In-situ hydrogen and oxygen measurment in H2/O2 reactor

Date: 2025-08-26
Tags: O2 AE Firesting O2 sensor H2 H2 Evolution in-situ Unisense H2 Sensor O2 evolution
Category: Protocol
Created by: Alexander Eith

Goal

Parrallel in-situ H2 and O2 quantification

Prerequisites and preparation

Experience with both H2 and O2 sensor

Introdcution to perform this type of experiment

Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle

Equipment - H2 UniAmp Single Channel System

Equipment - H2/O2 reactor

Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 1) (or another Firesting)

Equipment - Robust probe for liquid O2 measurment

Succesful and valid calibration of robust probe according to Protocol - Calibration of Trace range robust probe for O2 measurment

Desired photocatalytic system

Equipment - Advanced irradiation setup V1.0 I - ideally in semi permanent installed H2 setup

3 * GL14/NS14 transition piece

1 * GL18/NS14 transition piece, short

egg shaped stir bar

BOLA fittings: 1 * 10 mm, 2 * 4 mm, 1 * 3 mm

NS14 glas stopper

Safety

Everything stated in Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

Polarization needs to be 100 mV (will be adjusted automatically when connected to Equipment - H2 UniAmp Single Channel System and Equipment - H2 UniAmp Sensor

Be careful with H2 sensor: sensitive to touch, being turned upside down

Be careful with O2 sensor: glas fiber cables

Steps

Step number	Step description	Pictures
1. Build setup for H2 calibration	Fill the reactor with water (25 mL) Add the fitting BOLA fittings to to the sensors and connect them to the transition pieces: • H2 sensor (10 mm BOLA) • PT100 and PT1000 (4 mm BOLA) • O2 sensor (3 mm BOLA) Close them tightly Add the transition pieces to the reactor in that order, place all sensor in roughly the same hight • PT1000 goes into the middle - secure it with the Hodler for the H2/O2 reactor - is placed on the BOLA fitting • PT100 goes into the connection closest in the direction of stirring from the vlave • O2 sensor goes into the connection furthest away in the direction of stirring from the vlave • H2 sensor goes opposite the valve - pay spacial attention to the positioning: The sensor is placed in such a way, that the longer part of the needle prtoects the sensor in the direction of stirring	
2. H2 calibration	Analog to Protocol - Liquid phase calibration of H2 UniAmp sensor with following deviations: • Use H2/O2 reactor instead of NS14/GL14 flask • Measure O2 content during calibraiton - can be used to check, if H2 bubbling is working, O2 value should be not higher than 2 µM, better below 1 µM when calibration is performed • Take 1.000.000 ppm point as 2nd calibration point, for that bubble H2 from a ballon through the solution -- for details see Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014 • Bubble H2 till stable signal is reached • For successful calibration slop around 1.4 should be obtained	
3. Disasamble the reactor	Remove all sensors, leave them in the BOLA Fitting Put 3D printed protective cover on H2 sensor Wipe O2 sensor dry and add protective cover Place all sensor in secure places Leave the H2 sensor plugged in, all other sensors can be plugged out if desired, but that is not needed	

4. Clean and dry the reactor	Degrease the reactor Empty the water Dry the reactor in a drying oven till dry (recommended temperature 120 °C, so that it does not take to long) - also start the Drying oven early enough, so that it can preheat	
5. Sample preparation	Prepare sample as desired in seperate flask/vial	
6. Build setup for experiment	• Add desired LEDs and power sources and adjust to correct setting • Add reactor to irradiation setup • Add catalyst suspension/solution • Build setup as described in 2., but do not add the H2 sensor, close the open port with NS14 stopper	
7. Start degassing of reactor	• Start O2 logging • Add tubing for Argon sparging thorugh the valve • Start degassing - flow should be quite fast, but it should not splash around	
8. Degassing of reactor	Degas until stable O2 conc. of approx. 0 µM is obtained (at least 20 min, typically 30 to 45 min needed) Better wait 5 more minutes, to get better result	
9. Add H2 sensor	Move cannula for Ar sparging to gas phase Add H2 sensor in Ar counter flow - place in same position and orientation as before (details see 1.)	
10. Stop degassing	After closing wait 30 s to 1 min to see if signal increases storngly Remove Ar sparging tube from reactor and close the valve Wait approx. 3 min to see rate of increase (should be below 1 µM / 5 min, if higher than 1 µM / 2 min: see 10a.) Stop O2 logging	
10a. Touble shouting: Too high leakage	• Never degas with H2 sensor in place, so remove sensor before continue degassing • If longer degassing time does not help: Check all connections (NS14 glass joints and BOLA fittings) • If that does not help inform responsible person	
11. Pre-reaction baseline	Start H2 log and O2 log shortly after each other Take pre-reaction baseline (min. 15 min, but longer if needed: e.g. with linear increse in O2 total min. 20 min, non linear increase: min. 30 min)	

12. Irradaition	Start irradiaiton, also make comment in H2 software (UniSense) for irrad start and press add the second you turn on the light, also look the time of the O2 log in that moment Note down: • Start time of irrad • Start time of irrad according to H2 log down to the second • Start time of irrad relative to the O2 log For stopping irrad follow the same procedure	
13. Post-reaction baseline	Duration depend on type of experiment - wait appropiate amount of time, than stop the h2 and the O2 log	
14. Disasamble the reactor	1. Disconnect all sensors 2. Remove the H2 sensor, degrease the NS14 joint, clean the sensor with water and if needed a wet Kimtech wipe - do not use dry wipes and any other solvent than water - you can touch the glas part of the sensor, but do not put force on it, place it in 3D printed protective hull and clamp it in storage position 3. Remove the O2 sensor, degrease the NS14 joint, clean the with water and Kimtech wipes - never ever use other solvents than water, dry sensor with Kimtech wipe, remove BOLA fitting, add prtoective cap and store it in its box 4. Remove PT100, degrease the NS14 joint, clean the with water and Kimtech wipes, dry sensor with Kimtech wipe, remove BOLA fitting, and store it in its box 5. Reomve PT1000, therefore move 3D printed holder for the the reactor a bit to the front and clip out the BOLA fitting, degrease the NS14 joint, clean the with water and Kimtech wipes, dry sensor with Kimtech wipe, remove BOLA fitting, and store it in its box	
15. Clean H2/O2 reactor	Clean H2/O2 reactor as needed, avoid abrasive and glass attacking chemicals	

Linked resources

- Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 1)
- Equipment - Robust probe for liquid O2 measurment
- Equipment - Advanced irradiation setup V1.0 I
- Equipment - EPR, CEEC I lab K002
- Equipment - H2 UniAmp Sensor - Low range - 2.1 x 80 mm needle
- Equipment - H2 UniAmp Single Channel System
- Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle
- Equipment - H2/O2 reactor
- Protocol - EPR measurement on EPR, CEEC I lab K002
- Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014
- Protocol - Gas phase calibration of H2 UniAmp sensor
- Protocol - Hydrogen measurement with H2 UniAmp sensor (1 point gas phase measurement)
- Protocol - Hydrogen measurement with H2 UniAmp sensor (liquid or gas phase continous measurment)
- Protocol - Liquid phase calibration of H2 UniAmp sensor
- Protocol - Calibration of Trace range robust probe for O2 measurment

Unique eLabID: 20250826-841bc4c09cc0cb476e16989d31adb40bcb6434fd
Link: https://elab.water-splitting.org/database.php?mode=view&id=259