

Do you work well under pressure?

Introducing the

**Compre
Drive**

rh*d* instruments – flexible cell solutions

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CEO
Sales and Marketing

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CEO
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Jens Wallauer



Head of Product Management
Software Development

Christoffer Karlsson



Senior R&D Scientist

Michael Schöll



Mechanical Design
Engineer

Sebastian Kranz



International Sales
Manager

Electrochemistry experts and engineers with a passion
to develop innovative testing solutions for you

rh*d* instruments – flexible cell solutions

Microcell product line

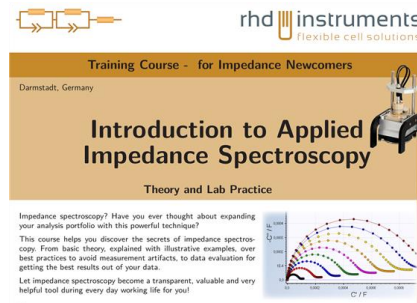


CompreDrive product line



Software and Services

RelaxIS3
Impedance Spectrum Analysis



rh*d* instruments – flexible cell solutions

2020

The CompreDrive press system is released

2018

On-site seminars on the topic of 'Impedance Spectroscopy'

2017

Move to our new headquarters in Darmstadt

2015

Coupling of electrochemistry with spectroscopy with the TSC Spectro

2014

RelaxIS: Impedance Spectrum Analysis joins the software portfolio

2014

Inception of the cooperation with Metrohm Autolab and Deutsche Metrohm

2013

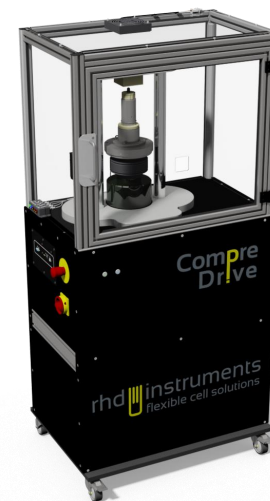
Official formation of rh*d* instruments GmbH & Co. KG

2012

Expansion of cell portfolio with the TSC Battery and TSC Surface

2011

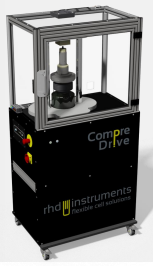
Commercial launch of the Microcell HC system



RelaxIS3
Impedance Spectrum Analysis



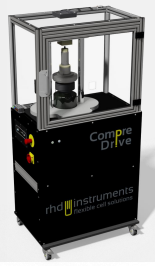
Motivation for CompreDrive development



State-of-the-art measuring systems: Needs in cutting-edge research

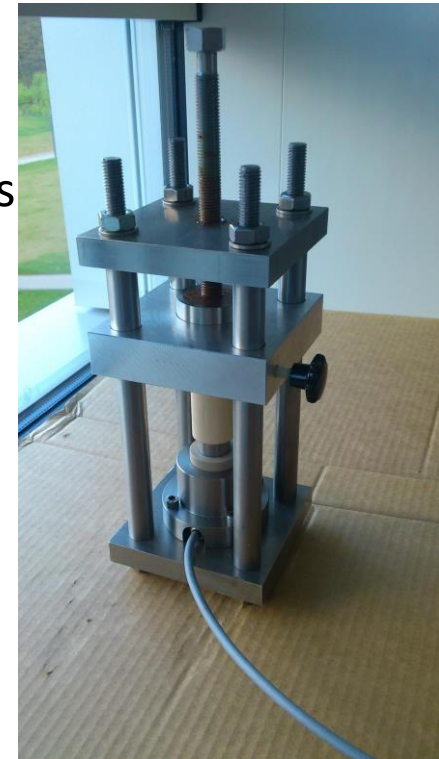
- ☐ Reproducible results
- ☐ Robust and easy to handle equipment
- ☐ Active force control and logging
- ☐ Active temperature control and logging
- ☐ Automation
- ☐ Fixed, standardized procedures
- ☐ Data management
- ☐ Safety of operation

Motivation for CompreDrive development

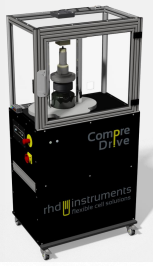


Issues in solid state electrochemistry:

- Home-built setups
- “Measuring Cells” with insufficient manufacturing tolerances
- Manual force regulation via hydraulic cylinder or screw
- Force direction inaccurate due to manufacturing tolerances



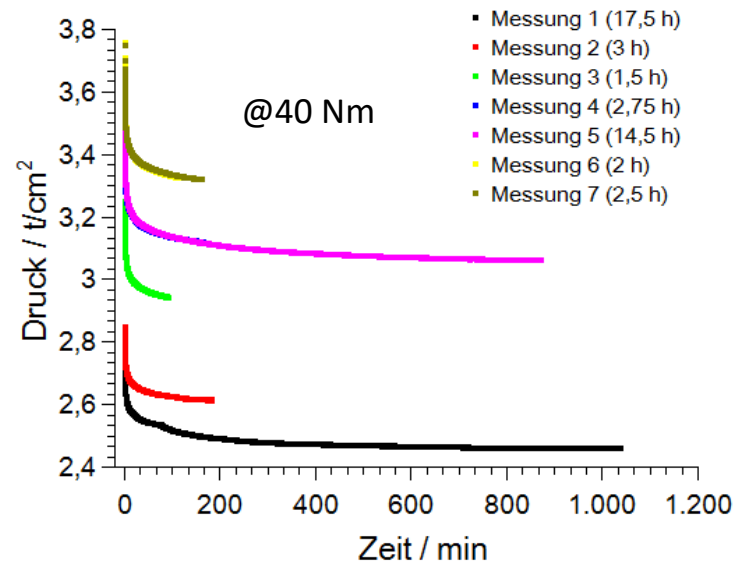
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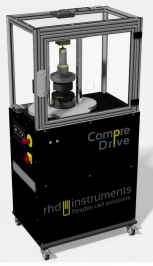
State-of-the-art

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- Force direction inaccurate due to manufacturing tolerances
- Often no force monitoring at all



