Electrochemical Impedance Spectroscopy using Programmable REGATRON G5 Power Supplies

Your report, straight from research!

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1 Introduction

Impedance spectroscopy is a measurement method widely used in the development processes of electrochemical systems. May it be batteries, supercapacitors, fuel cells, or, in this case, electrolyzers. Electrochemical Impedance Spectroscopy (EIS) measurements provide crucial information about the internal electrochemical processes in the cells. It is possible to identify where the main contributions of possible loss mechanisms occur in the assembly. This tool is well-suited for research on electrolysis cells but faces numerous challenges in the scale-up and adoption of measuring multiple stacked cells (stacks). The main motivations for the use of EIS at higher power ratings for the stack development process are:

- Understanding of the stack design
- Insights into the voltage disparities and differences between the cells
- Description of stack parts like the bipolar plates and their influence on performance
- Monitoring of measurement values that describe the state of health of the stack

This application note gives some insight into the challenges that arise in the implementation and how they have been solved using Power Electronics from REGATRON.

2 Testbench Overview

The first HyCentA test bench used for stack testing is called “David 1” and has the following specifications:

- PEM and AEM technologies
- Generic electrolysis stacks with a power rating of 1 to 15 kW
- Asymmetric pressure operation of the stacks
- Cathode pressure up to 160 bar
- Possibility of operation under explosive atmospheres on the anode/oxygen side (incl. concentration measurement)
- Voltage monitoring for each cell of the stack
- Multi-channel parallel EIS measurement for all cells of the stack
- Up to 24 cells
- EIS frequency range: 0,01 Hz – 10 kHz
- Online Hydrogen gas quality monitoring (according to ISO 14687:2019)

A second test bench at HyCentA with higher power is called “Goliath” with the following specifications:

- PEM and AEM technologies
- General electrolysis stacks with a power rating of up to 150 kW
- Asymmetric pressure operation of the stacks
- Cathode pressure up to 50 bar(rel), Anode up to 10 bar(rel)
- Possibility of operation under explosive atmospheres on the anode/oxygen side (incl. concentration measurement)
- Voltage monitoring for each cell of the stack
- Multi-channel parallel EIS measurement for all cells of the stack
- Up to 24 cells
- EIS frequency range: 0,01 Hz – 10 kHz
- Hydrogen gas quality monitoring with gas samples (according to ISO 14687:2019)

3 Topology

The EIS measurement setup is realized twice and consists of the following parts and equipment:

HyCentA electrolysis test bench: "David 1"

- Control Software developed by HyCentA, which handles the automated test execution and file storage
- 4.5 kW PEM electrolysis research stack
- Function generator: Tektronix AFG1062
- Digitizers: Dewesoft Sirius-8xSTG
- Power supply: REGATRON G5.UNV.18.80.676.M
- External current measurement: Danisense DS600ID

HyCentA electrolysis test bench: "Goliath"

- Control Software developed by HyCentA, which handles the automated test execution and file storage
- 30 kW PEM electrolysis prototype stack
- Function generator: Tektronix AFG1062
- Digitizers: Dewesoft Sirius-8xSTG
- Power supply multi-device network of 3 x G5.SRC.54.80.2028 connected in parallel with a total of 6084 A
- External current measurement: Danisense DR5000UX-10V/7500A

