

Supplementary Information

AMPERE: Automated modular platform for expedited and reproducible electrochemical testing

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S1: System overview

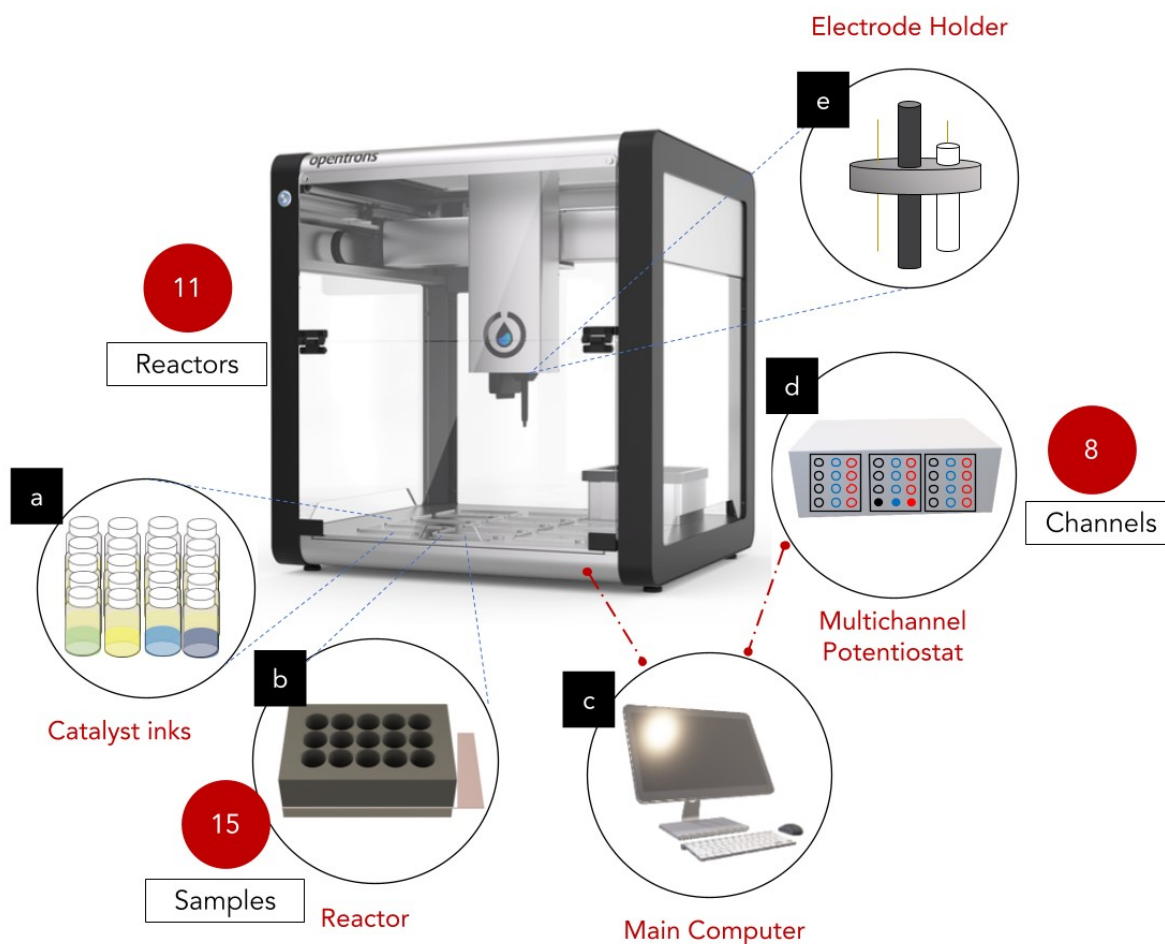


Figure S1. AMPERE system overview. a) catalyst ink vials for drop-casting. b) array reactor, you can fit up to 11 in the Opentrons OT-2. c) master computer interfacing with both the Potentiostat and Opentrons OT-2 through either usb or wireless connection. d) multi-channel potentiostat. e) electrode holder designed to hold both the counter electrode and reference electrodes, as well as a camera probe for imaging.

Table S1. Summary of the electrochemical high throughput platforms used in the literature. CV is cyclic voltammetry, CP is chronopotentiometry, CA is chronoamperometry, ECSA is electrochemical surface area, LSV is linear sweep voltammetry.

Reference	Platform	Speed up method	Current range (scale)	Testing techniques	Time/ experiment [sec]	# Demonstrated experiments	Operation mode
This work	Gantry system + array reactors	Automation Miniaturization Parallelization	mA	CV, LSV, CP, ECSA	870	168	Sequential/ Parallel
Gregoire <i>et al</i> ¹	Scanning droplet cell	Automation Miniaturization	μ A	CV, CA	4	5456	Sequential
Oh <i>et al</i> ²	Gantry system + array reactors	Automation Miniaturization	nA	CV	36	200	Sequential
Geroool <i>et al</i> ³	Array reactors	Miniaturization Parallelization	μ A	CV	20	96	Parallel

S2: Reactor design

Reactor design v1

The base plate of the reactor is CNC milled from a sheet of high-density polyethylene. According to the attached drawings, all the 3.5052mm screw holes (highlighted in red) are tapped to 6-32 UNC. The neodymium magnets are placed in the 4.763mm holes (highlighted in orange) with the north side facing up. The base plate is used to hold the substrate. We have used carbon paper and fluorine doped tin oxide (FTO) coated glass as substrates in this work but virtually any material that can conduct electricity on the surface can be used.

The top piece of the reactor is CNC milled from a sheet of polyether ether ketone (PEEK). The neodymium magnets are placed in the 4.763mm holes (highlighted in orange) with the south side facing down. The reactor is placed on top of the base plate, which is holding the substrate, aligned with the magnets and secured with the bolts. An O-ring (highlighted in green) is used to seal the cells and prevent electrolyte leakage. While strong magnets can be used to hold the reactor on top of the baseplate, we have found that they can sometimes loosen over extended periods, leading to

potential electrolyte leakage. To maintain sufficient compression and prevent leakage, we incorporated bolts into the design. We recommend using both magnets for initial alignment of the cell and bolts to ensure a secure compression.

