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SELECTIVE ELECTROCHEMICAL REDUCTION OF CO2 TO HIGH VALUE CHEMICALS

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DELIVERABLE REPORT

D1-4 – Technical Consistency Plan	
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DISSEMINATION LEVEL		
PU	*Public*	***X***
PP	*Restricted to other programme participants (including the Commission Services)*	
RE	*Restricted to a group specified by the consortium (including the Commission Services)*	
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NATURE OF THE DELIVERABLE		
R	*Report*	***X***
P	*Prototype*	
D	*Demonstrator*	
O	*Other*	

SUMMARY	
Keywords	*Techniques, Standards*
Abstract	*This report sets the standard operating parameters for testing electrochemical* CO_2 *reduction catalysts, gas diffusion electrodes, and membranes used in the SELECTCO2 project. The purpose of this report is to allow for a set of conditions all partners can use as a guide to ensure consistency among different partners. While these guidelines are primarily meant to be used for consistency among SELECTCO2 partners, the consortium welcomes other entities to use these parameters as well to achieve consistency throughout the entire electrochemical* CO_2 *reduction field. It should be noted that this is a living document and may be modified if deemed necessary by the consortium*
Public abstract for confidential deliverables	

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TECHNICAL CONSISTENCY PLAN

CONTENTS

1 INTRODUCTION

To achieve commercial viability for electrochemical CO_2 reduction to useful chemicals, a large number of parameters needs to be tested, analysed and then optimized. Given the complexity of these devices, a large number of entities with varying expertise needs to be able to work together in a consistent manner. In doing so, consistency in methods of experimental testing and data analysis is necessary.

This report first denotes a number of key parameters that are essential towards electrochemical CO_2 reduction and then produces a guideline of what condition or range of conditions should be used. From a catalysis standpoint, having consistent parameters will allow for a direct and straightforward way to compare the effectiveness of different catalysts to a given product. From an overall standpoint, having consistent parameters allows researchers focusing on different areas of these devices (catalysis, mass transfer, membranes, etc.) to interface their area of research with that of another area, thus allowing for the production of optimal devices.

The primary purpose of this Technical Consistency Plan (**TCP**) is to produce a document that the SELECTCO2 consortium members can use as a guideline when determining how to set up a test or analyse data. Furthermore, this also will be used by the SELECTCO2 consortium members to ensure consistent parameters are used when different work packages are interfacing with each other. This will be particularly important for interfacing experimental tasks with modelling tasks. An additional purpose of this report is to make it open to the public, which allows for other researchers and developers to use these protocols, thus allowing for a larger amount of data and knowledge using consistent parameters. It should be noted that electrochemical CO_2 reduction is a rapidly advancing field. To account for this, the TCP is a living document and may be updated in the future if new or modified parameters need to be denoted and standardized.

2 SCOPE

This report focuses on the testing related to the low TRL (TRL 2-4) type issues, which is the focus of the SELECTCO2 project. While low temperature electrochemical CO_2 reduction is not yet fully commercialized, these devices are similar enough to fuel cells and water electrolysis devices, that the field has a general idea of what a commercial device will encompass. Below is a simple cartoon of the interworking of what an optimal test-scale device is generally conceived to be.

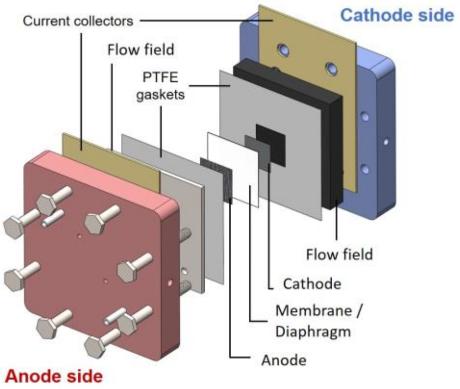

Figure 1: Cartoon of a membrane electrode assembly cell (modified from Ref [1])

Full scale commercial devices will roughly be a scaled up model of Figure 1 with stacks, power electronics, heat control, and other balance of plant issues. However, since SELECTCO2 only focuses on optimization the area between the two flow fields, the scope of this Technical Consistency Plan only relates to testing and analysis within this area. Specifically, this report provides consistency parameters for catalysts, gas diffusion layers, membranes & ionomers, flow fields, and reactor operating conditions.

