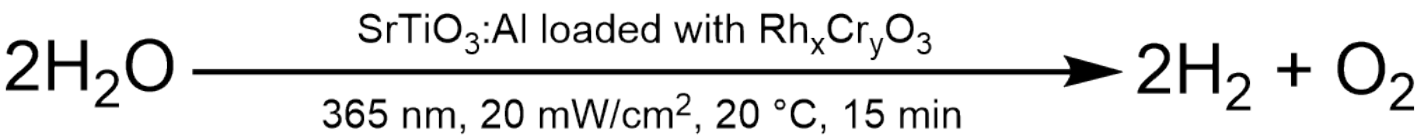
NB-326: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C (reproduction NB-325)

Date: 2025-10-28
Tags: O2 | Test | Calibration | NB | Firesting | O2 sensor | H2 | SrTiO3 | Unisense | RhCrO3:Al:SrTiO3 | H2 Sensor | temperature | In situ | Trace range robust oxygen sensor | photocatalysis | Unisense normal range
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-325: simultaneous detection of H_2 and O_2 evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:$SrTiO_3$ suspension (EA-358, 0.5 mg/mL), 365 nm LED, 20 mW/cm^2, 20 °C.

Reaction scheme



ChemDraw file linked: NB-326-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C
Similar experiments	SrTiO3 - NB-316: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Pressure [bar]	Concentration [mM]
milli-Q H_2O	/	/	/	/	/	/	/	0.998	25 + 25 (for calibration)	/	/
Al:SrTiO3 RhCrOx (EA-358)	SrTiO3 - EA-358: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	12.50	12.72	/	/	/	/	/

Hydrogen	1333-74-0	/	/	/	/	/	/	/	2 balloons (ca. 2 L)	approx. 1	/

Excel sheet for reagent calculation

/

Irradiation Parameters

Power measurement was performed using Power Meter - 843-R-USB + 919P-020-12 in Equipment - Advanced power measurment setup V1.0 I

Power measurement was performed in experiment Prep work - NB-324: Measuring power output of UHP-365 nm #4 with 18A-4 in advanced irradiation setup I I

	Name
Used Set-up	Equipment - Advanced irradiation setup V1.0 I
Irradiation setup number	Equipment - Irradiation setup 4 (CEEC II, E002)

	Light Source Name	**Power Source Name**	**Wavelength [nm]**	**Power Setting [mW]**	**Analog Setting [0.00 - 10.00]**
First light source	Light Source - UHP LED 365 nm-4	Power Sources - BLS-18000-14	365	22	0.07

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	20
Stirring speed [rpm]	500
Irradiation start: **1. Firesting [relative to start log]** **2. Unisense**	1. 603 s 2. 17:50:27

Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1534 s 2. 18:05:57

O_2/H_2 sensor equipment

	Equipment	Used protocol
Used Firesting	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)	Protocol - Operation of Firesting Fiber-Optic Oxygen Meter 2 Channel Software
Used O_2 sensor	Equipment - Robust probe for liquid O2 measurment	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor
Used H_2 sensor	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
28.10.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
	10:51-11:57	Conditioning of H2 sensor	**NB-326-Logger1** 1.8 mV at the end of polarization procedure	NB-326.ulog NB-326-Logger1.csv NB-326-Logger1.bmp
	ca. 14:30	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), done according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling.	/	/
	14:53	Start of O2 logging.	**NB-326-Ch2-1**	2025-10-28_145357_NB-326-Ch2-1.txt 2025-10-28_145357_NB-326-Ch2-1.png

	14:54	Start of H2 logging.	**NB-326-Logger2** offset -2 mV	NB-326.ulog NB-326-Logger2-calibration.csv NB-326-Logger2-calibration.bmp
	14:57	Degassing was started.	/	20251028_145926-degassing of water.jpg
	15:19	Introducing H2 sensor into the reactor under Ar flow.	/	/
	15:22	0 ppm was taken.	/	/
	15:30	H2 bubbling of the reactor was started	/	20251028_153053-purging with H2.jpg
	15:39	Introducing second H2 balloon.	/	20251028_154109-with 2nd H2 balloon.jpg
	15:50	1.000.000 ppm point was taken and calibration was saved	802 mV, slope: 1.026, 781.887 uM	20251028_155003-H2 table.jpg
	15:52	Stop of H2 logging.	/	/
	15:52	Stop of O2 logging.	/	/
	ca. 16:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	16:20	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	16:25	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	16:28-31	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251028_163114-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	16:40-50	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	ca. 16:55	Assembling the setup.	Currently, stopper instead of H_2 sensor, PT100, PT1000 and O_2 robust probe are inside the reactor immersed in the liquid phase	20251028_165756-before degassing and irradiation.jpg

