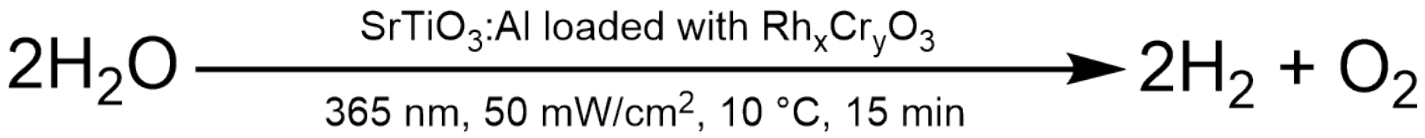
NB-359: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C (reproduction NB-351)

Date: 2025-11-24
Tags: O2 | Test | Calibration | NB | Firesting | O2 sensor | H2 | SrTiO3 | troubleshooting | Unisense | RhCrO3:Al:SrTiO3 | H2 Sensor | temperature | In situ | H2/O2 reactor | Trace range robust oxygen sensor | photocatalysis | Unisense normal range
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-351: 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, 50 mW/cm^2, **10 °C** (changing temperature relatively to reference conditions).

Reaction scheme



ChemDraw file linked: NB-351-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °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	12.58	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (ca. 1 L)	ca. 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-314: Measuring power output of UHP-365 nm #4 with 18A-4 in advanced irradiation setup

	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	56	0.19

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	10
Stirring speed [rpm]	500
Irradiation start: **1. Firesting [relative to start log]** **2. Unisense**	1. 600 s 2. 17:47:57
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1520 s 2. 18:03:17

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
24.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
	12:01-13:11	Conditioning of H2 sensor	**NB-359-Logger1** 1.6 mV at the end of polarization procedure	NB-359.ulog NB-359-Logger1-pre-polarization.csv NB-359-Logger1-pre-polarization.bmp
	13:30	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 10 °C) done according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling.	/	/
	13:40-53	Keeping the system under stirring to reach 10 °C (T control by PT1000).	/	/
	13:53	Start of O2 logging.	**NB-359-Ch2-1**	2025-11-24_135359_NB-359-Ch2-1.txt 2025-11-24_135359_NB-359-Ch2-1.png
	13:56	Degassing was started.	/	20251124_135652-degassing of water.jpg

	14:19	Start of H2 logging.	**NB-359-Logger2** offset -2 mV	NB-359.ulog NB-359-Logger2-calibration.csv NB-359-Logger2-calibration step.bmp NB-359-Logger2-2point calibration.bmp
	14:20	Moving cannula to gas phase above the liquid level.	/	/
	14:23	Introducing H2 sensor into the reactor under Ar flow.	/	/
	14:24	0 ppm was taken.	offset -1 mV	/
	14:32	Removing the previous 0 point, taking new 0 ppm.	/	/
	14:32	Degassing was stopped.	/	/
	14:33	H2 bubbling of the reactor was started.	Drop in H2 value at 14:41, but afterwards increased and could reach steady state.	20251124_143502-H2 bubbling.jpg
	14:47	1.000.000 ppm point was taken.	618 mV	20251124_144653-H2 table.jpg 2
	14:52	The previous point was deleted, new 1.000.000 ppm point was taken and calibration was saved	662 mV (stable for several minutes), slope: 0.771, 856 uM 14:54 - decrease in H2 signal, followed by stopping the logging.	/
	14:54	Stop of H2 logging.	/	/
	14:57	Stop of O2 logging.	/	/
	15:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	15:25	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	15:30	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	15:32-35	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251124_153508-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		

	15:40	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	15:45	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	/
	15:48-58	Keeping suspension at 10 °C, T control by PT1000.	/	/
	15:58	Start of O2 logging.	**NB-359-Ch2-2**	2025-11-24_155825_NB-359-Ch2-2.txt 2025-11-24_155825_NB-359-Ch2-2.png
	16:00	The degassing was started	/	20251124_160032-degassing of the suspension.jpg
	16:29	Cannula was transferred to gas phase, above the suspension.	/	/
	16:31	H_2 sensor was added in Ar counterflow.	/	/
	16:33	The degassing was stopped by removing the cannula and closing the valve.	/	/
	16:35	Stop of O2 logging.	/	/
	16:35	Start of O2 logging.	**NB-359-Ch2-3**	2025-11-24_163521_NB-359-Ch2-3.txt 2025-11-24_163521_NB-359-Ch2-3.png
	16:35	Start of H2 logging.	**NB-359-Logger3** High H2 signal after introducing sensor to the reactor.	NB-359.ulog NB-359-Logger3.csv NB-359-Logger3.bmp
	16:36	Stop of H2 and O2 loggings.	Removing the sensor from the reactor, introducing once again, checking cable management.	/
	16:42	Start degassing once again.	**NB-351-Ch2-4**	2025-11-24_164417_NB-359-Ch2-4.txt 2025-11-24_164417_NB-359-Ch2-4.png
	16:44	Moving cannula to gas phase above the liquid level.	/	/

