

NB-329: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) I I

Date: 2025-10-29

Tags: O₂ Test Calibration NB Firing
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

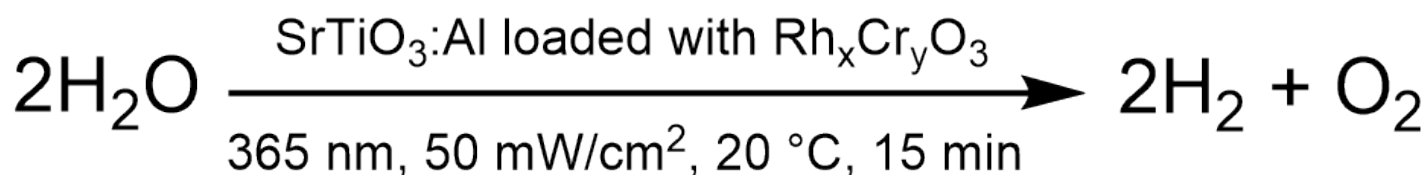
Status: Done

Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-316 - simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh_xCrO_y:Al:SrTiO₃ suspension (EA-358, 0.5 mg/mL), 365 nm LED, 50 mW/cm², 20 °C.

Reaction scheme



ChemDraw file linked: [NB-329-SrTiO3-photocatalytic H2O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	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 SrTiO₃ - NB-320: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) I
Similar experiments	SrTiO₃ - NB-313: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW

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 ₂ O	/	/	/	/	/	/	/	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	12.69	/	/	/	/	/
Hydrogen	/	/	/	/	/	/	/	/	2 balloons (ca. 2 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]	20

Stirring speed [rpm]	500
Irradiation start: 1. Firesting [relative to start log] 2. Unisense	1. 610 s 2. 19:47:02
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1562 s 2. 20:02:54

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 measurment	Protocol - In-situ hydrogen and oxygen measurment 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 measurment in H ₂ /O ₂ reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
29.10.2025		Calibration data was used from experiment SrTiO ₃ - NB-316: Liquid phase H ₂ and O ₂ of RhCrOx,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C.	/	/
		Sample preparation		
	18:25	Weighing EA-358 photocatalyst in a 50 mL vial.	/	/
	ca. 18:32	Addition of 25 mL H ₂ O to the vial via graduated cylinder.	/	/
	18:34-37	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	20251029_183725-suspension after vortex.jpg

		Continue in Protocol - In-situ hydrogen and oxygen measurement in H2/O2 reactor from step 6		
	18:40	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	/
	ca. 18:45	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	18:48	Start of O2 logging.	NB-329-Ch2-1	2025-10-29_184846_NB-329-Ch2-1.txt 2025-10-29_184846_NB-329-Ch2-1.png
	18:52	The degassing was started	/	20251029_185556-during degassing of the suspension.jpg
	19:28	Cannula was transferred to gas phase, above the suspension.	/	/
	19:32	H ₂ sensor was added in Ar counterflow.	/	/
	19:35	The degassing was stopped by removing the cannula and closing the valve.	/	/
	19:36	Stop of O2 logging.	/	/
	19:36	Start of O2 logging.	NB-329-Ch2-2	2025-10-29_193651_NB-329-Ch2-2.txt 2025-10-29_193651_NB-329-Ch2-2.png
	19:36	Start of H2 logging.	NB-329-Logger1	NB-329.ulog NB-329-Logger1.csv NB-329-Logger1.bmp
	19:36-47	Equilibration time.	/	/
	19:47	The irradiation was started	19:56 and 20:00 - change in H ₂ signal (no external impact on the system, most probably caused by attachment/detachment of bubble).	20251029_194850-after start of irradiation.jpg
	20:03	The irradiation was stopped.	/	/
	20:03-12	Equilibration time.	/	/

	20:12	Stop of O2 and H2 logging.	/	/
	ca. 20:15-30	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).	20251029_201328-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
29.10.2025	19:36	NB-329-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-329.ulog NB-329-Logger1.csv	NB-329-O2 and H2 curve.py	NB-329-Logger1.bmp NB-329-O2 and H2 curves.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	Clean response, but at 19:56 - change in signal intensity (decrease and afterwards increase), at 20:00 - again, most probably caused by bubble(s) attached/detached to/from the tip surface of the electrode, 52 uM H2 at the end of irradiation.
	18:48	NB-329-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-29_184846_NB-329-Ch2-1.txt	/	2025-10-29_184846_NB-329-Ch2-1.png	/	Degassing of the suspension.
	19:36	NB-329-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-29_193651_NB-329-Ch2-2.txt	NB-329-O2 and H2 curve.py	2025-10-29_193651_NB-329-Ch2-2.png NB-329-O2 and H2 curves.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	Changes in O2 value during irradiation, 42 uM O2 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 (50 mW/cm², 20 °C, 15 min) were performed.

H₂ level at the end of irradiation - 52 umol/L, O₂ level - 42 umol/L.

Linked experiments

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

SrTiO₃ - NB-319: Liquid phase H₂ and O₂ of RhCrO_x,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 RhCrO_x,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 \(CEEC II, E002\)](#)

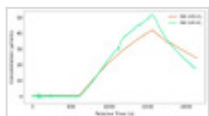
Protocol - [Getting hydrogen from hydrogen bottle in CEEC II E014](#)

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

Attached files

NB-329-O₂ and H₂ curves.png

sha256: b2678d1613fc48456e3dad22a827cac8a6c9c5fb9da805521ce2c73549285728



NB-329-O₂ and H₂ curve.py

sha256: 9181c042ca5c1fc696f2f66ac713ea5d80ba6785014993d3053707d22bb4ed9d

NB-329-SrTiO₃-photocatalytic H₂O splitting.cdxml

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NB-329-SrTiO₃-photocatalytic H₂O splitting.png

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20251029_201328-after irradiation.jpg

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20251029_185556-during degassing of the suspension.jpg

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20251029_183725-suspension after vortex.jpg

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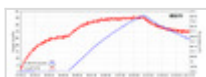
20251029_194850-after start of irradiation.jpg

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2025-10-29_193651_NB-329-Ch2-2.png

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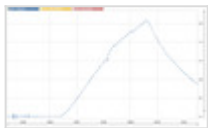


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NB-329-Logger1.bmp

sha256: 118b14fe146c4391889c9eb34c9f34f03a12733a97af4550c3ab222c17b15e4b



NB-329.ulog

sha256: 4b165abf4521ed092f34ebe940166048a10d54e12a981e201d49d2d3e74a819f

NB-329-Logger1.csv

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3299>