It should be noted that the parameters listed in here are what the SELECTCO2 consortium partners are striving to attain, however this project has a strong research aspect to it, so some experiments and analysis produced from this project may not always follow these parameters if deemed necessary. Furthermore, to ensure maximum productivity, these recommendations for operating parameters are related to finalized results in terms of deliverables, publications, or intra-consortium disseminations.

3 DISCUSSION

This section contains a point-by-point description of the areas of work where standard procedures, techniques or materials will be used.

3.1 Characterization

3.1.1 Catalysts

It is expected that all catalysts will be characterized by:

3.1.1.1 **Scanning electron microscopy** to show the size and morphology of the catalysts. The focus should be to show a representative area of the sample.

3.1.1.2 **X-ray diffraction measurements**. The measurements should denote how the experiment was taken (2θ, grazing incidence, etc.), what source of radiation was used (Cu, Co, etc.) All peaks should be labelled and either denoted what material and crystal facet they correspond to or denoted that these are unknown peaks.

3.1.1.3 Denoting the **amount of catalyst tested**. This will vary depending upon the type of catalyst tested. For particles it is expected that the mass loading is given (mg/cm^2), whereas for single crystal and thin films catalysts this will be given in (mg/cm^2 geometric), whereas for physical vapour or chemical vapour deposition this value will be given in thickness of catalysts layers (nm).

3.1.1.4 **Post-testing analysis**. Any technique that is used to measure the catalyst before testing should also be used after testing (provided the testing technique makes this possible). This is essential to ensuring that the catalyst is properly characterized and the effect of the testing are accurately monitored.

When possible and deemed necessary catalysts will also be characterized by:

3.1.1.5 **X-ray photoelectron spectroscopy**. If the sample is exposed to air after testing, the researcher should denote this, and give a perspective on how any potential surface oxidation from the environment may affect the analysis of the XPS.

3.1.1.6 **Inductively coupled plasma (ICP) analysis for catalysts loading**. Both ICP mass spectroscopy (ICP-MS) or atomic emission spectroscopy (ICP-AES) are excellent techniques for determining exact catalyst loadings. Thus it is strongly suggested that catalyst loadings of nanoparticles be validated with ICP techniques. Since this is a destructive technique it is suggested that two samples are produced, one for measuring loading before testing and one for measuring loading after testing.

3.1.1.7 **Electrochemically active area for surface area**. Electrochemically active surface area should be calculated for all metallic catalysts as noted in Clark et. al.[2] , in terms of cm^2, thus allowing for the possibility to determine parameters such as surface normalized current densities. If it is deemed that the catalysts is situated on a substrate in such a manner that the aforementioned capacitive approach denoted by Clark et.al would be invalid, the researchers should denote that. Due to the structure- and distribution- complexity of the carbon-based single site catalysts, quantification and identification of active sites will be done via the combination of various techniques, involving electrochemically active surface area, N_2 physisorption area, chemisorption, ICP-AES, X-ray photoelectron spectra (XPS), X-ray absorption spectra (XAS).

3.1.1.8

3.1.2 Gas Diffusion Layers

It is expected that all gas diffusion layers will be characterized by:

3.1.2.1 **Scanning electron microscopy** to show the morphology and porosity of the gas diffusion layer. The analysis will be performed in surface and cross-section to observe the homogeneity and layering of the structure.

3.1.2.2 **Contact angle measurements** to show hydrophobicity/hydrophilicity of the microporous layer.

3.1.2.3 **Conductivity measurements** to ensure effective electrical contact to the catalyst. This will be measured with probes suited for soft materials will be used for such purpose.

3.1.2.4 **Porosity measurements**. This will be done via Capillary Flow Porometer (PMI/Porous Materials Inc.). Through this technique it will be possible to measure:

- Gurley number: defined as the number of seconds required for 100 cubic centimeters (1 deciliter) of air to pass through 1.0 square inch of a given material at a pressure differential of 4.88 inches of water (0.176 psi)(ISO 5636-5:2003);
- Bubble point pressure: defined as the pressure required to initiate flow through the wet sample. Such pressure is related to the largest most constricted through pore in the material, and it is therefore an indication of material porous structure.