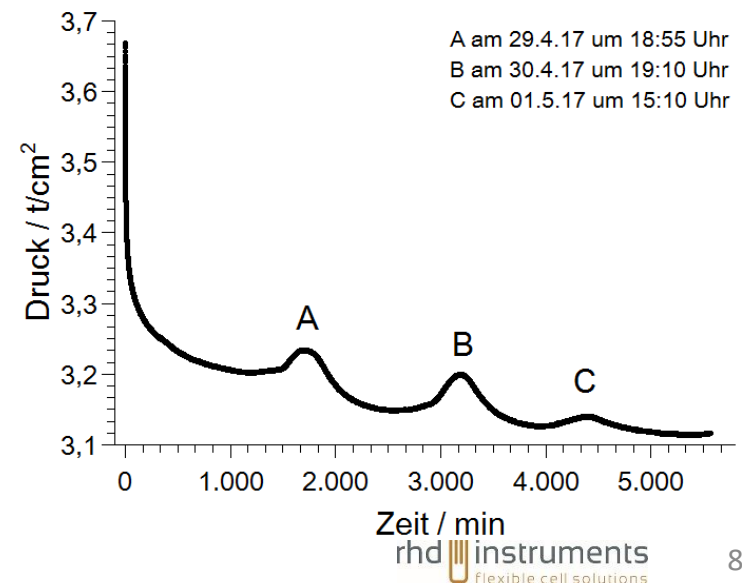
Motivation



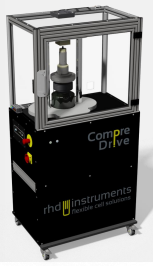
State-of-the-art

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- No active force regulation



Motivation



State-of-the-art

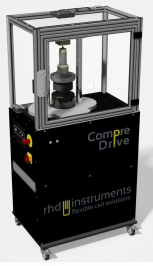
Issues in solid state electrochemistry:

- Home-built setups
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- Manual force regulation via hydraulic cylinder or screw
- Force direction inaccurate due to manufacturing tolerances
- Often no force monitoring at all
- No active force regulation
- No or insufficient temperature regulation

→ Inaccurate conditions

- Highly limited force range
- Limited flexibility
- No automation

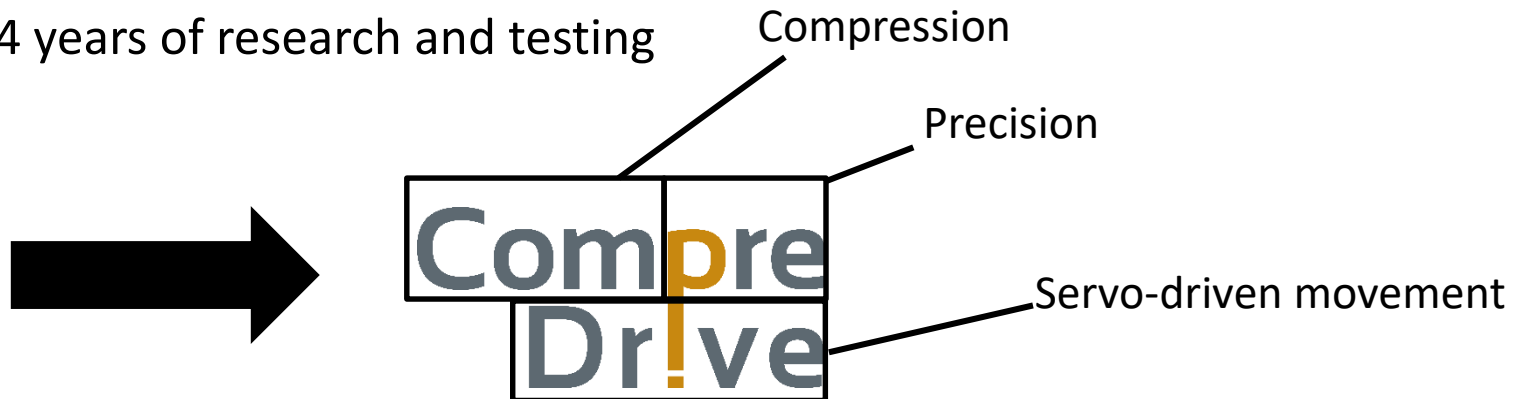
Motivation



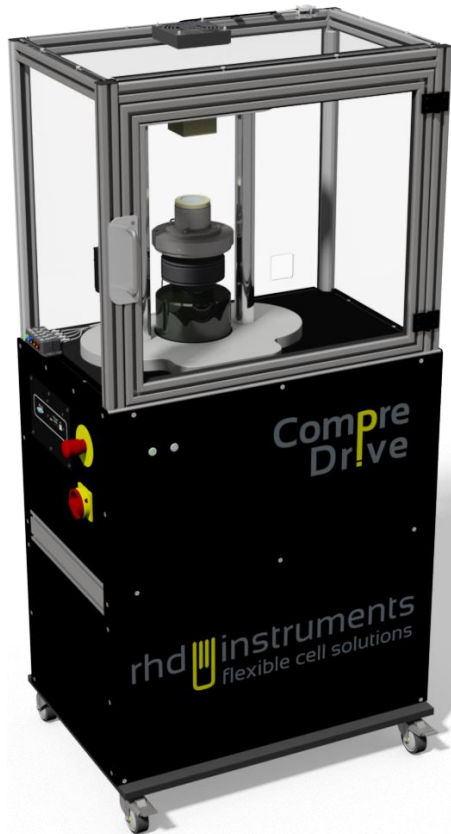
rhd's approach:

- Design of highly specific measuring set-up for solid state research
- Eliminate common drawbacks
- Enable (semi-)automated solid state measurements

