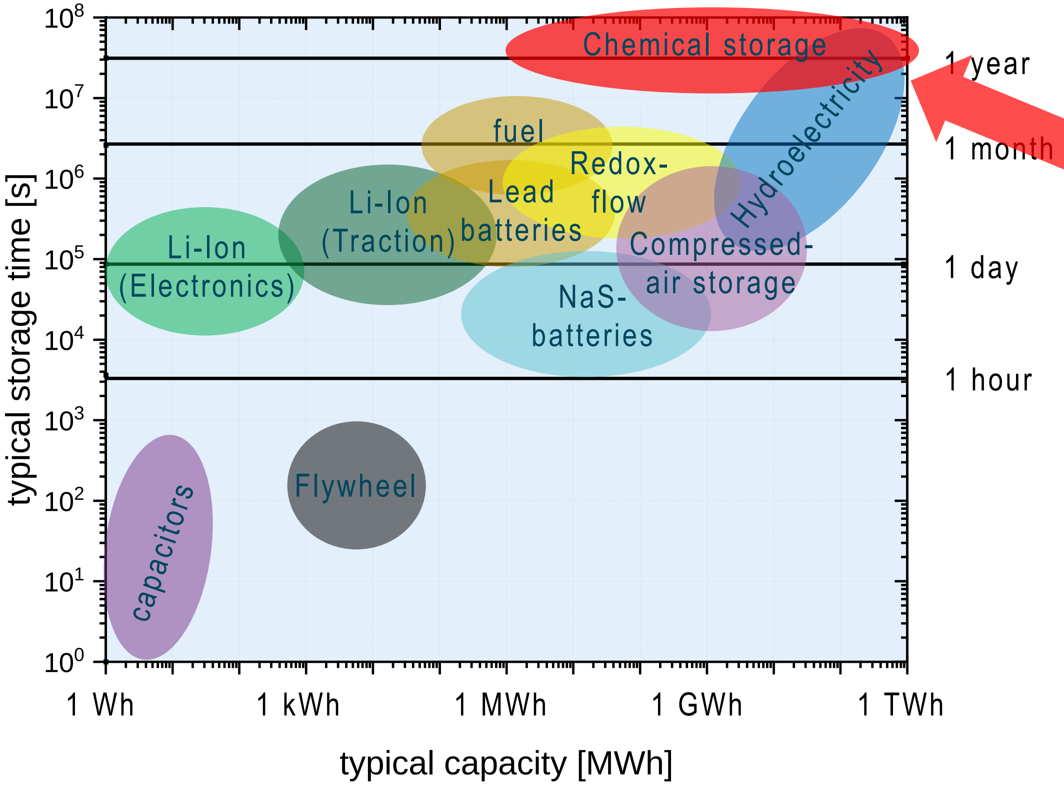
High throughput technology in electrochemistry

FZJ: B. Hecker, H. Tempel, D. Dogan, R.-A. Eichel
hte: A. Müller, F. Schneider, F. Huber, M. Dejmek, G. Wasserschaff

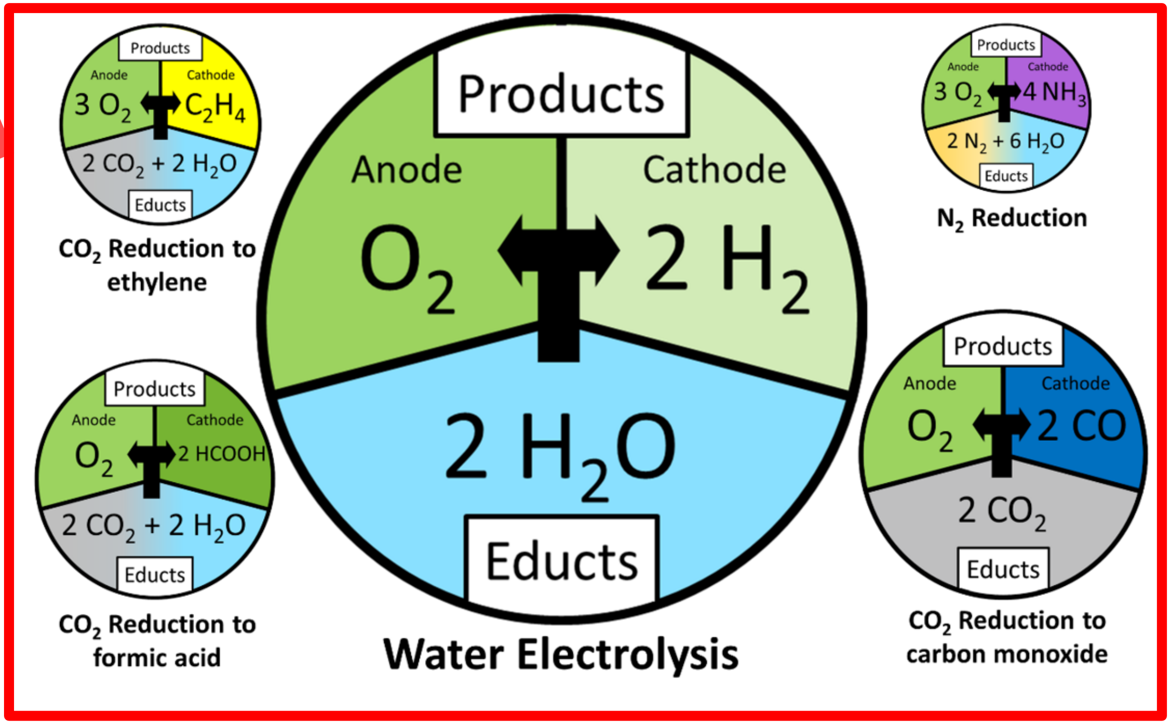
IEK-9, fundamental electrochemistry

Forschungszentrum Jülich

Different storage technologies for electrical energy



Different technologies arranged by their typical storage time and capacity [1]



Educts and Products for the water electrolysis and further electrochemical reactions that can be applied storing renewable electricity

[1] Adopted from: Schüth, Ferdi, and Rüdiger A. Eichel. "Energiespeicher für die Zukunft." *Physik Journal* 13.10 (2014): 31-36.

IEK-9, fundamental electrochemistry

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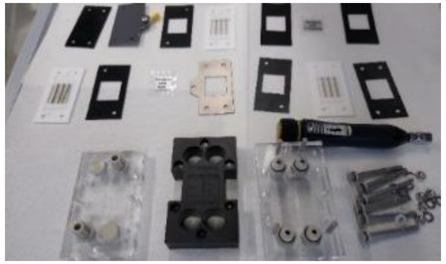
Different research fields:

Catalysts → **Electrode** → **Reactor** → **System** → **Process**

Applied cells



Research focus

Catalysts	Electrode	Reactor	System	Process
Fundamental chemistry	Increased reaction rate	Improving efficiency	Ensure Scalability	Economical application

Electrochemical catalyst research example:

CO_2 electrolysis to CO

Power to CO

CO_2 Electrolysis: $2\,CO_2 \xrightarrow{+\text{ Electricity}} 2\,CO + O_2$

Reduction of CO_2: $2\,CO_2 + 2\,H_2O + 4\,e^- \xrightarrow{[\text{Ag-nano-structures}]} 2\,CO + 4\,OH^-$

Selectivity

Activity

Durability

Oxidation of Water/ Oxygen evolution reaction: $4\,H_2O \xrightarrow{[\text{OER catalyst (Ni, Pt)}]} O_2 + 4\,H^+ + 4\,e^-$

Electrochemical catalyst research example:

CO_2 electrolysis to CO

Results by B. Hecker and E. Robens

Planning/ Organization	Synthesis/Preparation	Electrochemical measurements	Evaluation/ Interpretation

