Citation (APA)
Hursán, D., Pellumbi, K., Endrődi, B., Samu, A. A., Wessel, E., Pelzer, H., Józó, V., Burdyny, T., Janáky, C., & More Authors (2026). Toward Standardization in CO2 Electrolysis: A Round Robin Study. *Advanced Energy Materials*, *16*(21), Article e06808. https://doi.org/10.1002/aenm.202506808

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RESEARCH ARTICLE OPEN ACCESS

Toward Standardization in CO_2 Electrolysis: A Round Robin Study

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Received: 31 December 2025 | **Revised:** 9 March 2026 | **Accepted:** 23 March 2026

Keywords: carbon dioxide | carbon monoxide | electrocatalysis | electrolysis | KPI comparison | reproducibility

ABSTRACT

CO_2 electrolysis is an emerging technology for the sustainable production of fuels and chemicals. Its transition from laboratory-scale research to real-world application is strongly driven by both regulatory and strategic means, aimed at achieving net-zero greenhouse gas emissions. To meet this goal, accelerated progress in CO_2 electrolysis research and technological development is essential to ensure economic viability. This requires clear performance targets, reference materials, and standardized testing protocols that serve as a basis for reliable performance comparison within the CO_2 electrolysis community. To address this need, a Round Robin experiment was conducted involving well-established R&D entities in the field of CO_2 electrolysis. The objective was to identify and mitigate the main sources of experimental variability, thereby enhancing reproducibility. We found that especially the modes of temperature measurements and cell/anolyte heating alongside pressure fluctuations and overpressures during product analysis are considerable differences among labs, while adjustments to the initial electrochemical protocol helped in minimizing voltage spikes in changing operation. As a result of multiple measurement campaigns and in-depth discussions among participants, a recommendation for a standardized testing protocol and test setup requirements for CO_2 electrolyzers are provided.

1 | Introduction

CO_2 electrolysis research is undergoing a significant transition from predominantly catalyst-focused investigations conducted in laboratory environments toward more application-driven studies in high-performance membrane-based flow reactors. This coincides with the appearance of large-scale demonstrator CO_2 electrolyzer systems, proving the readiness of the technology for pilot and demonstration plant deployment. One of the major challenges that the CO_2 electrolysis industry must tackle is extending electrolyzer lifetime beyond 20000 h [1] to achieve economic viability. This requires material stability across system components, including electrodes, catalyst supports, membranes, cell hardware, and balance of plant components; in addition to optimized operating conditions to facilitate such stability over time. To accelerate this development, joint efforts between

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TABLE 1 | Summary of the activities carried out during the project.

Activity	Goal	Outcome
Alignment	Define experimental conditions based on the previous expertise of participating labs	First measurement protocol established
Cell production and validation	Provide standardized and comparable electrolyzer hardware and MEA to all partners	Electrolyzer and MEA components prepared and shipped to participating labs; factory acceptance tests completed
1st measurement campaign	Initial cross-lab reproducibility evaluation	Test setups optimized; experimental parameters refined for the 2nd measurement campaign
2nd measurement campaign	Improve cross-lab reproducibility and in-lab repeatability through parameter standardization	Electrochemical protocol refined for the 3rd campaign
3rd measurement campaign	Improve coherent performance across labs	Protocol validated and agreement reached on standardized testing method
In-depth analysis	Identify setup requirements and main reasons for experimental variability	Understanding of variability sources and setup standards

academic research institutes and industry players are needed [2]. A major hurdle is the lack of standardized testing protocols and reference materials within the CO_2 electrolysis community. Such uncertainty and variability inhibit research and commercial entities due to questionable comparability across various labs, thus requiring each institution to undertake its own individual optimization and exploration processes. These processes are costly from both a financial and a time perspective.

Although literature includes publications outlining practices for assembling and operating laboratory-scale CO_2 electrolyzers [3, 4], there remains considerable variation in cell designs, MEA/electrode components, chemicals used, and operating parameters—not only between labs but often within the same lab across various research projects/reports. Consequently, the true reason behind outstanding performance often remains obscured because of improper and frequently arbitrary benchmarking practices.

While academic research often focuses on improving one performance metric (e.g., cell voltage or Faradaic efficiency), this often connected with significant sacrifices of another metric. In industrial settings, key performance indicators (KPIs) must be holistically optimized. While these KPI targets are often also used as directions in academic research [5], standardized testing protocols are needed for a more complete comparison of the employed materials and methodologies. The main operating conditions—such as cell temperature, pressure, CO_2 inlet flow, relative humidity of the CO_2 inlet—largely influence cell performance, and their effects are well-studied topics. For instance, cell temperature has a pronounced effect on both cell voltage (decreasing with increasing temperature) and product selectivity. In general, higher temperature decreases CO_2RR selectivity vs. HER, while C_{2+} production is maximized on Cu electrodes at intermediate temperatures [6–8]. Both cathode pressure and differential pressure have been found to strongly impact CO_2 electrolysis performance, where elevated pressures generally improve mass transport, thereby affecting catalytic selectivity and improving energy efficiency [9–11]. Nevertheless, pressure remains mostly an uncontrolled parameter in laboratory research, introducing uncertainty to performance evaluation. Relative humidity influences electrolyzer performance by water being a proton source for both CO_2RR and HER, affecting membrane hydration and ionic conductivity, and also affecting precipitate formation. Fluctuations in relative humidity can induce mechanical stress in membranes and electrodes [12], compromising long-term stability, therefore its precise control is needed. Optimizing humidity [13] levels is also critical for maintaining proper cation balance at the cathode gas diffusion electrode (GDE) [14], thereby enhancing system longevity. Overall, controlled relative humidity helps to ensure consistent operating conditions and reproducible performance.

While cross-laboratory (Round Robin-type) experiments have been conducted for water electrolysis [15, 16] aiming to develop standardized testing protocols and benchmark systems—such efforts are still lacking in the field of CO_2 electrolysis. The current work, in the framework of the ROROTE project (Round-Robin Testing for CO_2 Electrolysis), was a European initiative designed to fill this gap by establishing a unified testing protocol for the CO_2 electrolysis community (Table 1).

