

“Optimisation of the mixed-acid online monitoring and control in stainless steel pickling plants”

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Content of Presentation

- › **Model based online concentration measurement –**

General overview of functionality and developments for HCl-Fe, H₂SO₄-Fe pickling acid systems

- › **Online concentration monitoring and control at stainless steel strip pickling lines –**

Overview of developments in RFCS Pilot project for HNO₃-HF-mixed acid systems

- › **Outlook**

Online-measuring technique for automated concentration supervision in acidic process bath

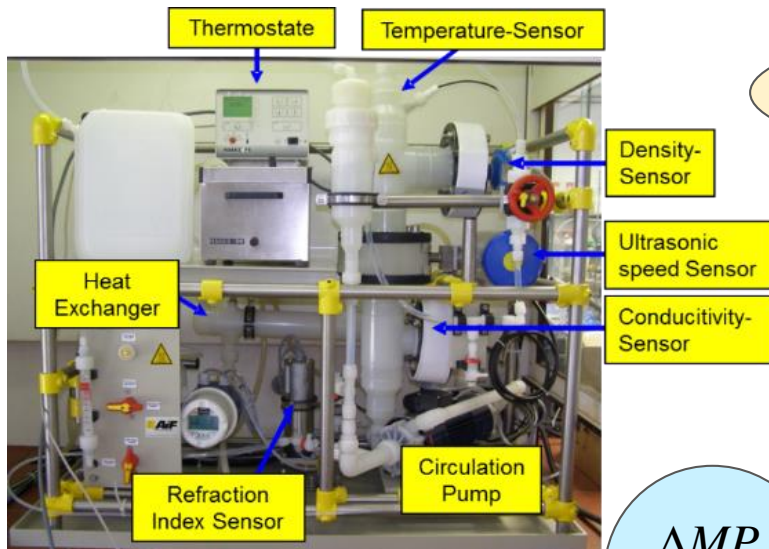
Initial Situation

- › Pickling by acid solutions is an important process step for fabrication of highly pure metal surfaces
- › Pickling bath supervision by manual process analysis → time and cost intensive
- › Not available online-concentration data → automated process control difficult
- › High product quality/plant-productivity demands fast adjustment and perpetuation of optimal set points of free acid and metal salts concentrations

Solution

- › Development of an operational online-measuring technique for continuous and simultaneous acid concentration analysis
- › Application of a model based analysis technique by physical parameter online measurement

Model-based online-concentration analysis for pickling acid solutions (DynAcid®) - Functionality



integral measuring method

operational online-measurement

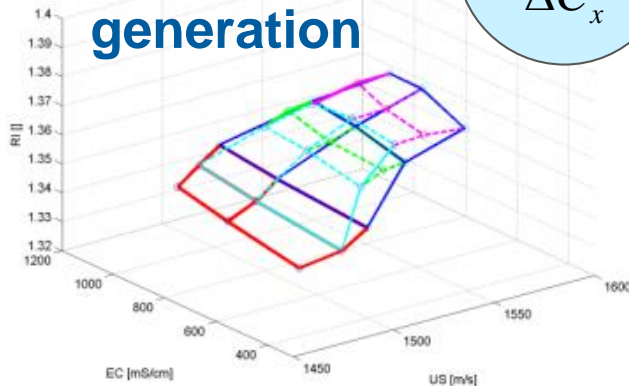
Physical parameter measurements
(T , v_{US} , λ_{el} optional RI or ρ)
continuously in acid solution

Mathematic-physical
concentration calculation model

Pickling bath concentrations (2 or 3)
continuously + simultaneously

$$\frac{\Delta MP_x}{\Delta C_x}$$

model database generation

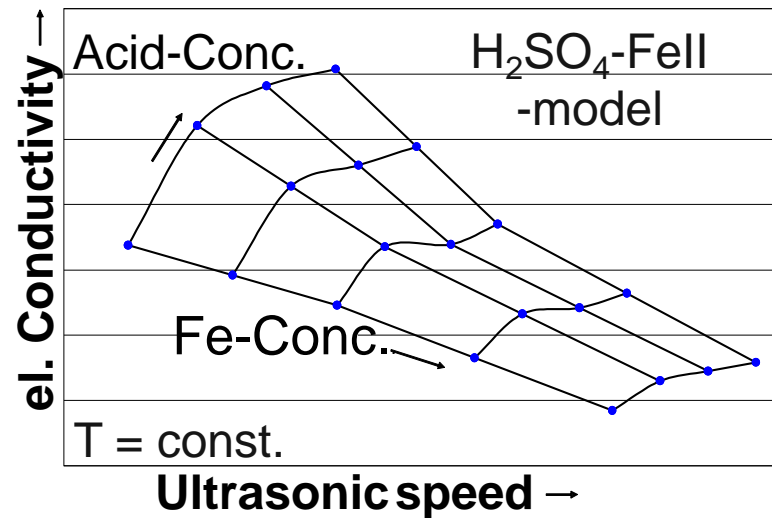


Development of concentration calculation models

Fundamental development of DynAcid®-Technique for HCl- and H₂SO₄-Fe pickling lines