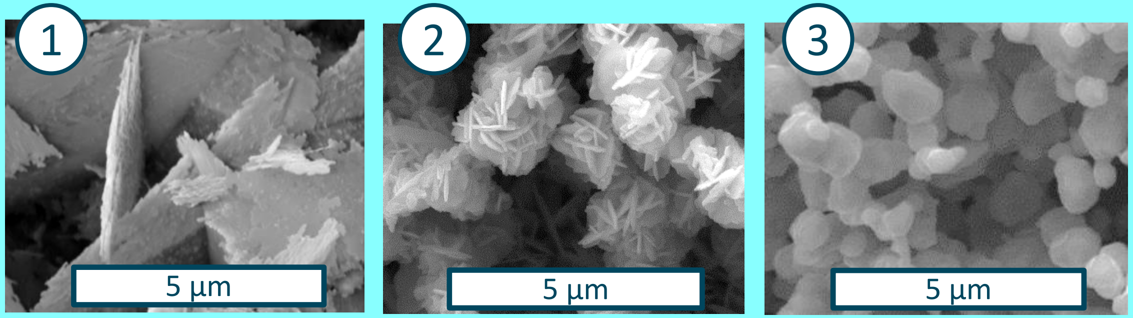
Goals:
Investigating catalytic effects of:

1. Reactive Edges
2. Mass transport effects
3. Electrochemical surface area

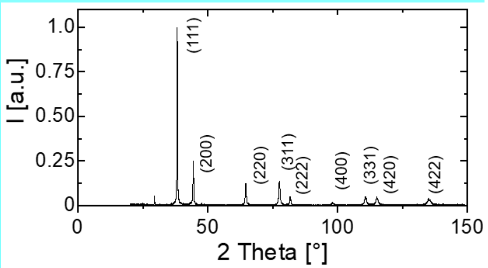
Literature research

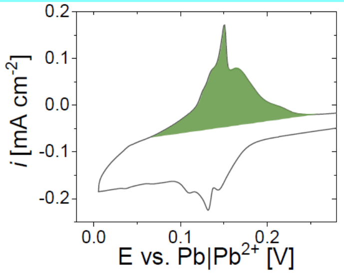
Theoretical estimations

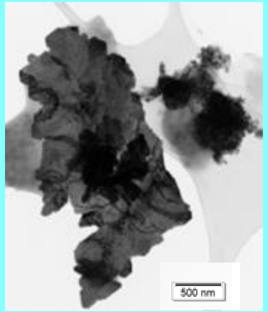
Planning lab experiments



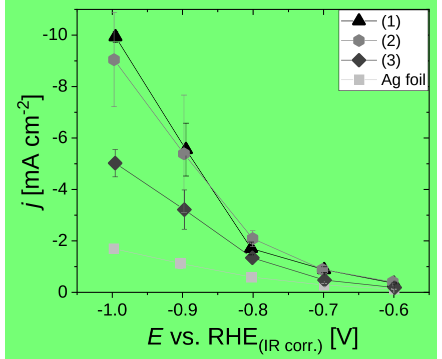
SEM images synthesized Ag catalysts



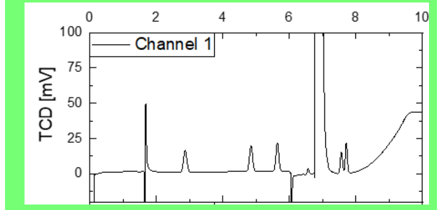




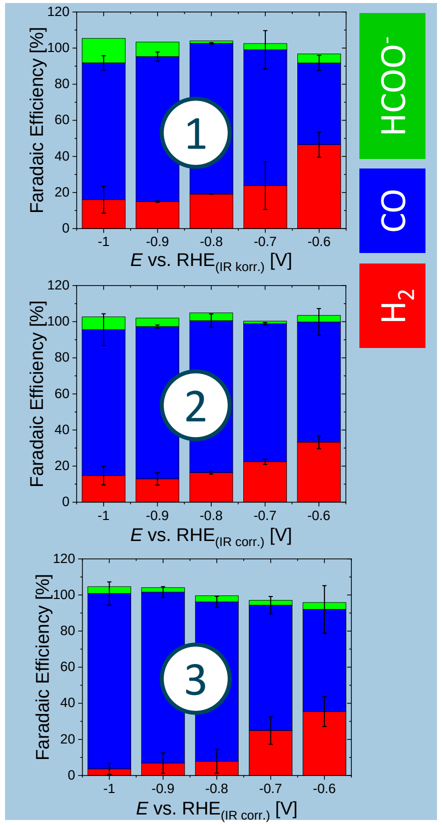
XRD 1 **Pb-UPD** 1 **TEM** 1



Current density



GC signal

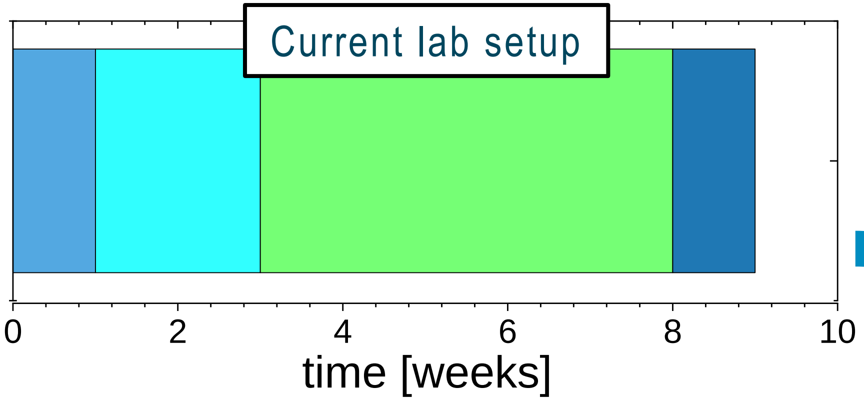


Electrochemical catalyst research

time frames performing a study

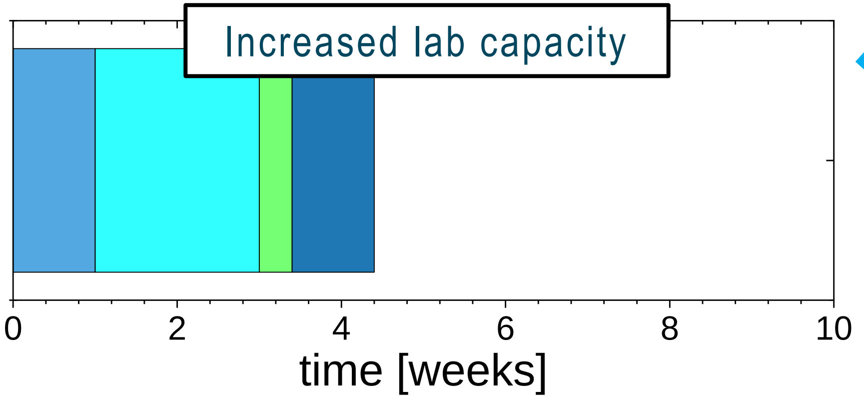
Planning/ Organization	Synthesis / Preparation	Electrochemical measurements	Evaluation/ Interpretation

Tuning the Selectivity for the CO_2 Reduction towards CO through specific Synthesis of Silver Catalysts with different Morphologies



~9 weeks

Similar studies using an increased capacity for electrochemical measurements



~4.5 weeks

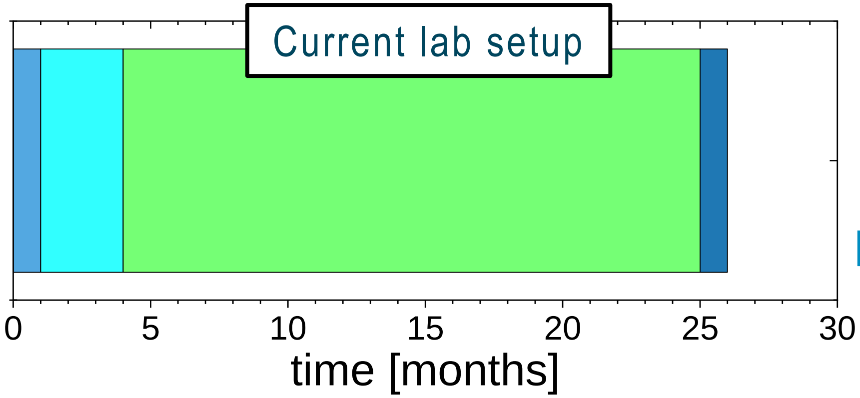
Timeframes used as an example based on experience