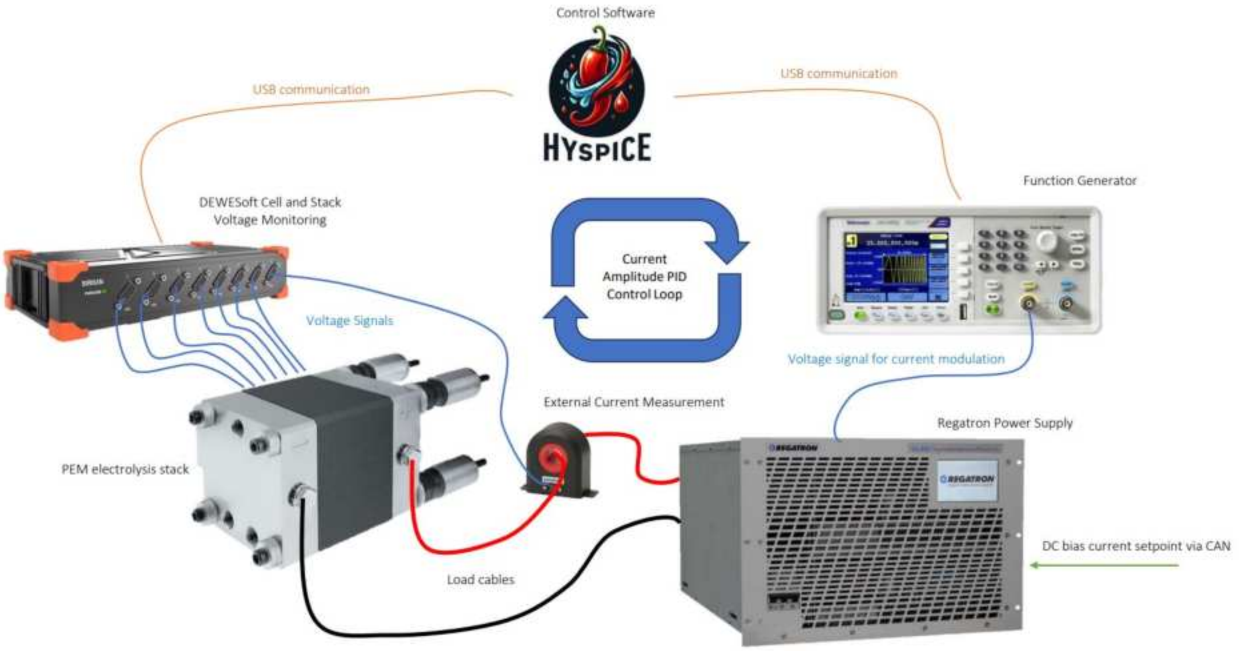

Figure 1: Topology of the setup. Pictures of products and the stack are exemplary.[1]

Figure 1 shows the topology of the measurement setup used. The control software handles the test execution, which consists of activating and controlling the function generator and the current and voltage measurement triggers. The REGATRON G5 power supply provides the DC operation current and additionally the measurement signal modulation. The stack is fixed on the test bench, where the produced gases are handled and the correct water flow is provided. A benefit of the REGATRON power supplies is that the DC offset and the signal modulation

[1] Stack Reference: H-Tec / QuestOne

are independently accessible. In this test setup, the DC offset is set via a CAN fieldbus, and the signal modulation is provided as an analog voltage signal with high resolution. The REGATRON G5 device handles both inputs and creates the desired DC and pulsating current as a modulated signal at the output. In this configuration, it is possible to use any device that has a CAN-bus interface for the current setpoint, and the signal generation device can be the one that suits best for the used automation application. The integration into a test bench and communication with a Programmable Logic Controller (PLC) can easily be done with the provided configuration tools.

4 Implementation

To execute an EIS measurement, some considerations must be made. Stack measurements are typically controlled in galvanostatic mode for better control of the power, as small changes in the voltage lead to big changes in the current drawn by the stack, especially with increased active surface area of the cells. The alternating current signal, which is superimposed on the DC stack current, should be as small as possible to ensure that the system behaves linearly and time-invariantly. This is necessary for further analysis steps, which incorporate a model fit of the measurement to an equivalent circuit that represents the electrochemical system and describes it in more detail. Based on that requirement, a second constraint arises in the actual measurement of the voltages of the stack and the individual cells. The respective levels of the voltage response need to be high enough to be measured adequately, which comes down to a good signal-to-noise ratio (SNR). Typical values are up to 5 % of the main current level of DC.

As stacks typically need larger current amplitudes for flawlessly measurable voltages than single cells, the problem of alternating magnetic fields and their influence on the voltage measurement needs to be considered. To minimise interference due to induction voltages in the supply cables, the area between the individual DC conductors should be kept small. This can be achieved using twisted pair sense cables. If the sense cable routing is not executed correctly, artifacts in the measurement occur that are not part of the cell impedance. This can lead to wrong interpretations.

The superimposing of such a measurement signal is a challenge for power electronics as it forces the signal through the impedance of the electrolyzer and the load cables. The load cables have an inductive behavior, which therefore should be kept as small as possible. This can be achieved by using low-inductive cables and/or minimizing the area that spans between the load cables. Alternatively, this can be achieved by twisting the load cables if possible. If the load cables have a very large cross-section due to the required current level, the cables should be routed in parallel with as little area in between as possible. Routing the load cables at the greatest possible distance to the voltage sense connections further helps to minimize the inductive coupling of artifacts. The overall impedance of the system, including the output impedance of the power supply, determines the shape and characteristics of the amplitude-damping curve, which represents the signal amplitude for every frequency measured. The favorable behavior is a constant amplitude independent of the frequency. Damping or amplification phenomena change the measurement quality:

- Excessive amplification can cause the system to leave the linear range.
- Excessive damping of the amplitude can lead to poor or unmeasurable voltages with poor SNR values.