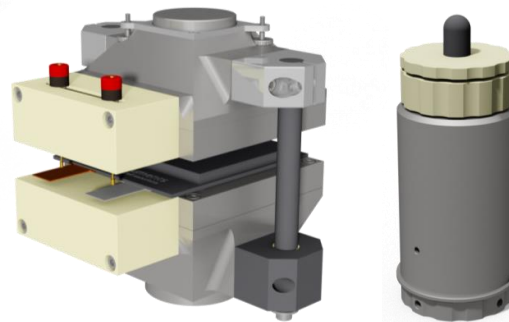
→ Over 4 years of research and testing



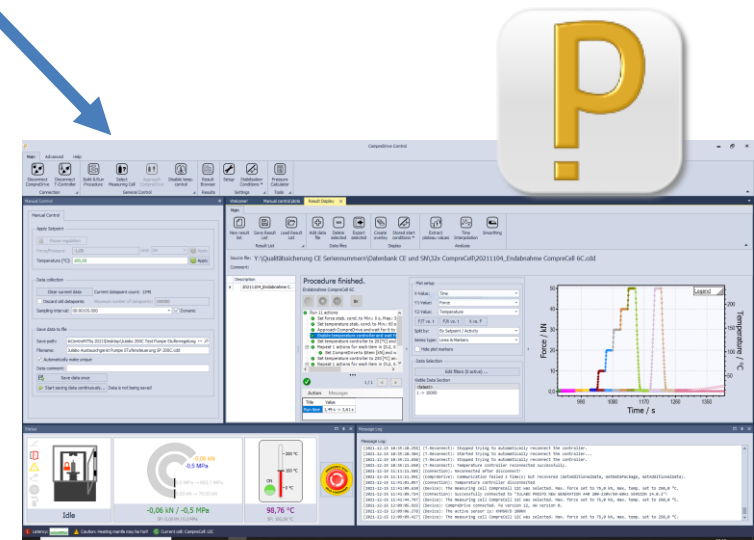
Compre Drive



1. CompreDrive

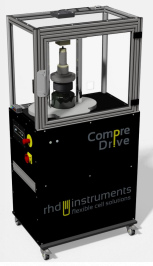


2. CompreCell

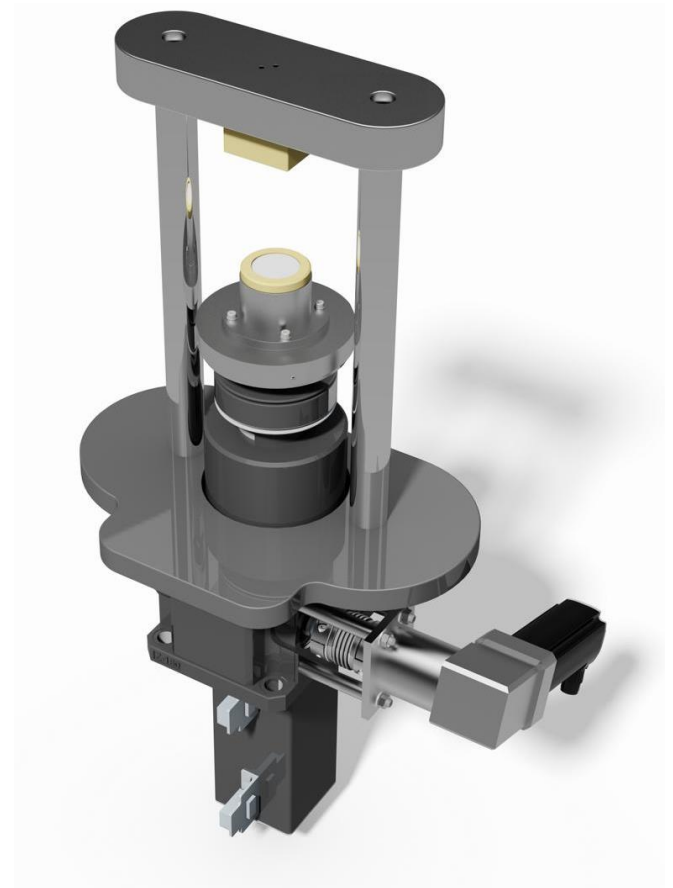


3. CompreDriveControl

1. CompreDrive

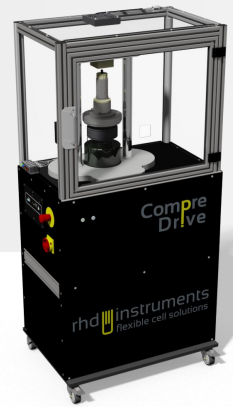
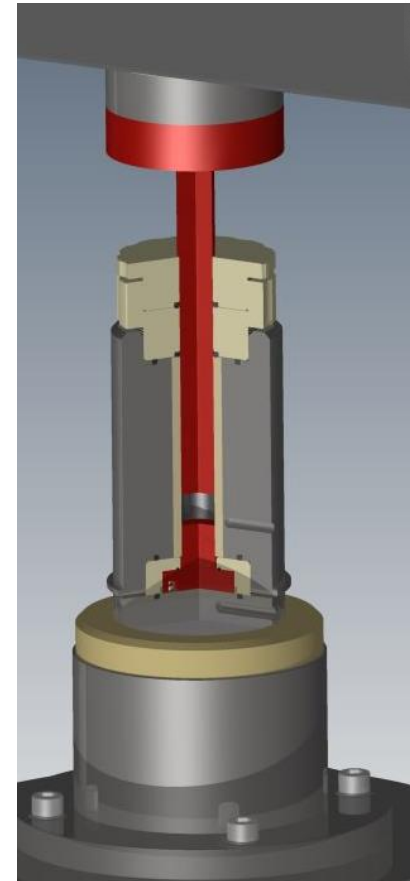


- Modular platform -> Different measuring cells available
- Main part: Force control unit
- High precision servo drive
 - minimum step width <10 nm
- 75 kN maximum force
 - 663 MPa @ 12 mm sample diameter
 - 1.7 GPa @ 6 mm sample diameter
 - Different force sensors available



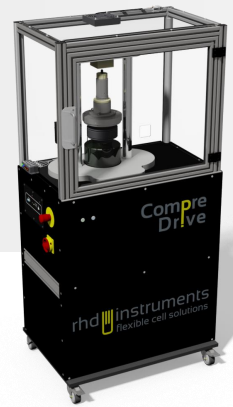
1. CompreDrive

- Application of force via spherical collet
 - Only uniaxial forces
- Electrical insulation by $\text{Al}_2\text{O}_3/\text{ZrO}_2$ ceramic
- Highly stressed parts made from hard-metal
 - Robust and durable