Electrochemical catalyst research

time frames performing a study

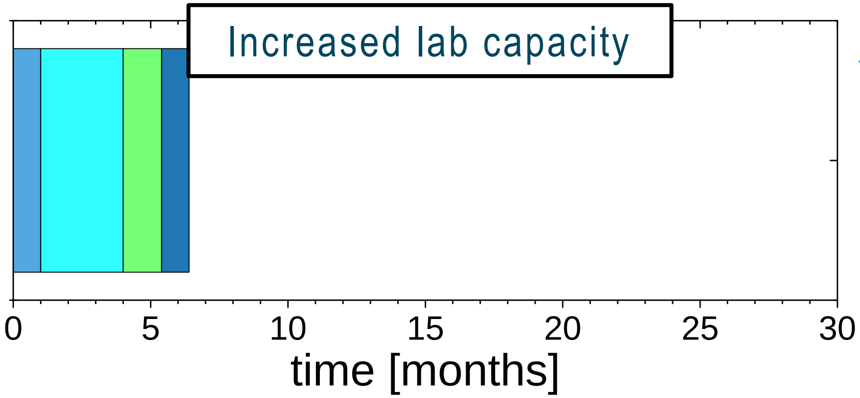
Planning/ Organization	Synthesis / Preparation	Electrochemical measurements	Evaluation/ Interpretation

Long term study including 16 experiments á 1000 h



~26 months (to slow, not applicable)

Similar studies using an increased capacity for electrochemical measurements



~6.5 months

Timeframes used as an example based on experience

Electrochemical catalyst research

Challenges in increasing the lab capacity

Electric supply/measurements

Gas supply

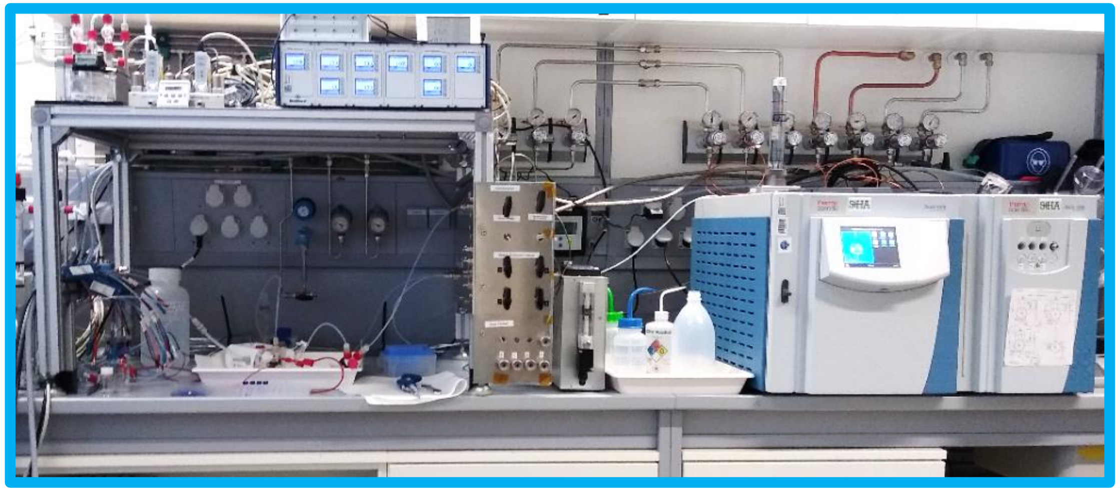
Liquid supply

Product quantification

Temperature control

pressure control

Electrochemical reactor/ cell



Lab setup for electrolysis experiments in Jülich

General challenges:

- Many parameters
- Broad range for the settings*
- High manual workload
- Hardly comparable
- Need for general test standards[1]
- High quality preparation/synthesis needed

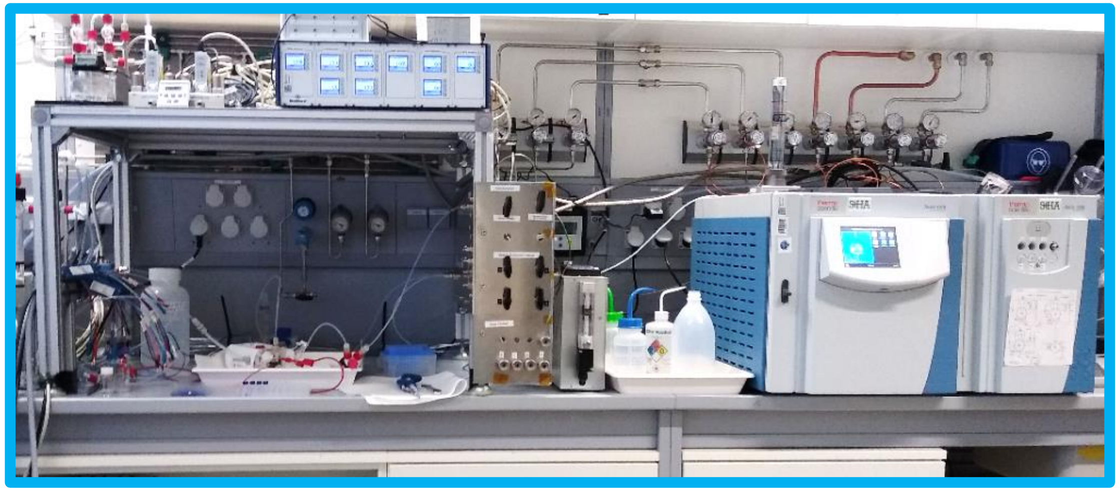
*Depending on the respective experiments/research goal

[1] Beck, Lorenz, et al. "Investigation and standardisation of electrolysers for green hydrogen production using the test infrastructure in Bremerhaven, Germany." *Journal of Physics: Conference Series*. Vol. 2257. No. 1. IOP Publishing, 2022.

Electrochemical catalyst research

Challenges in increasing the lab capacity

- Electric supply/measurements
- Gas supply
- Liquid supply
- Product quantification
- Temperature control
- pressure control
- Electrochemical reactor/ cell



Lab setup for electrolysis experiments in Jülich

Preexisting experience in high throughput application

High throughput development required

- Electric supply/measurements
- Gas supply
- Liquid supply
- Product quantification
- Temperature control
- pressure control
- Electrochemical reactor/ cell

Example for a high throughput system by hte

Electrochemical catalyst research

Performed tasks

Tasks

- Adjusting the liquid and gaseous supply
- Adjusting the product quantification
- Adjusting the temperature and pressure control
- Designing the electric supply
- Designing an electrochemical reactor
- *Preexisting experience in high throughput application*
- *High throughput development required*

