

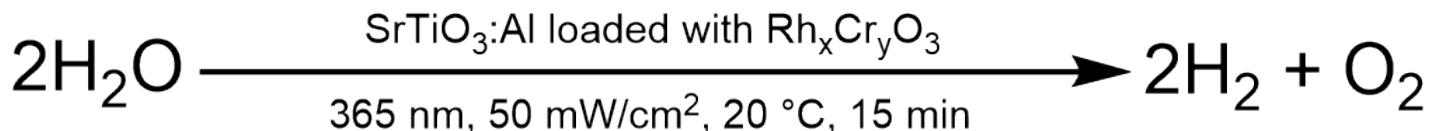
NB-336: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C V (reproduction NB-316)

Date: 2025-11-10
Tags: O₂ Test Calibration Future NB
Firesting O₂ sensor H₂ SrTiO₃
troubleshooting Unisense
RhCrO₃: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

Reproduction of NB-316: simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:SrTiO₃ suspension (EA-358 sample, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C, 15 min (reference conditions).

Reaction scheme



ChemDraw file linked: [NB-316-SrTiO₃-photocatalytic H₂O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	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
Similar experiments	SrTiO ₃ - NB-313: 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 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]	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	12.67	/	/	/	/	/	/
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 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-18000-14	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. 17:45:13
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1508 s 2. 18:00:21

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
10.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	11:29-12:50	Conditioning of H ₂ sensor	NB-336-Logger1 1.7 mV at the end of polarization procedure	NB-336.ulog NB-336-Logger1-pre-polarization.csv NB-336-Logger1-pre-polarization.bmp
	ca. 13:00-20	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 H ₂ bubbling.	Now PT100 is recognized with Firesting without problems, no errors	/

	13:46	Start of O2 logging.	NB-336-Ch2-1	2025-11-10_134636_NB-336-Ch2-1.txt 2025-11-10_134636_NB-336-Ch2-1.png
	13:46	Start of H2 logging.	NB-336-Logger2 offset -2 mV	NB-336.ulog NB-336-Logger2-calibration.csv NB-336-2-point calibration.bmp NB-336-Logger2-calibration step.bmp
	13:49	Degassing was started.	/	20251110_135012-degassing of water.jpg
	14:23	Moving cannula to gas phase, above liquid.	/	/
	14:25	Introducing H2 sensor into the reactor under Ar flow.	/	/
	14:26	0 ppm was taken.	/	/
	14:36	H2 bubbling of the reactor was started	First needle of the H2 balloon - no bubbling, Replacing the needle with the new one - start bubbling	/
	14:44	Introducing second H2 balloon.	/	20251110_144451-two H2 balloons.jpg
	14:52	1.000.000 ppm point was taken and calibration was saved	826 mV, slope: 1.043, 790.065 μ M	20251110_145047-H2 table.jpg
	14:53	Stop of H2 logging.	/	/
	15:00	Stop of O2 logging.	/	/
	ca. 15:20	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
	Sample preparation			
	16:35	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	16:42	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/

	16:42-45	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer , stage 4/6), covered with Al foil before further use.	breaking residual lumps manually with Smartspatula in the vial.	20251110_164831-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H2/O2 reactor from step 6		
	ca. 16:50	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	16:55	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	20251110_165652-before degassing and irradiation.jpg
	16:58	Start of O ₂ logging.	NB-336-Ch2-2	2025-11-10_165836_NB-336-Ch2-2.txt 2025-11-10_165836_NB-336-Ch2-2.png
	17:00	The degassing was started	/	20251110_170124-degassing of the suspension.jpg
	17:28	Cannula was transferred to gas phase, above the suspension.	/	/
	17:30	H ₂ sensor was added in Ar counterflow.	/	/
	17:34	The degassing was stopped by removing the cannula and closing the valve.	/	/
	17:34	Stop of O ₂ logging.	/	/
	17:35	Start of O ₂ logging.	NB-336-Ch2-3	2025-11-10_173512_NB-336-Ch2-3.txt 2025-11-10_173512_NB-336-Ch2-3.png
	17:35	Start of H ₂ logging.	NB-336-Logger3	NB-336.ulog NB-336-Logger3-during irradiation.csv NB-336-Logger3-during irradiation.bmp
	17:35-45	Equilibration time.	/	/