- › Basic measuring method developments (model database + cal. model)
- › Set-up of measuring technique, testing in laboratory + technical centre

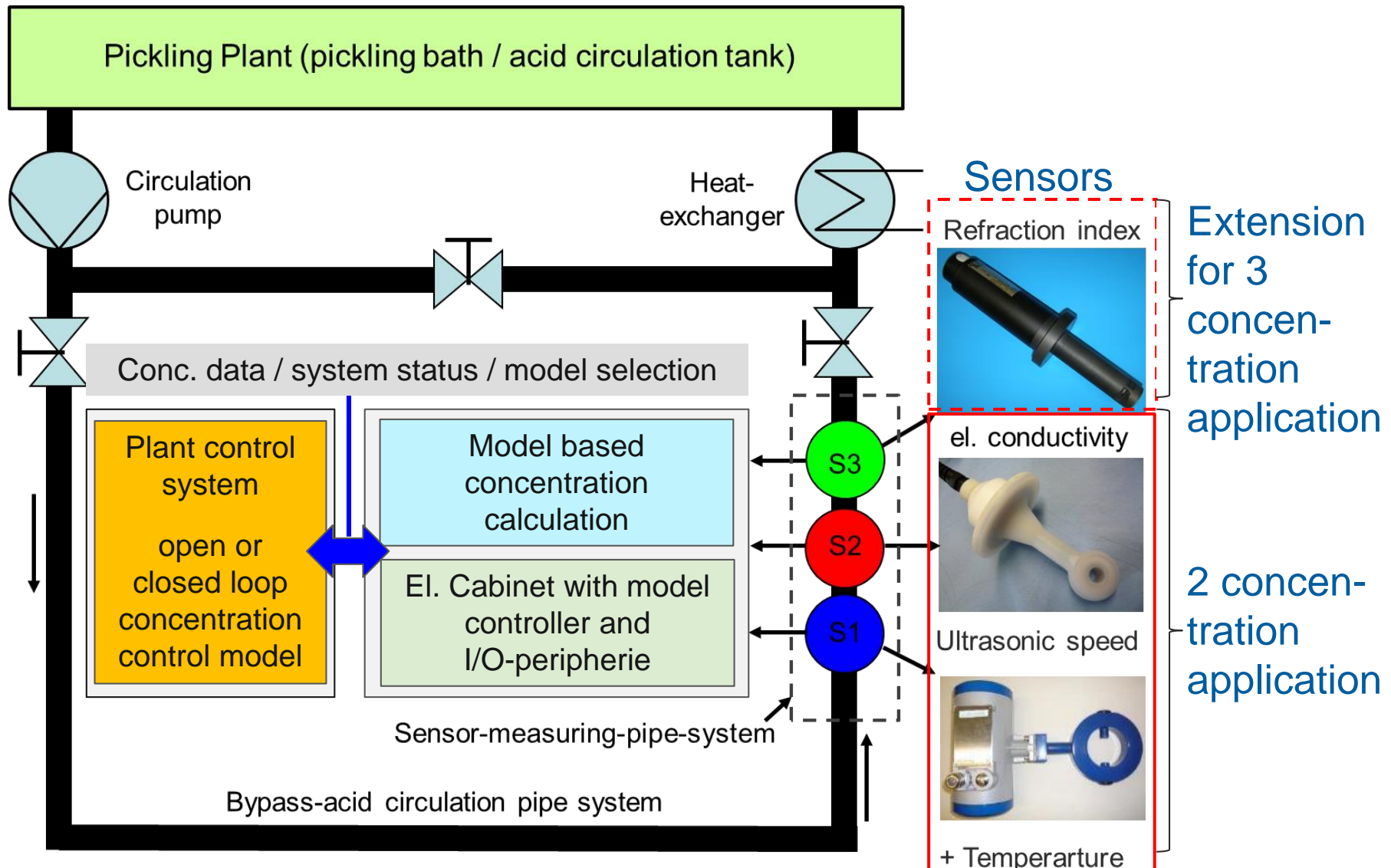
Set of characteristic curves from laboratory investigations