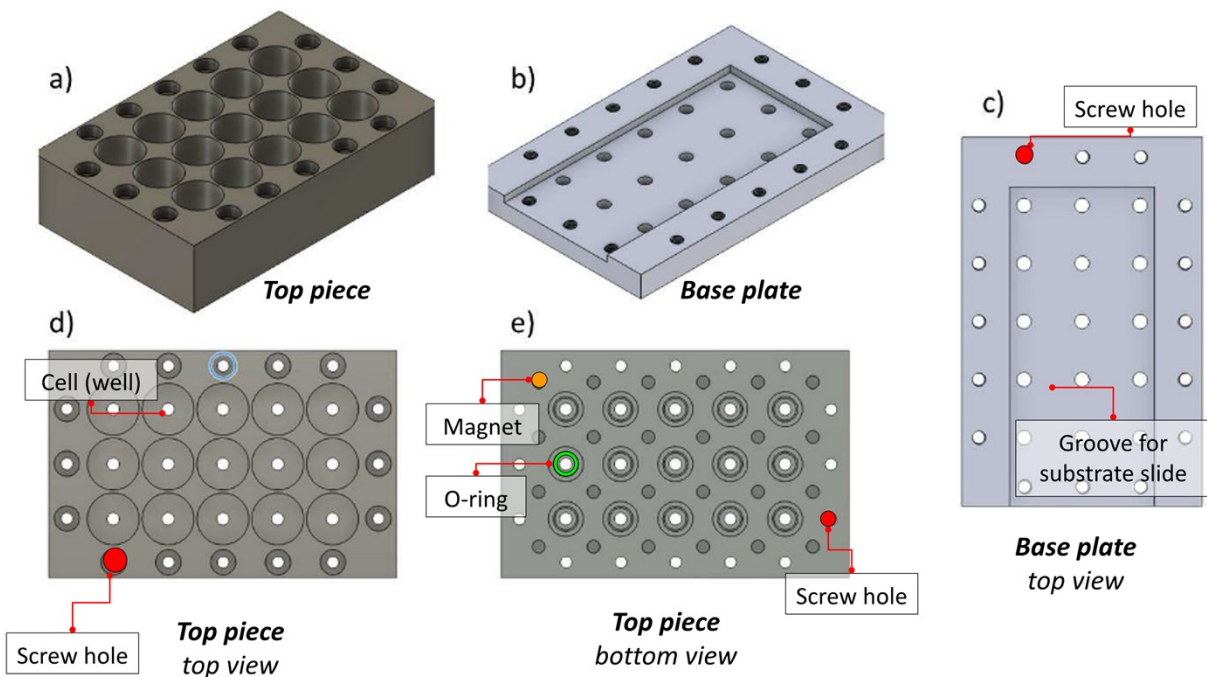


Figure S2. 3D CAD drawings of the array reactor. a) top piece with 15 wells carved in that can hold 5 mL of liquid each. the holes on the outside of the wells are for screws to tightent the structure. b) base plate that can house the FTO slide. holes on the frame of the plate are for screws and the holes in the base are for observing the samples from the bottom. c) top view of the base plate. d) top view of the top piece. e) bottom view of the top piece.

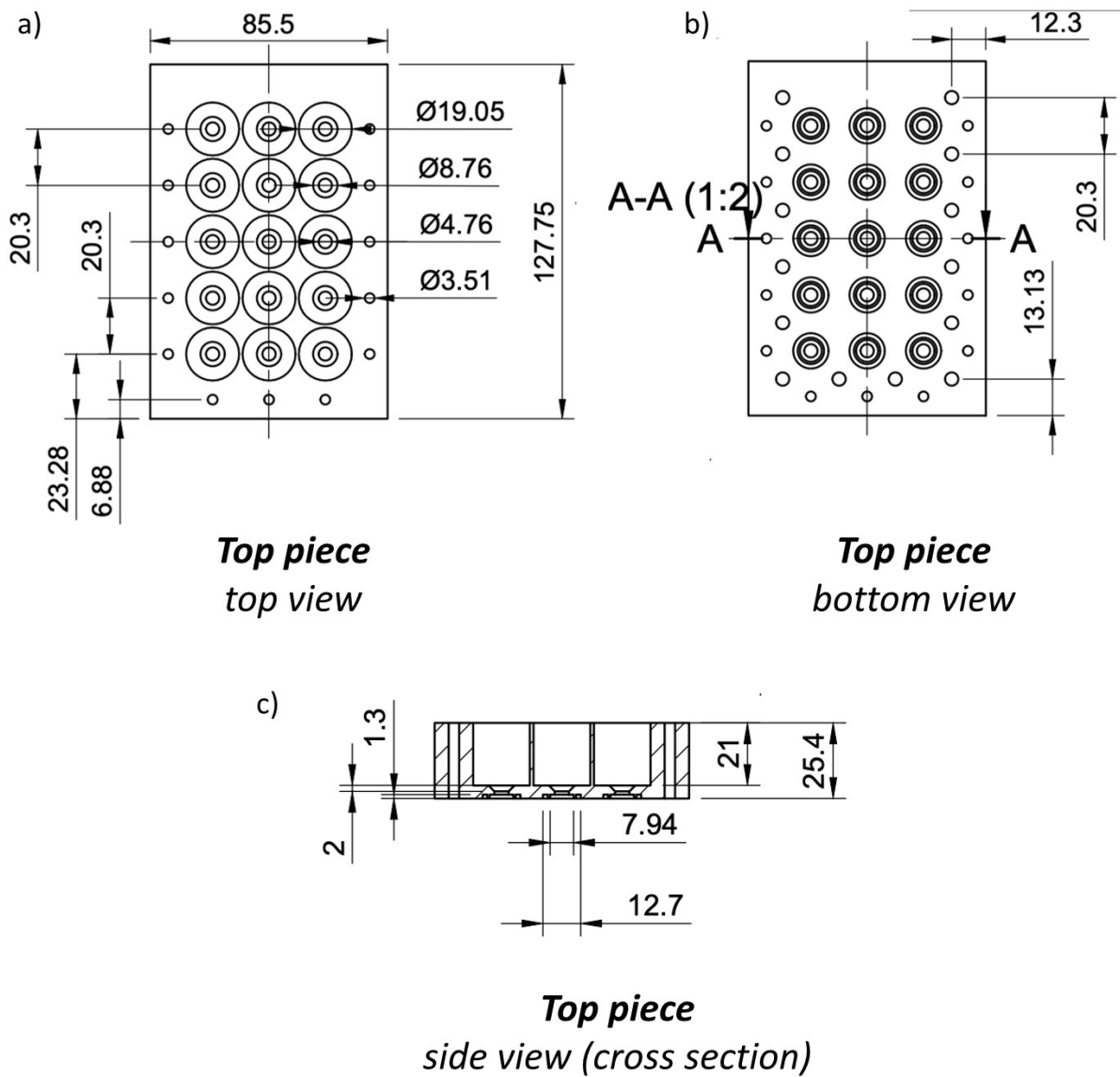
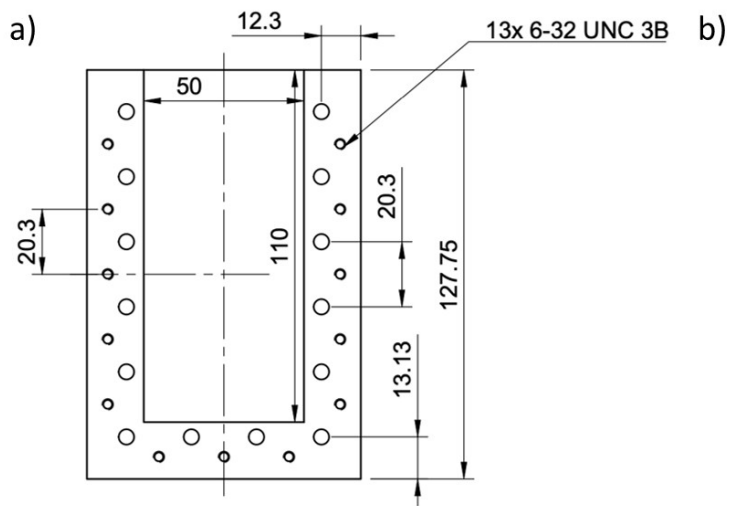
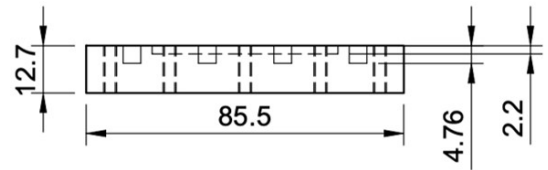


Figure S3. Detailed 2D drawings for the top piece. a) top view, b) back side of the base plate, top view, c) side view. All measurements are in mm.



