Electrochemical Protocols

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1. Platinum Electrodeposition
2. PEDOT:pTS Electrodeposition
3. Iridium Oxide Electrodeposition

**Platinum Electrodeposition**

Materials Needed:

* Deionized Water
  + - *Any supplier*
* Hydrochloric acid - HCl (37%)
  + - *HYDROCHLORIC ACID 37 % ANALAR NP REAG.PE - Analytical Grade*
* Ethanol (100%) or Isopropyl Alcohol
  + - *Any supplier*
* Chloroplatinic Acid 8% in H2O
  + - *262587 - Chloroplatinic acid solution from Sigma*
* Platinum wire
  + - *Any supplier*
* Potentiostat
  + - *Can be any model! But the model used in for these protocols was the PalmSens4*

Making the plating solution:

* Simply mix **1:1 8% chloroplatinic acid with deionized water** to make a 4% solution for electroplating
* Store the chloroplatinic acid solution in a sealed container as it is highly toxic

Electrodeposition Protocol:

* Pour **~100 mL of deionized water** and **ethanol** into containers
* Pipette **~10 mL of HCl** into a small glass beaker
* Pipette **~25 mL of chloroplatinic acid** into the deposition area
* Submerge the electrodes into ethanol and then put directly into the HCl
  + Note: the ethanol significantly increases the wettability of the small electrodes and thus is necessary for the desired etching of the electrodes with HCl
* Hold the electrodes in **HCl for ~30 seconds**
* Transfer the electrodes from the HCl to deionized water and rinse
* Transfer the electrodes to the chloroplatinic acid in the plating bath
* Once submerged and held in place, attach the working electrode clip from the potentiostat to the electrodes that will be plated on
* Submerge an addition piece of solid platinum to the plating bath and connect to the counter electrode clip
  + Note: in order to prevent hydrogen formation on the counter electrode the surface area of this platinum wire/piece must be approximately equal to the surface area of the electrodes you are plating onto
* Finally submerge the tip of the silver/silver chloride reference electrode into the bath and connect that to the reference clip
* Set the potentiostat to run in mixed mode with **4 cycles of 60 seconds** each cycle
* The current should be set to alternating positive and negative currents equal to **0.215 nA/um2** (multiply this value by the total area to be plated to get the final current to apply)
* After plating the electrodes should be disconnected from the potentiostat and rinsed in deionized water
  + Note: For ideal long-term coating stability it is necessary to store in deionized water for **24 hours** before drying completely
* If the deposition is successful, the electrodes should have significantly darkened in color to almost black grey color

**PEDOT:pTS Electrodeposition**

Materials Needed:

* Deionized Water
  + - *Any supplier*
* Hydrochloric acid - HCl (37%)
  + - *HYDROCHLORIC ACID 37 % ANALAR NP REAG.PE - Analytical Grade*
* Ethanol (100%) or Isopropyl Alcohol
  + - *Any supplier*
* PEDOT:pTS electroplating solution
  + Acetonitrile
    - *ACETONITRILE EMPLURA*
  + p-Toluenesulfonic acid monohydrate
    - *402885 - p-Toluenesulfonic acid monohydrate from Sigma*
  + EDOT monomer (3,4-Ethylenedioxythiophene)
    - *483028 - 3,4-Ethylenedioxythiophene from Sigma*
* Platinum wire
  + - *Any supplier*
* Potentiostat
  + - *Can be any model! But the model used in for these protocols was the PalmSens4*

Making the plating solution:

* Mix **25 mL of deionized water** with **25 mL of acetonitrile** in a glass beaker
* Stir in **430.5 mg pTS** (can be purchased from Sigma)
* Once the pTS has dissolved into the water/acetonitrile solvent, pipette in **534 uL of EDOT** solution
  + Note: the EDOT will form many globules at first, this is normal because it is not highly miscible in aqueous solutions.
* Stir the solution until the EDOT is fully mixed
  + Note: the solution will appear clear
* Store the PEDOT:pTS solution in a sealed container as it is highly toxic

Electrodeposition Protocol:

* Pour **~100 mL of deionized water** and **ethanol** into containers
* Pipette **~10 mL of HCl** into a small glass beaker
* Pipette **~25 mL of PEDOT:pTS plating solution** into the deposition area
* Submerge the electrodes into ethanol and then put directly into the HCl
  + Note: the ethanol significantly increases the wettability of the small electrodes and thus is necessary for the desired etching of the electrodes with HCl
* Hold the electrodes in **HCl for ~30 seconds**
* Transfer the electrodes from the HCl to deionized water and rinse
* Transfer the electrodes to the PEDOT:pTS plating solution in the plating bath
* Once submerged and held in place, attach the working electrode clip from the potentiostat to the electrodes that will be plated on
* Submerge an addition piece of solid platinum to the plating bath and connect to the counter electrode clip
  + Note: in order to prevent hydrogen formation on the counter electrode the surface area of this platinum wire/piece must be approximately equal to the surface area of the electrodes you are plating onto
* Finally submerge the tip of the silver/silver chloride reference electrode into the bath and connect that to the reference clip
* Set the potentiostat to run in chronopotentiometry mode for **450 seconds at 2 mA/cm2** (multiply this value by the total area to be plated to get the final current to apply)
* After plating the electrodes should be disconnected from the potentiostat and rinsed in deionized water
* If the deposition is successful, the electrodes should have significantly darkened in color to very deep blue/purple color

**Iridium Oxide Electrodeposition**

Materials Needed:

* Deionized Water
  + - *Any supplier*
* Hydrochloric acid - HCl (37%)
  + - *HYDROCHLORIC ACID 37 % ANALAR NP REAG.PE - Analytical Grade*
* Ethanol (100%) or Isopropyl Alcohol
  + - *Any supplier*
* Iridium oxide electroplating solution
  + IrCl4 
    - *Iridium(IV) chloride hydrate – 500 mg from Insight Biotechnology*
  + 30% Aqueous Hydrogen Peroxide
    - *H1009 - Hydrogen peroxide solution from Sigma*
  + Oxalic Acid
    - *241172 - Oxalic acid from Sigma*
  + Potassium Bicarbonate
    - *P9144 - Potassium bicarbonate from Sigma*
  + Wide range litmus paper
    - *Any supplier*
* Platinum wire
  + - *Any supplier*
* Potentiostat
  + - *Can be any model! But the model used in for these protocols was the PalmSens4*

Making the plating solution:

* Mix **50 mL of deionized water** to a glass beaker
* Dissolve **70 mg of IrCl4** into the water for **30 minutes**
  + Note: mild heat helps with the dissolving
* After the 30 minutes add **0.5 mL of 30% aqueous hydrogen peroxide (H2O2)** and stir for **10 additional minutes**
* Next add **250 mg of oxalic acid** to the solution and stir for another **10 minutes**
* Next ***slowly*** adjust the pH of the solution to **10.5** by stirring in **potassium bicarbonate** to the solution
* Confirm the pH with a test paper
  + Note: solution should be a yellowish-brown color
* Solution must be left covered at room temperature for **3 to 4 days to stabilize** at which time it will have changed to **deep purple** in color
* Store the iridium oxide solution in a sealed container as it is basic and will react explosively with acids

Protocol:

* Pour **~100 mL of deionized water** and **ethanol** into containers
* Pipette **~10 mL of HCl** into a small glass beaker
* Pipette **~25 mL of iridium oxide plating solution** into the deposition area
* Submerge the electrodes into ethanol and then put directly into the HCl
  + Note: the ethanol significantly increases the wettability of the small electrodes and thus is necessary for the desired etching of the electrodes with HCl
* Hold the electrodes in **HCl for ~30 seconds**
* Transfer the electrodes from the HCl to deionized water and rinse
* Transfer the electrodes to the iridium oxide plating solution in the plating bath
* Once submerged and held in place, attach the working electrode clip from the potentiostat to the electrodes that will be plated on
* Submerge an addition piece of solid platinum to the plating bath and connect to the counter electrode clip
  + Note: in order to prevent hydrogen formation on the counter electrode the surface area of this platinum wire/piece must be approximately equal to the surface area of the electrodes you are plating onto
* Finally submerge the tip of the silver/silver chloride reference electrode into the bath and connect that to the reference clip
* Set the potentiostat to run in cyclic voltammetry mode with a range from **-0.8 V to +0.7 V** and a **rate of 100 mV/s for 200 cycles**
* Repeat this step twice more for a total of **600 deposition cycles**
* After plating the electrodes should be disconnected from the potentiostat and rinsed in deionized water
  + Note: For ideal long-term coating stability it is necessary to store in deionized water for **24 hours** before drying completely
* If the deposition is successful, the electrodes should have significantly darkened in color to almost black blueish color