3.1.3 Anion exchange membrane (AEM) and ionomers (AEI)

It is mandatory that all AEM and AEIs in their pristine “as-synthesised” Cl^- forms be characterised:

3.1.3.1 With at least one spectroscopic technique (Raman, FT-IR ATR, and/or solid-state NMR).

3.1.3.2 For their **ion-exchange capacities**. This characterization will be done using precipitation titrations with aqueous $AgNO_3$ titrant on Cl^- form AEMs and AEIs and reported in mmol Cl^- anions per gram of dry material as described in detail in Wang *et al.*[3]

AEMs will also normally be characterized by:

3.1.3.3 Their **ionic conductivities**. The conductivities at 60 °C in water will be done with the following anion forms: Cl^-, HCO_3^{2-} (keep in mind that once converted to bicarbonate forms – the materials will naturally convert to a mixed HCO_3^-/CO_3^{2-} form). For measuring the conductivities of pure OH^- form AEMs at 60 °C at a relative humidity 95%, the method developed by Ziv *et al.* needs to be applied.[4] While most testing of catalysts will be done at 25 °C, the conductivity will be tested at 60 °C due to both practical reasons and to relate to the standard operating procedures already established in the AEM community for water electrolyzers. However, it should be noted that conductivities scale with temperatures, thus the same trends at 60 °C will be relevant at 25 °C. Conductivities at different temperatures can be provided (between 30 – 80 °C) where required, thus allowing for more relevant data when necessary for collaborations.

3.1.3.4 **Water uptakes and swelling**. These results are specific to a give AEM type, anion and temperature, thus this data will be taken with regards to the specific conditions of interest for a given set of experiments.

When required AEMs and AEIs (in Cl^- anions forms) will also be characterized by:

3.1.3.5 **Thermal analyses** (TGA (in air), DSC, DMA). These techniques will allow for measuring changes in crystallinity, glass-transition temperatures and thermal decomposition on-set temperatures.

Spectroscopic techniques, ion-exchange capacity, and conductivity measurements can also be applied to post-cell/device tested AEMs and AEIs if required to check for *in-situ* degradations.

3.2 Cyclic voltammetry

3.2.1 **The standard operating conditions for testing will be 1M $KHCO_3$, 18 MΩ water, 25 °C and at least 99.99% CO_2.** While these will be the base conditions, it is well known that the molarity, the cation, and the anion all influence CO_2 reduction, thus it is reasonable to vary these parameters. Nevertheless, a benchmark test should be used with standard operating conditions to relate any experiments to the standard conditions.

3.2.2 The **standard scan rate for cyclic voltammograms will be 20 mV/s.** Various tests may use other scan rates, but when trying to compare results between SELECTCO2 partners, 20 mV/s will be the default scan rate.

3.2.3 Cells made to test cyclic voltammograms should be **made from either Teflon, PEEK, PFA, graphite or inert passivating metals (Ti, Nb or Ta)**. If neutral or acidic electrolytes are used, glass is also a potential option.

3.2.4 When plotting data where a reference is used, the **data should be converted to either a Relative Hydrogen Electrode (RHE) or Standard Hydrogen Electrode (SHE) scale**. If known, the pH should be stated either in the text or figure caption to allow the reader to convert between the two. Since hydrogen is by-product and there is a large knowledge base around hydrogen evolution, there are many benefits to plotting the data versus RHE. However often the local pH is unknown, and thus plotting versus RHE is impossible. It is also known that many CO_2 reduction products scale with SHE rather than RHE (i.e. the rate limiting step is not a function of H^+), thus there are certain specific benefits of plotting versus SHE.

3.2.5 Cyclic voltammograms should **compensate for ohmic losses**, on three-electrode measurements, unless noting why not. Ohmic losses between the reference and working electrode can severely modify the results and lead to misinterperation of the data. Compensating for ohmic losses (via Impedance) allows for a more intrinsic understanding of the catalyst. It should be noted in full working devices the goal is to measure and decrease the total device voltage, and in this case the ohmic losses must be included.

3.3 Cathode loading (full devices)

3.3.1 The **standard catalyst loading for nanoparticles will be 2 mg/cm^2.** This is a parameter that will vary widely and be optimized, but to allow for uniform comparison among different SELECTCO2 partners and to help with modelling, 2 mg/cm^2 will be the default loading to be used as a standard.

3.3.2 The **standard catalyst loading for sputtered films will be 100 nm.** This is a parameter that will be optimized, but to allow for uniform comparison among different SELECTCO2 partners and to help with modelling, 100 nm will be the sputtering thickness of catalysts onto gas diffusion layers.

3.3.3 The standard catalyst loading produced via an integrated catalyst-GDL production will be a 1-7 mg/cm^2 dispersed evenly throughout a 10-100 μm GDL. This is a parameter that will be optimized, but to allow for uniform comparison among different SELECTCO2 partners, but the aforementioned conditions will be used as a standard benchmark condition.

