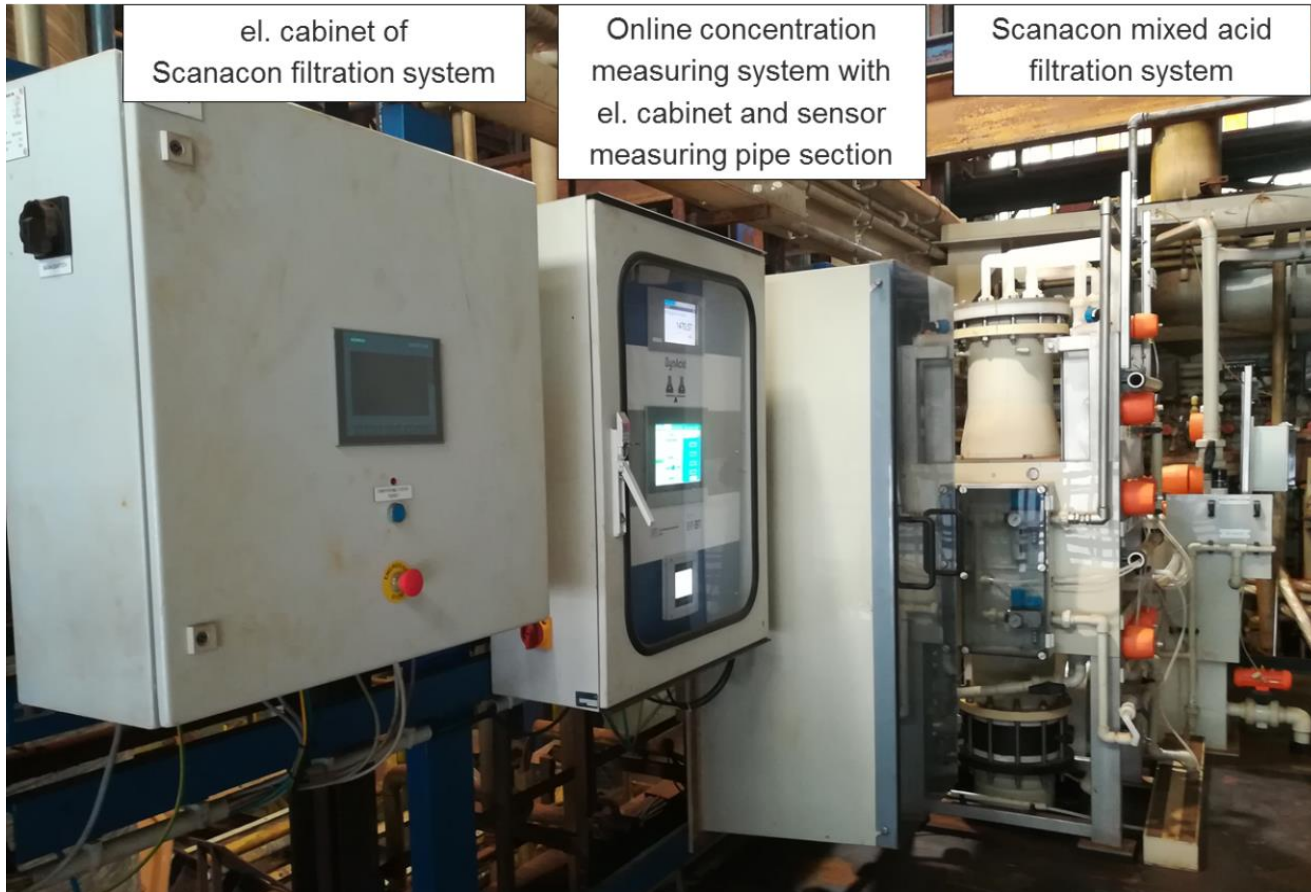
# Concentration monitoring and control at stainless steel wire rod mixed acid pickling (Project MACO Pilot)



Source: Scanacon

# Integration of concentration measuring system / filtration technique at the DEW mixed acid wire rod pickling plant

## › Installation of the equipment



# Summary of current main project results

- › Successful installation and commissioning of filtration technique and online concentration measuring system
- › The pickling tank concentration monitoring frequency can be increased from about 8 h (by chemical at-line analysis) to about 2,5 h by application of the online analysis.
- › The combination of at-line analysis and online-analysis enables a significant improvement of the pickling process control (faster adjustment of bath concentrations by open-loop correction dosage of acids + reduction of critical process conditions / better management of autocatalytical process conditions)
- › Online analysis system functionality and analyses accuracy depends on operation conditions (e.g. higher gas bubble accumulation in sample stream after filtration technique, low flow rates, temperature oscillations > 10 K/h during pickling tank switching ...)
- › At present, after calibration deviations between online analysis and BFI laboratory reference analysis < +/- 5 g/L for free HF and total metals and < 10 g/L for free HNO<sub>3</sub> concentration (under ideal measuring conditions)

## Tests and Optimisation of measurement equipment

- › Further optimisation works and tests at the pickling plant applications → 12/2019
- › Further optimisation of concentration calculation models → 12/2019
- › Testing of pickling programme management model tool (developed by University of Oviedo) → 12/2019

Thank you for your kind attention !

Open for discussion...