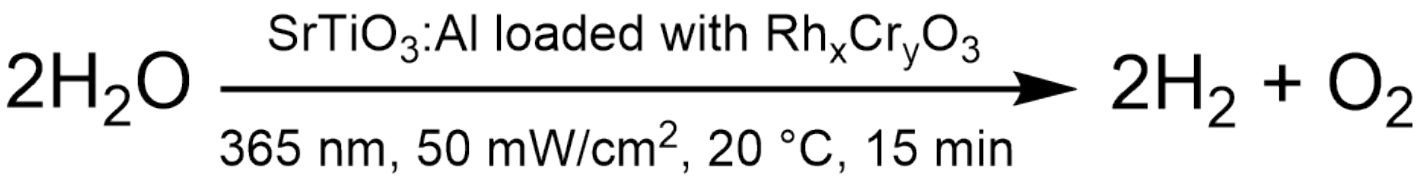
NB-331: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reprodution NB-316) I I I

Date: 2025-11-03
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 | Liquid phase detection
Category: SrTiO3
Status: Done
Created by: Nadzeya Brezhneva

Objectives

Third reproduction of NB-316 - 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, 20 °C.

Reaction scheme



ChemDraw file linked: NB-329-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-329: 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 I 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-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
Similar experiments	/

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	12.53	/	/	/	/	/
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-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]	20

Stirring speed [rpm]	500
Irradiation start: **1. Firesting [relative to start log]** **2. Unisense**	1. 600 s 2. 18:36:13
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1515 s 2. 18:51:28

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
03.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
	11:20-12:21	Conditioning of H2 sensor	**NB-331-Logger1** 1.78 mV at the end of polarization procedure	NB-331-Logger-pre-polarization.bmp NB-331-Logger1-pre-polarization.csv
	13:30-40	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.	/	/

	13:56	Start of O2 logging.	**NB-331-Ch2-1**	2025-11-03_135629_NB-331-Ch2-1.txt 2025-11-03_135629_NB-331-Ch2-1.png
	13:57	Start of H2 logging.	**NB-331-Logger2**	NB-331-Logger2.bmp NB-332-Logger2.csv
	13:59	Degassing was started.	/	20251103_140014-degassing of water.jpg
	14:36	Moving cannula to the gas phase above the suspension.	/	/
	14:37	Introducing H2 sensor into the reactor under Ar flow.	/	/
	14:39	0 ppm was taken.	/	/
	14:48	H2 bubbling of the reactor was started	14:48 no H2 purging, GL14 cap needs to be replaced.	/
	14:54	Stop of O2 and H2 loggings.	/	/
	ca. 15:00-15:10	Change of GL14 cap	/	/
	15:21	Start O2 logging.	**NB-331-Ch2-2**	2025-11-03_152144_NB-331-Ch2-2.txt 2025-11-03_152144_NB-331-Ch2-2.png
	15:23	Start H2 logging.	**NB-331-Logger3**	NB-331-Logger3-2 point calibration.bmp NB-331-Logger3-calibration step.bmp NB-331-Logger3-calibration.csv
	15:24	Start degassing.	/	/
	16:08	Moving cannula to gas phase above the suspension.	/	/
	16:10	Introducing H2 sensor under Ar flow.	/	/
	16:19	Removing cannula, closing the valve.	/	/

	16:20	Introducing first H2 balloon.	/	20251103_162050-first H2 balloon.jpg
	16:26	Introducing second H2 balloon.	/	20251103_162709-with the 2nd baloon.jpg
	16:33	1.000.000 ppm point was taken and calibration was saved	852 mV (removed and at 16:35 854 mV was taken), slope: 1.072, 796 uM	20251103_163319-H2 table.jpg
	16:36	Stop of H2 logging.	/	/
	16:36	Stop of O2 logging.	/	/
	ca. 16:50	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	17:15	Weighing EA-358 photocatalyst in a 50 mL vial.	/	/
	17:25	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	17:26-29	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	Milky white suspension	20251103_173021-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	ca. 17:30	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	
	17:35-40	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	
	17:44	Start of O2 logging.	**NB-331-Ch2-3**	2025-11-03_174410_NB-331-Ch2-3.txt 2025-11-03_174410_NB-331-Ch2-3.png
	17:46	The degassing was started	/	20251103_174646-degassing of the suspension.jpg
	18:18	Cannula was transferred to gas phase, above the suspension.	/	/
	18:21	H_2 sensor was added in Ar counterflow.	/	20251103_182720-after introducing H2 sensor.jpg
	18:24	The degassing was stopped by removing the cannula and closing the valve.	/	/
	18:25	Stop of O2 logging.	/	/