Base plate
top view

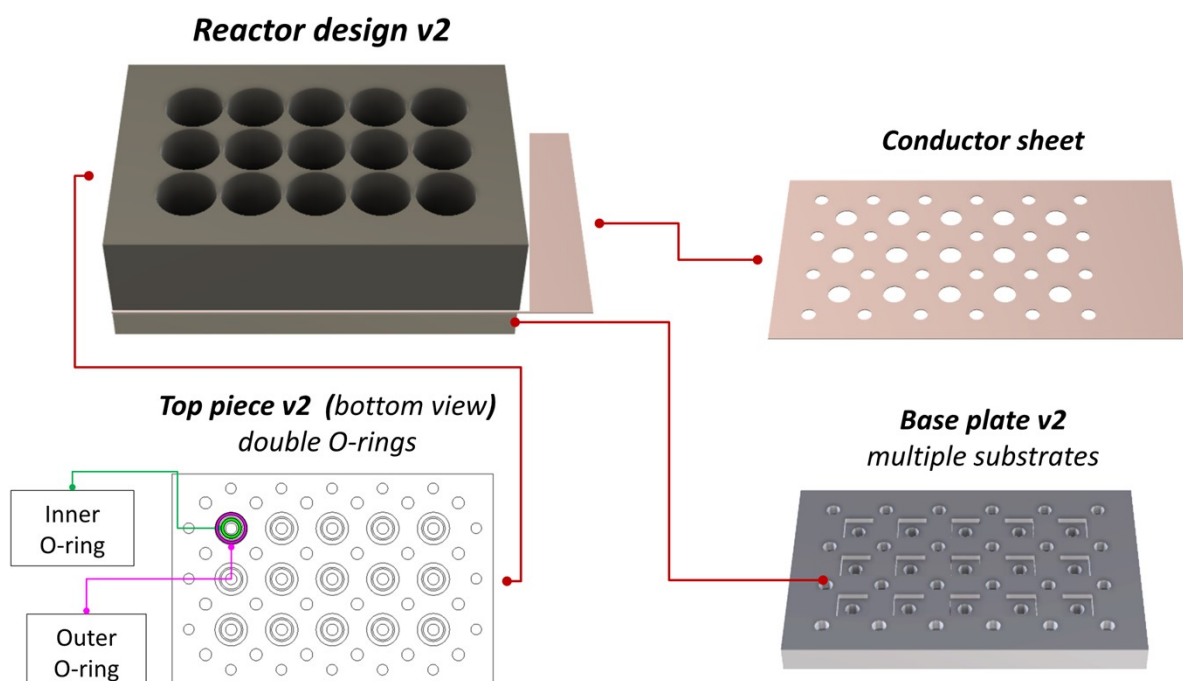


Base plate
side view (cross section)

Figures S4. Detailed 2D drawings for the base plate. a) top view and b) side view. All measurements are in mm.

Reactor design v2

This design allows the use of individual substrates instead of a single substrate slide. A conductor sheet is sandwiched between the top piece and the base plate to enable electron conduction through the top surface of the substrates. In addition to the inner O-ring used in the previous design, an additional outer O-ring (highlighted in purple) is added to push the conductor sheet against the substrate and maintain sufficient compression for electrical conduction.



Figures S5. Alternative design of the reactor that allows the use of multiple individual substrates. A conductor sheet is used to conduct electrons through the top surface of the substrate to the potentiostat.

*Top piece
top view*



*Top piece
bottom view*



Figures S6. Images of the top piece of the alternative reactor design with double O rings. The inner O-ring is used to seal the electrolyte cell and prevent leakage while the second outer O-ring is used to push the conductor sheet for electrical conduction.

Table S2. Equipment and component information

Item	Manufacturer	Use
OT-2 Robot	Opentrons	Liquid handling, both metal oxide ink and electrochemical solution
VSP-3e Potentiostat	Biologic	Perform electrochemical characterization
Ag/AgCl Reference Electrode	CH Instruments	electrochemical characterization
VS20 replacement platinum wire	Sigma-Aldrich	electrochemical characterization
PEEK Sheet, 6" x 6" x 1"	McMaster-Carr	Reactor
HDPE Sheet, 6" x 6" x 1/2"	McMaster-Carr	Baseplate
Neodymium Magnet Magnetized Through Thickness, 3/16" Thick, 3/16" OD	McMaster-Carr	Securing reactor to baseplate
Black-Oxide Alloy Steel Socket Head Screw 6-32 Thread Size, 1-1/8" Long	McMaster-Carr	Securing reactor to baseplate
EPDM O-Ring 1/16 Fractional Width, Dash Number 008	McMaster-Carr	Seal cell in reactor
EPDM O-Ring 1/16 Fractional Width, Dash Number 012	McMaster-Carr	Pushed the conductor sheet against the substarte

Electrode holder design v1

In this work, we made an electrode holder that attaches to the OT-2 pipette head to move between samples during electrochemical testing. We crafted a bracket that securely clasps around one of the OT-2 pipettes using plastic screws, enabling the attachment of electrodes at the holder's end as shown in the figure below. During the experiment, the pipette head remains fully extended and does not retract. The holder is manually installed and removed following the sample preparation for electrochemical testing. This design enables the attachment of multiple electrodes to the bracket's end allowing parallel electrochemical tests.

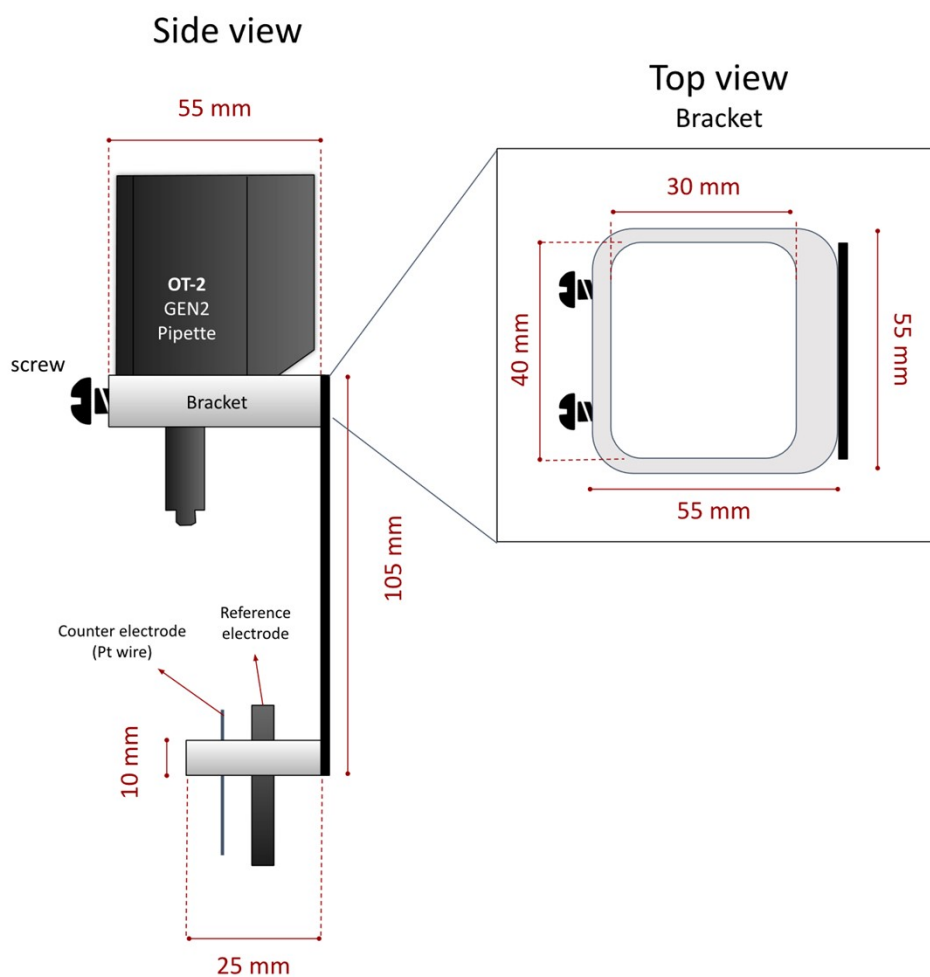
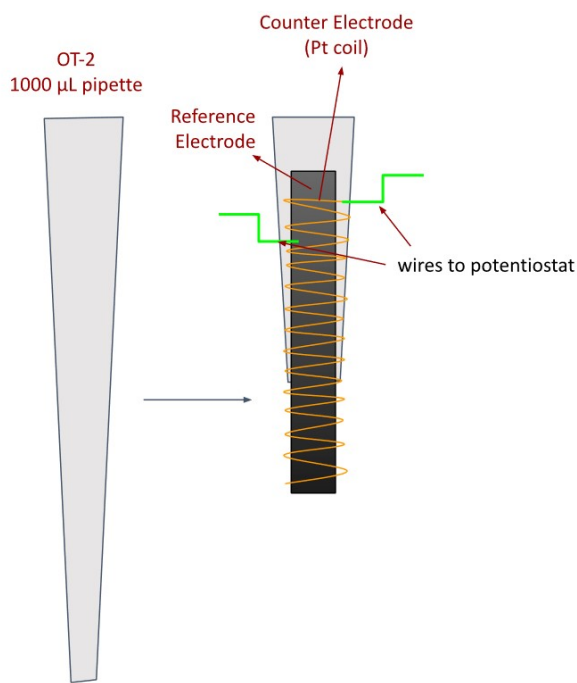