	17:45	The irradiation was started	/	20251110_174514.after start of irradiation.jpg
	18:00	The irradiation was stopped.	/	/
	18:00-10	Equilibration time.	/	/
	18:10	Stop of O ₂ and H ₂ 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).	/

Analysis

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

Used calibration for UniSense: NB-336-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
10.11.2025	11:29	NB-336-Logger1	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-336.ulog NB-336-Logger1-pre-polarization.csv	/	NB-336-Logger1-pre-polarization.bmp	/	Conditioning of sensor.
	13:46	NB-336-Logger2	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-336.ulog NB-336-Logger2-calibration.csv	/	NB-336-2-point calibration.bmp NB-336-Logger2-calibration step.bmp	/	2-point calibration, slope 1.043

	17:35	NB-336-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-336.ulog NB-336-Logger3-during irradiation.csv	NB-336-O2 and H2 curves.py	NB-336-Logger3-during irradiation.bmp NB-336-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	Sharp increase and decrease at the beginning and end of irradiation.
	13:46	NB-336-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-10_134636_NB-336-Ch2-1.txt	/	2025-11-10_134636_NB-336-Ch2-1.png	/	Degassing followed by calibration of H2 sensor.
	16:58	NB-336-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-10_165836_NB-336-Ch2-2.txt	/	2025-11-10_165836_NB-336-Ch2-2.png	/	Degassing of the suspension.
	17:35	NB-336-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-10_173512_NB-336-Ch2-3.txt	NB-336-O2 and H2 curves.py	2025-11-10_173512_NB-336-Ch2-3.png NB-336-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	Change in O2 value during irradiation, very slow O2 decrease rate after switching irradiation off.

Results

Reproduction of NB-316 was performed. 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, reference conditions) were performed.

Sharp increase/decrease in the H₂ signal after start and end of irradiation, Slow decrease rate of O₂ in post-irradiation equilibration.

Future recommendations

Old procedure	Problem	Suggested new procedure
/	Sharp increase in H ₂ signal.	Adjust the position of H ₂ sensor in the reactor.

Linked experiments

SrTiO3 - NB-313: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 0.5 mg/mL), 365 nm, 50 mW

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

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

Linked resources

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](#)

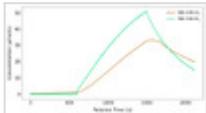
Protocol - [Liquid phase calibration of H2 UniAmp sensor](#)

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

Attached files

NB-336-O2 and H2 curves.png

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NB-336-O2 and H2 curves.py

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20251110_135012-degassing of water.jpg

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20251110_144451-two H2 balloons.jpg

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20251110_145047-H2 table.jpg

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20251110_164831-suspension after vortex.jpg

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20251110_165652-before degassing and irradiation.jpg

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20251110_170124-degassing of the suspension.jpg

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20251110_174514.after start of irradiation.jpg

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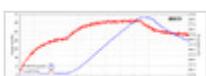


2025-11-10_173512_NB-336-Ch2-3.txt

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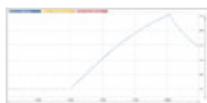


NB-336-Logger3-during irradiation.csv

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NB-336-Logger3-during irradiation.bmp

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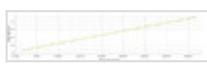


NB-336-Logger2-calibration.csv

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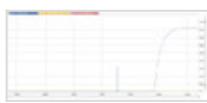
NB-336-2-point calibration.bmp

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NB-336-Logger2-calibration step.bmp

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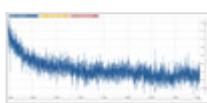


NB-336-Logger1-pre-polarization.csv

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NB-336-Logger1-pre-polarization.bmp

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NB-336.ulog

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3403>