3.3.4 The **standard catalyst loading for single-site catalysts will be 2mg/cm^2.** This is a parameter that will vary widely and be optimized, but to allow for uniform comparison among different SELECTCO2 partners and to help with modelling, 2 mg/cm^2 will be the default loading to be used as a standard.

3.4 Gas diffusion layer (GDL)

3.4.1 Initially the **standard gas diffusion layer will be a Freudenberg H14C9**. This will be used to compare data among SELECTCO2 partners and to allow for a commercial GDL to be used allowing researchers outside the consortium compare with SELECTCO2 results. It should be noted though that one of SELECTCO2 objectives focuses on improving gas diffusion layers, and thus it is highly likely that as this project progresses either this standard will be switched, or an additional standard will be used based on gas diffusion layers produced by DENO.

3.4.2 The standard **gas diffusion layer thickness will be 180 μm** (which is the thickness of the Freudenberg H14C9). Again, this parameter will be varied to optimize its performance. However, it is important to start with a standard thickness because gasket need to be developed in accordance with this thickness.

3.4.3 The porosity and hydrophobicity are set values in the Freundenberg H14C9 GDL's, thus standard values do not need to be set for these. However, if in the future it is determined a new standard will be used, a standard hydrophobicity and porosity should be denoted in an updated version of this protocol (provided that these are variable parameters).

3.5 Membranes & ionomers

In the electrochemical CO_2 reduction community there is nowhere close to a unified consensus on the optimal way to separate the anode and cathode, though it is generally believed that some type of ion-exchange membrane should be used. Ion exchange membranes can be broken down into 3 types of membranes: cation exchange membranes, anion exchange membranes, and bipolar membranes, which basically consist of a cation and anion exchange membrane bound together. While only anion exchange membranes and ionomers will be optimized in the SELECTCO2 project, potentially all 3 types of membranes may be used since each approach has their advantages and disadvantages. For project efficiency, it is not suggested that all 3 types of membranes be tested for each experiment, however it is recommended that when comparing common catalysts or gas diffusion layers the same membrane should be used as a basis. Below is a description of the standard membranes to be used for each case.

3.5.1 For experiments where anion exchange membranes are used, initially the **standard anion exchange membrane used will be a Sustainion –X37-50 Grade RT**. This will be used to compare data among SELECTCO2 partners and to allow for a commercial GDL to be used allowing researchers outside the consortium compare with SELECTCO2 results. It should be noted though that one of SELECTCO2 objectives focuses on improving anion exchange ionomers, and thus it is highly likely that as this project progresses either this standard will be switched, or an additional standard will be used based on anion exchange membranes produced by US. It is expected that prior to testing, the loosely bound anions in the membrane will be exchanged to that of the anions used in the electrolyte. Since the standard electrolyte is 1M $KHCO_3$, this would entail that membranes will be pretreated in a carbonate salt solution (e.g. $KHCO_3$) prior to actual testing to ensure all residual anions are removed and will not influence the results.

3.5.2 For experiments using cation exchange membranes, **the standard cation exchange membrane used will be Nafion 212**. The SELECTCO2 project does not look to improve cation exchange membranes because this is already a highly developed field, so it is highly unlikely that this standard will be changed throughout the course of the project. It is expected that prior to testing the cations in the membrane will be exchanged to that of the cation used in the electrolyte. Since the standard electrolyte is 1M $KHCO_3$, this would entail that Nafion 212 membranes will be pretreated in a potassium salt solution (e.g. $KHCO_3$) prior to actual testing to ensure all residual cations are removed and will not influence the results.

3.5.3 For experiments using bipolar membranes, **the standard bipolar membrane used will be Fumasep FBM-PK**. The SELECTCO2 project is not currently planning on looking to improve bipolar membranes, however this is a new and advancing field, so there may be the potential for newer standards to be used in the future. If it is decided to change standards, this document will be updated accordingly. It is expected that prior to testing both the cation and anions from the bipolar membrane will be replaced with K^+ and HCO_3^-, respectively.

3.5.4 **Standard testing will not initially involve ionomers**. However ionomer production and optimization is a part of the SELECTCO2 project so it is highly probable that a standardized procedure for type and loading of ionomer with catalysts. While the ionomers produced in SELECTCO2 will be non-commercial there will be comparative commercial standards (e.g. from Dioxide Materials) that can potentially be used as standards.