Figure 2: Wiring recommendations for correct measurements[2]

[2] Stack Reference: H-Tec / QuestOne

5 Measurements

The following chapters discuss a measurement on the testbench “David1” and a measurement with “Goliath”.

5.1 Measurements with David 1

The measurements executed at David1 were carried out by manually correcting the amplitude values of the modulation signal because, at some frequencies, the setup tends to over-amplify or damp the signal. The setup is used to evaluate controller parameters that allow a steady operation for DC and proper signal quality for lower frequency signals, as well as fast and high enough modulation in the higher frequency range. The controller parameters evaluated are suited for the application of capacitive loads like electrolysis stacks and are specific to every individual connected impedance, including the cable inductances. For applications at higher power levels (>18 kW), multiple device configurations, and highly different load impedances, the parameters need to be re-evaluated.

The following modulation controller parameters allow a stable operation at lower frequencies and are fast enough for proper signal generation at higher frequencies:

- Current Kp: 0,5
- Current Tn: 5 µs

The standard parameters for the DC current controller that work best for DC setpoints are:

- Current Kp: 0,15
- Current Tn: 8 µs

A very critical part, as mentioned in Chapter 4, is a sufficient SNR to ensure proper signal detection. Therefore, a result in the most critical measurement point at the highest possible frequency is presented. The setup is able to realize sufficient modulation at this operation point.

Measurement results for a PEM Stack 4.5 kW:

- Amplitude setpoint: 4 A
- Stack Current Amplitude @10kHz: 2.47 A
- Damping: 20*log10(4/2,47) = 4.18 dB
- Stack Voltage Amplitude: 137 mV

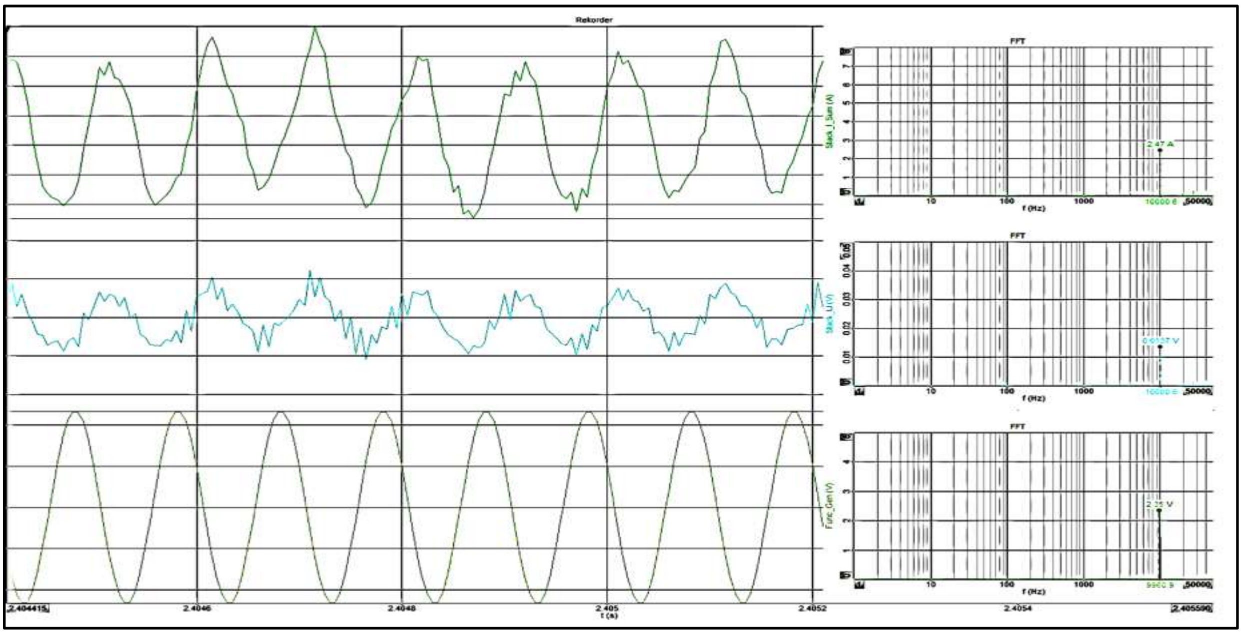

Figure 3: Time and frequency domain plots of current modulation at 10 kHz

Figure 3 presents the modulated signals at 10 kHz in the time and frequency domains. See the stack current (light green) and the stack voltage (blue). Both are slightly overlaid with noise, which cannot be avoided. As the measurement is executed in a current-controlled operation, the measured sine signal in the voltage needs to be sufficiently above the noise floor in the frequency spectra to be evaluated reliably and correctly. This condition is fully met. As a reference, the input voltage signal of the function generator is also depicted in the graphic. (darker green, below)