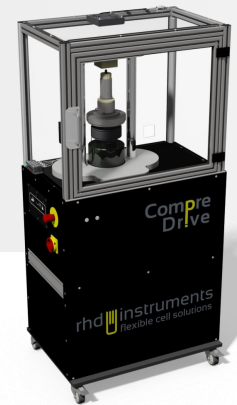


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1. CompreDrive



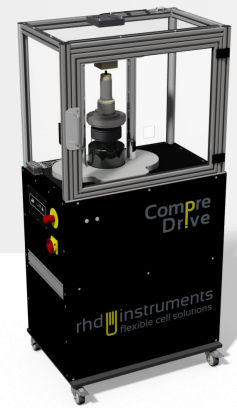
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- Highly stressed parts made from hard-metal
 - Robust and durable
- Universal connection capabilities for all potentiostat/galvanostat models
- Safety first!
 - Safety glass housing with open door detection
 - Electromechanical door lock
 - Self checks, SW based limits, User input checks,



1. CompreDrive

Temperature regulation add-ons

- High precision Pt100 temperature sensor
- 3 different configurations:



No active heating



CompreDrive

Electrical heating
RT \rightarrow +250°C



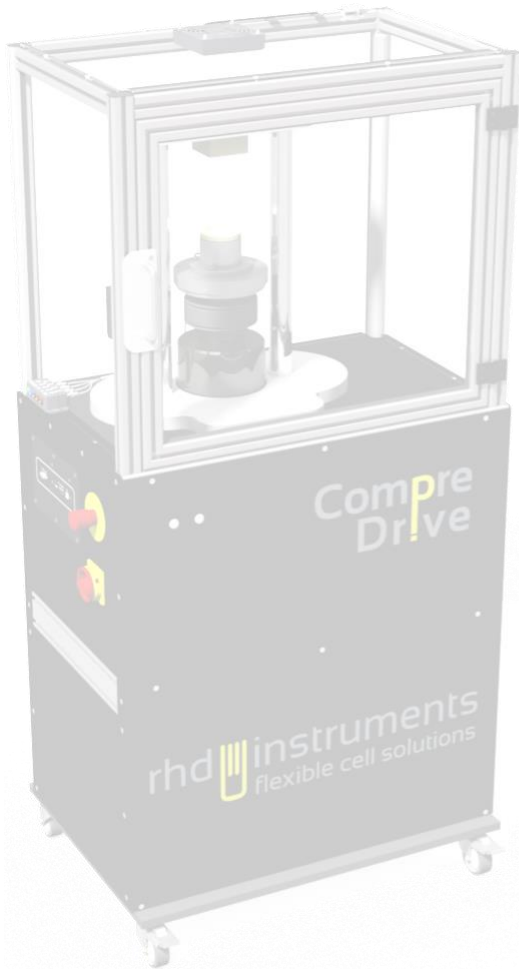
CompreDrive H

Fluid heating and cooling*
-40°C \rightarrow +250°C

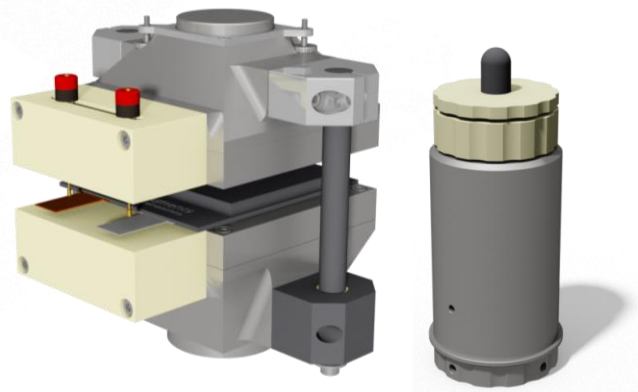
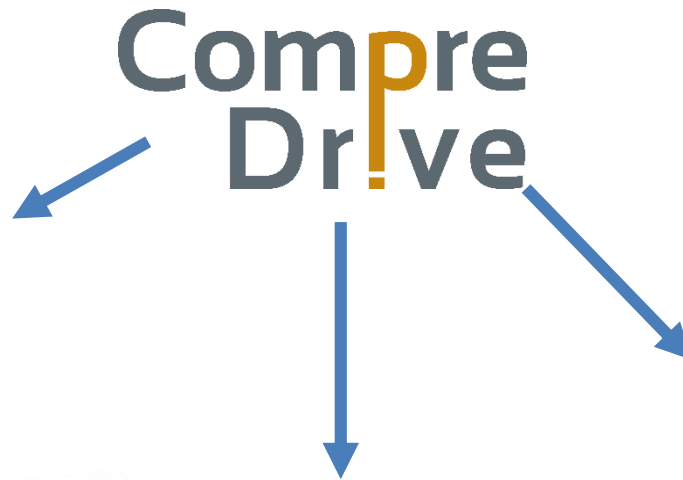
*lab scale refrigerated circulator required