Electrochemical catalyst research

Performed tasks

Tasks

Adjusting the liquid and gaseous supply

Adjusting the product quantification

Adjusting the temperature and pressure control

Designing the electric supply

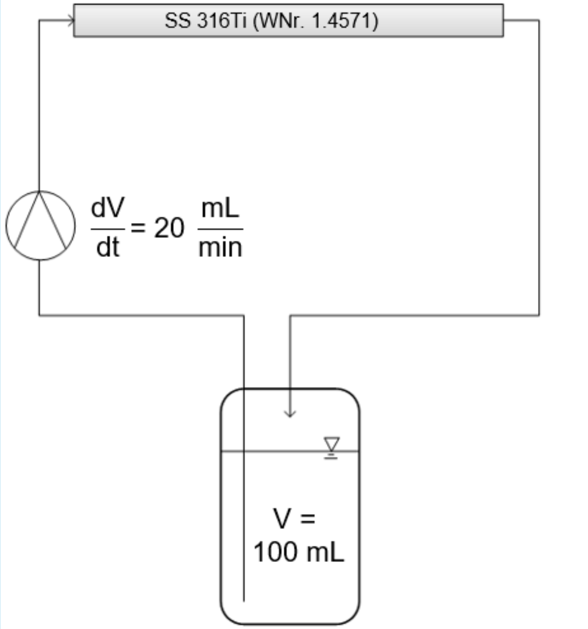
Designing an electrochemical reactor

Preexisting experience in high throughput application

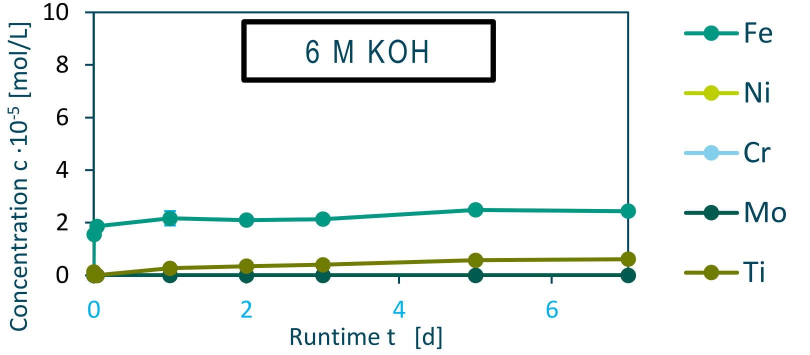
High throughput development required

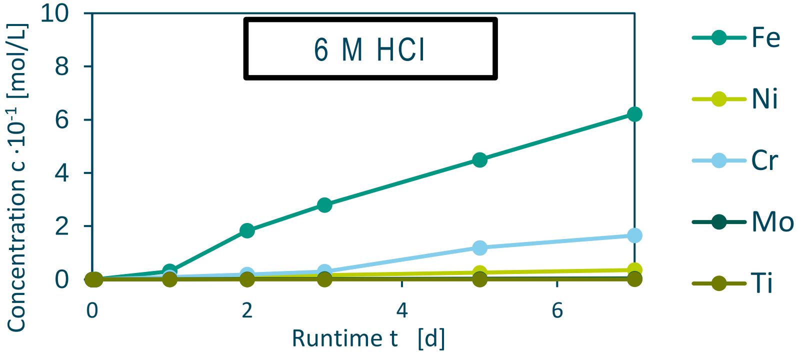
Metal ions dissolved from stainless steel using different electrolytes

$A_{contakt,SS316Ti}$ = 20 cm², l = 15,9 cm, d_o = 6 mm



$c_{metall,max} = 10^{-6}$ mol L^{-1}





Experiments by D. Dogan

→ Using polymer materials instead of steel components for the liquid supply

Electrochemical catalyst research

Performed tasks

Tasks

Adjusting the liquid and gaseous supply

Adjusting the product quantification

Adjusting the temperature and pressure control

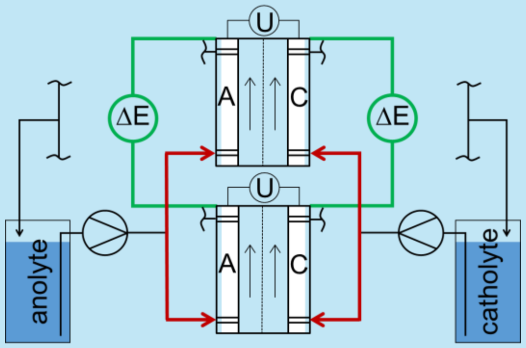
Designing the electric supply

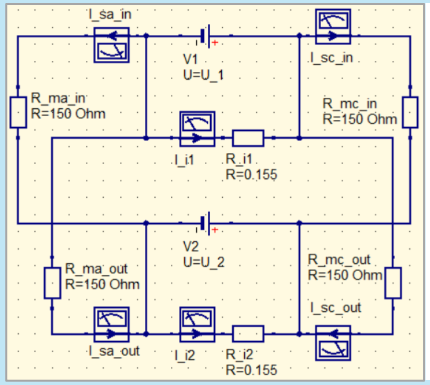
Designing an electrochemical reactor

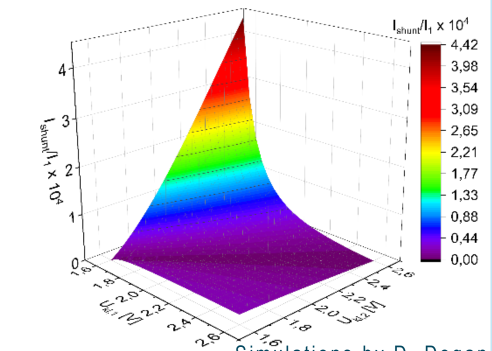
Preexisting experience in high throughput application

High throughput development required

Shunt current between parallel operating electrochemical cells with ionic connection







Cell-to-cell shunt current is caused by potential difference between adjacent electrodes

Computer model in Quite Universal Circuit Simulaton (QUCS):

Simulation of relative shunt current for 2-cell-model:

→ Ensure ionic and electric isolation between the electrochemical cells

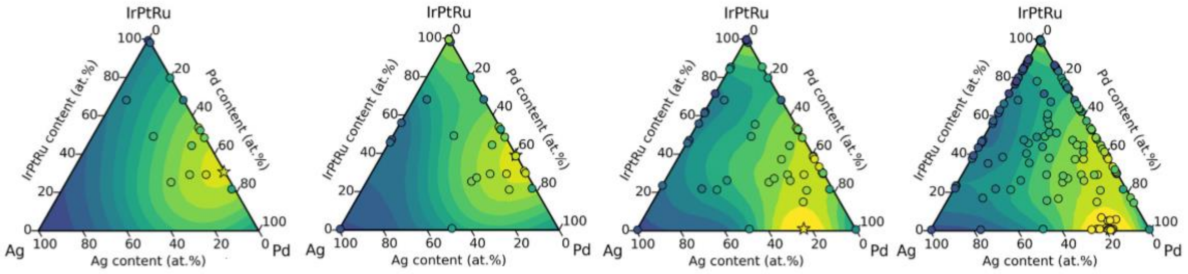
Electrochemical catalyst research

Possible advance applications

Using advanced data evaluation

e.g. creating multidimensional heat maps for the electrochemical performance depending on multiple parameters

15 samples | 28 samples | 54 samples | 150 samples



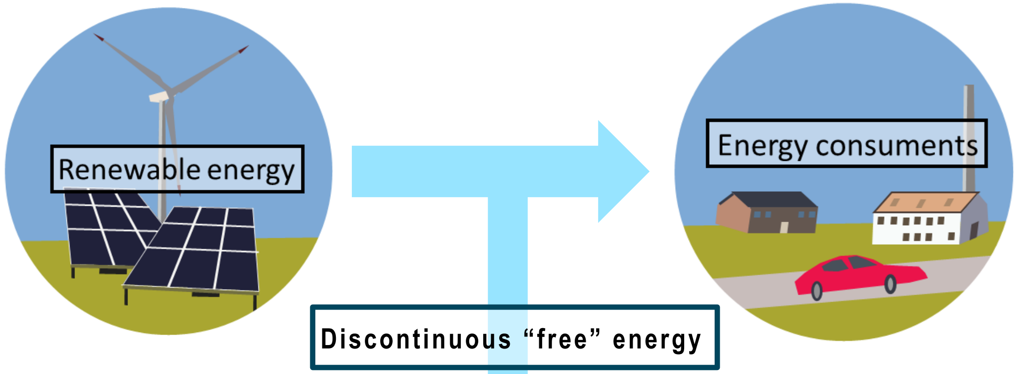
Excample: ORR catalyst performance depending on the composition

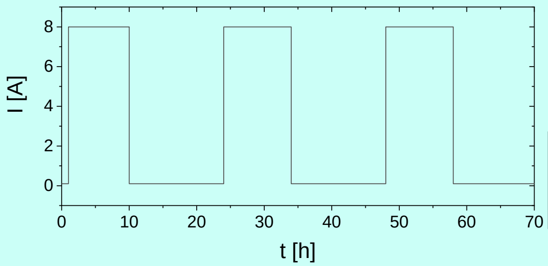
Pedersen, Jack K., et al. "Bayesian Optimization of High-Entropy Alloy Compositions for Electrocatalytic Oxygen Reduction." *Angewandte Chemie* 133.45 (2021): 24346-24354.