Mobile measuring system test and demonstration equipment

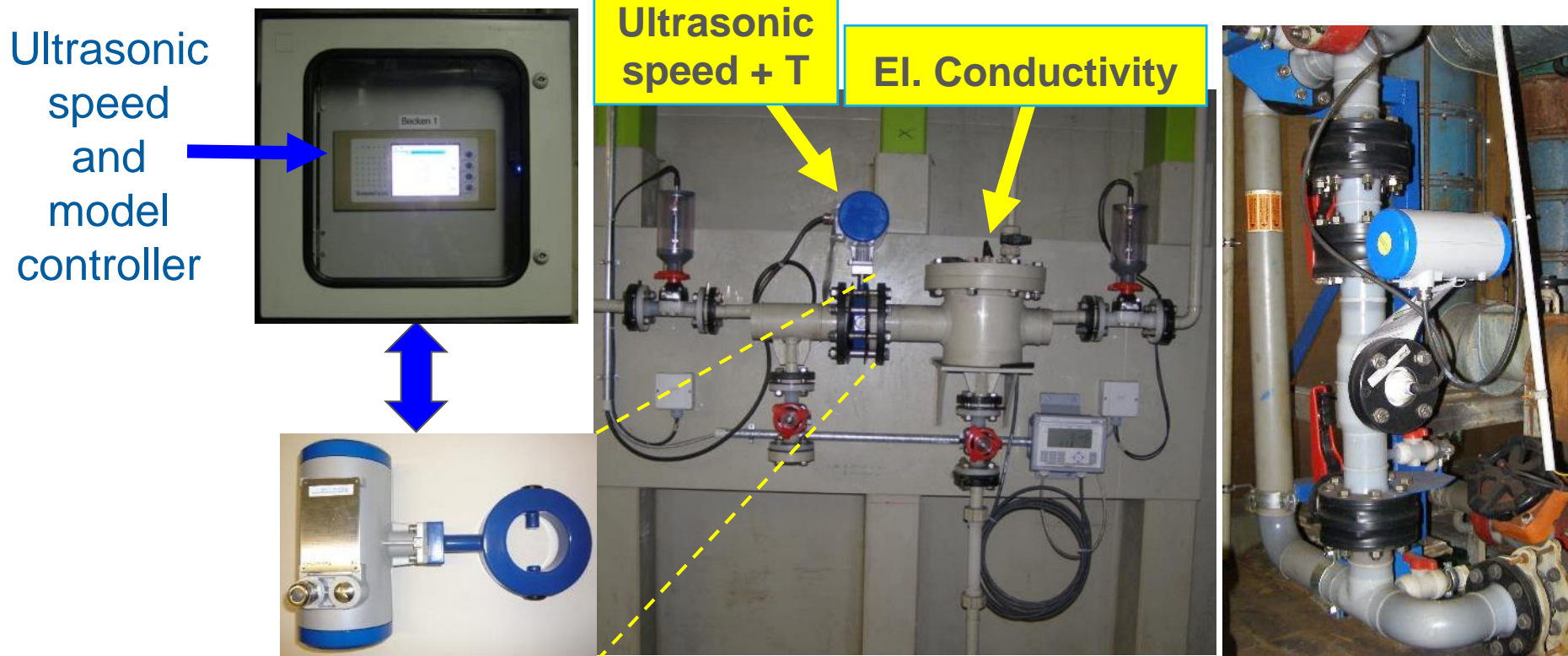


Concept for operational online measuring system integration at pickling plants



Operational installation of DynAcid[®]-Technique for HCl- and H₂SO₄-Fe pickling lines

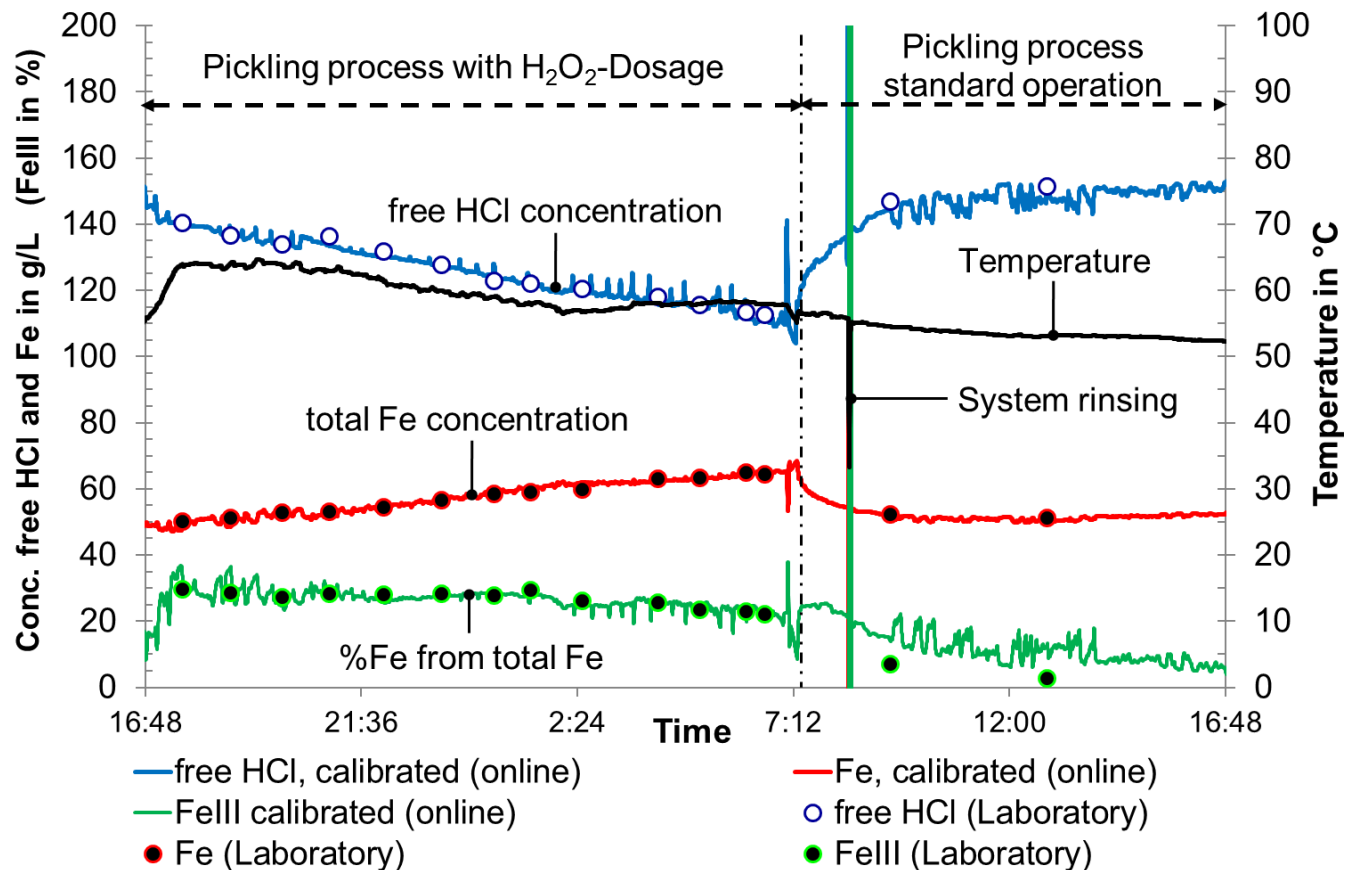
› Examples of operational installations and commissioning