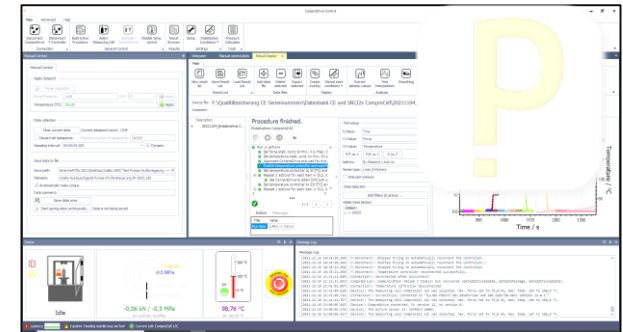
CompreDrive HC



1. CompreDrive



2. CompreCell



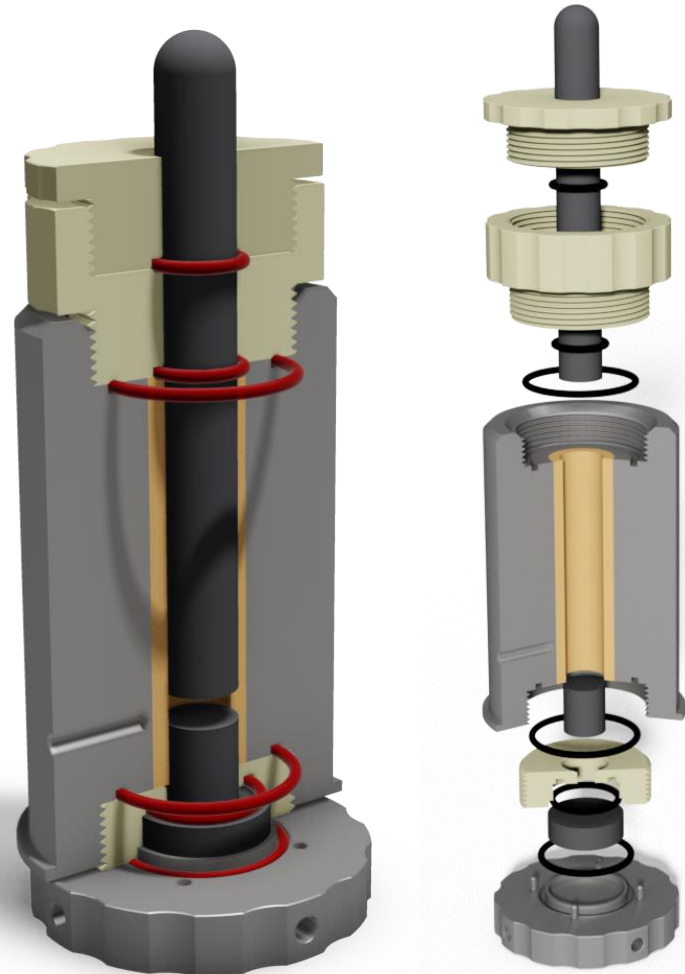
3. CompreDriveControl

2. CompreCell



Example 1: CompreCell 12C

- Designed for powders and disc-like samples
- Airtight (He leakage rate $< 10^{-7}$ mbar·l·s⁻¹)
- Stainless steel body
- Insulation sleeve: Al₂O₃
- Sample diameter: 12 mm
- Maximum sample height: 10 mm
- Maximum Force: 75 kN (=663 MPa)
- Temperature Range: -40 → +250°C
- Plate-plate geometry (2 electrodes)



2. CompreCell



Different Labs – different needs

Max. pressure: 1,7 GPa



CompreCell:

12C

Diameter:

12 mm

Insulation:

Al_2O_3



6C

6 mm

Al_2O_3



12P

12 mm

PEEK*/PEI**

* up to 150 °C

** up to 200 °C

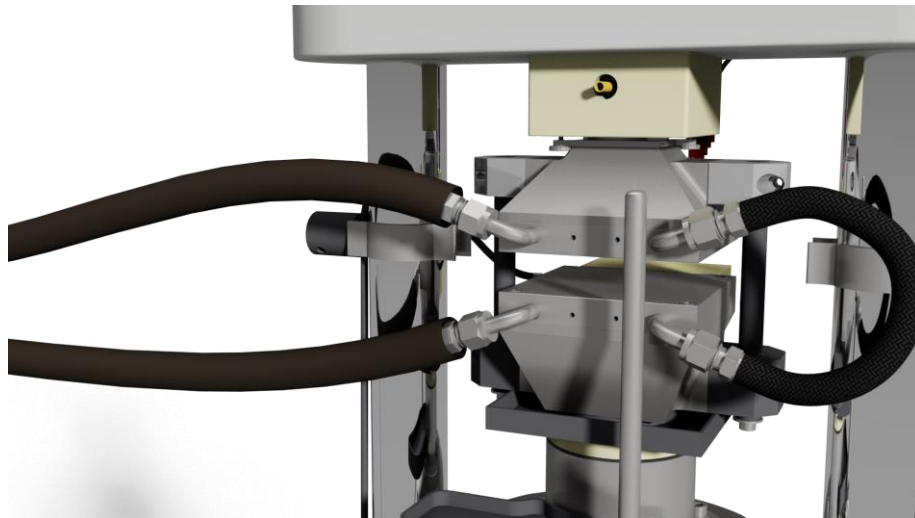
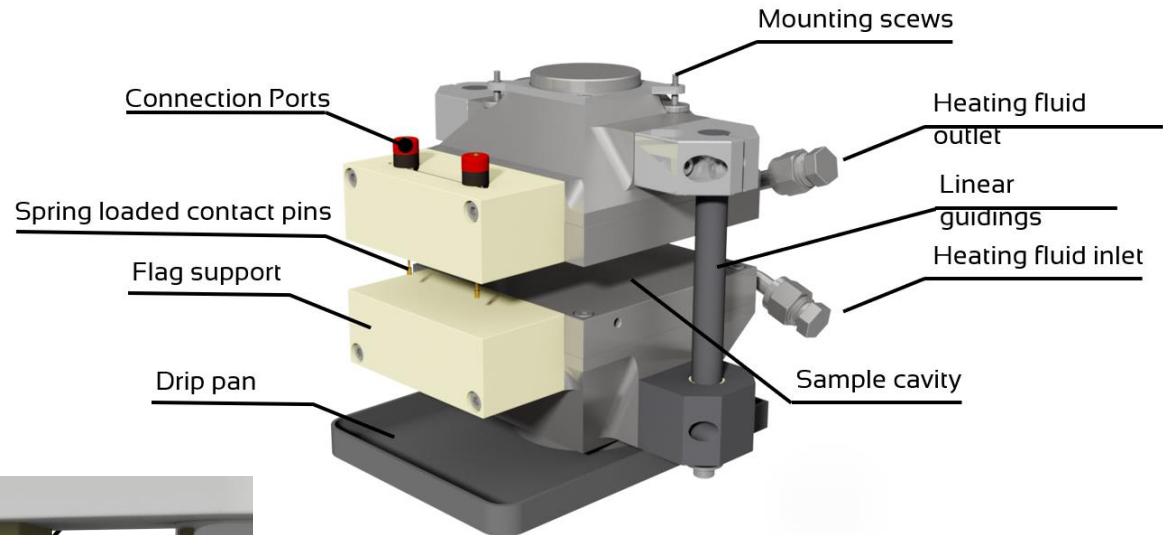
→ Custom cells available! ←

Recent developments for CompreDrive



CompreCell Pouch 10S HC

- Designed for pouch-bag samples
- 10cm x 10cm sample cavity
- Solid stainless steel body
- Temperature range: 0-100 °C
- Both plates with fluid channels