3.6 Anode (full devices)

3.6.1 The standard anode used in this project will be a De Nora 'DN351' anode. Oxygen evolution anodes are well established technology and DENO is a worldwide expert in making these type of electrodes. As part of the SELECTCO2 project DENO will be supplying the partners with these electrodes, thus allowing for a commercial product to be used as a standard anode. However, the DENO anode is not marketed towards research scale electrolysis, which entails most researchers outside the SELECTCO2 consortium will not have access to these electrodes. In situations where the SELECTCO2 partners are trying to provide a standard that researchers can use to directly compare their data to work produced from SELECTCO2, a Dioxide Materials 'Anode electrode for carbon dioxide electrolyzer' will be used.

3.7 Reactor Design

3.7.1 General

3.7.1.1 The **standard geometric surface area of cathode will be 5cm^2.** Lower activity areas may be used to compare to previous results or to analyze the effects of cathode size, but to allow for uniform comparison among different SELECTCO2 partners and to help with modelling, 5cm^2 will be the default active surface area to be used.

3.7.1.2 The **standard geometric surface area of anode will be 5 cm^2**. While lower sizes may be used to correspond to smaller cathode sizes (to balance pressure issues) or varied to note how variations in anode to cathode size effect performance, 5 cm^2 will be the default value, and will be the size typically used when necessary for modelling experiments.

3.7.1.3 The **standard pressure will be 1 bar.** In some cases this may be varied to investigate its effect, but a 1 bar standard test will always be employed to allow the results to be comparable.

3.7.1.4 In all moderate and high-current density experiments (> 1 mA/cm^2) **the standard measurement technique for gaseous products will be via gas chromatography**. For low current density experiments (<1 mA/cm^2), mass spectrometry may also be considered a standard technique to measure gas products. For liquid products either HPLC, NMR, or static headspace gas chromatography will be considered valid techniques for measuring products.

3.7.1.5 The **standard measurement technique for outlet flow rate of the electrolysis cell will be measured using a bleed line of an inert standard (such as N_2)** (as shown in Figure 2), which acts as an in-line calibrator for the mass flow rate into the gas chromatograph. A calibrated volumetric mass flow metre may also be used as an alternative method if necessary.

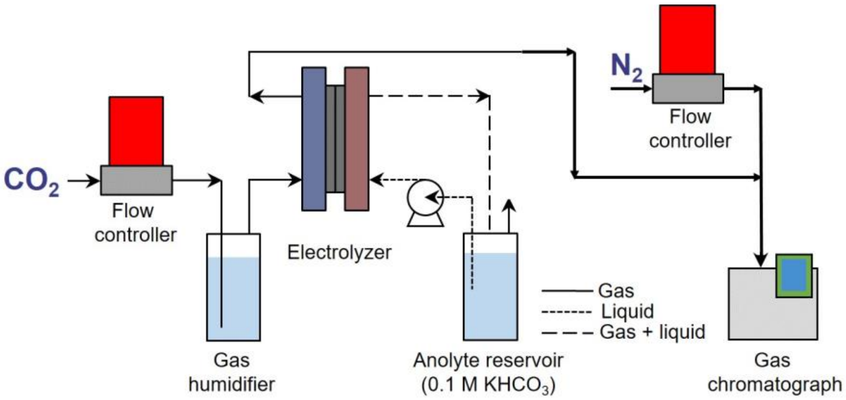

Figure 2. The schematic illustration of the electrolyzer (MEA) for CO_2 reduction. This is a modification of a figure in Ref 1.

3.7.2 Zero gap reactors

This refers to no catholyte layer between cathode and membrane (also known as MEA reactors).

3.7.2.1 The **standard zero-gap CO_2 electrolyzer reactor will be purchased from Dioxide Materials.** This allows for uniform comparison among different SELECTCO2 partners and to help with modelling.

3.7.2.2 The **standard flow fields will be Stainless Steel cathode flow field and Titanium anode flow field from the standard reactors purchased from Dioxide Materials**. This allows for uniform comparison among different SELECTCO2 partners and to help with modelling.

3.7.2.3 The **standard tightening process will tighten the bolts with the torque wrench in a star pattern first at 2 Nm and then at 4 Nm**. This allows for uniform comparison among different SELECTCO2 partners and to help with modelling.

3.7.3 Liquid-gas phase reactors

This refers to a fixed gap between electrodes and membrane.