Testing advanced on/off or transient process conditions

Discontinuous supply

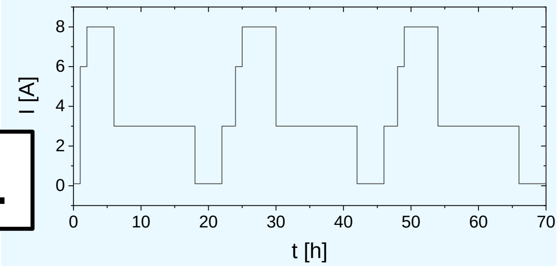
Discontinuous demand





On/off power supply

VS.



Transient power supply

hte

Overview

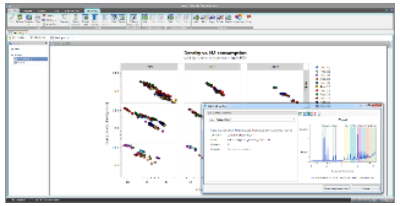
- Intro
- Cat screening via high throughput methodologies
- Transfer of high-advanced testing towards electrochemistry
- Case study: Joint target FZJ & hte (press release) and integration of analytics
- Conclusion

Overview

Intro	Cat screening via high throughput methodologies	Transfer of high-advanced testing towards electrochemistry	Case study: Joint target FZJ & hte (press release) and integration of analytics	Conclusion

Our business offerings

From customer‘s challenges to customized solutions

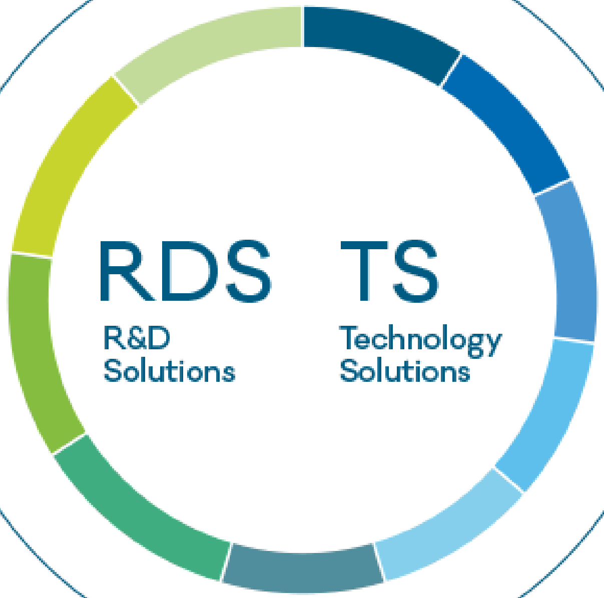


Material synthesis & testing

Catalyst & Process development

Scientific Consulting

Technical Services for Industrial Customers



Automated Test Equipment

Laboratory Automation

Workflow Analysis / Tech. Consulting

Software Solutions

hteControl4 Experiment Automation

myhte4 Scientific Data Warehouse

hte

Overview

Intro

Cat screening via high throughput methodologies

Transfer of high-advanced testing towards electrochemistry

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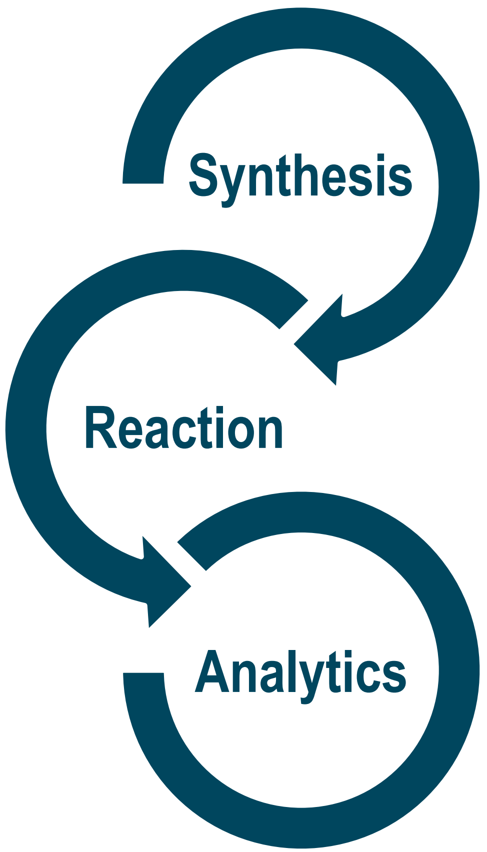
Conclusion

Catalyst screening using high throughput methodologies

- Combinatorial Synthesis
- Combinatorial Reaction
- Combinatorial Analytics



High-throughput screening

Stages in screening & maturity

Stage 1	Planar substrates Maximum number Qualitative analytics	✓ Diffusion
Stage 2	Real particles Reduced number Detailed analytics	✓ Diffusion ✓ Convection
Stage 3	Real reactors Single Reactor Detailed analytics	✓ Diffusion ✓ Convection ✓ Heat conduction

Catalyst vs electrocatalyst screening

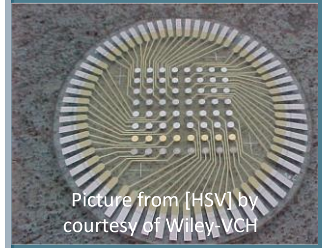
Combinatorial reaction

Stage 1

Picture from [HSV] by courtesy of Wiley-VCH

Microfluidic Screening reactor in the sealing press with adsorption pads

High-throughput Screening in Heterogeneous Catalysis [BER]

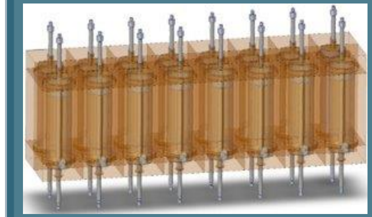


Picture from [HSV] by courtesy of Wiley-VCH

Sputtered wafer with 64-element addressable electrode array on a 3” quartz wafer

High-throughput Screening in Heterogeneous Catalysis [STR]

Stage 2



16-fold fixed-bed screening reactor for DME

pubs.acs.org/acscatalysis

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DOI: 10.1021/acscatal.9b00765, ACS Catal. [MKI]

Picture from [HSV] by courtesy of Wiley-VCH

16-channel rotating disc electrode for electrocatalysts

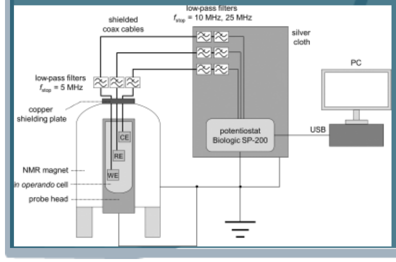
High-throughput Screening in Heterogeneous Catalysis [HSV]

Stage 3

https://en.wikipedia.org/wiki/Catalytic_converter

1-fold engine simulator for regenerability study on exhaust catalysts

Catalysts 2015, 5, 1770-1796; doi:10.3390/catal5041770 [MID]



Electrochemical cell for in operando NMR

https://doi.org/10.5194/mr-2-265-2021 [RAE]

- Intro
- Cat screening via high throughput methodologies
- **Transfer of high-advanced testing towards electrochemistry**
- Case study: Joint target FZJ & hte (press release) and integration of analytics
- Conclusion

Challenges

How do we get this into the lab?