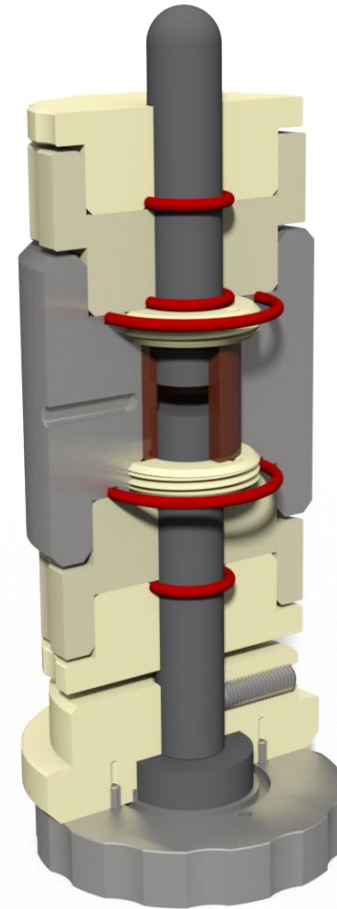
- Adjustable gold-plated contacts
- Maximum sample height: 20 mm
- Maximum Force: 75 kN
- Fixed to CompreDrive main unit

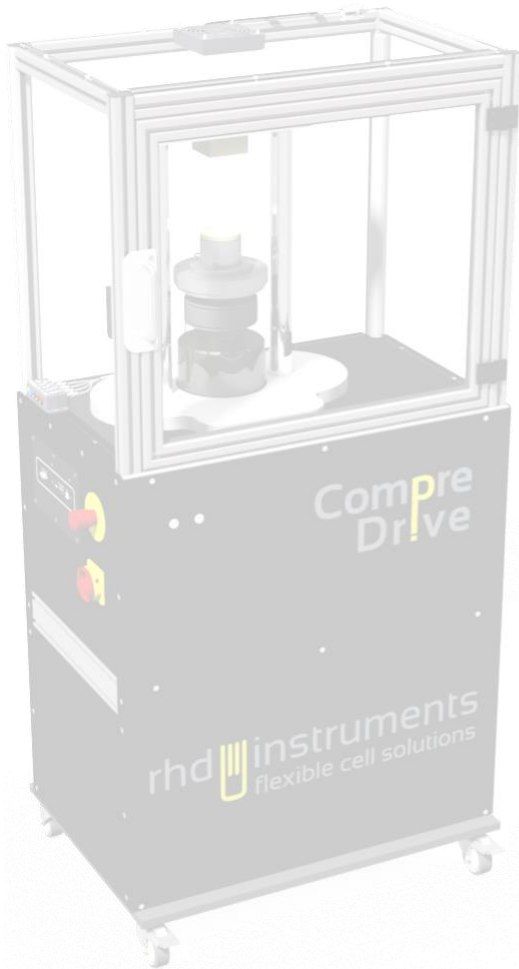
Recent developments for CompreDrive



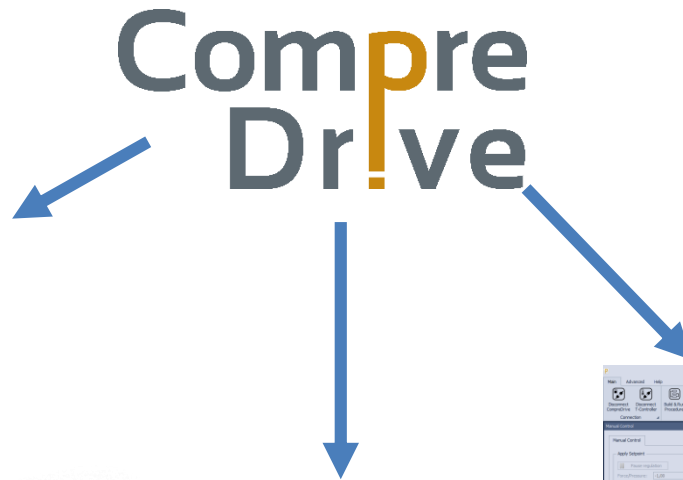
Double-Piston CompreCell

- Optimized for ASSB preparation
- Moveable cell body
- Air tight
- Compatible with H/HC add-on
- Variable insulation sleeves available
- Same temperature and pressure ratings like standard CompreCells