Figure S7. A drawing of a design for an electrode holder that attaches to the Opentrons OT-2 Gen 2 pipettes with dimensions.

Electrode holder design v2

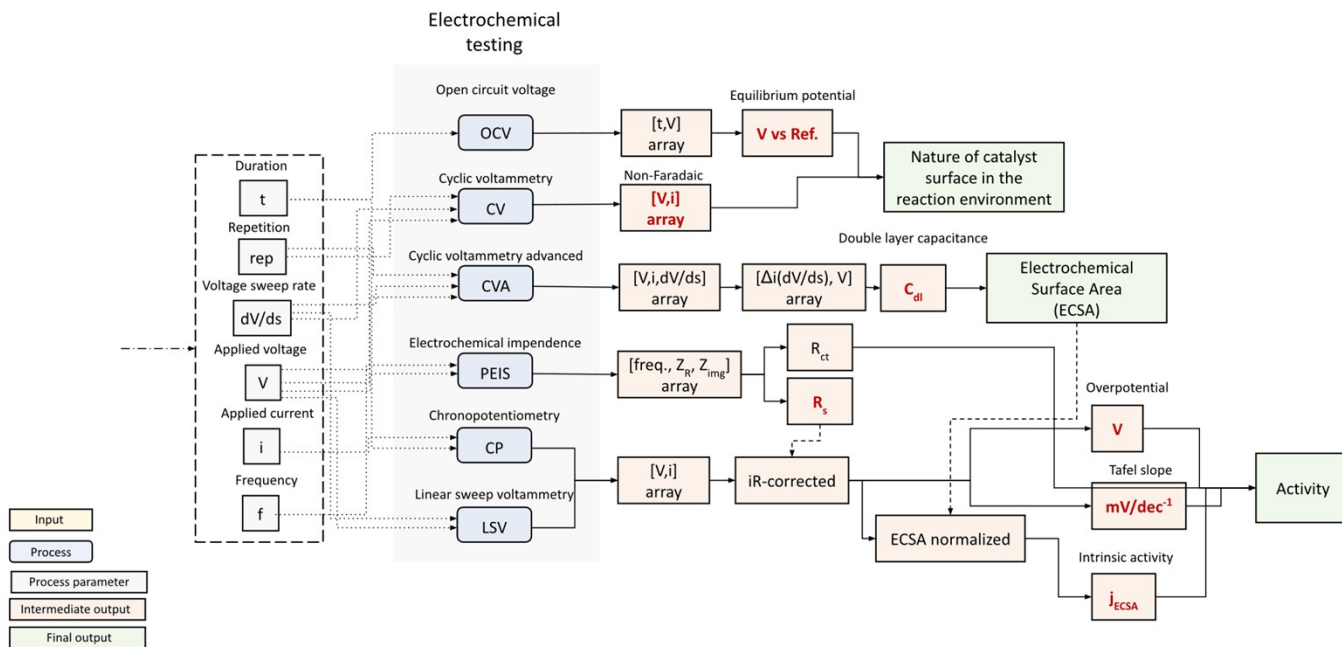
To facilitate automatic handling of the electrode holder and enable seamless transitions between sample preparation and electrochemical testing without manual intervention, we developed an alternative mechanism. In this design, electrodes are carried in a 1000 μL pipette tip instead, as illustrated in the figure below. The tip can be installed in the pipette rack and be picked up automatically by the OT-2 platform when needed like a regular pipette tip. While this mechanism was not employed to generate results in this work, it was successfully tested and proved capable of supporting autonomous experiments and remote operation. Using a multi-pipette head this could be extended to multiple electrode holders operating in parallel and feeding to multi-channel potentiostat to increase throughput of electrochemical testing.



Figures S8. A drawing of an alternative design for an electrode holder that is carried by an Opentrons OT-2 Gen 2 pipette 1000 μL tip with dimensions.

S4: Potentiostat connection

The electrochemical tests are conducted using a Biologic VSP-3e, following a multistep protocol that is implemented on the EC-lab[®] software, as depicted in **Figure S7**.



Figures S9. A block diagram showing the electrochemical testing protocol.

Communication and triggering mechanism

The triggering mechanism employed in this study is illustrated in Figure S8. This represents a straightforward method for setting up a communication protocol between the OT-2, the master computer, and the Biologic potentiostat avoiding the need for extensive coding or complex modifications. It's important to note that there may be other effective methods for establishing this communication that we have not investigated. Additionally, using different equipment, such as substituting the OT-2 with another pipetting or gantry system, or replacing the Biologic potentiostat with a different model, would require alternative communication and triggering protocols. The current protocol functions as follows:

1. Initialization

- EC-Lab® is opened and the following protocol is loaded and ran:

```
Technique : 1  
External Application  
trig_pot.exe  
  
Technique : 2  
Open Circuit Voltage (OCV)  
  
Technique : 3  
Cyclic Voltammetry Advanced (CVA)  
  
Technique : 4  
Potentio Electrochemical Impedance Spectroscopy (PEIS)  
0V  
  
Technique : 5  
Linear Sweep Voltammetry (LSV)  
  
Technique : 6*  
Linear Sweep Voltammetry (LSV)  
  
Technique : 7*  
Chronopotentiometry (CP)  
  
Technique : 8*  
Linear Sweep Voltammetry (LSV)  
  
Technique : 9  
External Application  
end_pot.exe  
  
Technique : 10  
Loop  
Go to technique 1 and repeat n times 15
```

** We employ a three-step process for the accelerated stability test, which includes LSV, CP, and another LSV. For a more detailed explanation of this technique, please refer to Section S5: Electrochemical Testing in our documentation.*

EC-Lab® runs the first technique: “*trig_pot.exe*” (Code block 1). It basically holds proceeding to the next technique before a *trigger* file is sent to the master computer in the <*user dir*>.