2 | Alignment Activities

At the beginning of the project, an online kick-off meeting was organized with the participation of the six involved laboratories. The goal was to identify the critical operating conditions that, based on previous experience of the participants, must be fixed for the first measurement campaign to obtain reproducible data. These parameters were the following: anolyte composition, inlet CO_2 flow rate and humidity, cell (cathode) temperature, and cathode back pressure. Questions on the pre-conditioning of the electrolyzer prior to the electrochemical protocol were also discussed and cleared. The same electrolyzer cell and MEA components, manufactured by single entities, were used by all partners throughout the project, as detailed in Sections S1 and S2. To avoid any inconsistency, the membrane pretreatment protocol

TABLE 2 | Experimental conditions for the three measurement campaigns. Changes between the first and second campaign are highlighted in light green, while changes between the second and third campaign are highlighted in dark green.

	First measurement campaign	Second measurement campaign	Third measurement campaign
Cell temperature	60°C	60°C	60°C
Membrane surface area / pretreatment solution volume ratio (cm^2/cm^3)	Not specified	1.2 cm^2 / cm^3; typically 1 piece of A4 sheet in 500 cm^3 1 M CsOH solution	1.2 cm^2 / cm^3; typically 1 piece of A4 sheet in 500 cm^3 1 M CsOH solution
Typical duration between completing membrane pretreatment and beginning cell assembly	Not specified	Use the membrane within 24–48 h after completion of pretreatment (i.e., max. 72 h from start of activation)	Use the membrane within 24–48 h after completion of pretreatment (i.e., max. 72 h from start of activation)
Membrane rinse before cell assembly	Not specified	Soak it in water for 5 min before cell assembly	Soak it in water for 5 min before cell assembly
Volume of anolyte	Not specified	200 cm^3	200 cm^3
Flow rate of CO_2	CO_2 excess = 6, i.e. 35, 100, 175 cm^3 / min CO_2 at 100, 300 and 500 mA cm^{-2}	CO_2 excess = 6, i.e. 35, 100, 175 cm^3 / min CO_2 at 100, 300 and 500 mA cm^{-2}	CO_2 excess = 6, i.e. 35, 100, 175 cm^3 / min CO_2 at 100, 300 and 500 mA cm^{-2}
Thickness of the PTFE gasket used to adjust cathode compression	300 µm* see also Supporting Information	300 µm	300 µm
Anolyte temperature at the start of anolyte circulation	Not specified	60°C	60°C
Flow rate of anolyte circulation	60 ± 20 cm^3 min^{-1}	65 ± 5 cm^3 min^{-1}	65 ± 5 cm^3 min^{-1}
CO_2 flow during cell heating?	Yes	Yes	Yes
Dry or humid CO_2 is used during heating?	Humid	Humid	Humid
CO_2 flow rate during heating	Not specified	35 cm^3 min^{-1}	35 cm^3 min^{-1}
Heat-up time	15 min with the application of 10 mA cm^{-2} current	45 min heating without bias + 15 min conditioning at 10 mA cm^{-2}	45 min heating without bias + 5 min conditioning at 10 mA cm^{-2}
Cell temperature when starting the electrochemical experiment	Not specified	>58°C	>58°C
PTFE cell holder used?	Not specified	Yes	Yes
Cathode temperature measured?	Was not required, however, the possibility was given for that	Yes (preferably logged too)	Yes (preferably logged too)
Anolyte pH / conductivity measurement during the experiment?	Not specified	Preferred, but not required	Preferred, but not required
Anolyte temperature measured?	Not specified	Preferred, but not required	Preferred, but not required
When to change the CO_2 flow changed between the current steps?	During the first current increase step, or the last current decrease step	During the first current increase step, or the last current decrease step	During the first current increase step, or the last current decrease step
Humidifier temperature	55°C	60°C	60°C
Anolyte salt Formula	CsOH converted into $CsHCO_3$ by CO_2 bubbling	$CsHCO_3$	$CsHCO_3$
CsOH/$CsHCO_3$ supplier	Not specified	Sigma–Aldrich	Sigma–Aldrich
CsOH/$CsHCO_3$ grade	Not specified	>99.9	>99.9
Anolyte concentration	0.05 M	0.05 M	0.05 M