5.2 Measurements with Goliath

The measurements taken with testbench Goliath require a higher-level effort to correct the amplitude of the drive signal (AGC) for the modulation. During a measurement, the modulation amplitude should always stay within a given bandwidth over the entire frequency range. The data acquisition software computes the required current amplitude via a live FFT calculation and sends it to the controller.

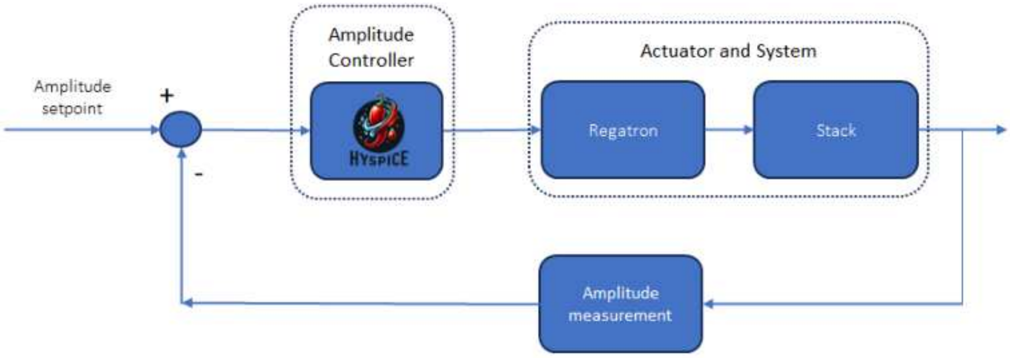

Figure 4: Amplitude controller schematic

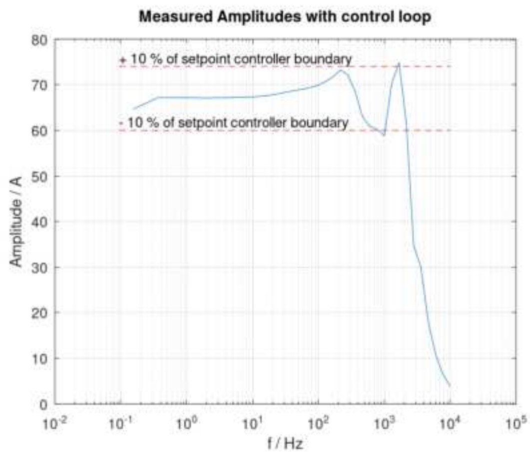

Figure 5: Modulation amplitude v/s frequency

The typical amplitude response curve plotted v/s frequency is depicted in Figure 5. The implemented controller sets the amplitude within an error band that can be defined. At some frequencies, the setup tends to over-amplify the signal. It was observed that higher frequencies were damped more strongly. The controller keeps the amplitude within the error band until a frequency of >2 kHz. Above this threshold, the attenuation effect is so pronounced that it leads to a substantial reduction in signal amplitude, even if the controller sets the signal to the maximum output level. This characteristic can be minimized by making the inductance between the power supply and the stack as small as possible., Different parameter settings also make big differences in the amplitude response. In contrast to the lower power levels exhibited by the David1 testbench, the current configuration at Goliath does not facilitate the acquisition of suitable controller parameters enabling both stable operation in a steady state and adequate signal modulation in the high-frequency range. A solution to this problem is a parameter change during the measurement, which is not implemented today.

The controller parameters for the measurement shown in Figure 5 are:

- Current Kp: 0,5
- Current Tn: 4.75 µs

Remark: In this measurement, a parameter change was not required.

6 Interpretation

The amplitude response affects the measurement quality and result, but does not need to be completely constant for the whole frequency range. As the impedance is calculated by dividing voltage by current, with less current, there is less voltage, and therefore, the calculated impedance is the same. As long as the assumption of a Linear Time Invariant (LTI) system holds, these differences do not matter.