- The “*OT_program.ipnyb*” is started on the OT-2 local Jupyter host and ran.
- The OT-2 robot moves to the first sample and pauses. It will wait before moving to the

next sample until it is triggered again.

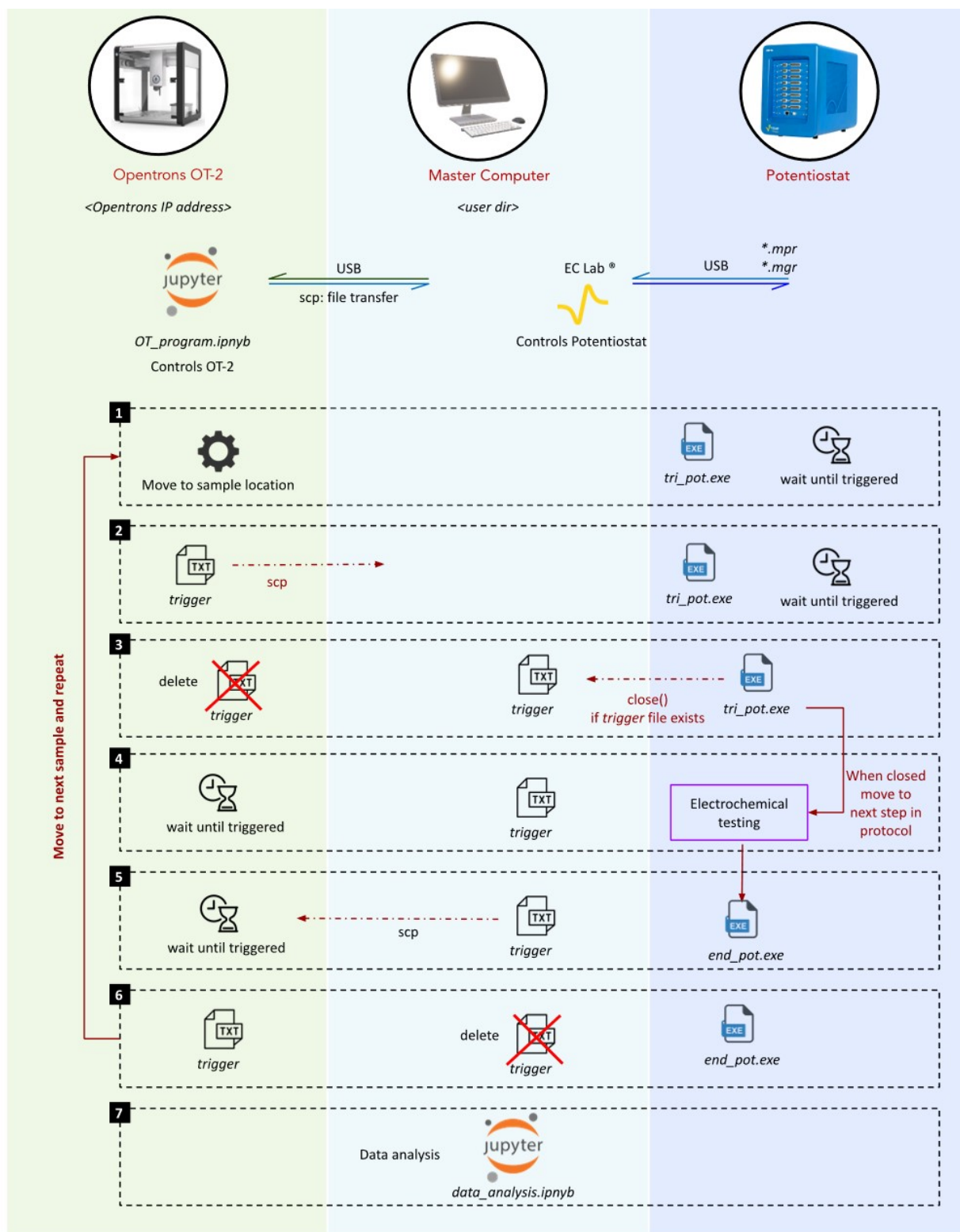
- The “*OT_program.ipnyb*” sends a trigger file from the local Jupyter notebook host to the master computer and then deletes the file locally.
- To learn about how OT-2 ssh files to the master computer check: <https://support.opentrons.com/s/article/Setting-up-SSH-access-to-your-OT-2>

2. Execution of Electrochemical Test:

- Once the *trigger* file is received at the master computer. “*trig_pot.exe*” closes and technique 2 in the protocol is started.
- EC-Lab® goes through technique 2 to 6 to complete the electrochemical test.

3. Final Steps and Looping:

- In technique 7, “*end_pot.exe*” is launched (Code block 2).
- This sends the *trigger* file back to the OT-2 via the master computer and then deletes it from the master computer.
- This triggers the OT-2 to move to the next sample. The process repeats until all samples are processed.



Figures S10. A diagram explaining the triggering mechanism.

Code 1: `trig_pot.exe`

The software is written in python and then converted to an .exe package using PyInstaller:

<https://pyinstaller.org/>

```
# Import necessary libraries
import tkinter as tk
import subprocess
import sys
import os

# Create a tkinter window and a canvas
root = tk.Tk()
canvas1 = tk.Canvas(root, width=300, height=300)
canvas1.pack()

# Define the function to run when the button is clicked
def run(path):

    print("waiting for trigger...")

    # Read the IP address from a text file
    with open(path+"ip_address.txt", "r") as file:
        ot2_ip_address= file.readline()

    # Continuously check for the existence of a file named 'trigger'
    while not os.path.exists("trigger"):
        continue

    # Use scp command to copy trigger file
    subprocess.run(
        ["scp", "-i", <ot2_ssh_key>, ot2_ip_address+":/trigger", path]
    )

    # exist the program
    sys.exit()

# Call the run() function
run(<user dir>)

# Start the tkinter event loop
root.mainloop()
```

Code 2: end_pot.exe

The software is written in python and then converted to an .exe package using PyInstaller:

<https://pyinstaller.org/>

```
# Import necessary libraries
import tkinter as tk
import subprocess
import sys
import os

# Create a tkinter window and a canvas
root = tk.Tk()
canvas1 = tk.Canvas(root, width=300, height=300)
canvas1.pack()

# Define the function to run when the button is clicked
def run(path):

    print("moving to next measurement...")

    # Read the IP address from a text file
    with open(path+"ip_address.txt", "r") as file:
        ot2_ip_address= file.readline()

    # Use scp command to copy trigger file
    subprocess.run(
        ["scp", "-i", <ot2_ssh_key>, path+"trigger", ot2_ip_address+": /"]
    )

    # If the 'trigger' file is found, remove it and exit the program
    if os.path.exists(path+'trigger'):
        os.remove(path+'trigger')
        sys.exit()

# Call the run() function
run(<user dir>)

# Start the tkinter event loop
root.mainloop()
```

S5: Electrochemical testing

Electrode washing protocol

Our electrodes were washed between experiments by immersing them repeatedly in a nitric acid solution followed by DI water, reducing residual metal contaminants. To assess the effectiveness of our cleaning protocol, we carried out ICP measurements on the electrolyte solutions after the reactions. The results demonstrated minimal traces of contaminants, with Ru detected at 5.7 ppb [± 5.031 ppb] in solutions used with Ir samples, and Ir at 9.2 ppb [± 10.342 ppb] in solutions used with Ru samples. These levels are near the detection limits of the equipment, which are 3 ppb for Ru and 10 ppb for Iridium. Despite these minimal traces, we advise replacing electrodes when switching materials or after a designated number of uses to maintain the highest levels of safety and precision.