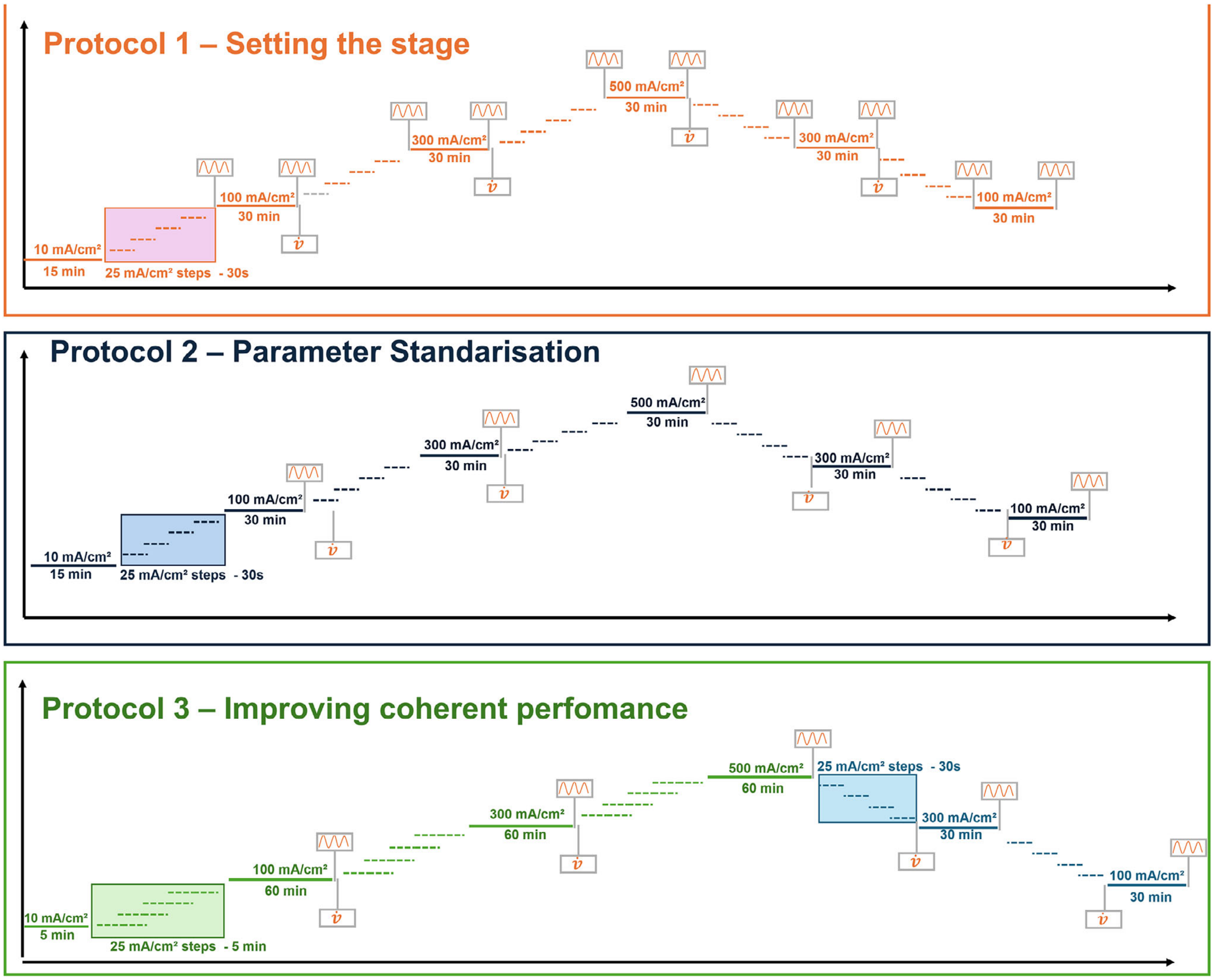

FIGURE 1 | Electrochemical protocols used in the three measurement campaigns of the project.

was also fixed. The experimental parameters were thoroughly discussed between participants, and the ones that were considered to be the most relevant were fixed. These are summarized in Table 2.

An electrolysis protocol (Protocol 1 in Figure 1) was created by incorporating suggestions from all participants. Some of the key aspects were the following:

- *Cell heat-up*: During the equilibration of the cell to the target temperature, apply a small current (10 mA cm^{-2}) to the cell.
- *Current ramp-up*: Instead of applying a large current density directly, increase the current density gradually in 25 mA cm^{-2} steps each held for 30 s in order to let the cell thermally, mechanically, and electrochemically condition. Although industrial electrolyzer systems typically employ a continuous constant rate current ramp during startup, stepwise current increases are preferred in laboratory environments, as they enable more reliable acquisition of polarization curves and Tafel data [17, 18].
- *Electrochemical impedance spectroscopy (EIS)*: Perform galvanostatic EIS at the start and end of each tested current density (100, 300, and 500 mA cm^{-2}) at 10 mA RMS (root mean square) over a frequency range of 1 Hz to 100 kHz. Having EIS incorporated in our protocol could serve as a diagnostic tool, helping to deconvolute voltage losses (ohmic, kinetic, mass transport) and revealing MEA, cell/stack, and system-related failures [19, 20].
- *Product analysis*: If not continuous, take samples after 30 min of electrolysis, prior to the EIS step.
- To maximize the impact of the investigation, test lower current densities (100 and 300 mA cm^{-2}) twice to assess whether cells behave similarly during downturn or exhibit hysteresis effects.

The partners also agreed on what data they will share as follows:

- All electrolysis data, such as FE, $U_{Cell,}$ and EIS. All data was stored in a shared database.
- Set-up information, scheme, and photograph of the set-up (including the used instruments and their calibration method).
- Formula for the calculation of FE.

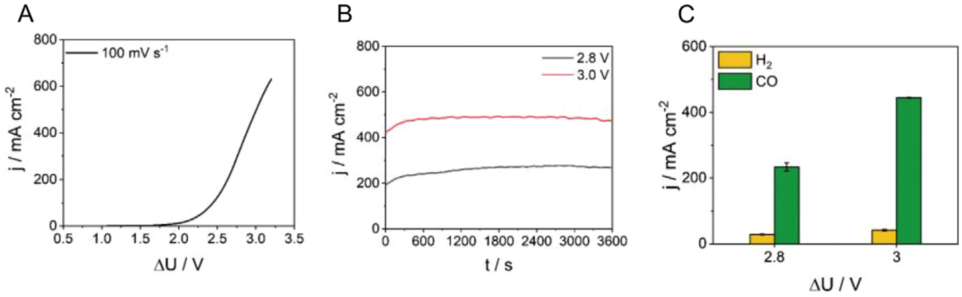

FIGURE 2 | Factory acceptance test results of the electrolyzer cell sent to Lab-1.

- Any operando analytics, such as temperature variation, pressure changes during electrolysis.

3 | Cell Production and Validation

The electrolyzer cell used in this project was supplied by eChemicles, with an active area of 8 cm^2. Prior to distribution to project partners, each cell underwent pretesting to verify its functionality. A detailed test report and assembly manual were provided alongside the cell. The general considerations for cell assembly are provided in Section S2. The pre-testing consisted of linear sweep voltammetric (LSV) and chronoamperometric (CA) characterization applying the conditions listed in Section S3. The results—including current densities from LSV and CA tests, and product distribution (CO/H_2 ratio) during CA—are summarized in Table S1. The performance graphs of the electrolyzer cell sent to Lab-1 are shown in Figure 2 as an example. The variation among the various cells stayed within the specified limits, but their minor contribution to the interlab variations cannot be excluded.

4 | The First Measurement Campaign

In the first measurement round, all the participating laboratories performed initial tests and optimized their test setups for the employed electrolyzer cell, incorporating the previously reported cathodes fabricated by Fraunhofer UMSICHT [21] and the Ir-coated anodes, and PTFE gaskets provided by eChemicles. The same batch of PiperION membrane was used for all the experiments in the project to ensure consistency. During this round, in-lab repeatability was not evaluated because operational parameters were not always consistent due to the ongoing setup optimization process. The laboratories encountered various challenges, including back pressure from the analytical setup (e.g., gas chromatography connection), temperature control issues, and unclear cell assembly instructions (e.g., the method for tightening screws, gasket thickness to be used). Therefore, for the first campaign, only the best-performing experiment (i.e., the lowest achieved cell voltage)—obtained with the optimized setups—from each participant was compared. One representative dataset obtained at Lab-6 is shown in Figure 3, while a comparative bar plot presenting the main KPIs (cell voltage, selectivity, and high frequency resistance, considered as series cell resistance, R_{cell}) obtained in the different labs is provided in Figure 4.