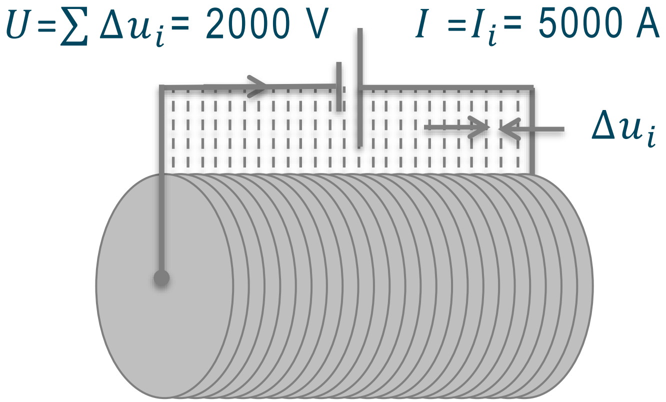
Sunfire Pressurized Alkali Electrolyzer

- Power consumption: P = 3 x 3.2 MW
- Voltage (estim.): U = 2000 V
- Current (estim.): I = 5000 A
- Net hydrogen production: 2,230 Nm^3/h
- Water consumption: 1.9 m^3/h

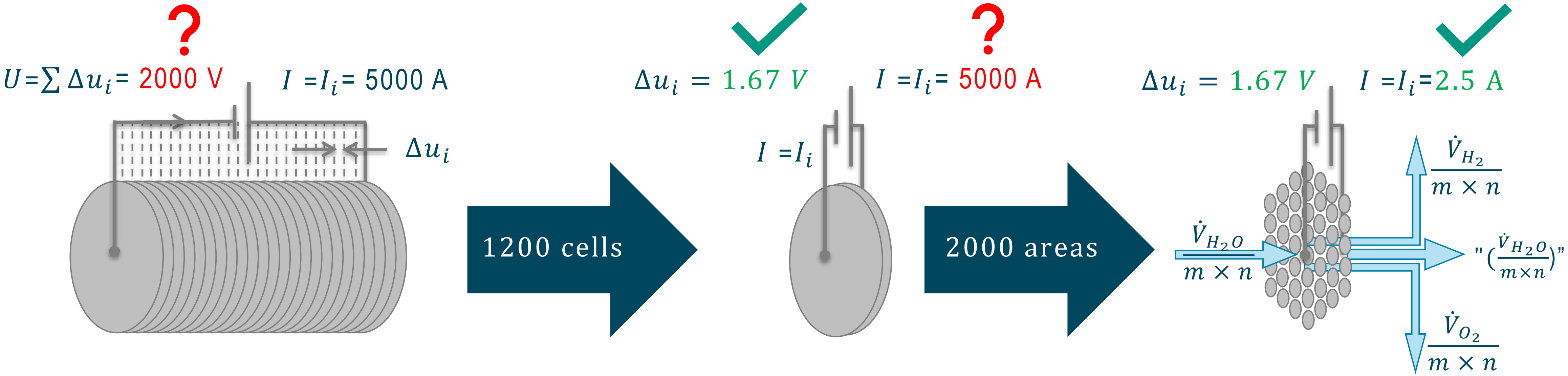
https://www.sunfire.de/en/news/detail/demo4grid-project-partners-successfully-install-a-3-2-mw-pressurized

Challenges

Applied voltage



- Voltage: U = 2000 V
- Current: I = 5000 A
- Current density: i = 0.5 A/cm²
- Cell number: n = 1200
- Cell area: O = 1 m²



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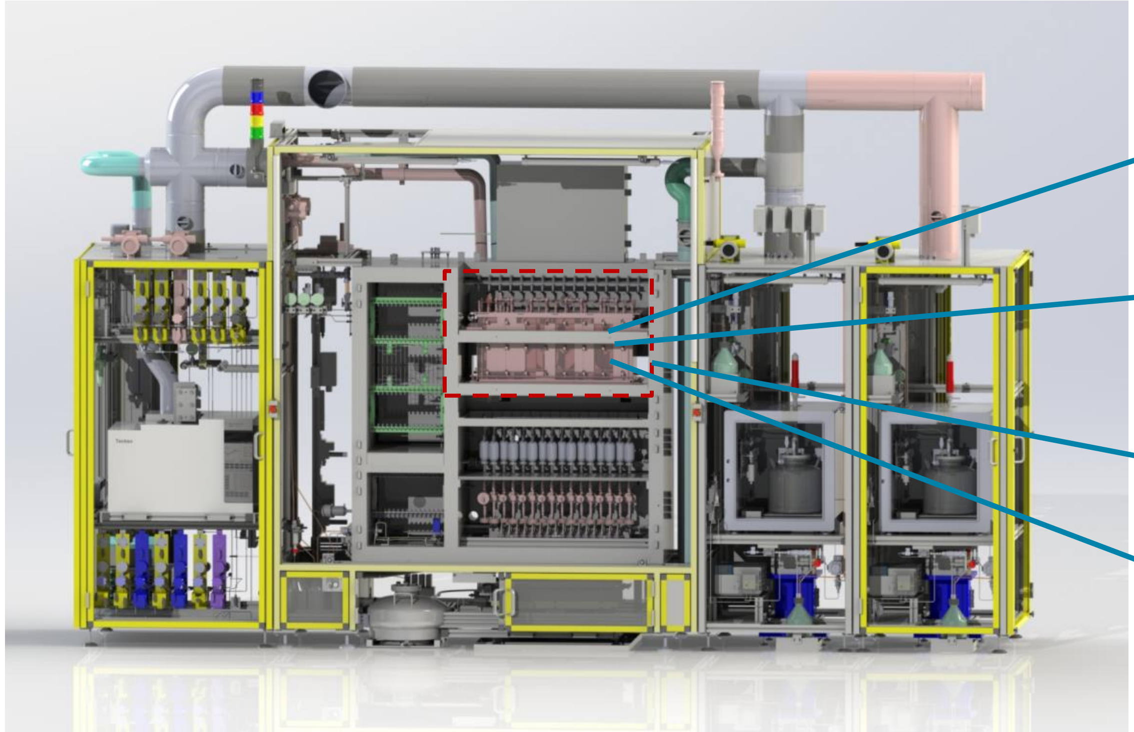
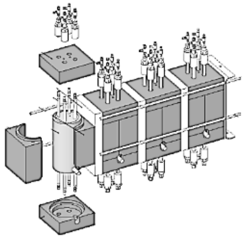
High throughput platform

Transferring knowhow from existing screening platform for catalysis



Modular hte test system



Flow Reactors

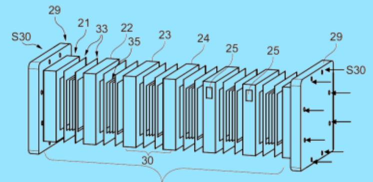
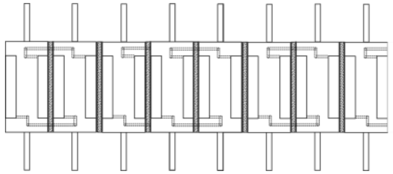
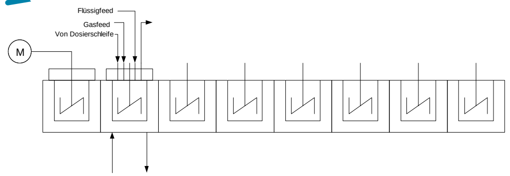