Measuring technique application at pickling lines

Special development of online measuring technique for HCl-FeII-FeIII stainless steel pickling bath applications

- › Continuous concentration monitoring and FeIII-FeII-ratio adjustment by H₂O₂-dosage
- › Operational demonstration at dip-tank HCl-wire rod pickling plant



- › **Calibration** of model based online analysis systems **on operational measuring conditions** by laboratory reference analysis
 - differences between artificial model solutions and pickling acids
 - differences in measuring system set-ups
 - specific operational temperature and concentration ranges
- › **Measuring conditions of acid media:** As possible low gas bubble and sludge accumulation, flow rate $> \sim 6 \text{ m}^3/\text{h}$, $dT_{\text{sample}} < 10 \text{ K/h}$,
- › **Measurement accuracy:** Deviations to reference analysis mainly $\sim < 5 \text{ g/L Fe}$ and $< 8 \text{ g/L HCl /H}_2\text{SO}_4$
- › **Maintenance** – depending on operational application:
 - Sensor-(pipe section) rinsing from solid matter (\sim every 2 to 7 days)
 - Validation of basic functionalities (\sim 2 to 4 times/year)

Commercial availability of DynAcid® online measuring technique for H₂SO₄- and HCl-pickling bath applications

Sales and distribution + commissioning and support

- BFI Betriebstechnik GmbH (Düsseldorf)
- SensoTech GmbH (Magdeburg-Barleben)
+ Ultrasonic speed sensor and model controller technique

Specially offered measuring technique service options by BFI

- Consulting concerning system installation and operation
- System commissioning on-site the operation locations
- System calibration on operation conditions by reference analysis
- System maintenance (online sensor dismounting, cleaning and validation, failure analysis and repair support)
- System demonstration installations

Overview DynAcid®-Installations by BFI + BFI-Betriebstechnik GmbH (inclusive commissioning)

- HCl-pickling lines (each 2 systems)
 - DEW (Germany, dip-tank wire rod pickling), ~ 2006 →
 - TKSE (Germany, strip pickling line), ~ 2007 →
 - Turkey (Iskenderun, strip pickling line), ~ 2011 →
 - South Korea (Pohang, strip pickling line), ~ 2012 →
- H₂SO₄-pickling lines
 - TKS (Germany, strip pickling line) → 4 systems, ~ 2008 →
 - TK (Germany, strip pickling line) → 2 systems, ~ 2009 → ~ 2015
(facility closed)

MACO-Pilot – Optimisation of the mixed-acid online monitoring and control in stainless steel pickling plants

RFCS Project (07/2016 – 12/2019)

- › Optimisation of the **online concentration measuring technique for different HF-HNO₃-mixed acid** pickling plant applications
 - calculation model enhancement, system set-up, sensor-lifetime
- › **Operational installations and optimisation of prototype systems** at stainless steel strip and wire rod pickling plants
- › **Improvement of the pickling process operation** at stainless steel pickling plants
 - Closed loop control model, pickling programme management tool

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high performance stainless steel

