

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

Date: 2025-10-30
Tags: O₂ Test Calibration NB Firesting O₂ sensor H₂ SrTiO₃ Unisense RhCrO_x:Al:SrTiO₃ H₂ Sensor temperature In situ H₂/O₂ reactor Trace range robust oxygen sensor photocatalysis Unisense normal range

Category: SrTiO₃

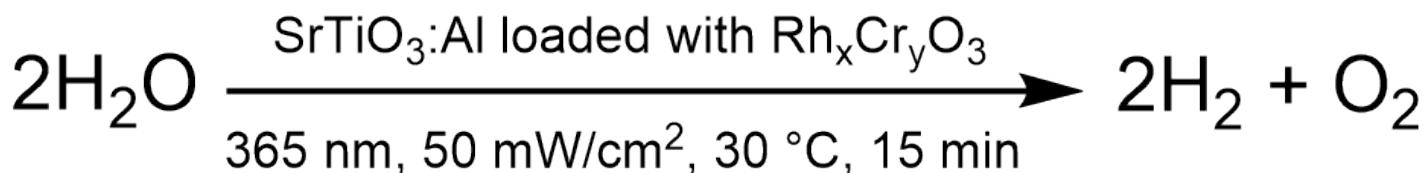
Status: Done

Created by: Nadzeya Brezhneva

Objectives

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, 50 mW/cm², 30 °C.

Reaction scheme



ChemDraw file linked: [NB-330-SrTiO₃-photocatalytic H₂O splitting.cdxml](#)

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

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: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	12.41	/	/	/	/	/

Hydrogen	/	/	/	/	/	/	/	/	2 balloons (ca. 2 L)	ca. 1	/
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Excel sheet for reagent calculation

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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-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-18000-1 4	365	56	0.19

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	30
Stirring speed [rpm]	500
Irradiation start: 1. Firesting [relative to start log] 2. Unisense	1. 601 s 2. 18:25:20

Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1520 s 2. 18:40:39
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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
30.10.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	10:58-11:57	Conditioning of H ₂ sensor	NB-330-Logger1 2 mV at the end of polarization procedure	NB-330.ulog NB-330-Logger1-pre-polarization.csv NB-330-Logger1-pre-polarization.bmp
	12:45-55	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 30 °C) done according to Protocol - Liquid phase calibration of H₂ UniAmp sensor with H₂ bubbling.	/	/
	14:42	Start of O ₂ logging.	NB-330-Ch2-1	2025-10-30_144208_NB-330-Ch2-1.txt 2025-10-30_144208_NB-330-Ch2-1.png

	14:42	Start of H2 logging.	NB-330-Logger2 offset -2 mV	NB-330.ulog NB-330-Logger2-calibration.csv NB-330-Logger2-calibration curve.bmp NB-330-Logger2-2 point calibration.bmp
	14:44	Degassing was started.	14:49 - moving PT100 sensor closer to double walled beaker	20251030_144557-degassing water.jpg
	15:38	Introducing H2 sensor into the reactor under Ar flow.	Offset -4 mV	/
	15:56	0 ppm was taken.	/	/
	15:57	H2 bubbling of the reactor was started	/	20251030_155759-calibration with H2 balloon.jpg
	16:03	Introducing second H2 balloon.	/	20251030_160417-after introducing 2nd balloon.jpg
	16:21	1.000.000 ppm point was taken and calibration was saved	984 mV, slope: 1.324, 743.616 μ M	20251030_162136-H2 table calibration.jpg
	16:23	Stop of H2 logging.	/	/
	16:23	Stop of O2 logging.	/	/
	ca. 16:25-30	Deassembling the setup, drying the reactor with acetone and compressed air .	/	
	Sample preparation			
	17:10	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid	/
	17:12	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	17:14-17	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer , stage 4/6), covered with Al foil before further use.	/	20251030_171729-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		

	17:20	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	ca.17:25	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	/
	17:20-39	Stirring of the suspension at 30 °C.	/	/
	17:39	Start of O ₂ logging.	NB-330-Ch2-2	2025-10-30_173934_NB-330-Ch2-2.txt 2025-10-30_173934_NB-330-Ch2-2.png
	17:42	The degassing was started	/	20251030_174229-during degassing.jpg
	18:07	Cannula was transferred to gas phase, above the suspension.	/	/
	18:09	H ₂ sensor was added in Ar counterflow.	/	20251030_181222-after introducing H2 sensor.jpg
	18:13	The degassing was stopped by removing the cannula and closing the valve.	/	/
	18:15	Stop of O ₂ logging.	/	/
	18:15	Start of O ₂ logging.	NB-330-Ch2-3	2025-10-30_181518_NB-330-Ch2-3.txt 2025-10-30_181518_NB-330-Ch2-3.png
	18:15	Start of H ₂ logging.	NB-330-Logger3	NB-330.ulog NB-330-Logger3-during irradiation.csv NB-330-Logger3-during irradiation.bmp
	18:15-25	Equilibration time.	/	/
	18:25	The irradiation was started	/	20251030_182544-after start of irradiation.jpg
	18:40	The irradiation was stopped.	/	/
	18:40-50	Equilibration time.	/	/
	18:50	Stop of O ₂ and H ₂ logging.	/	/

