

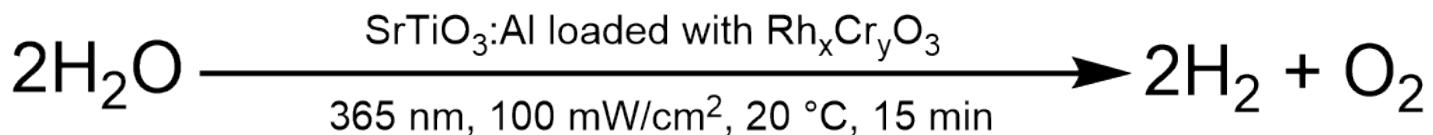
NB-318: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm², 20 °C

Date: 2025-10-21
Tags: O₂ Test Calibration NB Firesting
O₂ sensor H₂ SrTiO₃ Unisense
RhCrO_x: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

Simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh_xCr_yO₃ 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:

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO ₃ - NB-316: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C SrTiO ₃ - NB-315: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 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 ₂ O	/	/	/	/	/	/	/	0.998	25.00	/
Al:SrTiO ₃ RhCrO _x (EA-358)	SrTiO ₃ - EA-358: Modification of Al:SrTiO ₃ (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	12.50	12.77	/	/	/	/

Excel sheet for reagent calculation

/

Irradiation Parameters

Power measurement was performed using [Power Meter - 843-R-USB + 919P-020-12](#) in [Equipment - Advanced power measurement 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. 600 s 2. 01:03:09
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1525 s 2. 01:18:34

O₂/H₂ 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 O ₂ measurement	Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor
Used H ₂ sensor	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
20.10.2025		Calibration from experiment SrTiO ₃ - NB-320: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C (reproduction NB-316) I was used.		
		Sample preparation		
	23:05	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid	/
	23:15	Addition of 25 mL H ₂ O to the vial via graduated cylinder, covering with Al foil before further use.	/	/
	23:15-18	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251020_231915-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor from step 6		
21:10.2025	ca. 0:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s).	/	/
	0:05	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	20251021_000834-before degassing and irradiation.jpg

	0:10	Start of O2 logging.	NB-318-Ch2-1	2025-10-21_001003_NB-318-Ch2-1.txt 2025-10-21_001003_NB-318-Ch2-1.png
	0:13	The degassing was started	/	20251021_001540-degassing of the suspension.jpg
	ca. 0:40	Cannula was transferred to gas phase, above the suspension.	/	/
	0:44	H ₂ sensor was added in Ar counterflow.	/	/
	0:48	The degassing was stopped by removing the cannula and closing the valve.	/	/
	0:52	Stop of O2 logging.	/	/
	0:53	Start of O2 logging.	NB-318-Ch2-2 Leakage rate ca. 0.05 uM in 1.5 min	2025-10-21_005309_NB-318-Ch2-2.txt 2025-10-21_005309_NB-318-Ch2-2.png
	0:53	Start of H2 logging.	NB-318-Logger1	NB-318.ulog NB-318-Logger1.csv NB-318-Logger1.bmp
	0:53-1:03	Equilibration time.	/	/
	1:03	The irradiation was started	A bit step-like behaviour in O ₂ curve	20251021_010323-after start of irradiation.jpg
	1:18	The irradiation was stopped.	/	/
	1:18-28	Equilibration time.	/	/
	1:28	Stop of O2 and H2 logging.	/	/

	ca. 1:30	Deassembling the setup, cleaning the reactor.	<p>Tips of the sensors and reactor were covered with attached photocatalyst particles.</p> <p>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).</p>	20251021_013206-after irradiation.jpg
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Analysis

Used calibration for Firesting: [20250611-BOLA-fitting-liquid-phase-trace-oxygen-sensor-H2-O2 reactor.ini](#)

Used calibration for UniSense: NB-315-Logger3

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
21.10.2025	0:53	NB-318-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-318.ulog NB-318-Logger1.csv	NB-318-O2 and H2 curve.py	NB-318-Logger1.bmp NB-318-O2 and H2 curves.png	/	Clean response of H2 sensor, ca. 92 uM H2 at the end of irradiation.
20.10.2025	0:10	NB-318-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-21_001003_NB-318-Ch2-1.txt	/	2025-10-21_001003_NB-318-Ch2-1.png	/	Degassing of the suspension.
	0:53	NB-318-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-21_005309_NB-318-Ch2-2.txt	NB-318-O2 and H2 curve.py	2025-10-21_005309_NB-318-Ch2-2.png NB-318-O2 and H2 curves.png	/	O2 curve during irradiation, O2 value ca. 75 uM at the end of irradiation.

Results

Simultaneous H₂ and O₂ measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O₂/H₂ photoreactor under 365 nm irradiation (100 mW/cm², 20 °C, 15 min) were performed .

H₂ level at the end of irradiation - 92 umol/L, O₂ level - 75 umol/L.

Linked experiments

SrTiO₃ - NB-315: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C

SrTiO₃ - NB-316: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 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](#)

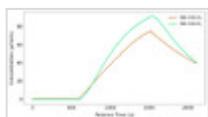
Protocol - [Liquid phase calibration of H₂ UniAmp sensor](#)

Protocol - [In-situ hydrogen and oxygen measurement in H₂/O₂ reactor](#)

Attached files

NB-318-O2 and H2 curves.png

sha256: 6bf279fdb2760cd7bf6ee45bf61d484fc62ac7fbbca1d2e0cc7806d02ccdc66c



NB-318-O2 and H2 curve.py

sha256: f29a54633e064d412a4574769d8c1d150d21604e778027dde24b3848d2baaec3

Unisense-NB-318-screenshot.png

sha256: 6da5772f16b4ca8201360746a763a7556344eaad169fdfbf8e10623fdf640eff



NB-318-SrTiO₃-photocatalytic H₂O splitting.png

sha256: 7408e07503f6e62c3e9b953bcd65664c695e64e9805ca7b6def802182c54f077



NB-318-SrTiO₃-photocatalytic H₂O splitting.cdxml

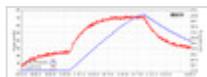
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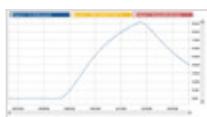
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NB-318.ulog

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NB-318-Logger1.bmp

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NB-318-Logger1.csv

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20251020_231915-suspension after vortex.jpg

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20251021_000834-before degassing and irradiation.jpg

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20251021_001540-degassing of the suspension.jpg

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20251021_013206-after irradiation.jpg

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20251021_010323-after start of irradiation.jpg

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3240>