**Nitrogen electrochemical fixation checklist**

 This checklist applies to N2 electrocatalytic activation to N-containing products (N-products) such as ammonia, nitric acid, urea, etc.

|  |  |  |  |
| --- | --- | --- | --- |
| Request | Yes? | N/A | Response(state where this information can be found in the text or why this is not applicable) |
| 1. Quantification of N-contaminations |
|  | 1.1. Labware |
|  | Details of the labware cleaning process |[ ] [ ]  Click or tap here to enter text. |
|  | Quantitative confirmation of the effectiveness of the employed cleaning procedure. e.g. recommended practice: please keep the electrolyte inside the sealed electrochemical cell with no potential applied for a period of time exceeding the duration of the longest N2 activation experiments reported. Please then analyze the electrolyte solution for the NO*x* and NH3/NH4+ contents before and after this treatment. |[ ]  ☐ | Click or tap here to enter text. |
|  | 1.2. Gas purity  |
|  | Please quantify the amount of NH3, N2O, NO and NO2 contaminants in 14N2, 15N2 and Ar gas supplied into the cell through analysis of the gas supplies and solutions through which these gasses were passed through. |[ ]  ☐ | Click or tap here to enter text. |
|  | If the amount of impurities > 1 mol.% of the amount of N-product produced during the N2 activation tests, both 14N2 and 15N2 must be purified and please provide: |  |  |  |
|  |  | Experimental set-up and full details (including gas flow rate, volume of the gas, the reagents used for purification, purification time, detection method for each contamination etc) for the purification process |[ ]  ☐ | Click or tap here to enter text. |
|  |  | Please quantify the NH3, N2O, NO and NO2 contaminants after the purification process. |[ ]  ☐ | Click or tap here to enter text. |
|  | When a secondary gas such as O2, H2, CO2 or other is also involved in the N2 (co-)activation, the purity of this secondary gas must be quantified and purification must be applied following the same procedure as those for the N2 gas. |[ ]  ☐ | Click or tap here to enter text. |
|  | 1.3 Electrolyte purity |
|  | Please report the purity of every reagent |[ ]  ☐ | Click or tap here to enter text. |
|  | Please quantify the amount of NO2-, NO3-, NH3/NH4+ contamination in each component of the electrolyte solution separately (e.g. electrolyte salt and solvent) and in the resulting electrolyte solution. |[ ]  ☐ | Click or tap here to enter text. |
|  | Details of the measurements (volume of the electrolyte, detection method). |[ ]  ☐ | Click or tap here to enter text. |
|  | 1.4. Electrode purity  |
|  | Please quantify the overall N-content in the electrodes used in the experiment. |[ ]  ☐ | Click or tap here to enter text. |
| 2. Control experiment |
|  | 2.1. Control experiments without any applied potential |
|  | Please provide control experiments under N2 (and other reacting gas for case of co-activation) without any applied potential immediately prior to the N2 activation experiments.  |[ ]  ☐ | Click or tap here to enter text. |
|  | 2.2. Ar control experiments  |
|  | Experiments with applied potential/current under Ar (and other reacting gas for case of co-activation) must be performed at least three times immediately prior to the N2 activation experiment and the resulting N-product along with relevant N-contaminant (NH3/NH4+/NOx) must be quantified. Ar control tests can be undertaken in experiments not immediately preceding the N2 activation tests in the cases where such tests induce irrevocable degradation of the key system components. |[ ]  ☐ | Click or tap here to enter text. |
|  | 2.3. Effect of membrane  |
|  | Please provide details of the membrane cleaning procedure  |[ ]  ☐ | Click or tap here to enter text. |
|  | To exclude the effect of the membrane, a test in an electrolyte solution with deliberately added N-product (for example, NH3/NH4+ for the N2 electrochemical reduction studies or NOx for the N2 electrochemical oxidation studies) under Ar must be performed to confirm that the membrane is not accumulating NH3/NH4+/NO*x*. In another test, the membrane must be kept in contact with N-product-free electrolyte solution for a period of time longer than the N2 activation tests, and the concentration of NH3/NH4+/NO*x* must be monitored as a function of time. |[ ]  ☐ | Click or tap here to enter text. |