3.7.3.1 The **standard liquid catholyte layer between the cathode and the ion exchange membranes will be 2 mm thick.** For tests where higher cell efficiencies are desired, we will reduce this thickness to 1 mm using an interchangeable flow channel that can be easily switched in and out. A catholyte chamber with this distance will be produced by SELECTCO2 partner TUD, and distributed to the relevant parties (as part of Milestone 1). This parameter may be varied and optimized as part of the SELECTCO2 project, so it is possible this standard may change in the future. If this is the case, the TCP will be updated accordingly.

3.7.3.2 **If a cell with a catholyte is used it should be denoted whether it is a flow over or flow through design.** A flow over design is where a gas flows over the gas diffusion layer (GDL) and does not penetrate the GDL. A flow through design has the gas penetrate the GDL and leave in the same stream as the catholyte. Since both designs have advantages and it is currently clear which is optimal, both will be employed during this project. Modelling will be done on both approaches and if it is determined (with corresponding experiments) that one approach is clearly optimal, this section will be updated and a standard approach will be set.

3.7.3.3 It will be **standard to press the anode GDE directly onto the membrane.** On rare occasions when it is determined a gap should be used between the GDE and anode, the standard distance will be 2mm.

3.8 Testing

3.8.1 The **standard operating temperature will be 25 °C.** While this is the standard operating temperature, WP3 will often operate at 60-80 °C since these elevated temperature studies are a significant focus of the WP. However when possible room temperature test will also be done to allow comparability to other WP's.

3.8.2 The **standard CO_2 inlet flow will be 50 mL/min.** This parameter will be varied often by both experimentalists and those modeling to help in understanding conversion and optimize performance. While 50 mL/min will be the default value, it is expected different flow rates will be used throughout the project, potentially leading to a new standard (and thus updated TCP).

3.8.3 The **standard relative humidity of the inlet CO_2 will be 100%.** This parameter will be varied often by both experimentalists and those modeling to help in understanding conversion and optimize performance. While 100% relative humidity will be the default value, it is expected different flow rates will be used throughout the project, potentially leading to a new standard (and thus updated TCP).

3.8.4 The **standard anolyte reservoir** will be 100 ml. This parameter may be varied to either dilute or concentrated the effects of the anolyte electrolyte, but 100 ml will act as the standard value.

3.8.5 The **standard test duration will either be 1 hr, 4hr, 24 hr, 100 hr, or 200 hr.** Given that some catalysts/gas diffusion layers/membranes will be more stable than others, it is prudent to have a different standard duration testing. However by narrowing these test durations to a few select lengths, this will allow more comparable data among the SELECTCO2 partners.

3.8.6 **Operating potential, current density and product selectivity will be recorded for all experiments**. For experiments where multiple current densities are tested using the same catalyst, it is also suggested that current density sweeps are produced from 0 mA/cm^2 to the mass transfer limited regime of CO_2 (i.e. where H_2 evolution starts to dominate) at 50 mA/cm^2 intervals. Forward and reverse scans (low current to high current and high current to low current) are suggested as well when perform sequential tests.

For reactors with a fixed gap between electrodes and membrane

3.8.7 The **standard catholyte reservoir** will be 100 ml. This parameter may be varied to either dilute or concentrated the effects of the anolyte electrolyte, but 100 ml will act as the standard value.

3.8.8 For reactors using the flow over approach, the **standard catholyte flow tests will take place at 1 mL/min.** The liquid flow rate may be increased as needed to prevent significant gas from crossing over into the liquid catholyte channel. In addition since this may effect product selectivity, and WP2, WP3, and WP4 all focus on different products, it may be determined in the future that different flowrates are necessary dependent upon what product is desired.

3.8.9 For reactors using the flow through approach, the **standard catholyte flow tests will take place at 100 mL/min.** Since this may effect product selectivity, and WP2, WP3, and WP4 all focus on different products, it may be determined in the future that different flowrates are necessary dependent upon what product is desired.

4 CONCLUSIONS AND FUTURE WORK

In conclusion this report sets out the techniques that will be used to characterize catalysts, gas diffusion layers, and membranes. It also denotes the standard operating procedures and standard materials that will be used as part of the SELECTCO2 project. While these procedures and materials will be modified to improve performance, this report is useful in that it provides a standard set of conditions allows for benchmarking and comparing results between partners as well as providing a starting point for modeling the reactors. Since this is a living document, it will be modified periodically to account for new developments both within the project and from the electrochemical CO_2 reduction field in general.

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6 APPENDIX

None