For the comparative plots, we chose to report the CO/H_2 ratio instead of Faradaic efficiencies because both significant (±10%) overestimation and underestimation of the $\sum$FE value were observed (Figure S2). This highlights a widespread challenge within the CO_2 electrolysis community. We mostly attribute this observation to non-quantitative product analysis and/or inaccurate outlet flow measurements (note the difficulty tied to measuring the volumetric flow rate of gas mixtures of varying composition). Similarly, it is important to pay attention to the employed calibration gases as they should match the expected vol% of the different CO_2-electrolysis for different cell sizes and operating conditions. Scaling Faradaic efficiencies to force their sum to 100% is also not an appropriate solution, as it can mask underlying inaccuracies in product quantification and the formation of possible alternative CO_2 reduction products.

Since Ag is known to be a selective electrocatalyst for CO production, liquid-phase product analysis was not performed. The formation of other products, however, cannot be completely ruled out. For example, small amounts of formate might be expected [22], but its quantification in AEM CO_2 electrolyzers is challenging due to its anionic nature, which can lead to membrane crossover and anodic re-oxidation.

While CO:H_2 ratios provide a practical comparison metric, they can be misleading at high CO selectivities (FE_{CO} > 95%), where small variations in FE result in disproportionately large changes in the ratio (Figure S2). This limitation should be considered when interpreting performance trends. Overall, a combination of presenting all three routes (faradaic efficiency, CO/H_2 ratio, CO vol% in total gas mix) allows for a more complete and sound presentation of the obtained data.

At the lowest current density (100 mA cm^{-2}), cell voltages—read at the end of the constant current testing phases—varied between 2.6 and 2.8 V across the different laboratories, corresponding to a 200 mV difference (Figure 4). At 500 mA cm^{-2}, the difference between the highest and lowest measured cell voltage increased to 700 mV, indicating that operation at higher current densities introduces greater measurement uncertainty. The dataset could be grouped into two categories: Labs 4, 5, and 6 reported significantly lower cell voltages compared to Labs 1, 2, and 3. Interestingly, this trend correlated with the applied heating method. As for the practicalities, a major difference in the typical cell heating method among partners was identified. While Lab-1 and Lab-3 placed the entire electrolysis setup in a heated oven, Lab-4 and Lab-5 tempered the cell using the heated anolyte. Lab-2 employed a combined approach: both

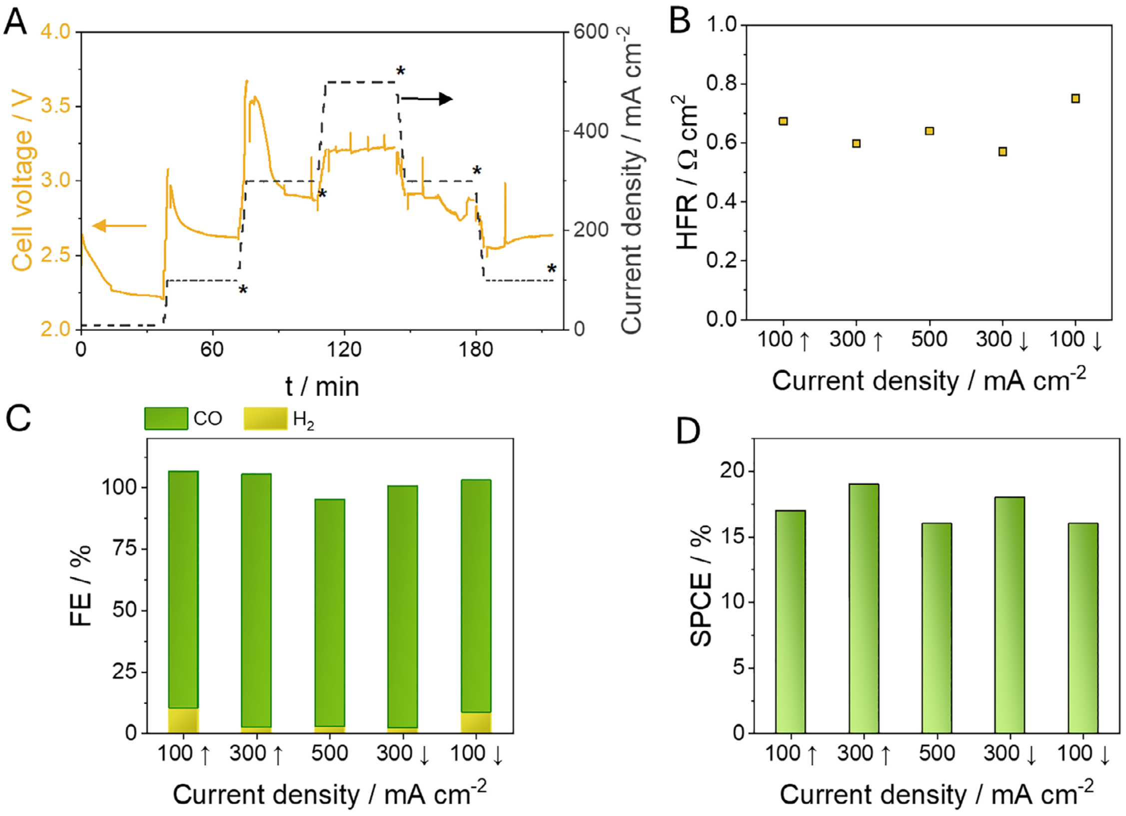

FIGURE 3 | Representative dataset of the first measurement campaign, measured at Lab-6. (A): Voltage and current profile. The asterisk shows where the EIS spectra were taken. (B): High frequency resistance measured at the end of the constant current testing phases in both the upward and downward directions. (C): Faradaic efficiency (FE) for CO and H_2. (D): Single pass conversion efficiency (SPCE). The experimental conditions are listed in Table S3.