	16:56	Introducing H2 sensor under Ar flow.	Immediately after immersing the sensor in the suspension - signal OK, but afterwards again the signal value was high (maybe problems with the position of the cable)	/
	16:56	Removing cannula, closing the valve.	/	/
	ca. 17:07	Stop of O2 logging.	/	/
	17:07	Start of O2 logging.	**NB-351-Ch2-5**	2025-11-24_170748_NB-359-Ch2-5.txt 2025-11-24_170748_NB-359-Ch2-5.png
	17:08	Start of H2 logging.	**NB-351-Logger4**	NB-359.ulog NB-359-Logger4.csv NB-359-Logger4.bmp
	17:13	Stop of O2 logging.	H2 logging showed not stable signal of the sensor after immersion in the suspension	/
	17:20	Stop of H2 logging.	/	/
	17:22-36	Degassing of the suspension (cannula above the suspension).	/	/
	17:37	Start of O2 logging	**NB-351-Ch2-6**	2025-11-24_173757_NB-359-Ch2-6.txt 2025-11-24_173757_NB-359-Ch2-6.png
	17:37	Start of H2 logging.	**NB-351-Logger5**	NB-359.ulog NB-359-Logger5-during irradiation.csv NB-359-Logger5-during irradiation.bmp
	17:37-47	Equilibration time.	/	/
	17:47	The irradiation was started	/	/
	18:03	The irradiation was stopped.	/	/
	18:03-13	Equilibration time.	/	/
	18:13	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).	20251124_181412-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-359-Logger2

for feasibility, loggings used for calibration and in photocatalytic tests are highlighted with green.

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
18.11.2025	12:01	NB-359-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-359.ulog NB-359-Logger1-pre-polarization.csv	/	NB-359-Logger1-pre-polarization.bmp	/	Pre-polarization of H2 sensor.
	14:19	NB-359-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-359.ulog NB-359-Logger2-calibration.csv	/	NB-359-Logger2-calibration step.bmp NB-359-Logger2-2point calibration.bmp	/	Calibration of H2 sensor, at 10^6 ppm signal 662 mV, slope 0.771
	16:35	NB-359-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-359.ulog NB-359-Logger3.csv	/	NB-359-Logger3.bmp	/	High signal after introducing H2 snsor to the suspension --> needs to be eliminated.
	17:08	NB-359-Logger4	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-359.ulog NB-359-Logger4.csv	/	NB-359-Logger4.bmp	/	Not stable signal after immersing the sensor in the suspension.
	17:37	NB-359-Logger5	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-359.ulog NB-359-Logger5-during irradiation.csv	NB-359-O2 and H2 curve.py	NB-359-Logger5-during irradiation.bmp	SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C	H2 evolution during irradiation.
	13:53	NB-359-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-24_135359_NB-359-Ch2-1.txt	/	2025-11-24_135359_NB-359-Ch2-1.png	/	Degassing of water followed by calibration of H2 sensor.

	15:58	NB-359-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-24_155825_NB-359-Ch2-2.txt	/	2025-11-24_155825_NB-359-Ch2-2.png	/	Degassing of the suspension (eliminating problems)
	16:35	NB-359-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-24_163521_NB-359-Ch2-3.txt	/	2025-11-24_163521_NB-359-Ch2-3.png	/	Degassing of the suspension (eliminating problems).
	16:44	NB-359-Ch2-4	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-24_164417_NB-359-Ch2-4.txt	/	2025-11-24_164417_NB-359-Ch2-4.png	/	Degassing of the suspension (eliminating problems).
	17:07	NB-359-Ch2-5	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-24_170748_NB-359-Ch2-5.txt	/	2025-11-24_170748_NB-359-Ch2-5.png	/	Degassing of the suspension (successful).
	17:37	NB-359-Ch2-6	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-24_173757_NB-359-Ch2-6.txt	NB-359-O2 and H2 curve.py	2025-11-24_173757_NB-359-Ch2-6.png	SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C	O2 evolution during irradiation.

Results

Reproduction of NB-351: 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 (50 mW/cm^2, 10 °C**,** 15 min) were performed.

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

SrTiO3 - NB-330: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C

SrTiO3 - NB-334: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C (reproduction NB-330)

SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C

Linked resources

Equipment - VWR® VV3, Vortex Mixer

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

Equipment - Advanced power measurment chamber V1.0 I

Equipment - Irradiation setup 4 (CEEC II, E002)

Light Source - UHP LED 365 nm-4

Power Sources - BLS-18000-1 4

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

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

Attached files

NB-359-O2 and H2 curves.png
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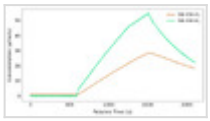

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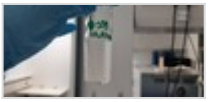

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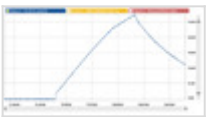

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NB-359-Logger2-calibration.csv
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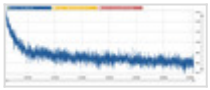

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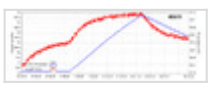

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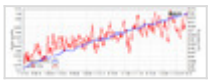

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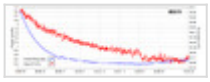

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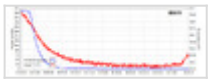

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Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3542