	17:00	Start of O2 logging.	**NB-326-Ch2-2**	2025-10-28_170045_NB-326-Ch2-2.txt 2025-10-28_170045_NB-326-Ch2-2.png
	17:03	The degassing was started	/	20251028_170354-degassing of the suspension.jpg
	17:32	Cannula was transferred to gas phase, above the suspension.	/	/
	17:35	H_2 sensor was added in Ar counterflow.	/	/
	17:38	The degassing was stopped by removing the cannula and closing the valve.	/	20251028_174044-after introducing H2 sensor.jpg
	17:40	Stop of O2 logging.	/	/
	17:40	Start of O2 logging.	**NB-326-Ch2-3**	2025-10-28_174023_NB-326-Ch2-3.txt 2025-10-28_174023_NB-326-Ch2-3.png
	17:40	Start of H2 logging.	**NB-326-Logger3**	NB-326.ulog NB-326-Logger3-during irradiation.csv NB-326-Logger3-during irradiation.bmp
	17:40-50	Equilibration time.	/	/
	17:50	The irradiation was started	/	20251028_175536-after start of irradiation.jpg
	18:05	The irradiation was stopped.	/	/
	18:05-15	Equilibration time.	/	/
	18:16	Stop of O2 and H2 logging.	/	/

	ca. 18:20	Deassembling the setup, cleaning the reactor.	Tips of the sensors and reactor were covered with attached photocatalyst particles. Tip: After preliminary cleaning with sticks, wipes, the residual particles attached to the walls of the reactor could be removed by sonication - fill the reactor with water and place it in ultrasonic bath for ca. 20 s (Eco mode).	20251028_182055-after irradiation.jpg

Analysis

Used calibration for Firesting: 20250611-BOLA-fitting-liquid-phase-trace-oxygen-sensor-H2-O2 reactor.ini

Used calibration for UniSense: NB-326-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
28.10.2025	10:51	NB-326-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-326.ulog NB-326-Logger1.csv	/	NB-326-Logger1.bmp	/	
	14:54	NB-326-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-326.ulog NB-326-Logger2-calibration.csv	/	NB-326-Logger2-calibration.bmp	/	
	17:40	NB-326-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-326.ulog NB-326-Logger3-during irradiation.csv	NB-326-O2 and H2 curve.py	NB-326-Logger3-during irradiation.bmp NB-326-O2 and H2 curves.png	SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C	
	14:53	NB-326-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-28_145357_NB-326-Ch2-1.txt	/	2025-10-28_145357_NB-326-Ch2-1.png	/	
	17:00	NB-326-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-28_170045_NB-326-Ch2-2.txt	/	2025-10-28_170045_NB-326-Ch2-2.png	/	
	17:40	NB-326-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-28_174023_NB-326-Ch2-3.txt	NB-326-O2 and H2 curve.py	2025-10-28_174023_NB-326-Ch2-3.png NB-326-O2 and H2 curves.png	SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C	

Results

Simultaneous H_2 and O_2 measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O_2/H_2 photoreactor under 365 nm irradiation (20 mW/cm^2, 20 °C**,** 15 min) were performed.

H_2 level at the end of irradiation - ca. 17.5 umol/L, O_2 level - ca. 17.1 umol/L.

Linked experiments

SrTiO3 - NB-316: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

SrTiO3 - NB-318: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C

SrTiO3 - NB-322: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C (reproduction NB-318)

SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C

Linked resources

Equipment - VWR® VV3, Vortex Mixer

Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)

Equipment - Irradiation setup 4 (CEEC II, E002)

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

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

Attached files

NB-326-O2 and H2 curves.png
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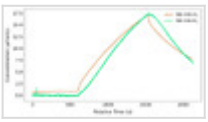

NB-326-O2 and H2 curve.py

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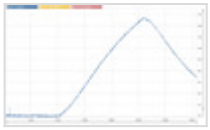

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20251028_174044-after introducing H2 sensor.jpg
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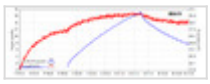

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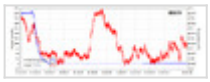

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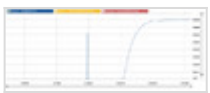

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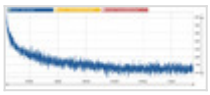

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