Best practices for OER experiments

EC-MS Technical Note #16

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Introduction

The ***EC-MS Application Note #4*** gives a thorough introduction on the benefits of using the EC-MS for OER studies. In this document, we list some best practices recommended to obtain the best possible results with your Spectro Inlets EC-MS.

Experiment preparation

- For any materials investigated it is crucial for the user to ensure that the tested material can sustain the electrochemical environment, *e.g.* the catalyst material and/or support may dissolve under OER conditions. Note, catalyst oxidation (which may also change conductivity) is included in the total measured current by the potentiostat, while the MS is insensitive to such effects. We recommend EC-MS users to confer with *Pourbaix* diagrams [1] for all the elemental constituents of the investigated materials.
- Prior any experiment it is recommend for users to clean the EC-MS cell according to ***Cleaning Procedures EC-MS Technical Note #12***. Always store electrolytes in clean glassware/PFA bottles and refrain from using alkaline electrolytes more than a day old, as CO_2 uptake from the ambient air may change electrochemical responses.
- Characterization of the catalyst is recommended to be done both prior and after any prolonged electrochemical experiment. This can be done most easily electrochemically (*e.g.* by CO-stripping [2], see ***CO-stripping Technique EC-MS Application Note #1***), or from the double-layer capacitance. For the latter, we encourage users to record initial and final *pseudo* stable CVs (at varying scan rates) as such EC-MS data provide essential information of change in activity and relative catalyst area.

EC-MS specific considerations

- Use standard chips in acid and neutral conditions, and non-aqueous chips in alkaline electrolyte (do not use highly concentrated alkaline electrolyte or HF).
- Note, for alkaline OER using carbon-based catalyst/supports, any CO_2 formed will react with the electrolyte producing (bi)carbonates, which vapour pressure is too high for detection by the EC-MS. Hence, full quantification by O_2 calibration is recommended for such studies.
- Match scan rate and bandwidth filter ensuring minimum feedback waves in the CV is observed, for more info on this important issue see ***Potentiostat instability** EC-MS Technical Note #3*.
- Use CVs to identify redox processes onset/offset potentials and see if insights match *Poubaix* diagrams [1] of catalyst/support constituents. This provides the user with insights on material transitions (and stability). CVs taken at various scan rates can be used for area evaluation. Note, repeated oxidation/reduction of the catalyst may roughen the catalyst or completely detach/dissolve electrode materials.
- For quantitative EC-MS evaluation of OER performance (see ***EC-MS quantification** EC-MS Application Note #2*) conducting potentiostatic measurements (with evaluation of series resistance) is recommended, as doing so eases and improve accuracy of the charge integration. Repeated redox events from cycling/stepping may increase dissolution. We recommend stepping potentials using (using the EC-lab ”SPEIS” technique). This approach renders, unless too large currents are accessed (< 0.3 mA) or too poorly conducting electrolyte is used, reproducible results. We rely on post-correction for the IR-compensation of the potential, consequently reproducing exact potential set-points is difficult.

References

[1] K. A. Persson, B. Waldwick, P. Lazic, and G. Ceder, “Prediction of solid-aqueous equilibria: Scheme to combine first-principles calculations of solids with experimental aqueous states,” *Phys. Rev. B*, vol. 85, p. 235 438, 23 Jun. 2012. DOI: `10.1103/PhysRevB.85.235438`.

[2] A. López-Cudero, A. Cuesta, and C. Gutiérrez, “Potential dependence of the saturation CO coverage of Pt electrodes: The origin of the pre-peak in CO-stripping voltammograms. part 1: Pt (111),” *Journal of Electroanalytical Chemistry*, vol. 579, no. 1, pp. 1–12, 2005. DOI: `10.1016/j.jelechem.2005.01.018`.