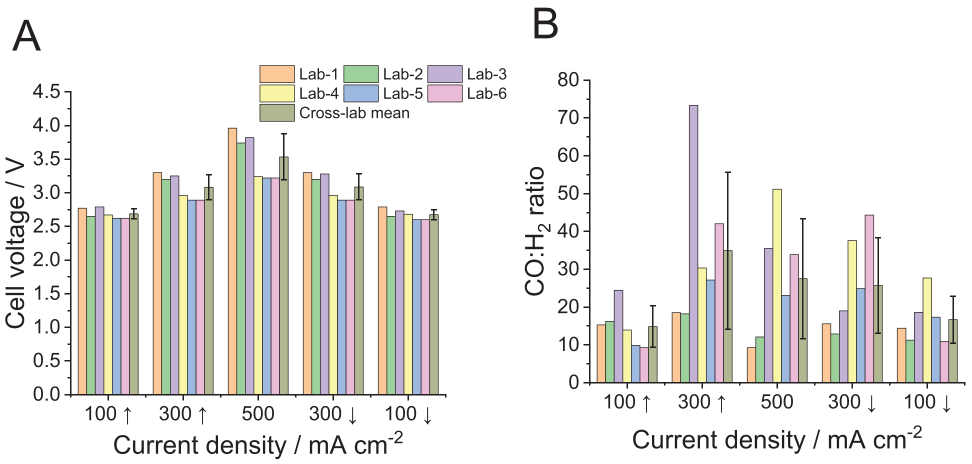

FIGURE 4 | Cell voltages (A) and selectivity (B) obtained in the first measurement round in the different laboratories (as recorded 30 min after setting the given current density). The cross-lab average was calculated by averaging the respective values measured in the individual labs. Error bars represent the standard deviation of these values. Note that the gasket thickness at the cathode side and electrolyte concentration varied across labs. For details, see Table S3.

the cell and the humidifier were placed in an oven, while a heating jacket was used for the external anolyte. Additionally, Lab-2 employed an anolyte concentration of 0.1 M $CsHCO_3$ in their single-run 1st campaign. Although it is well known that the heating method can significantly influence CO_2 electrolysis performance [8], we decided not to standardize this aspect, as this would have required major system modifications for some partners (see the process flow diagrams of the setups in Section S3). Laboratories that tempered the cell using only the heated anolyte achieved lower cell voltages than those that placed the entire setup (including the cell) in a heated oven. Notably, setting the anolyte or oven temperature to the nominal measurement temperature (e.g., 60 °C in the present study) does not necessarily result in the same actual cell temperature. Heat losses in the tubing and cell body, together with the heat generated by the electrochemical reaction, often cause substantial deviations from the intended temperature. As a result, when heating through the anolyte loop, a higher anolyte setpoint temperature is typically required to compensate for thermal losses before the fluid reaches the cell. In contrast, when operating in an oven, the cell may exceed the target temperature due to limited heat dissipation and the additional internal heat produced during electrolysis (Figure S3). For these reasons, temperature control should be based on the actual cell, ideally cathode, temperature (or, if direct measurement is not possible, on the anolyte inlet temperature as close to the cell as feasible), rather than relying solely on the nominal temperature of the heating system.

In terms of selectivity, all groups achieved a CO:H_2 ratio greater than 9 at each current density. In this case, no direct correlation between the heating method and the selectivity was revealed. The observed large deviations in the CO:H_2 ratio correspond to minor variations in the Faradaic efficiencies of CO (provided that only CO and H_2 are the formed products). Specifically, the CO:H_2 ratio of 70, measured for Lab-3, corresponds to a CO Faradaic efficiency of approximately 99%, while the ratio of 18 measured for Lab-2 under the same conditions (300 mA cm^{-2}) corresponds to about 94%. Such differences can easily arise from typical measurement uncertainties between laboratories—especially analytical instrument calibration or gas flow measurement challenges during higher current density operation. In general, any electrolyzer cell achieving CO Faradaic efficiency above 90% (CO:H_2> 9) is regarded as a high-performing system within the community.

After completing the initial measurement round, detailed operating parameters were collected from each partner using a questionnaire (Section S5). This revealed major inconsistencies among the tests performed in different laboratories. For example, the heat-up time required for the cell to reach 60 °C varied significantly and was generally longer than the 15 min specified in the original protocol. Additionally, some laboratories began pumping room-temperature anolyte into the cell, while others used preheated anolyte. This difference could influence membrane and ionomer hydration as well as thermal gradients, thereby affecting initial performance.

Preparation of $CsHCO_3$ anolyte from CsOH also raised concerns. Conductivity and pH measurements were performed in some laboratories to assess the completion of the acid–base reaction between CsOH and CO_2. Large deviations in the time were observed (ranging from 15 min to 48 h), which may stem from differences in CsOH quality (e.g., degree of carbonation) or variations in CO_2 flow rate and/or the volume of CsOH solution treated. Finally, to eliminate this source of variability during the project, a consensus was reached to use $CsHCO_3$ purchased directly from a supplier rather than converting CsOH in-house. Using CsOH, however, is significantly more cost-effective, making it the preferred option in industrial settings and budget-conscious laboratories. Therefore, we strongly recommend establishing clear purity criteria for CsOH-derived $CsHCO_3$ anolyte in future protocols.

EIS measurements conducted at the beginning of the galvanostatic phases in the upward steps were not reliable, due to the rapidly and highly changing cell voltage. Therefore, only the EIS spectra at the end of the galvanostatic steps were used for analysis. The measurement protocol was also modified accordingly (Figure 1- Protocol 2).

5 | The Second Measurement Campaign

To improve both cross-laboratory and in-laboratory reproducibility, several parameters were fixed for the second measurement campaign that had been left open (or not explicitly clarified) during the first round (see Table 2). These fixed parameters were selected based on the combined expertise of all participating laboratories, taking into account their potential benefits for performance and reproducibility, as well as the effort required to implement the changes. The latter consideration ensured that these adjustments would not necessitate major modifications to the experimental setup, since one of the main goals of the project was to propose the basic recommended setup requirements for reproducible CO_2 electrolysis experiments. For instance, harmonization of the heating mode was not required—as this would demand major setup modifications—even though major differences were pointed out with regard to this during the initial measurement campaign. On the other hand, fixing the anolyte volume was not expected to significantly affect short-term electrolyzer performance, but this change could be carried out relatively easily by each participant.

The following parameters were fixed for the second campaign: (i) ratio of membrane surface area / pretreatment solution volume, (ii) minimum and maximum time elapsed between membrane pretreatment and measurement, (iii) using $CsHCO_3$ directly instead of preparing it from CsOH, (iv) temperature of anolyte at start of anolyte circulation (v) cell heating with humidified CO_2 flow (vi) heat-up time fixed to 45 min (vii) cell temperature when starting the electrochemical experiment (viii) PTFE cell holder to be used during the tests (could affect heat-management). These are detailed in Table 2, the changes compared to the 1st measurement round are highlighted in light green.