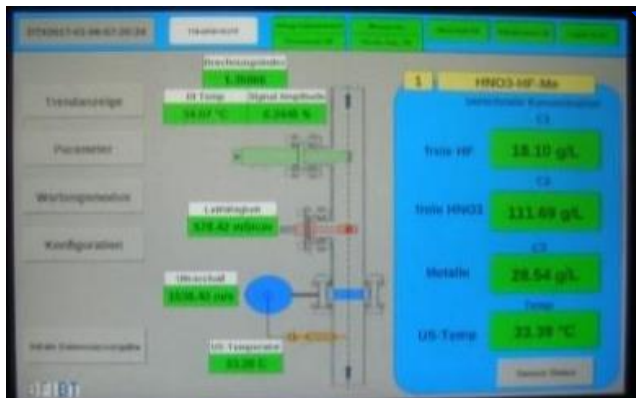
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Optimisation of online measuring system set-up for HNO_3 -HF mixed acid applications

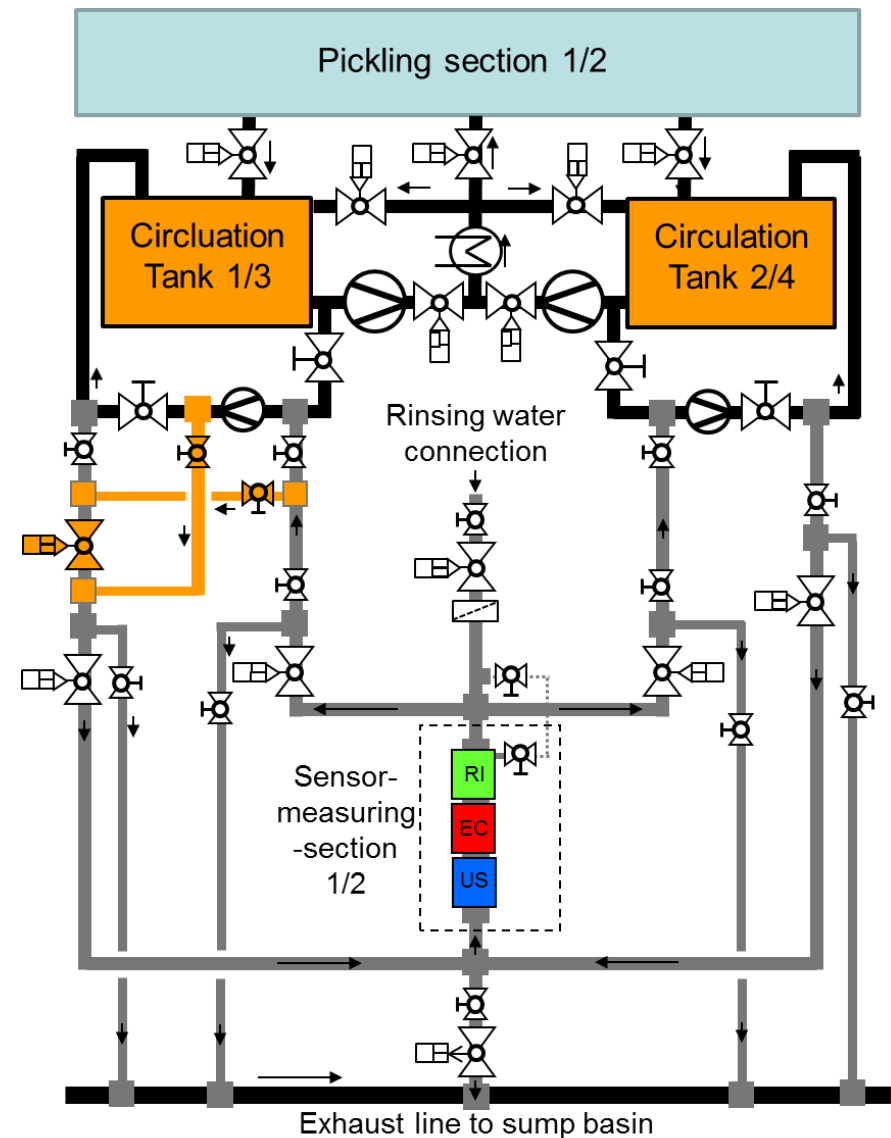
- › New types of sensors for refraction index and ultrasonic speed measuring
- › New el. cabinet model-controller and physical parameter sensor-measuring pipe set-up

Main Data Screen



Integration of online concentration measuring prototype systems – example for a modern strip pickling line

- 2 acidic pickling sections with 2 acid circulation and storage tanks per section
- Fast pickling programme change by switching between 2 different bath concentrations (e.g. ferritic / martensitic)
- Installation of 1 online measuring system per section
- Supervision of acid circulation tanks by automatically controlled switching
- Automatically controlled sensor measuring pipe water rinsing