Accelerated electrochemical stability test

We use an accelerated stability test protocol to evaluate the electrochemical stability of catalysts in oxygen evolution reaction (OER)⁴. The protocol starts by ramping the voltage from OCV to 1.55 V (vs RHE) at a scan rate of 20 mV/s, holding current for 5 minutes, followed by a reverse voltage sweep from 1.55 V (vs RHE) back to OCV. We measured Ir/Ru metal dissolution at the end of each experiment and used that to evaluate the stability of the catalyst.

Parallel testing

To parallelize testing and ensure independent electrochemical measurements, we recommend not using a single working electrode across different experiments at the same time. Instead, our modular design of the setup allows for each reactor (comprising 15 electrochemical cells) to have its own dedicated potentiostat channel. With this setup, the number of parallel experiments corresponds to the number of reactors that can be loaded into the robot. Although the robot can accommodate 11 independent reactors, we are limited by the number of available potentiostat channels, which is eight in this work. Therefore, in this configuration, the maximum parallelization achievable per AMPERE device is eight, which makes acceleration in general constrained by the lesser of either the number of available potentiostat channels or the number of reactors that can be accommodated.

In cases where acceleration is needed for a smaller number of independent experiments (e.g., fewer than 15 experiments which would only require one reactor) and each measurement still taking a

significant amount of time, we suggest using our second reactor design (described in S2: Reactor design) for parallelization. This design features isolated conductors for each cell, row, or column, enabling independent connections and testing. For example, by connecting each column (which includes three samples) to one potentiostat channel, we can effectively use five channels at once. We can now conduct five parallel measurements (covering all five samples in a single row), thereby speeding up the process by a factor of five.

It is important to note that we have not tested parallel experiments in either configuration in our current work. However, given that each experiment in the configuration is designed to be independent and isolated, we anticipate that this setup should function as intended.

S6: Materials

The received commercial Ruthenium Black was found to be fully oxidized under XRD testing. Therefore, in our experiments, we treated the commercial Ruthenium Black as a RuO₂ sample, and the commercial Ruthenium Black subjected to a three-hour annealing at 300 °C under forming gas as a Ru sample. The XRD patterns of commercial Ruthenium Black before and after annealing are depicted in Fig. S7.

Table S3. materials information

Material name	Product name/Manufacturer	Post-treatment
RuO ₂	Ruthenium Black, High Surface Area (57080273) / FuelCell Store	NA
Ru	Ruthenium Black, High Surface Area (57080273) / FuelCell Store	Annealing 3 hours at 300 °C under forming gas (H ₂ :Ar =5:95) to produce Ru
IrO ₂	Iridium Oxide, High Surface Area (11080001) / FuelCell	NA
Ir	Iridium, High Surface Area (57080249) / FuelCell	NA

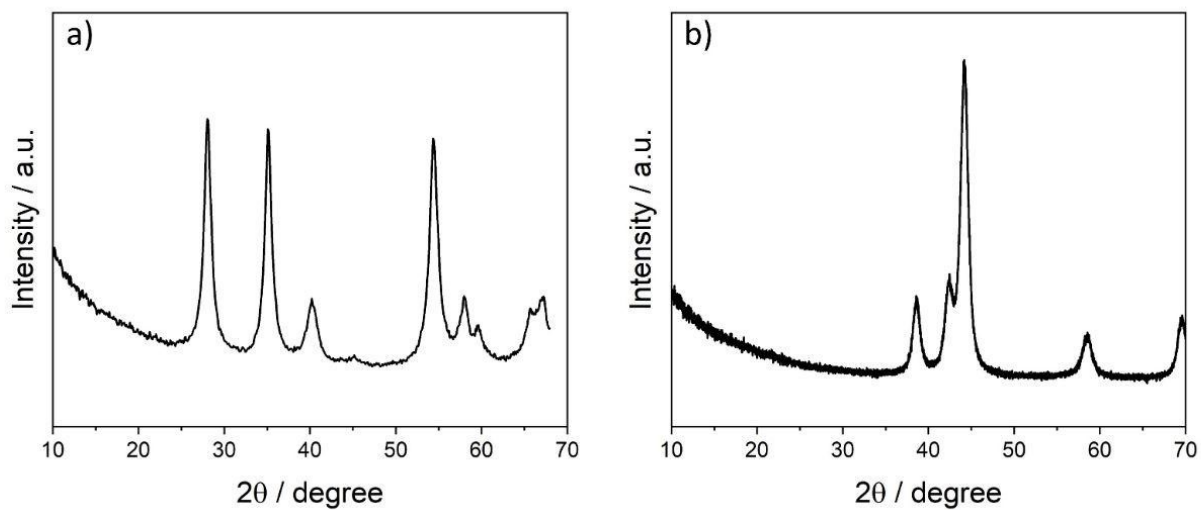


Figure S11. XRD pattern of commercial Ruthenium Black a) before and b) after Annealing 3 hours at 300°C under forming gas ($H_2:Ar = 5: 95$).

S7: Powder weighing and ink preparation

Powder weighing

All solid powders are weighed and dispersed using Chemspeed's gravimetric dispensing powder module. Catalyst powders are placed into separate powder container. The powders are then dispensed in tubes according to the target dosage, preparing for the subsequent catalyst ink solution preparation in Opentrons OT-2.



Figure S12. Image of the powder dispensing module within the Chemspeed system.

Catalyst ink preparation

Catalyst inks are prepared in 3 mL vials. A 5 cm x 10 cm plate was made to hold 30 liquid vials. The vials, which have pre-weighed powders using Chemspeed's powder dispenser (check powder weighing section), are taken to the Opentrons OT-2 platform for catalyst ink preparation. OT-2 uses the 1000 μ L pipettes to add organic solvents and water to each of the vials. Then, it picks up 20 μ L pipette to carefully transfer precise amounts of Nafion to the vials. Afterwards, the plate is transferred to a water bath and sonicated at room temperature for 30 minutes to homogeneously disperse the catalyst ink mix. Then, the plate is returned back to OT-2 for dropcasting. Using a fresh 20 μ L pipetting tip, a specific amount of the ink is aspirated from the vials and deposited in designated reactor wells. To prevent the spread of the ink beyond the desired dropcasted area, we employ O-rings to tightly compress the substrate. This method ensures that the ink remains

confined within the O-ring boundary limiting its spread both laterally and through the thickness of the substrate. The reactor is then moved to an oven to dry the dropcasted films at for 2 hours at 70 °C. All pipette tips are disposed and replaced between each of the runs to avoid cross contamination.

S8: Metal dissolution measurements

Inductively coupled plasma optical emission spectrometry (ICP-OES) analysis was conducted using an iCAP PRO XP, paired with an iSC-65 Autosampler to facilitate sequential automated measurements. Calibration of the instrument was performed using Ru/Ir standards (SPC Science, 1000 ppm) diluted in an aqueous solution to concentrations of 0.01, 1, and 50 ppm. The sample solutions were measured in their original electrolyte state following an electrocatalytic reaction.

