Iridium grows a hydrous oxide layer on its surface when it is electrochemically activated in an electrolyte. The oxide is porous and can assume several oxidation states which gives it a very high charge capacity as well as the ability to deliver large amounts of charge for stimulation applications [1, 2, 3]. The porous nature of the oxide also increases the effective surface area of the electrode site resulting in a decrease in impedance, a desirable characteristic for some recording applications [2, 4]. This material is also exceptionally resistant to dissolution and corrosion during in vitro and in vivo stimulation [5].

The process by which iridium is electrochemically oxidized is known as activation. Because of reliability and ease of application, the activation method used with our probes and discussed here is cyclic voltammetry. A standard three electrode scheme is used to perform cyclic voltammetry (Figure 1). The voltage waveform is applied to a counter electrode (usually a large platinum electrode) in the electrolyte. The iridium site is the working electrode and provides a return current path. A saturated calomel electrode (SCE) provides a reference (Fisher Scientific, catalog # 13-620-52). The voltage on the counter electrode is cycled between anodic and cathodic potentials while the site is exposed to an electrolyte.

![Figure 1: Three electrode configuration for cyclic voltammetry and activation](image)

The process of activation has several variables that can be changed to achieve iridium oxide that maintains consistent electrical characteristics. We have found that a high-quality oxide is formed when the following activation procedure is used:

1. Use an electrolyte of 0.3 M Na2HPO4.

2. (Optional) Initially hold sites at potentials of -3.0 V and 2.5 V for approximately three minutes each to remove any oxide that has formed, essentially cleaning the metal.

3. Cycle the activation potential between -0.85 and 0.75 Volts. These limits are usually wide enough to grow an oxide but narrow enough to remain within the water window. If you choose to vary these limits, as a rule the voltage limits should be set approximately 100 mV inside of the water window (as determined by the high current peaks on the CV).
4. Use a square wave (0.5 - 1 Hz) to activate. This allows the metal to remain at the critical levels for hydrous oxide formation (0.75 V) and inner oxide reduction (-0.85 V) for a longer time than a ramp wave would. Holding the potential at these levels will allow a more complete oxide formation and reduction and also reduce the number of potential cycles needed to grow the oxide.

5. Activate to a limit of no more than 30 mC/cm² as determined by cyclic voltammetry (the area under the CV curve). This measurement and calculation should be made every 50-100 activation cycles in order to determine when the desired charge capacity is reached.

References