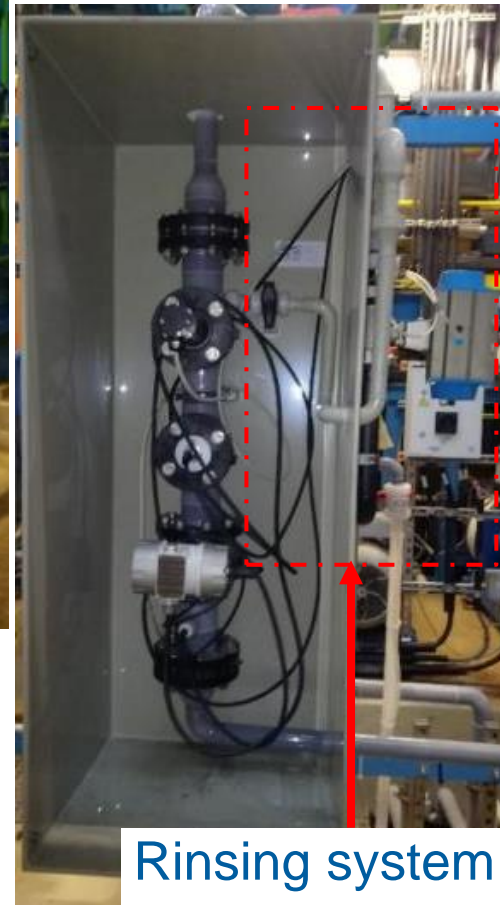


Example for realised online concentration measuring prototype system installation at strip pickling line



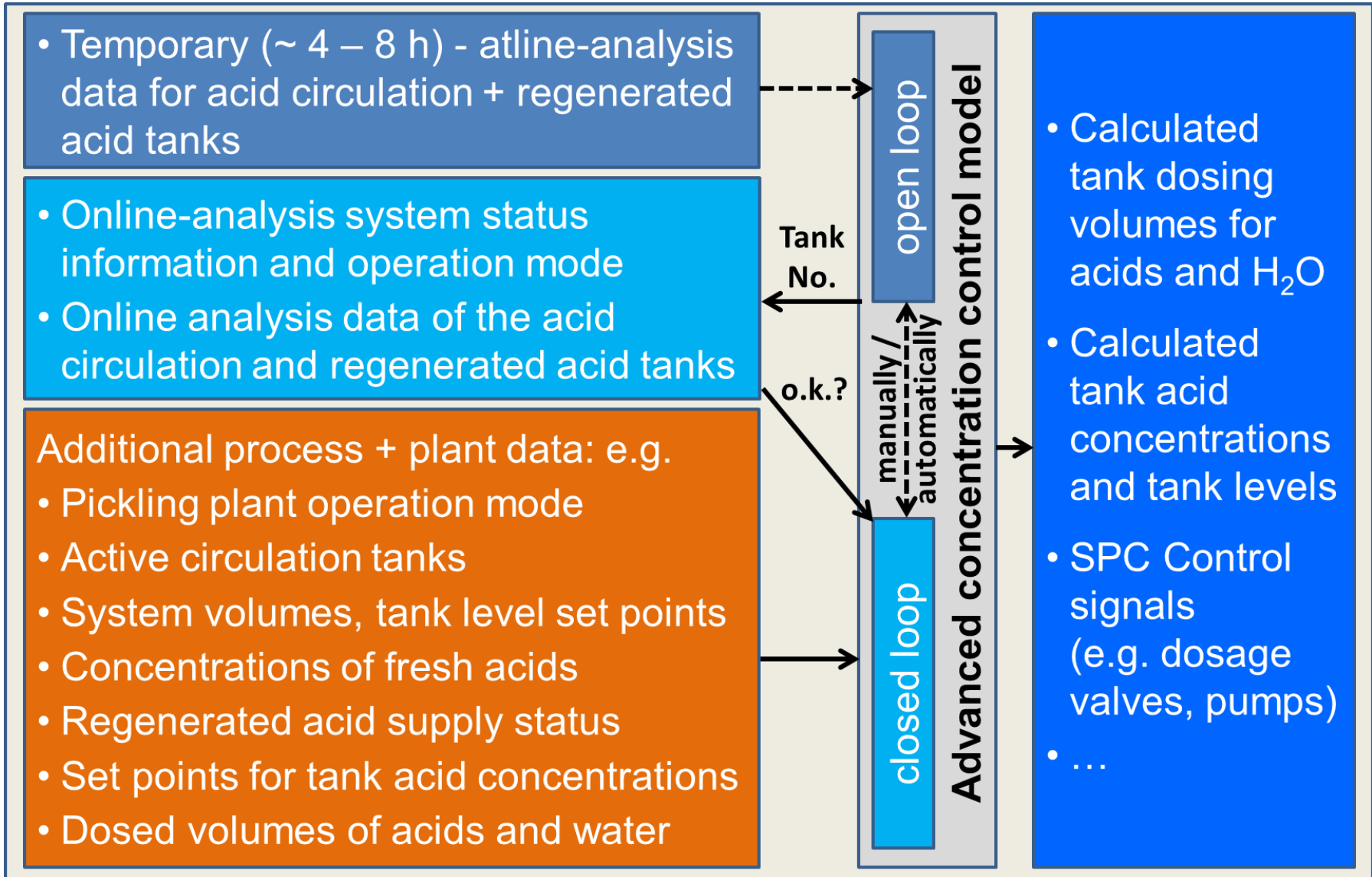
Integration of an online measuring systems at strip pickling line