S9: Experimental results

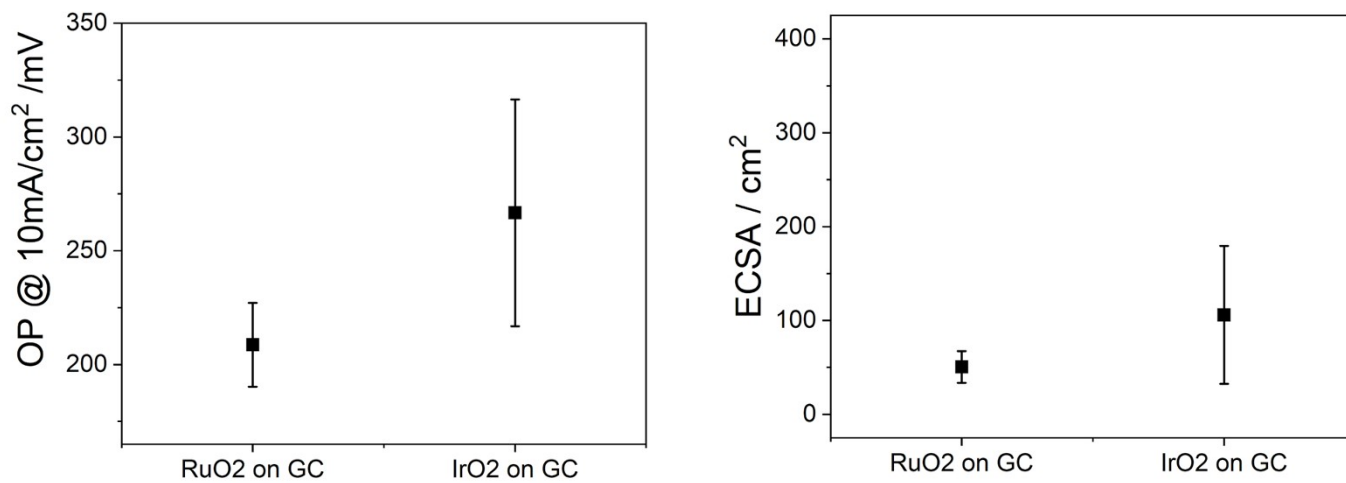


Figure S13. Measured overpotential (left) and ECSA (right) at 10 mA.cm⁻² for RuO₂ and IrO₂ samples deposited on glassy carbon (GC).

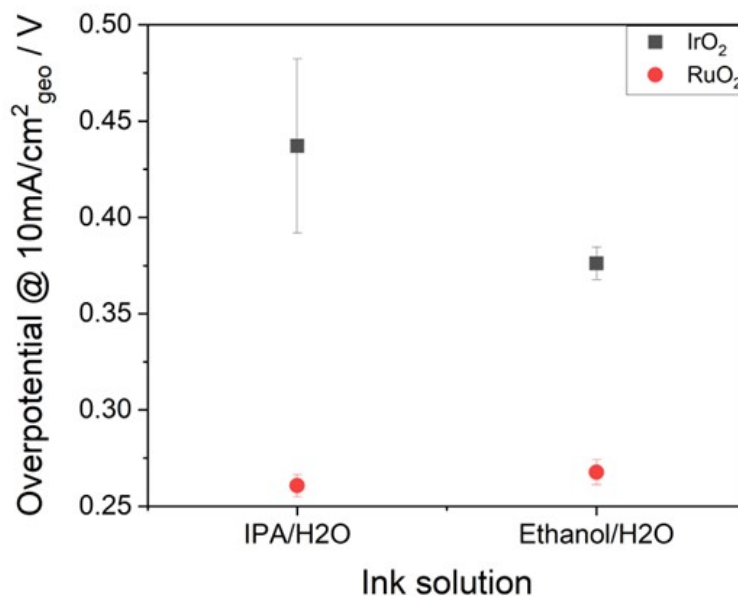


Figure S14. Measured overpotential at 10 mA.cm⁻² for RuO₂ and IrO₂ samples deposited FTO using IPA vs Ethanol as an organic solvent.

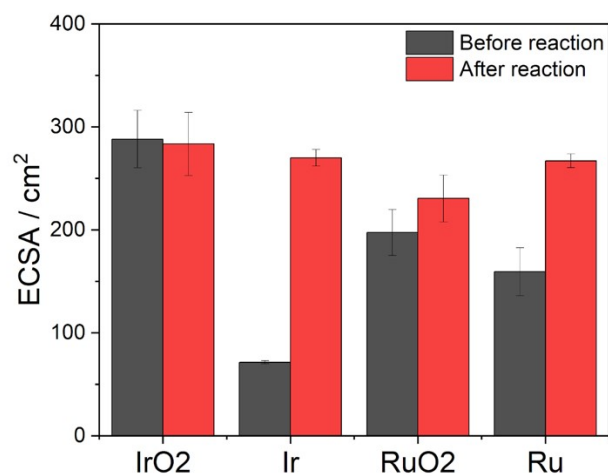


Figure S15. ECSA measurements for IrO₂, Ir, RuO₂, and Ru before and after activity and stability tests under OER.

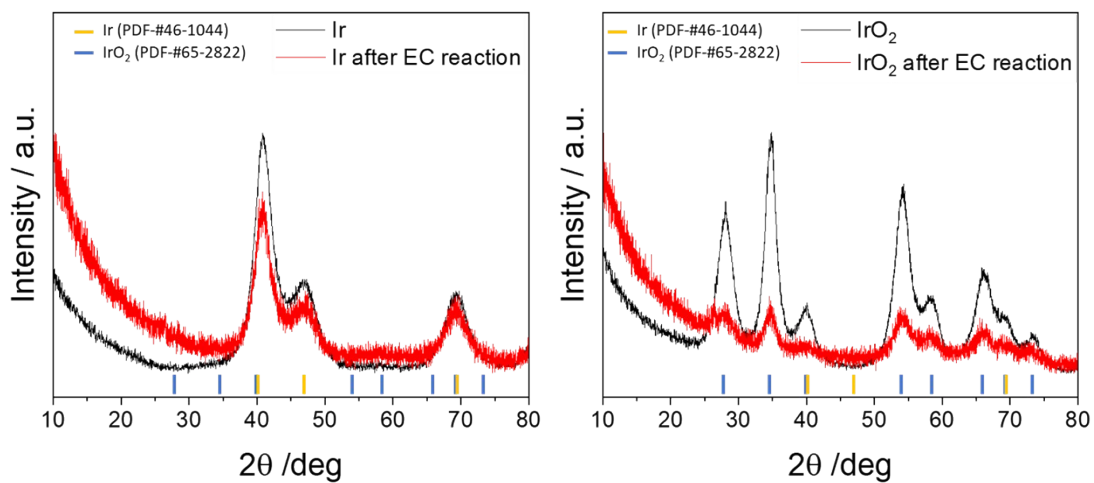


Figure S16. XRD patterns for Ir (left) and IrO₂ (right) before and after activity and stability tests under OER.