The result of an EIS measurement is typically a Nyquist plot, which represents the locus curve of the impedance in the component representation (real, imaginary) for multiple frequencies as depicted in Figure 7. Due to the layer assembly of the cells with non-electron-conducting membranes, the electrolysis cells show a capacitive behavior. With an Equivalent Circuit (EQC) evaluation as displayed in Figure 6, the curves can be interpreted in further detail using this model. The influence of the inductance of the load cables can be kept small if the position of the voltage measurement is correct. The equivalent circuit consists of the parts that are described in Table 1. A small inductance that can be assigned to the cell itself is present and cannot be avoided. Certain points and shapes of the resulting Nyquist plot (Figure 7) can be assigned to specific electrical and electrochemical parts present in the equivalent circuit. These parts are the result of internal resistance, reactions, and mass transportation phenomena. The method of building an equivalent circuit is only permissible if the assumption of an LTI system is allowed, as all the parts of the equivalent circuit are based on an LTI behavior. The results of these locus curves shall support and accompany measurements like polarisation curves and support theories about the internal processes and stack design choices.

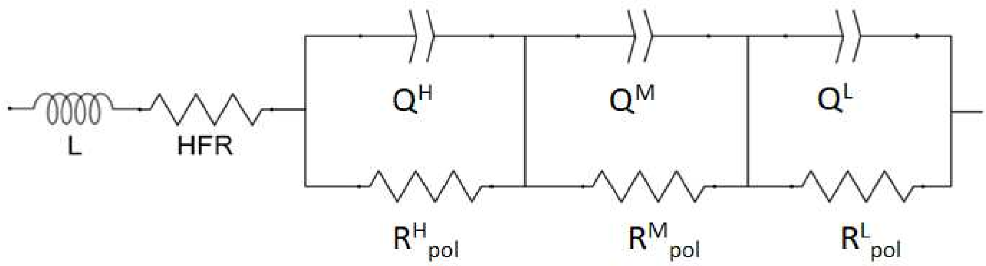

Figure 6: Equivalent circuit of an electrolysis cell [3]

Table 1: Description of the occurring resistances in the EQC

Parameter	Unit	Description
RelΩ	Ω·cm2	Electronic resistances of electron-conducting components
RionΩ	Ω·cm2	Ionic resistance of the membrane
HFR	Ω·cm2	High frequency resistance consisting of RionΩ + RelΩ
RHpol	Ω·cm2	Polarisation resistance of the high-frequency reaction kinetics
RMpol	Ω·cm2	Polarisation resistance of the medium frequency reaction kinetics
RLpol	Ω·cm2	Polarisation resistance of the low-frequency reaction kinetics
QH	$S \cdot s^n \cdot cm^{-2}$	CPE (Constant Phase Element) of the high-frequency reaction kinetics
QM	$S \cdot s^n \cdot cm^{-2}$	CPE of the medium frequency reaction kinetics
Q^L	$S \cdot s^n \cdot cm^{-2}$	CPE of the low-frequency reaction kinetics
L	nH	Inductance of the cell
Z_Q	$S \cdot s^{-n}$	Reactive Impedance of a CPE

The CPEs (Constant Phase Elements) describe the deviation of the impedance from ideal behavior (i.e., true capacitive behavior) of the capacitances in real electrochemical cells. The less they act like a real capacitor, the greater the factor n goes from 0 to 1. A circuit with R and CPE in parallel with a very unideal capacitor (e.g., 0.7) has a characteristic shape in the Nyquist plot: The occurring semicircle will be depressed (the center of the circle is underneath the real axis). The more "ideal" the behavior, the less depressed.

Figure 7 depicts a Nyquist plot representation of a prototype PEM stack, wherein the individual cells of the stack are illustrated. The imaginary part is shown in negative for better readability. The cells exhibit comparable curves, yet some display a shift along the real axis. High frequencies are shown in the left section of the plot, low frequencies are located on the right side. The intersection of the curves with the real axis is where the HFR can be read. Clearly, the cells have different amounts of HFR resistance. This can be attributed to uneven contact resistances from cell to cell and is an indicator of a potentially bad stack design, bad assembly, or faulty cells.