	18:26	Start of O2 logging.	**NB-331-Ch2-4**	2025-11-03_182612_NB-331-Ch2-4.txt 2025-11-03_182612_NB-331-Ch2-4.png
	18:26	Start of H2 logging.	**NB-331-Logger4**	NB-331-Logger4-during irradiation.bmp NB-331-Logger4-during irradiation.csv
	18:26-36	Equilibration time.	/	/
	18:36	The irradiation was started	/	20251103_183622-after start of irradiation.jpg
	18:51	The irradiation was stopped.	/	/
	18:51-19:01	Equilibration time.	/	/
	19:01	Stop of O2 and H2 logging.	/	/
	19:03	Deassembling the setup, cleaning the reactor.	Tips of the sensors and reactor were covered with attached photocatalyst particles, bubbles on the surface of the sensor tips. 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).	20251103_190250-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-331-Logger3

For feasibility, loggings used in calibration and photocatalytic test are highlighted in green

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation

03.11.2025	11:10	NB-331-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-331-Logger1-pre-polarization.csv	/	NB-331-Logger-pre-polarization.bmp	/	Pre-polarization.
	13:57	NB-331-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-331-Logger2.csv	/	NB-331-Logger2.bmp	/	Unsuccessful H2 purging --> replacing of GL14 cap required.
	15:23	NB-331-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-331-Logger3-calibration.csv	/	NB-331-Logger3-2 point calibration.bmp NB-331-Logger3-calibration step.bmp	/	2-point calibration, slope: 1.072, 796 uM at 10^6 ppm H2
	18:26	NB-331-Logger4	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-331-Logger4-during irradiation.csv	NB-331-1.py	NB-331-Logger4-during irradiation.bmp	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	During irradiation.
	13:56	NB-331-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-03_135629_NB-331-Ch2-1.txt	/	2025-11-03_135629_NB-331-Ch2-1.png	/	First degassing of water.
	15:21	NB-331-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-03_152144_NB-331-Ch2-2.txt	/	2025-11-03_152144_NB-331-Ch2-2.png	/	Second degassing and calibration of H2 sensor.
	17:44	NB-331-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-03_174410_NB-331-Ch2-3.txt	/	2025-11-03_174410_NB-331-Ch2-3.png	/	Degassing of the suspension.
	18:26	NB-331-Ch2-4	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-03_182612_NB-331-Ch2-4.txt	NB-331-1.py	2025-11-03_182612_NB-331-Ch2-4.png	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	Photocatalytic test (initial rate of O2 formation is faster than H2, afterwards close t 1:1 ratio).

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 (50 mW/cm^2, 20 °C**,** 15 min) were performed, reference conditions.

initial rate of O2 formation (first minutes of irradiation) is faster than H2, afterwards close to 1:1 ratio, some steps in O2 curve, should be monitored afterwards.

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-319: 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)

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

SrTiO3 - NB-329: 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 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-331.ulog
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NB-331-O2-H2-curve.png
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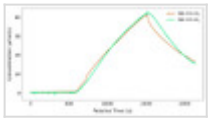

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NB-331-Logger4-during irradiation.csv
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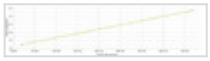

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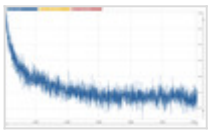

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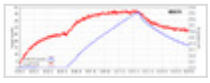

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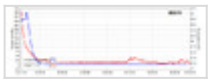

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