In the second measurement campaign, the electrochemical protocol remained the same as for the first campaign. In this measurement round, three laboratories carried out experiments, but all six were actively involved in the data analysis and discussions. Having the setups optimized, the participating three labs were able to perform parallel experiments to evaluate in-lab repeatability as well. The results are summarized in Figure 5. The cell voltages at 100 mA cm^{-2} in the upwards ramp were within a range of 110 mV (2.72–2.83 V), and both in-lab repeatability (standard deviation of the parallel measurements performed within one lab) and cross-lab reproducibility (standard deviation of the average values measured across laboratories) were comparable. At higher current densities, however, reproducibility decreased in both cases, with cross-lab deviations ($\Delta U_{\text{cross-lab}}$ = 1.01 V) exceeding in-lab ($\Delta U_{\text{in-lab}}$ = 0.75 V) variations at 500 mA cm^{-2} current density. The relatively large cross-lab deviation at high current densities can be partially rationalized by the

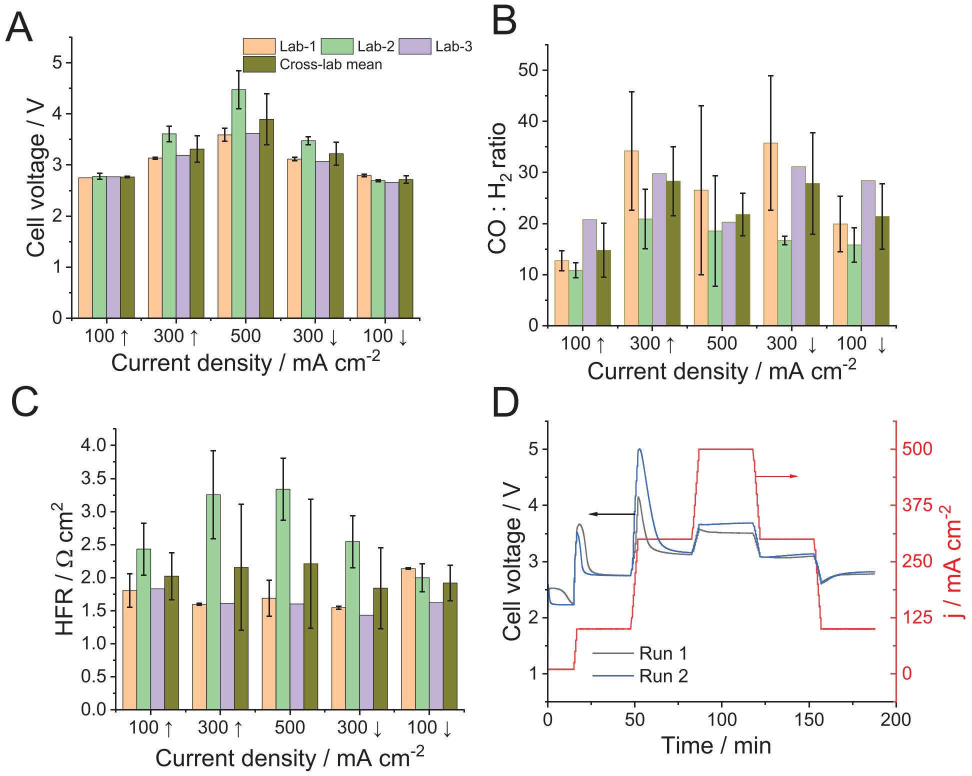

FIGURE 5 | Results of the second measurement round, where three laboratories participated (Lab-1, Lab-2, Lab-3). Cell voltages (A) CO:H_2 ratios (B), and high frequency resistance values (C) obtained in the second measurement round in the different labs. Voltage and current profiles measured at Lab-1 (D). Error bars within a single laboratory represent the mean and standard deviation of at least two parallel measurements performed under identical conditions. The cross-laboratory mean was calculated as the average of the mean values obtained from each individual laboratory, while the error bars for this metric represent the standard deviation of those individual laboratory means.

significant deviations of the measured high frequency resistance (HFR) values (Figure 5C), which varied between 1.5 and 3.3 Ω cm^{-2} at 500 mA cm^{-2} in the different laboratories. Indeed, the HFR-values showed correlation with the measured cell voltages (and also with the initial "peak" height upon increasing the current density- Figure 5D; Figure S7). The variation in the HFR values may originate from membrane conductivity issues or significant interfacial contact resistance at the GDL/membrane interface. The former could result from differences in the applied heating method and/or other setup-specific differences impacting thermal gradients within the cell, which may also influence relative humidity (e.g., lengths of tubing, types of connectors with accidental cold points) and, consequently, membrane conductivity. Assembly errors could further contribute to increased interfacial contact resistance (e.g., slight misalignment of MEA components), leading to higher HFR values. Notably smaller variation was observed during the ramp-down measurements, indicating that after an initial "break-in" period, the behavior is much more similar for the various setups.

The CO:H_2 ratios remained consistently high, above 10 at all current densities. However, Lab-2, which had the highest cell voltage, reported the lowest CO-production selectivity values as well, implying that in addition to the increased ohmic resistance, the increased voltage might also stem from increased CO_2 transport resistance or kinetic overpotential (possibly originating from slight differences in temperature and/or humidification), shifting selectivity toward H_2 evolution.

