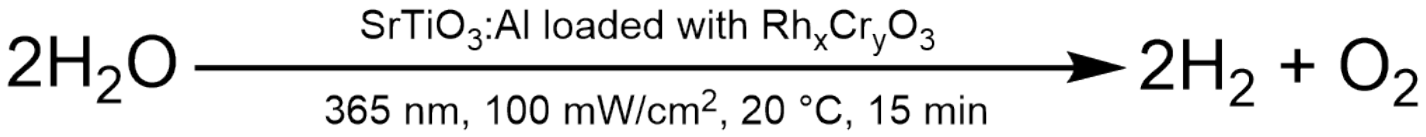
NB-322: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C (reproduction NB-318)

Date: 2025-10-27
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 NB-318: 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, **100 mW/cm²**, 20 °C (changing intensity relatively to reference experiment).

Reaction scheme



ChemDraw file linked: NB-322-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	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
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]	Concentration [mM]
milli-Q H_2O	/	/	/	/	/	/	/	0.998	25	/
Al:$SrTiO_3$ 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	12.37	/	/	/	/

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-317: Measuring power output of UHP-365 nm #4 with 18A-4 in advanced irradiation setup 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	113	0.39

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. 613 s 2. 22:07:52
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1536 s 2. 22:23:16

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
27.10.2025		Calibration from experiment SrTiO3 - NB-320: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reprodution NB-316) I was used.		
		Sample preparation		
	20:25	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid	/
	20:27	Addition of 25 mL H2O to the vial via graduated cylinder, covering with Al foil before further use.	/	/
	20:48-51	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251027_205218-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	21:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	21:05	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	20251027_210553-before degassing.jpg

	21:06	Start of O2 logging.	**NB-322-Ch2-1**	2025-10-27_210616_NB-322-Ch2-1.txt 2025-10-27_210616_NB-322-Ch2-1.png
	21:10	The degassing was started	/	/
	21:51	Cannula was transferred to gas phase, above the suspension.	/	/
	21:53	H_2 sensor was added in Ar counterflow.	/	/
	21:55	The degassing was stopped by removing the cannula and closing the valve.	/	/
	21:57	Stop of O2 logging.	/	/
	21:57	Start of O2 logging.	**NB-322-Ch2-2**	2025-10-27_215740_NB-322-Ch2-2.txt 2025-10-27_215740_NB-322-Ch2-2.png
	21:57	Start of H2 logging.	**NB-322-Logger1**	NB-322.ulog NB-322-Logger1.csv NB-322-Logger1.bmp
	21:57-22:08	Equilibration time.	/	20251027_220036-after introducing H2 sensor.jpg
	22:08	The irradiation was started	/	20251027_220824-after start of irradiation.jpg
	22:23	The irradiation was stopped.	/	/
	22:23-33	Equilibration time.	/	/
	22:33	Stop of O2 and H2 logging.	/	/

	ca. 22:40	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).	20251027_223530-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-320-Logger6

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
27.10.2025	21:57	NB-322-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-322.ulog NB-322-Logger1.csv	NB-322-O2 and H2 curve.py	NB-322-Logger1.bmp NB-322-O2 and H2 curve.png	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	Clean response of H2 sensor, ca. 90 uM H2 at the end of irradiation.
	21:06	NB-322-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-27_210616_NB-322-Ch2-1.txt	/	2025-10-27_210616_NB-322-Ch2-1.png	/	Degassing of the suspension.
	21:57	NB-322-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-10-27_215740_NB-322-Ch2-2.txt	NB-322-O2 and H2 curve.py	2025-10-27_215740_NB-322-Ch2-2.png NB-322-O2 and H2 curve.png	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	O2 curve during irradiation, O2 value ca. 79 uM at the end of irradiation.

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 (100 mW/cm^2, 20 °C**,** 15 min) were performed (reproduction NB-318).

H_2 level at the end of irradiation - 90 umol/L, O_2 level - 79 umol/L.

Linked experiments

SrTiO3 - NB-313: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 0.5 mg/mL), 365 nm, 50 mW

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-320: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reproduction NB-316) I

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 - Liquid phase calibration of H2 UniAmp sensor

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

Attached files

NB-322-O2 and H2 curve.png
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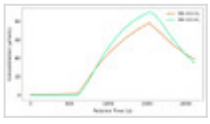

NB-322-O2 and H2 curve.py
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NB-322-SrTiO3-photocatalytic H2O splitting.png
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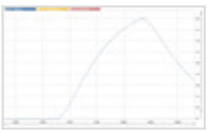

NB-322-Logger1.csv
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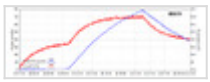

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NB-322.ulog
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