AE-JSC-321: Manufacturing and calibration of new 10 mL HTE with sensor spots I

Date: 2024-07-11

Tags: O2 JSC AE Calibration HTE

Status: Done

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Literature/reference experiments

| Literature | 1 |
|--------------------|---|
| Reproduction | 1 |
| Related experiment | Experiment - MLB-118: Production of NS14 cap with O2 and temperature optical sensor spots Experiment - JSC-622: HTE vial for O2 measurements Experiment - AE-300: Calibration of oxygen sensor spot in the 10 mL HTE vial |

Reagents

| Name | CAS Number / Experiment Number | concentration [M] | Equivalents | Molar mass [g/mol] | Density (g/ml) | Volume [ml] |
|---------------------------------|---|----------------------|-------------|-----------------------|----------------|----------------|
| Sodiumhydrogen carbonate | Experiment - AE-323: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O (AE-323-1) | 0.685 | 1 | 84.06 | / | 0.85 |
| Sodium carbonate (anhydrous) | Experiment - AE-323: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O (AE-323-2) | 0.3143 | / | 105.99 | 1 | 0.85 |
| Milli-Q water | 7732-18-5 | 1 | 1 | 18.02 | 1 | 6.8 |

Procedure/observations

The lights in the lab were turned off when working with the sensor spots

Done by AE, unless other indicated

| Date | Timo | Step | Observations |
|------|------|------|--------------|
| Date | Time | Steh | Observations |

| 11.07 | 14:00 | A PyroScience trace range oxygen sensor spot as well as a PyroScience optical temperature sensor spot were glued into a HTE glas vial provided by Michael Ringleb (silicone glue from PyroScience) (done by JSC) | Green O2 sensor spot and white temperature sensor spot |
|-------|---|--|--|
| | | The glue was allowed to harden at rt in the dark | |
| | 15:45 | Two 3D printed adapter to hold the optical fiber cables (printed by Michael Ringleg, dimensions: width 12 mm, height 8 mm, diameter of inner hole 2.55 mm, ca. 15 adapters were printed, two were selected that fit the optical fiber cables well) were glued on the outside of the vial (silicone glue from PyroScience) | White optical fiber cable adapters |
| | | The glue was allowed to harden at rt in the dark | |
| 15.07 | .07 The optical fibers were connected to the adapters and a Therma Pt100 was placed inside the vial on the same height as the sensor spots. | | T setup.jpg |
| | 11:22 | The vial was filled with water and both solutions | |
| | 11:29 | A 1-point calibration of the temperature sensor spot was performed using the Therma Pt100 as reference | |
| | 11:30 | the Therma Pt100 was removed from the vial and a slit septum cap was screwed onto the HTE vial. | |
| | 11:34 - 11:40 | A 2-point calibration of the oxygen sensor spot was performed. For this, Through the septum cap two needles were inserted (one in the liquid phase, one in the gas phase) and through the liquid phase needle, air was bubbled through the water phase with a balloon. Once a steady state was reached, the upper calibration point was set. | air setup.jpg |
| | 11:41 | The balloon was removed and the liquid phase needle was connected to argon, bubbling argon through the water phase | |
| | 12:20 | Once a steady state was reached, the lower calibration point was set | |
| | 12:20 | Both needles were removed from the vial and a log was started | |
| | 12:22 | The log was stopped. | AE-322-1.png AE-322-1.txt |
| | | The instrument configuration was saved. | 240702_Instruments_Settings_HTE_Vial_Liquid_Phase.ini |

Analysis

| Date | Time | Sample name | Analysis method | Analytical device | Solvent | Raw Data | Processed Data | Interpretation |
|-------|-------|-------------|-------------------------|---|---------|--------------|----------------|----------------|
| 15.07 | 12:20 | AE-322-1 | Optical O2 detection | Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel | H2O | AE-322-1.txt | AE-322-1.png | |

Results

Manufacturing and calibration worked, signal intensity is ok (approx. 240 for O2, when complete degassed, T approx. 130)

Linked experiments

- MLB-118: Production of NS14 cap with O2 and temperature optical sensor spots
- JSC-622: HTE vial for O2 measurements
- AE-300: Calibration of oxygen sensor spot in the 10 mL HTE vial
- AE-323: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O

Attached files

T-setup.jpg

sha256: e637919dc1f19084937c1d7924dd527c5219d180c4e44308429b17228cb30a3d



air-setup.jpg

sha256: 43ec43f102aeba44d752280b1872f05c17ef97041ed8c6415764dbe150916aef



AE-322-1.png

sha256: e1a59688904b7b2de27d3ee6e742798ebe8cbdee372de7ed0e40493fc20ef05c



AE-322-1.txt

sha256: b76b8bdb22ec6107c23ce9b9e45595b3d43926832c6723643926198850a53d9d

240717 Instruments Settings HTE Vial Liquid Phase.ini

sha256: 31fd349961696a03e80857196c3464a3d0feb313adc31517136475e70c7c507a



 $\label{thm:condition} \begin{tabular}{ll} Unique e LabID: 20240711-d3e9426d41b902b2cd8837a9f726ef8b8a933a7a Link: https://elab.water-splitting.org/experiments.php?mode=view&id=1203 and the condition of the co$