A common observation was the occurrence of significant voltage spikes during current ramp-up. Interestingly, these spikes varied in magnitude even among replicas within the same laboratory and, in some cases, exceeded 5 V (Figure 5D; Figure S3) and stabilized only very slowly to cell voltages values one could expect between 3 and 4 V. Notably, the phenomenon appeared only when increasing the current from 10 to 100 and from 100 to 300 mA cm^{-2}, while at 500 mA cm^{-2} the spikes were absent or minimal. Although this transient voltage behavior has not been thoroughly studied in the CO_2 electrolysis literature, a similar phenomenon was observed in water electrolysis. Transient water starvation was reported to result in voltage overshoots in AEM water electrolyzers [23]. Another plausible explanation is thermal shocks within the MEA. As the current rises, localized temperatures may increase rapidly inside the MEA. If this heat cannot dissipate efficiently (particularly when the cell is housed in a heated oven), thermal gradients may develop, placing mechanical and/or chemical stress on components, for example, through uneven membrane hydration. In this case, its short-term effect could be the observed transient voltage increase, potentially caused by localized and reversible membrane dry out, which would temporarily increase the ohmic resistance of the cell [24–26]. Over longer timescales, however, such an effect may lead to even more severe degradation processes, including pinhole formation on the membrane. Nevertheless, understanding this mechanistic phenomenon requires detailed in-situ probing of the MEA, and lies beyond the scope of this paper. It represents, however, an intriguing avenue for future research, as it could

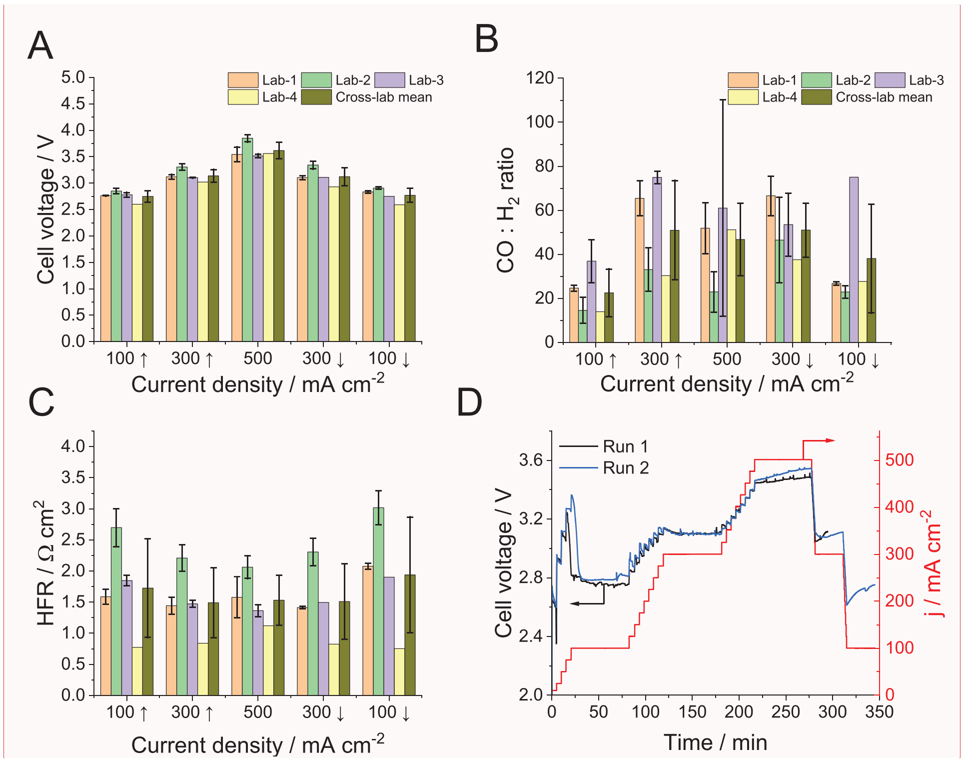

FIGURE 6 | Cell voltages (A) CO:H_2 ratios (B), and high frequency resistance values (C) obtained in the third measurement round in the different labs. Error bars within a single laboratory represent the mean and standard deviation of at least two parallel measurements performed under identical conditions. For Lab-3, during the current ramp-down steps, only one set of experimental data was available. The cross-laboratory mean was calculated as the average of the mean values obtained from each individual laboratory, while the error bars for this metric represent the standard deviation of those individual laboratory means. Voltage and current profiles measured at Lab-3 (D).

significantly influence electrolyzer start-up and conditioning protocols.

6 | The Third Measurement Campaign

In the third measurement round, the experimental conditions remained unchanged; only the electrochemical protocol was modified (Table 2 and Figure 1). The primary objective was to enhance cross-laboratory consistency and measurement stability, with particular emphasis on reducing or eliminating the voltage spikes observed during current steps. Participants agreed that these spikes most likely originated from the rapidly changing reaction microenvironment during the current ramp-up. To mitigate this issue, the hold time at each current step was extended. Specifically, the hold time at the 25 mA cm^{-2} current steps was increased from 30 s to 5 min, allowing longer conditioning before the galvanostatic measurement periods.

This modified sequence successfully reduced voltage spikes (Figure 6D). In this round, spikes were only observed before the 100 mA cm^{-2} step, and the system remained stable at higher current densities. Beyond improved stability, cell voltage values also decreased slightly across all laboratories compared to the second campaign. With standardized measurement conditions and extended conditioning time, therefore both in-lab repeatability and cross-lab reproducibility improved regarding cell voltage and HFR values as well (the only exception is the HFR measured at 100 mA cm^{-2} in the down-ramp, Figure 6). The raw EIS spectra are presented in Figures S4–S6. Since the number of participating laboratories differed in the second and third measurement campaigns, the improvement in crosslaboratory reproducibility was evaluated using only the dataset from Lab-1, Lab-2, and Lab-3, thereby excluding data from Lab-4. This comparison is shown in Figure S9.

Specifically, at 500 mA cm^{-2}, the maximum deviation in cell voltages within one lab was ($\Delta U_{\text{in-lab}}$) 140 mV, while the cross-lab deviation ($\Delta U_{\text{cross-lab}}$) was 370 mV. The variation in HFR values across laboratories was also lower compared to the 2nd measurement round (0.72 vs. 1.96 Ω cm^2 at 500 mA cm^{-2}), as the HFR for Lab-2 significantly decreased. Through this thorough parameter standardization, the performance variations between the labs that employed anolyte or oven heating also diminished. The measured cell voltage values of Lab-4 (anolyte heating) were comparable with those of Lab-1 and Lab-3 (oven heating). The large error bars observed in the CO:H_2 ratios (Figure 6B) are attributed primarily to the limited number of replicate measurements. We cannot fully rule out, however, experimental instability at higher current densities (under not fully optimized conditions) as has been reported previously for similar CO_2 electrolyzer systems [14].

To further elaborate on the origin of voltage spikes during the current ramp-up, a control experiment was conducted in Lab-2, in which the most severe spikes have been observed in the 2nd measurement campaign. Therefore, the ramp-up to the target current density was performed in potentiostatic mode instead of galvanostatic mode. That way, the magnitude of the voltage spikes is minimized by design (Figure S4). Also, lower cell voltage and the lowest HFR values were reached among the measurements performed in Lab-2 compared to the 3rd measurement campaign. This experiment shows that the operating mode can influence measurement stability by lessening the influence of voltage spikes on the measurement.