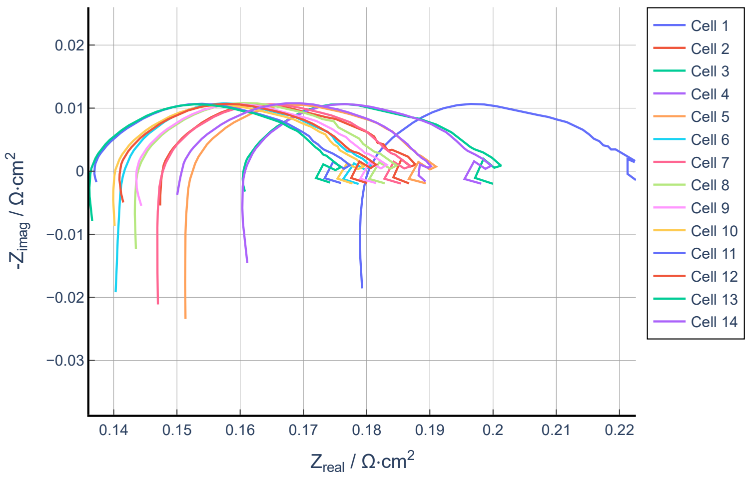

Figure 7: Nyquist plot of a PEM stack with 14 cells

The semicircular shapes observed in the occurring curves indicate that the polarization resistances of the electrochemical reactions within the individual cells are similar. The noise of the measurement in the low-frequency section comes from thermodynamic changes in the stack during the measurement of low frequencies, which induce changes in the voltage. A precise temperature control of the stack is of great importance in this part of the measurement.

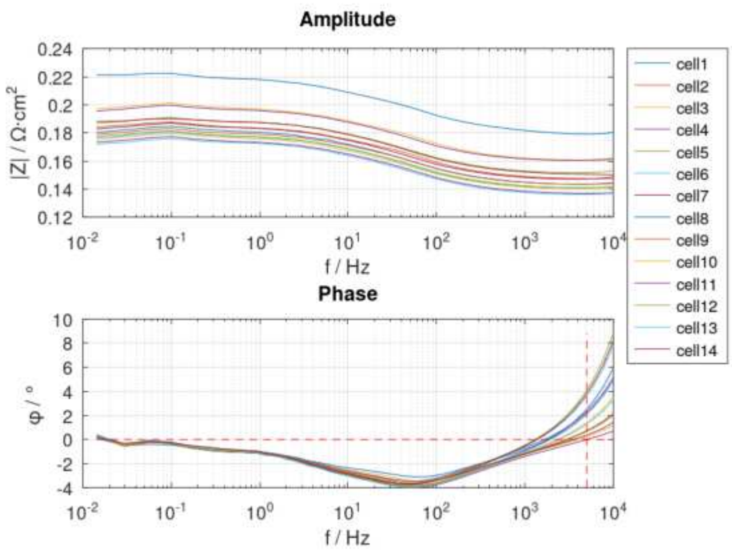

Figure 8: Bode plot of a PEM stack with 14 cells

Figure 8 shows the Bode-plot representation of the measurement, which has the advantage that the frequency dependence of the impedance is included directly in the plot. The zero crossing of the phase in the high-frequency range corresponds to the locus curve crossing the real axis, with the imaginary part being zero. This measurement value is of great importance for the characterization of such electrochemical systems. The red dotted crossing in the phase plot indicates the zero crossing at the highest frequency at 5 kHz. These zero crossings occur at frequencies ranging from 1 kHz to 5 kHz, indicating that signal modulation up to 10 kHz is adequate for such analysis.

7 Conclusions

The realization of EIS measurements with REGATRON devices in combination with appropriate data acquisition works well and is reliable at HyCentA. The devices are easy to operate and dispose of all the necessary functions that are needed for this use case. A significant advantage of the device is the ability to output both the DC operation point and the modulated signal as a single unit. There is no need for external modulators or other equipment. Therefore, the signal quality is very high as there are no interferences with other power electronics. Very important is the possibility to provide the analog signal with an external function generator, which allows for more freedom in the implementation of the setup and the automation of the measurement. The separation of the DC operation setpoints and the AC signal is of great importance for the integration into test benches that have fieldbus communication interfaces. Furthermore, the signal-to-noise ratio of the modulation increases with this separation as the analog signal does not need to provide the high DC offset too.

HyCentA is Austria's only extra-university research institution that focuses exclusively on hydrogen technologies. Founded in 2005, it conducts international research and development projects with industry and universities covering all aspects of hydrogen technologies, including production, distribution, storage, and application. HyCentA disposes of expertise in engineering, simulation, testing, measurement techniques, and education in this field and collaborates with the University of Technology in Graz on educational programs.

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