| 3. Quantification of N-product  |
|  | 3.1. Quantification using more than one method |
|  | Apart from quantification of N-product(s) by conventional spectrophotometry or ion chromatography, the N-product(s) must be quantified using a direct method *e.g.* NMR for NH3/NH4+, and either NMR or mass-spectroscopy or other similarly suitable techniques for other N-products (e.g. NOx or urea); satisfactorily consistent results from different analytical methods must be demonstrated. For the 1H NMR analysis, data must be collected in a deuterated solvent that has no labile deuterium, such as d6-DMSO, with appropriately adjusted pH to ensure reproducible ammonia protonation. |[ ]  ☐ | Click or tap here to enter text. |
|  | 3.2. Calibration |
|  | Please provide experimental details including purity of the quantification reagents and data for the calibration process using standard N-product solution for quantification. |[ ]  ☐ | Click or tap here to enter text. |
|  | 3.3. Isotope labeling experiment |
|  | Please perform 15N2 activation tests as a function of time under the same conditions as the key 14N2 activation tests and report 15N- and 14N-product yields along with the 15N and 14N contaminants (NH3/NH4+/NOx) for at least 2 (preferably, ≥3) distinctly different time-points.  |[ ]  ☐ | Click or tap here to enter text. |
|  | The N-product from the 14N2 and 15N2 activation tests must be quantified using NMR for NH3/NH4+, and using either NMR or mass-spectroscopy or other similarly suitable technique for other N-products (e.g. NOx or urea).  |[ ]  ☐ | Click or tap here to enter text. |
|  | Satisfactory agreement between N-product yield rates and faradaic efficiency in the 14N2 and 15N2 activation experiments must be demonstrated at each time-point.  |[ ]  ☐ | Click or tap here to enter text. |
|  | The results of control experiments as in 2.1 and 2.2 immediately prior to the 15N2 activation must be reported. |[ ]  [ ]  | Click or tap here to enter text. |
| 4. N2 activation test with a fixed N2 volume |
|  | Please undertake experiments in a closed system with a small, fixed volume of 14N2 and 15N2 gas (and other reacting gas for case of co-activation) for a period of time that is sufficient to produce N-product in the amounts vastly (at least by an order of magnitude) exceeding the quantified amount of the NO*x* and NH3/NH4+ contaminants present in the system (including in the electrolyte solution, gas phase, electrodes, etc.). The N-product must be quantified and plotted against time. The experimental set up and details must be included. |[ ]  ☐ | Click or tap here to enter text. |
| 5. Data reporting |
|  | 5.1. Key metrics  |
|  | Surface area of the electrode and the mass loading of the active material.  |[ ]  ☐ | Click or tap here to enter text. |
|  | N-product yield normalized to geometric area, mass, and where possible to electrochemical surface area at a given potential vs. RHE.  |[ ]  ☐ | Click or tap here to enter text. |
|  | Faradaic efficiency and N-product yield rate as a function of the test conditions (at multiple potentials and/or current densities). |[ ]  ☐ | Click or tap here to enter text. |
|  | Long-term stability of the faradaic efficiency and N-product yield rate. The duration of the tests must be > 24 h. |[ ]  ☐ | Click or tap here to enter text. |
|  | 5.2 Reproducibility  |
|  | At least three (preferably more) independent repeats of each key experiment (including all 15N2 activation tests, controls and long-term experiments) must be performed, with all relevant experimental details reported. Results must be reported in a manner that provides a quantitative measure of the reproducibility, most commonly as mean ± one standard deviation. |[ ]  ☐ | Click or tap here to enter text. |
|  | 5.3 Data processing  |
|  | In electrochemical studies, voltammetric analysis must be undertaken in the cycling mode and evolution of the data must be discussed and demonstrated for key experiments. |[ ]  ☐ | Click or tap here to enter text. |