Sensor measuring pipe system

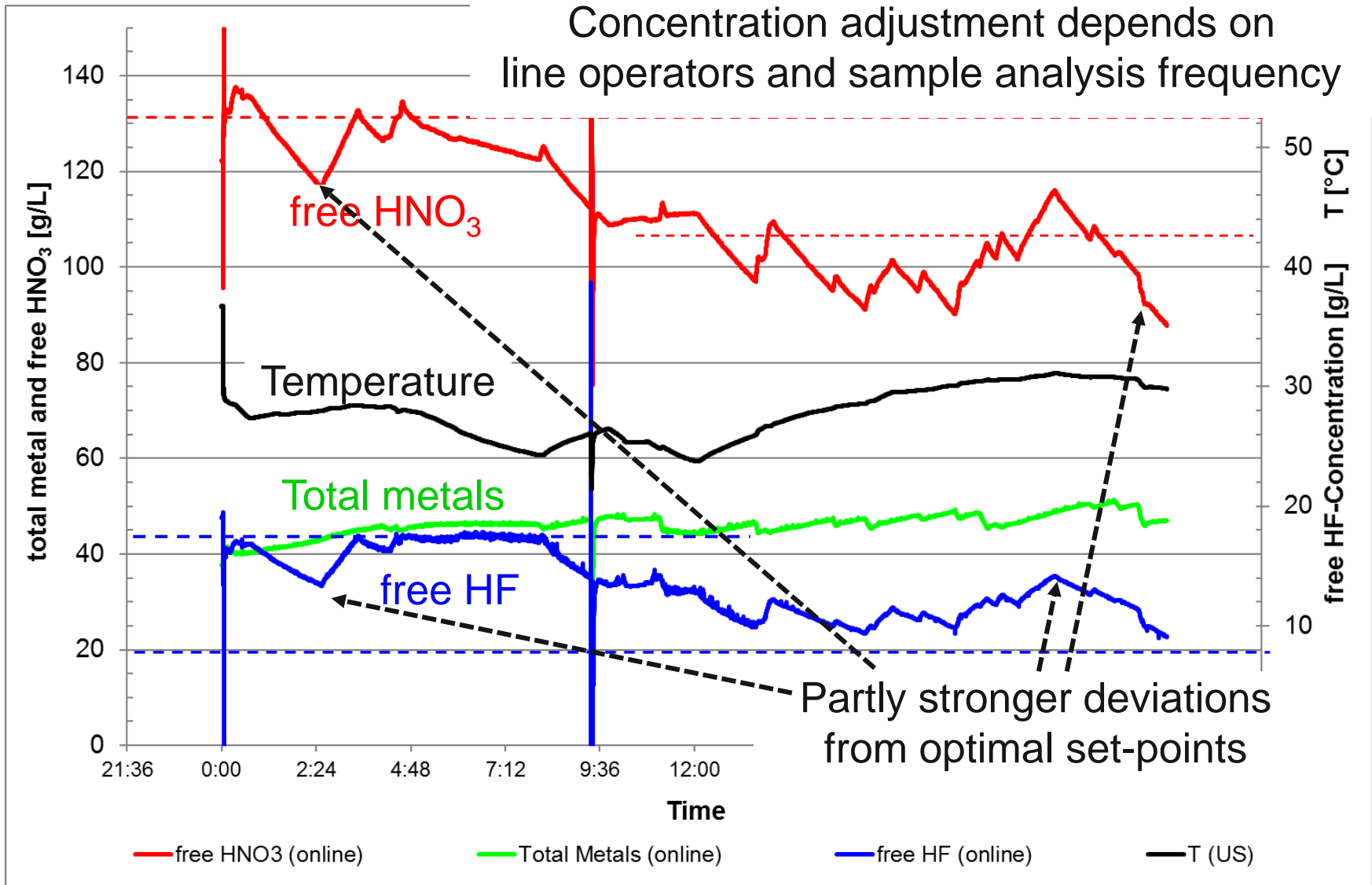


Connection to acid circulation system

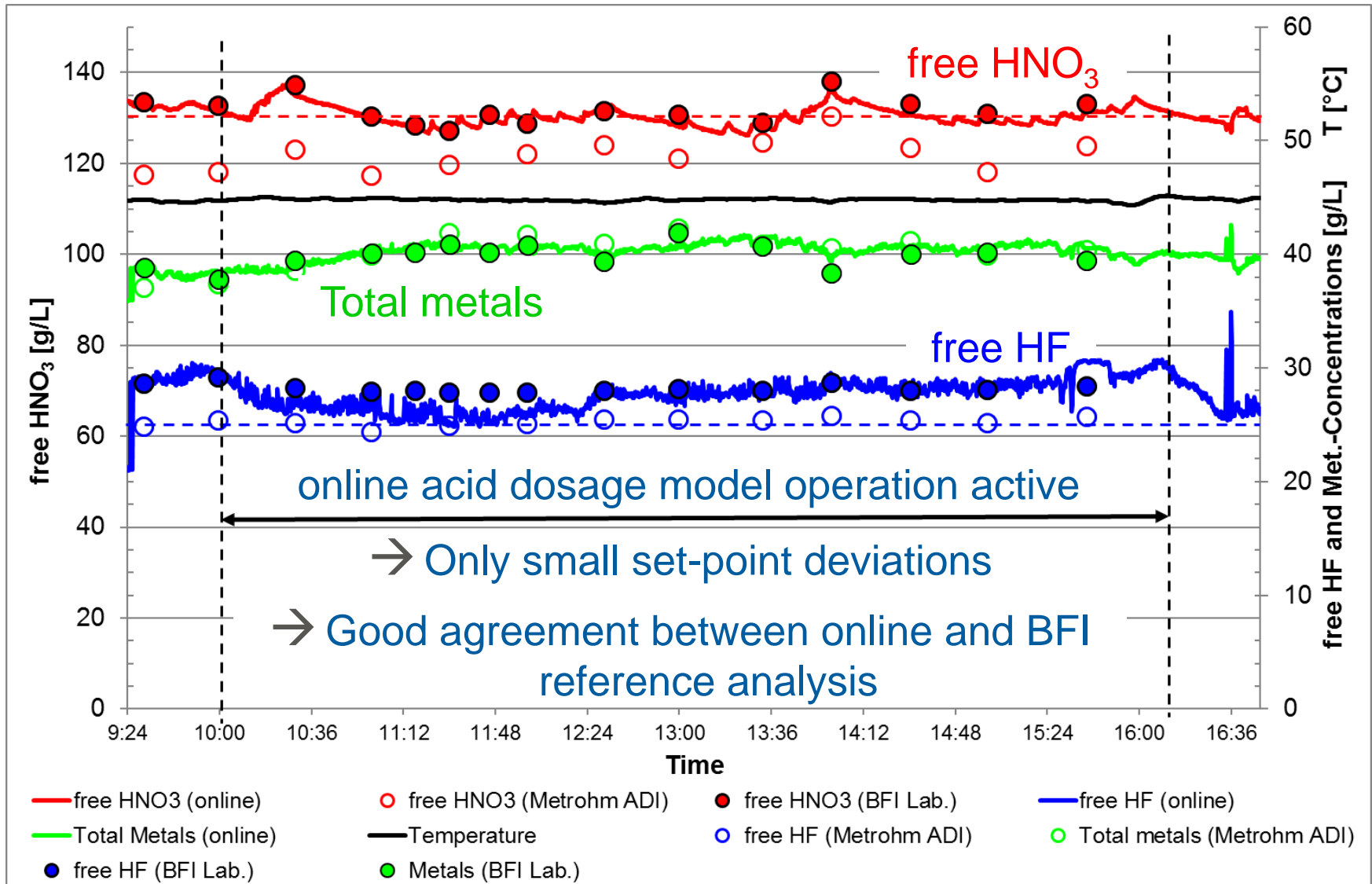
Example of advanced closed-loop concentration control model for strip pickling lines



Concentration monitoring and control at strip pickling line by manually operated pickling bath concentration control



Concentration monitoring and control at strip pickling line by automatic online pickling bath concentration control



Summary of current MACO Pilot results

- › Optimised online-measuring system prototype set-up
- › Successful installations of online measuring systems at strip and wire rod pickling plants
- › First testing of closed-loop online concentration control model for strip pickling process shows good results
- › Online measuring system functionality and analyses accuracy depends on operation conditions (e.g. gas bubble and sludge accumulation)
- › At present, deviations between online analysis and BFI laboratory reference analysis mainly $< \sim 5 \text{ g/L}$ for free HF + total metals and $< \sim 10 \text{ g/L}$ for free HNO_3 concentration
- › Enhanced information concerning online sensors operational life-time experiences (at present $\sim 2 - 6$ years)

RFCS Project MACO Pilot

- › Further optimisation works and tests at the pickling plant applications
→ 12/2019
- › Further optimisation of concentration calculation models → 12/2019
- › Realisation of marketable measuring technique on basis of the experiences and prototype system set-up, 1/2020 →

Ongoing online measuring system application developments

- › H_2SO_4 -Zn-Fe acid solution mixtures (recovery of Zinc from steel scrap)
- › HNO_3 - H_2SO_4 -Zn acid mixtures (surface treatment of Zn products)
- › ...

Thank you for your kind attention !

Open for discussion...

Contact

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