	ca. 19:00	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).	20251030_185122-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-330-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
30.10.2025	10:58-11:57	NB-330-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-330.ulog NB-330-Logger1-pre-polarization.csv	/	NB-330-Logger1-pre-polarization.bmp	/	2 mV at the end of polarization
	14:42-16:23	NB-330-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-330.ulog NB-330-Logger2-calibration.csv	/	NB-330-Logger2-2 point calibration.bmp NB-330-Logger2-calibration curve.bmp	/	Calibration with 1.000.000 ppm pint: slope of 1.324
	18:15-50	NB-330-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-330.ulog NB-330-Logger3-during irradiation.csv	NB-330-O2 and H2 curve.py	NB-330-Logger3-during irradiation.bmp NB-330-O2 and H2 curve.png	/	worked, clean signal response, 60 uM at the end of irradiation
	14:42-16:23	NB-330-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-30_144208_NB-330-Ch2-1.txt	/	2025-10-30_144208_NB-330-Ch2-1.png	/	O2 measurements during H2 calibration (degassing with Ar and purging with H2 afterwards).
	17:39-18:15	NB-330-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-30_173934_NB-330-Ch2-2.txt	/	2025-10-30_173934_NB-330-Ch2-2.png	/	Degassing of the photocatalyst suspension.
	18:15-50	NB-330-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-30_181518_NB-330-Ch2-3.txt	NB-330-O2 and H2 curve.py	2025-10-30_181518_NB-330-Ch2-3.png NB-330-O2 and H2 curve.png	/	worked, 47 umol/L 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, **30 °C**, 15 min) were performed.

H₂ level at the end of irradiation - 60 umol/L, O₂ level - 47 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-318: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm², 20 °C

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

Linked resources

Equipment - [VWR® VV3, Vortex Mixer](#)

Equipment - [Firesting Fiber-Optic Oxygen Meter 2 Channel \(Firesting 2\)](#)

Equipment - [Advanced power measurement 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](#)

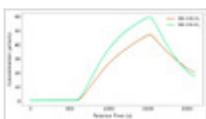
Protocol - [Liquid phase calibration of H₂ UniAmp sensor](#)

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

Attached files

NB-330-O2 and H2 curve.png

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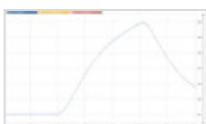


NB-330-O2 and H2 curve.py

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NB-330-Logger3-during irradiation.bmp

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NB-330-Logger3-during irradiation.csv

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NB-330-SrTiO3-photocatalytic H2O splitting.cdxml

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NB-330-SrTiO3-photocatalytic H2O splitting.png

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20251030_144557-degassing water.jpg

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20251030_155759-calibration with H2 balloon.jpg

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20251030_160417-after introducing 2nd balloon.jpg

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20251030_162136-H2 table calibration.jpg

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20251030_171729-suspension after vortex.jpg

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20251030_174229-during degassing.jpg

sha256: f74f18c682868836489a19bc8dff81be66c6bd092a538655304ebcb87b8a512



20251030_181222-after introducing H2 sensor.jpg

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20251030_182544-after start of irradiation.jpg

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20251030_185122-after irradiation.jpg

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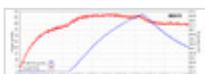


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2025-10-30_173934_NB-330-Ch2-2.txt

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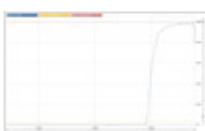
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NB-330-Logger2-calibration curve.bmp

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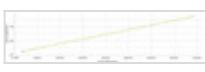


NB-330-Logger2-calibration.csv

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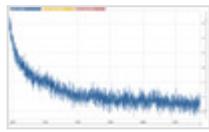
NB-330-Logger2-2 point calibration.bmp

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NB-330-Logger1-pre-polarization.bmp

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NB-330-Logger1-pre-polarization.csv

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NB-330.ulog

sha256: 6af05451a1cbd6c152c5341d2d1c2bc80fbb3acb6a407383899d2a5c41c62e0f



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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3300>