Overall, the parameter standardization across the three measurement campaigns led to notable improvements in both in-lab repeatability and cross-lab reproducibility. The remaining variations in cell voltages, HFR values, and selectivity are likely attributable to other laboratory-specific factors outside the scope of the current harmonization effort (e.g., tubing lengths, connector types influencing humidification levels, specific device calibration levels both at the input stage as well as quantification level, and gas inlet temperatures) or, in minor amounts, to MEA-to-MEA differences and cell hardware variability. These aspects could be systematically investigated in a dedicated follow-up study.

7 | Final Conclusions and Recommendations

Upon analyzing the results of all three campaigns together as a whole, the main observations and recommendations are the following:

The heating method has a significant influence on cell performance. Anolyte heating, compared to using a heated oven, appears to be a simpler and potentially more effective approach for laboratory experiments, as it may allow better heat management and easier heat dissipation. However, the most critical factor is to control the temperature based on the cell (cathode) temperature measured as close as possible to the MEA rather than the heating system temperature. This is essential because the oven or anolyte temperature—which are in many experiments the controlled values—can differ substantially from the actual MEA temperature, where the electrochemical reactions occur. Notably, in real, industrial-size stacks, no heat is provided, in contrast, efficient heat removal is the task. This latter is usually performed through the anolyte circuit, therefore this kind of thermal management brings it closer to industrial implementation [27].

Allowing the system to stabilize during ramp-up (by applying a slow current increase and/or using small step sizes) is beneficial for adapting to the dynamically changing reaction microenvironment. This approach helps to prevent issues such as local overheating and the associated voltage peaks, which in turn ensures more reliable performance under transient conditions. Also, changing the ramp-up setting from CP-steps to a CA-approach could ease the voltage peak issues.

EIS analysis is a valuable diagnostic tool for identifying voltage losses and distinguishing their origin. Performing measurements under non-stationary conditions, such as at the beginning of the electrolysis process when conditions are rapidly changing and current–voltage behavior is unstable, can, however, introduce significant uncertainties. For better reproducibility, it is recommended to conduct EIS measurements at the end of the testing period in short-term experiments.

Electrolyte composition is a crucial factor in AEM CO_2 electrolysis, impacting ionic conductivity, membrane and ionomer hydration, cathode cation balance, among others. Hydroxide and bicarbonate electrolytes are most commonly used as anolytes. However, hydroxide gradually converts into bicarbonate due to CO_2 crossover from the cathode, introducing unintended uncertainty in experiments—particularly during the initial phase—caused by a gradual change in solution pH. To avoid this, it is recommended to pre-convert hydroxide salts into bicarbonate by bubbling CO_2 into the solution. It is essential to verify the completeness of this acid–base reaction through pH and conductivity measurements.

Precise product quantification and accurate gas flow measurements can be challenging in industrially relevant electrolyzer setups and at high current densities. Even experienced laboratories often struggle to achieve a complete charge balance when determining Faradaic efficiencies. In addition, pressure fluctuations and overpressures during product analysis are considerably different if various laboratories, significantly affecting not just the product analysis but also the operation of the electrolyzer cell. Therefore, it is critically important to report the methodology used for gas flow measurement and product analysis in detail. In addition, all relevant performance metrics—such as individual and total Faradaic efficiencies, product ratios, and single-pass conversion efficiency—should be reported, and the most appropriate metric should be selected for fair comparison across studies.

Human factors—such as manual electrode coating, cell assembly, and non-automated measurement execution—can introduce significant variability in electrolysis experiments. This variability cannot be fully eliminated in either academic research or industrial R&D. To minimize its impact, comprehensive protocol descriptions and strict adherence to them are essential for benchmarking studies, including detailed explanations for process steps, even those often considered trivial (e.g., bolt tightening).

Lastly, variability in the electrolyzer cells and MEA components themselves—even when produced following identical protocols and subjected to rigorous quality control checks—cannot be fully excluded, adding an additional source of divergence in the experimental data. In addition to these points, we compiled a standardized test protocol (see Section S12), which can be used by any laboratory, and the obtained results can be compared to those published in this study. If the cell or the MEA components change, adjustments in the protocol might be necessary to achieve the optimal performance. For initial benchmarking, however, the protocol can still be employed.

In summary, the standardization of CO_2 electrolysis protocols and benchmarking practices is a critical step toward bridging the gap between exploratory research and industrial application. The main motivation behind academic research is to improve performance, whereas in industry, reliability and stability are usually prioritized over achieving record-breaking KPIs. The current initiative to standardize CO_2 electrolysis—bringing together academic and industrial stakeholders—aims to bridge this gap and foster joint research efforts, ultimately accelerating industrial deployment. Finally, we believe that with CO_2 electrolysis being a highly complex and dynamic system, standardization efforts should also be constantly adjusted as the achieved KPIs progress. In this sense, our current work should be viewed as an initial building block in a larger system toward industrial deployment.

Acknowledgements

This work has received funding from the European Union's Horizon Europe research and innovation programme under grant agreement No. 101202712, as well as the Pioneer Center for Accelerating P2X Materials Discovery (CAPeX), DNRF grant number P3. Project no. RRF-2.3.1-21-2022-00009, titled National Laboratory for Renewable Energy, has been implemented with the support provided by the Recovery and Resilience Facility of the European Union within the framework of Programme Széchenyi Plan Plus. U.-P. A. gratefully acknowledges funding by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) under Germany's Excellence Strategy—EXC2033–390677874 (RESOLV). P.S. acknowledges partial funding by the Bundesministerium für Forschung, Technologie, und Raumfahrt (BMFTR) under the DISCO2 Verbundvorhaben JETHYLEN (FKZ 033RC045B).

Conflicts of Interest

Dorottya Hursán, Angelika A. Samu, and Csaba Janáky are employees/shareholder of eChemicles, and declare the following competing financial interest(s): eChemicles is scaling up its patented CO_2 electrolyzer technology to provide an environmentally and economically sustainable alternative to fossil fuel-based chemicals.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.

Supporting File: aenm70880-sup-0001-SuppMat.docx.