Fig. 3

Electrochemistry



Membrane Technology

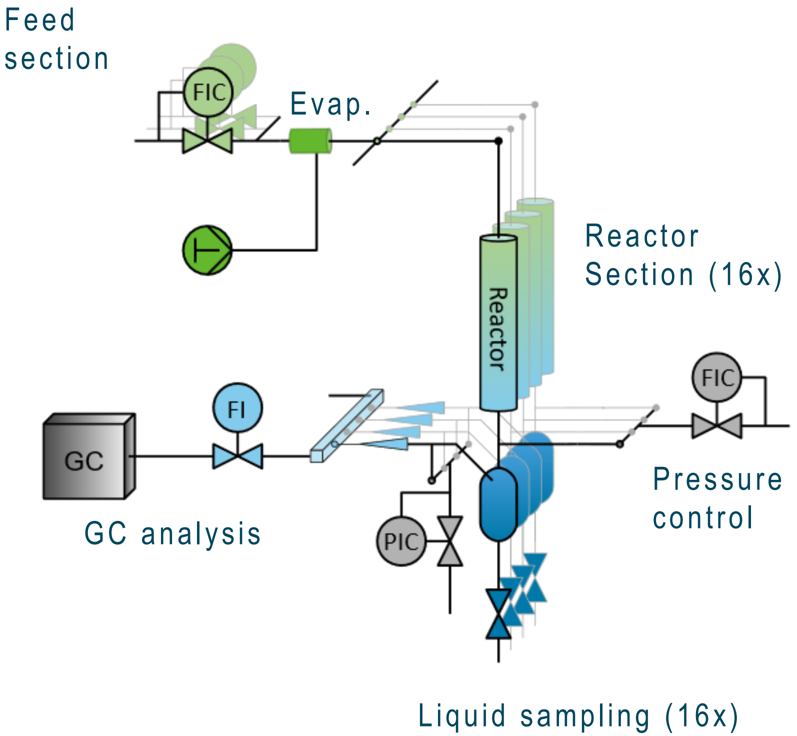


Micro Tank Reactors

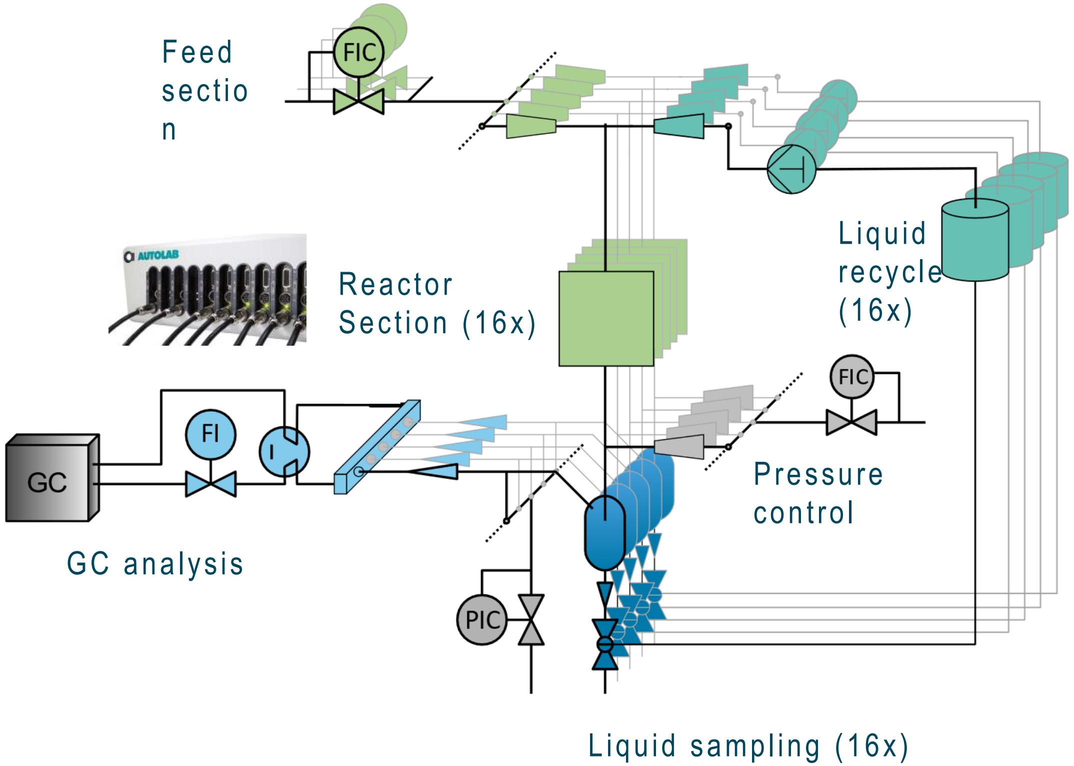
Product management

Process flow diagram

Heterogeneous Catalysis



Heterogeneous electro-Catalysis



Product management

Base patent on high throughput electrochemistry test equipment

(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG

(19) Weltorganisation für geistiges Eigentum
Internationales Büro

(43) Internationales Veröffentlichungsdatum
18. März 2021 (18.03.2021)

WIPO | PCT

(10) Internationale Veröffentlichungsnummer
WO 2021/048375 A1

(51) Internationale Patentklassifikation: Schmitz, Alexander, Postfach 33 05 23, 80065 München

Ansprüche:

1. Vorrichtung zur Untersuchung von chemischen Prozessen, aufweisend: einen Ofen (10) mit wenigstens einer Ofenkammer (11), einen Stapelplattenreaktor (20) mit einer Mehrzahl von nebeneinanderliegenden plattenförmigen Formkörperbausteinen (21, 22, 23, 24) und wenigstens einer Zuleitung (20a, 20b) für ein Edukt und wenigstens einer Ableitung (20c, 20d) für ein Produkt, und eine Anschlussvorrichtung zum Anschluss des Stapelplattenreaktors (20) an wenigstens eine ofenseitige Zuleitung (10a) eines Eduktes und wenigstens eine ofenseitige Ableitung eines Produktes (10c), wobei die Ofenkammer eine Aufnahmevorrichtung (40) aufweist, die derart ausgestaltet ist, dass sie die Mehrzahl von nebeneinanderliegenden Formkörperbausteinen (21, 22, 23, 24) des Stapelplattenreaktors (20) aufnehmen kann, wobei die Mehrzahl von Formkörperbausteinen (21, 22, 23, 24) des Stapelplattenreaktors (20) in der Aufnahmevorrichtung (40) derart gestapelt aneinander liegen, dass sie eine Mehrzahl von Reaktorkammern (31) mit jeweils einem Zulauf (30a, 30b) für ein Edukt und wenigstens einem Ablauf (30c, 30d) für ein Produkt, sowie einer von einer reaktiven Vorrichtung und einer nicht reaktiven Vorrichtung (35) aufweisen, die jeweils einen Einzelreaktor (30) bilden, wobei die Aufnahmevorrichtung (40) eine Pressvorrichtung (45) aufweist, die derart ausgestaltet ist, dass sie die Mehrzahl der gestapelt aneinander liegenden Formkörperbausteine (21, 22, 23, 24) des Stapelplattenreaktors (20) in Richtung der Stapelrichtung verpressen kann, wobei die Mehrzahl von Reaktorkammern (31) wahlweise parallel und/oder in Reihe geschaltet sein können.

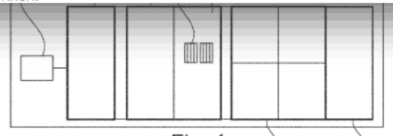


Fig. 1

WO 2021/048375 A1

(57) **Abstract:** The invention relates to a device, stacked plate reactor and to a method for investigating chemical processes to be carried out simultaneously or almost at the same time on a large number of functional element variations of the process parameters.

(57) **Zusammenfassung:** Vorrichtung, Stapelplattenreaktor und Verfahren zur Untersuchung von chemischen Prozessen, um zeitgleich oder in einem engen zeitlichen Zusammenhang an einer großen Anzahl von funktionellen Elementen Variation der Prozessparameter vorgenommen werden können.