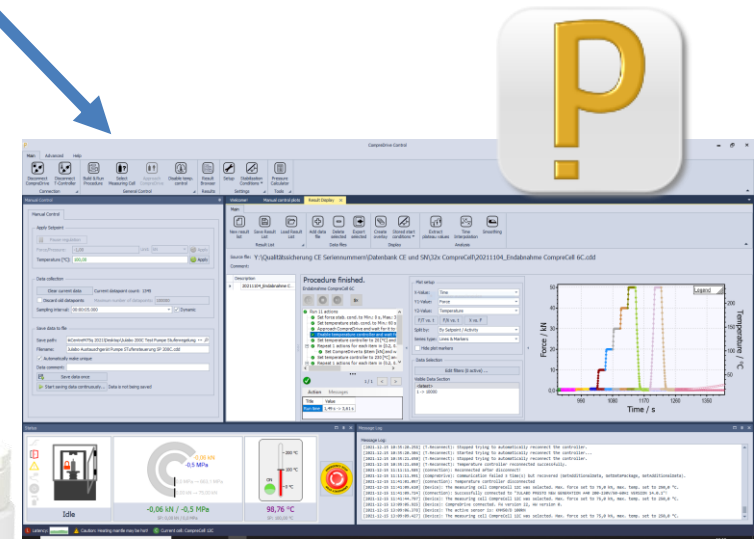




1. CompreDrive



2. CompreCell

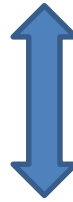


3. CompreDriveControl

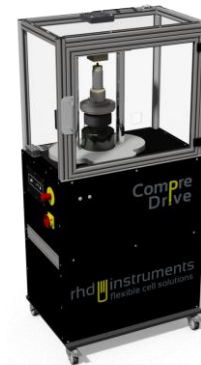
3. CompreDriveControl



Potentiostat / Galvanostat

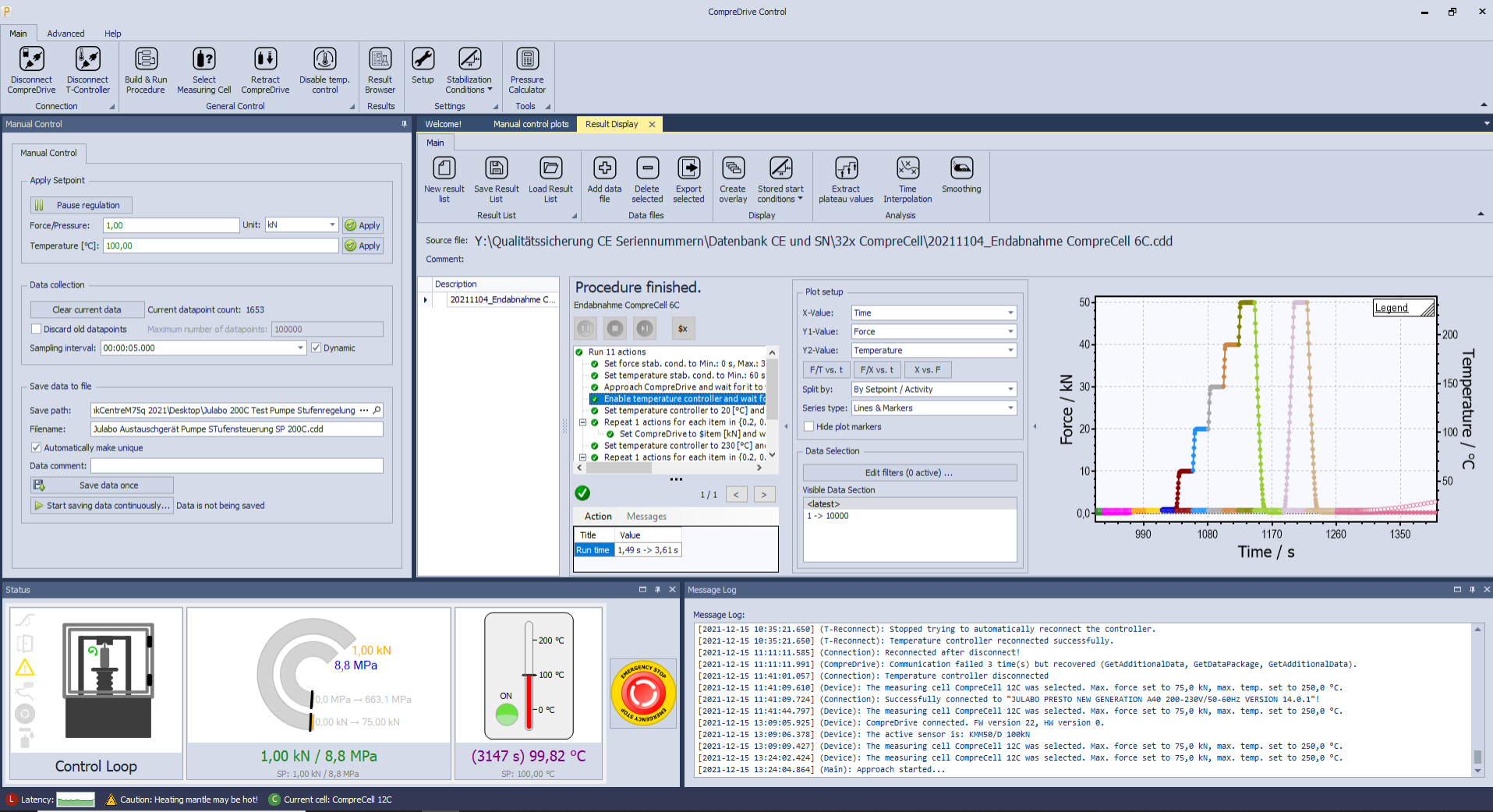


Temperature control



Pressure control

3. CompreDriveControl



3. CompreDriveControl



Procedure Editor - Force & temperature ramp with waiting time (New)

Main

Save changes Check Procedure Return from action Change Procedure Settings Undo Redo Delete Selected Actions Show/Hide Procedure Tree

Procedure Editor

Path to current action: ↑ Root

Run 9 actions

- Request a list of numbers from the user; store it in \$temperatures
- Request a list of numbers from the user; store it in \$forces
- Request a number from the user; store it in \$waittime
- Approach CompreDrive and wait for it to finish
- Enable temperature controller and wait for it to be ready
- Repeat 2 actions for each item in \$temperatures; Store current item in \$temperature
 - Set temperature controller to \$temperature [°C] and wait for stability
 - Repeat 4 actions for each item in \$forces; Store current item in \$force
 - Build text: Apply {force} kN at {temperature} °C...; store it in \$text
 - Show message \$text in Message log
 - Set CompreDrive to \$force [kN] and wait for stability
 - Wait for \$waittime seconds
 - Set temperature controller to 25 [°C] and wait for stability
 - Disable temperature controller and wait for it to be off
 - Retract CompreDrive and wait for it to finish

Toolbox

Simple Advanced

Drag & Drop actions to the list to use them

- CompreDriveControl
 - CompreDrive
 - Set Force/Pressure Setpoint
 - Approach CompreDrive
 - Retract CompreDrive
 - Pause CompreDrive Force Regulation
 - Unpause CompreDrive Force Regulation
 - Enable ramp & set ramp speed
 - Disable force ramp
 - Change force stab. conditions
 - Restore force stab. cond. to global settings
 - Temperature Controller
 - Set Temperature Setpoint
 - Enable temperature controller
 - Disable temperature controller
 - Change temperature stab. conditions
 - Restore temp. stab. cond. to global settings
 - Triggers
 - Hardware
 - Setup hardware triggers
 - Pulse hardware OUT trigger
 - Wait for hardware IN trigger
 - Software
 - Set software trigger
 - Wait for software trigger
 - Miscellaneous
 - Get device/system value
 - Sampling rate setup
 - Wait for stability

Actions (executed successively):

1. Request a list of numbers from the user; store it in \$temperatures
2. Request a list of numbers from the user; store it in \$forces
3. Request a number from the user; store it in \$waittime
4. Approach CompreDrive and wait for it to finish
5. Enable temperature controller and wait for it to be ready
6. Repeat 2 actions for each item in \$temperatures; Store current item in \$temperature [...]
7. Set temperature controller to 25 [°C] and wait for stability
8. Disable temperature controller and wait for it to be off
9. Retract CompreDrive and wait for it to finish