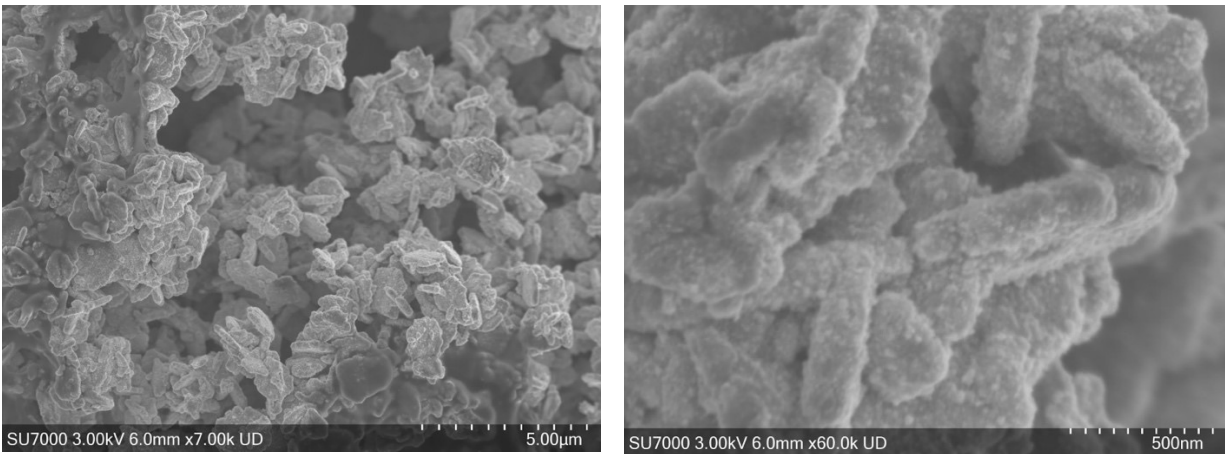


Figure S17. SEM images of Ir before activity and stability tests under OER.

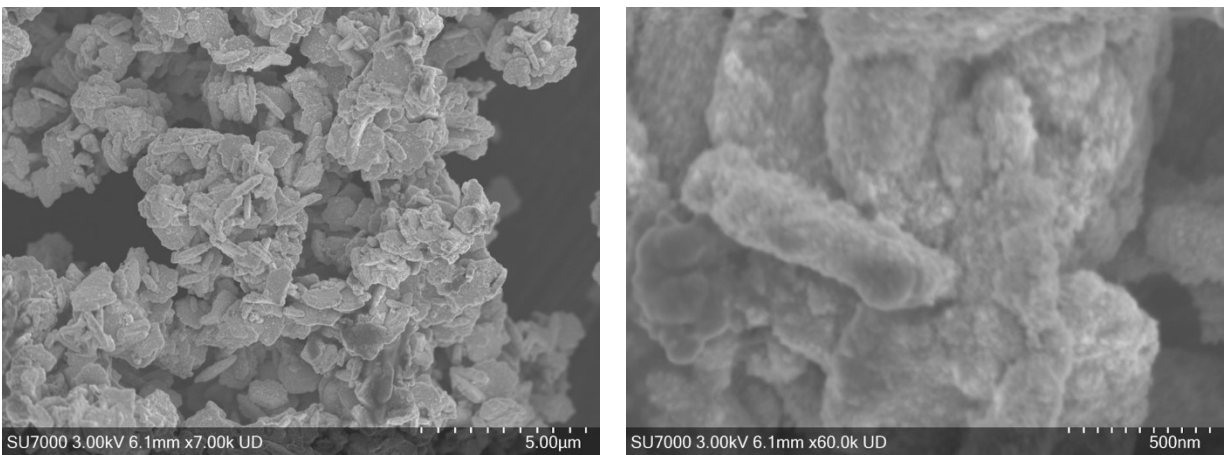


Figure S18. SEM images of Ir after activity and stability tests under OER.

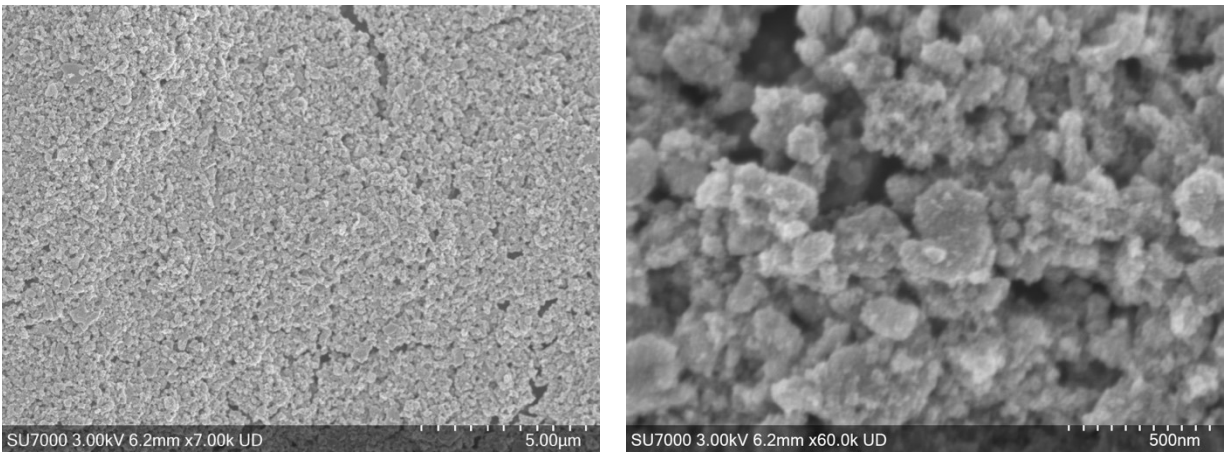


Figure S19. SEM images of IrO₂ before activity and stability tests under OER.

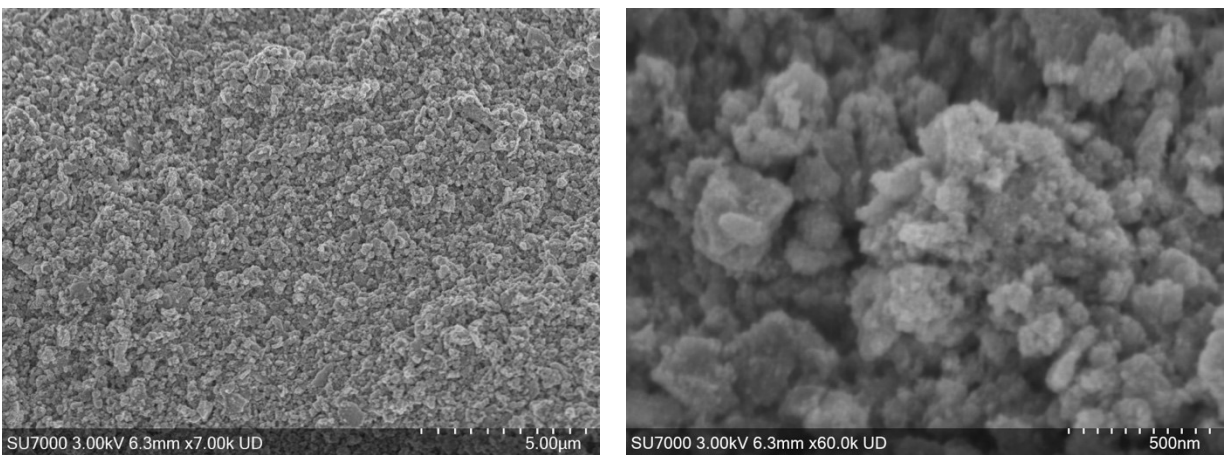


Figure S20. SEM images of IrO₂ after activity and stability tests under OER.

- 1 J. M. Gregoire, C. Xiang, X. Liu, M. Marcin and J. Jin, *Review of Scientific Instruments*, DOI:10.1063/1.4790419.
- 2 I. Oh, M. A. Pence, N. G. Lukhanin, O. Rodríguez, C. M. Schroeder and J. Rodríguez-López, *Device*, 2023, **1**, 100103.
- 3 B. H. R. Gerroll, K. M. Kulesa, C. A. Ault and L. A. Baker, *ACS Measurement Science Au*, 2023, **3**, 371–379.
- 4 J. Edgington, A. Deberghes and L. C. Seitz, *ACS Appl Energy Mater*, 2022, **5**, 12206–12218.