[Fortsetzung auf der nächsten Seite]

MAIWALD INTELLECTUAL PROPERTY

In accordance with your standing instructions, we will not remind you of the 30-months and 31-months deadlines and take no action regarding the entry into national/regional phases unless specifically instructed otherwise.

1. **Executive Summary**

Claims 1 to 9 and 16 to 20 are considered as being novel and based in an inventive step over the cited prior art.

2. **Subject matter of the present invention**

The present invention relates to device (claim 1) for investigating chemical processes having an oven, a stacked reactor with a plurality of shaped reactor composition blocks an inlet and an outlet, wherein the oven has a receptacle for receiving the shaped reactor composition blocks and a pressing unit for pressing the stacked shaped reactor composition blocks, wherein the reactor chambers formed by the shaped reactor composition blocks can be functionally arranged in series or in parallel.

This allows chemical tests at the same time or within a tight time frame correlation on a large number of functional elements under variation of the process parameters.

The invention further relates to a stacked reactor (claim10) to be used in the device according to claim 1 as well as a method (claim 16), which corresponds to the device of claim 1.

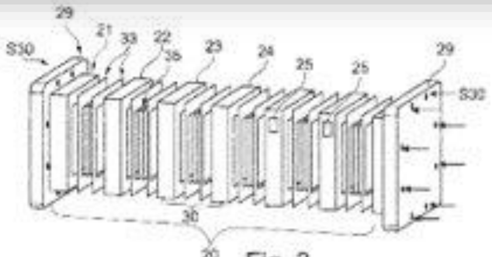


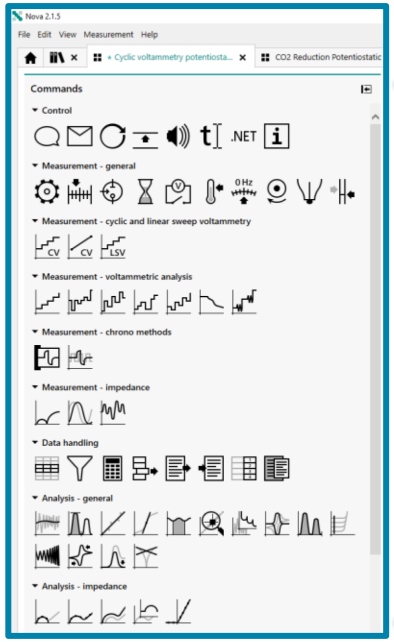
Fig. 3

- **No IP** on **single-fold** equipment possible
- **Bench-scale** as **established** market for test equipment
- Targeting **high thoughput electrochemistry test equipment** with **generic cell housing** for electrolysis, fuel cells and flow batteries
- Targeting test equipment on **component to device to system** level
- Focusing on **quantitative data** rather than qualitative ultra-high throughput screening
- Combining a certain degree of **parallelization** and **flexibility**

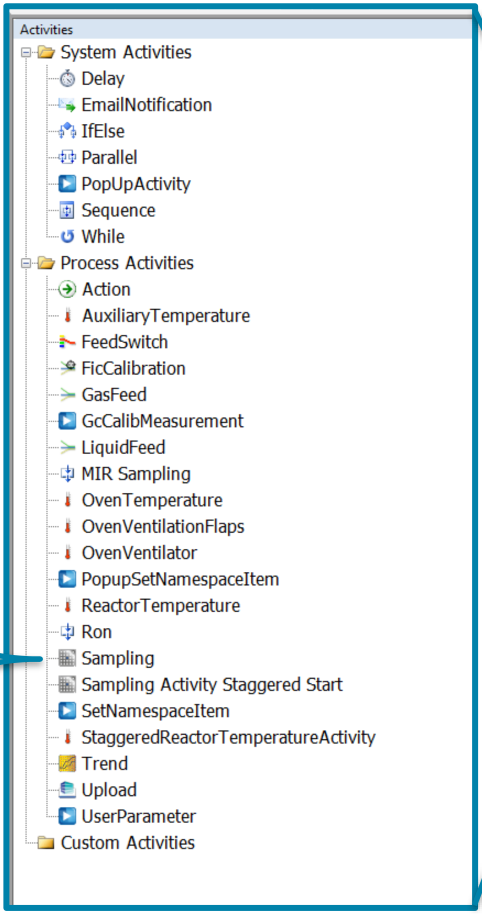
Analytics

Workflow setup & Metrohm interaction

Metrohm

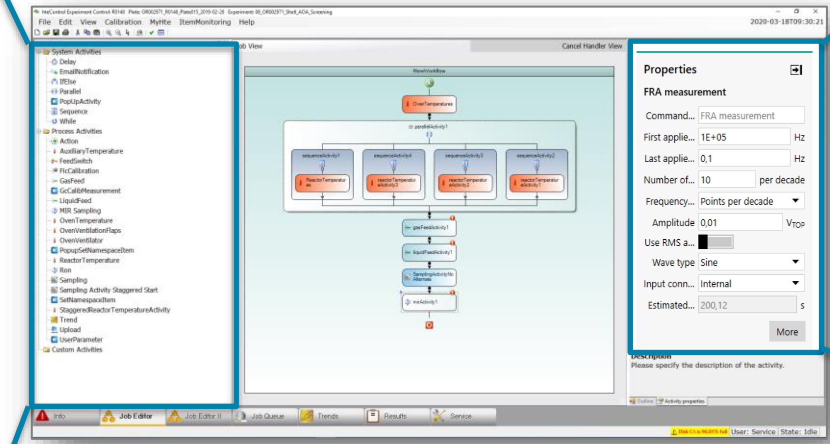


List of commands

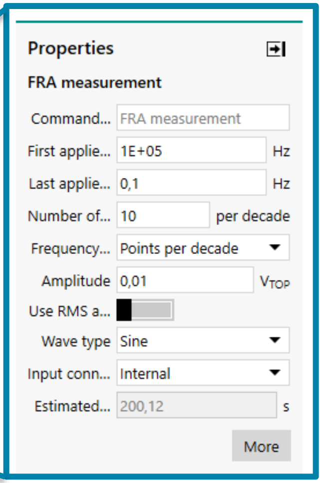


List of activities

Recipe Controller



Metrohm



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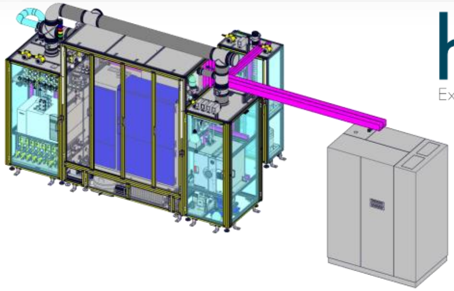

hte Control4
Experiment Automation

hte

Overview

Intro	Cat screening via high throughput methodologies	Transfer of high-advanced testing towards electrochemistry	Case study: Joint target FZJ & hte (press release) and integration of analytics	Conclusion

Jülich Application Knowledge PLUS hte Engineering provides a New Research Tool for Electrolysis

Press Release September 2022

hte – the high throughput experimentation company was awarded the contract by Research Center Jülich to provide a high throughput test system for electrolysis. This unit will enable hte to apply its expertise and strong technology platform for high throughput testing for catalytic processes to expand into the field of electrocatalysis.

"We selected hte because of its proven expertise in the design, construction and implementation of reactor systems combined with fast and efficient online analytics and a fully integrated software solution. We are really looking forward to working together to significantly enhance our R&D output in the field of electrocatalysis," **says Prof. Rüdiger Eichel from Research Center Jülich.**

QUESTIONS?

hte GmbH | Kurpfalzring 104 69123 Heidelberg, Germany T +49 6221 7497 0 info@hte-company.com www.hte-company.com