Description: Double-click actions to edit them

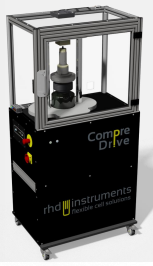
3. CompreDriveControl



CompreDriveControl main features

- Full control of forces/pressures, temperatures, electrochemistry (optional)
- Manual control and fully automated procedures
- Logging, visualization and analysis of all available data
- Results browser, export functions (graph, text)
- Read and set external setpoints: Synchronization with other programs via a simple http interface or hardware triggers

Motivation for CompreDrive development



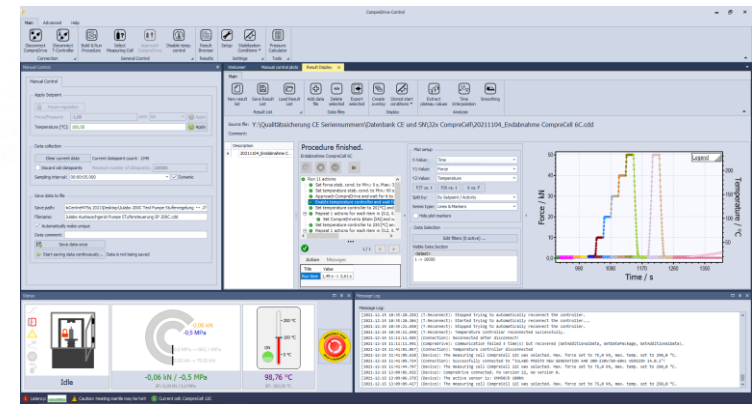
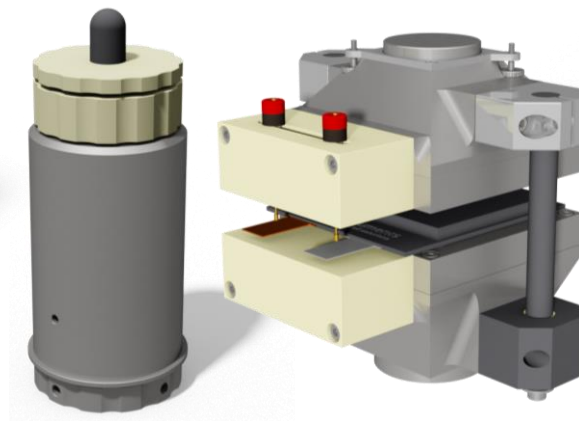
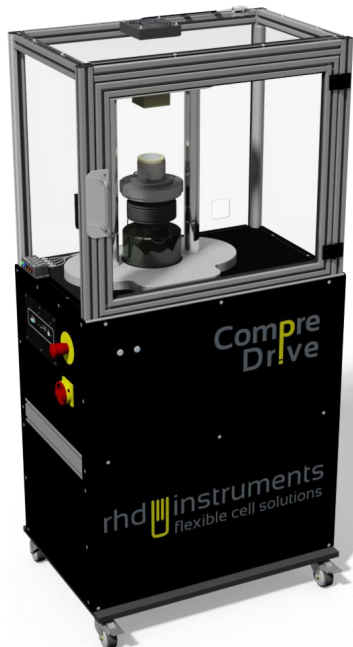
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- ✓ Data management
- ✓ Safety of operation

Compre Drive

- New experimental horizons in solid state electrochemistry -

Keep the pressure up!



Summary



- **Mechanical force up to 75 kN (1.7 GPa @ 6mm sample diameter)**
- **Temperature range -40°C <-> +250°C**
- **Active(!) force and temperature control**
- **Modular system allows for extension and customization**
- **Airtight measuring cells**
- **Extensive control software**
- **Fully automated electrochemical measurements of solid samples**

Available sensors



100 kN sensor: accuracy of the whole system is within ± 0.1 kN.

10 kN sensor: linearity error is <0.5 % full scale according to the sensor data sheet (0.05 kN)

2 kN sensor: linearity error is <0.2 % full scale according to the sensor data sheet (0.004 kN)

For the 100 kN sensor, you would get 0.1 % error.

The difference here is, that this is the standard sensor, and we check every single unit with a second calibration sensor in the system instead of a measuring cell.

The sensors are typically much better than the limit given in the datasheet.

In case of the 100 kN sensor, we do not use full scale.

Measured value amplifier has 24 bit resolution

Available sensors



Minimum force available

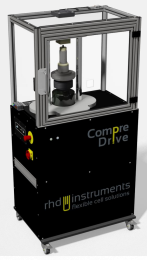
In general: 0.15 % of full scale, absolute minimum 30 N.

100 kN sensor: minimum force 150 N

10 kN sensor: minimum force 30 N

2 kN sensor: minimum force 30 N

The CompreDrive by rhd instruments



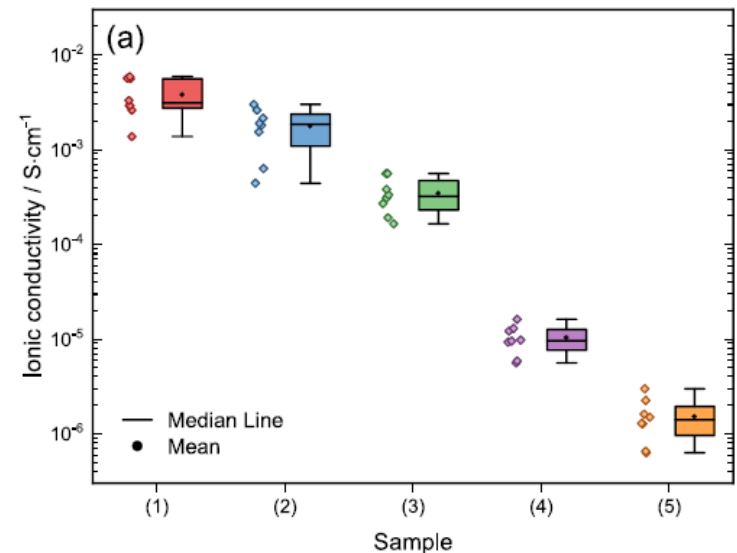
The importance of pressure and temperature control

Round-robin study by S. Ohno et al.

- 5 different solid electrolytes
- 8 labs around the world
- Determining ionic conductivity σ

Results:

- σ deviation: One order of magnitude
- Results highly dependent on local sample processing



S. Ohno et al., *ACS Energy Lett.* 2020, 5, 3, 910–915

→ Precise pressure and temperature control during sample preparation crucial