

NB-348: Liquid phase H₂ and O₂ of RhCrO_x:Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C VII (reproduction NB-316)

Date: 2025-11-13
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

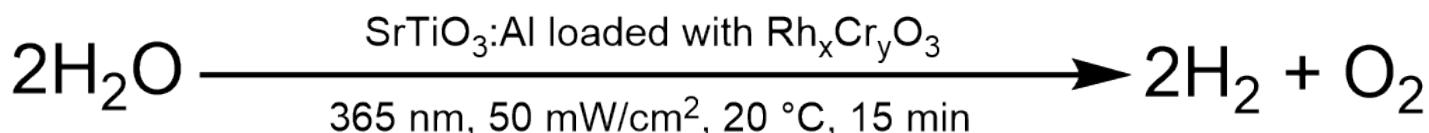
Status: Done

Created by: Nadzeya Brezhneva

Objectives

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	Inventor y number	Amount [mmol]	Equivale nts	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Pressure [bar]	Concentrati on [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)	/	/	/	12.5	12.45	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (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 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. 603 s 2. 17:37:25
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1520 s 2. 17:52:42

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
13.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	12:14-14:00	Conditioning of H ₂ sensor	NB-348-Logger1 ca. 1.8 mV at the end of polarization procedure	NB-348.ulog NB-348-Logger1-pre-polarization.csv NB-348-Logger1-pre-polarization.bmp
	14:10	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.	/	/

	14:33	Start of O2 logging.	NB-348-Ch2-1	2025-11-13_143340_NB-348-Ch2-1.txt 2025-11-13_143340_NB-348-Ch2-1.png
	14:33	Start of H2 logging.	NB-348-Logger2 offset - 2 mV	NB-348.ulog NB-348-Logger2-calibration.csv NB-348-Logger2-calibration step.bmp NB-348-Logger2-2point calibration curve.bmp
	14:36	Degassing was started.	/	20251113_143712-degassing of water.jpg
	15:02	Moving cannula to gas phase above the liquid.	/	/
	15:05	Introducing H2 sensor into the reactor under Ar flow.	/	/
	15:10	0 ppm was taken.	/	/
	15:17	Removing cannula, closing the valve.	/	/
	15:18	H2 bubbling of the reactor was started	/	20251113_151843-H2 bubbling.jpg
	15:38	1.000.000 ppm point was taken	846 mV	/
	15:42	Deleting the previous point, taking the new one, calibration was saved.	847 mV, slope: 1.074, 788 μ M	20251113_153640-H2 table.jpg
	15:43	Stop of H2 logging.	/	/
	15:43	Stop of O2 logging.	/	/
	ca. 16:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
	Sample preparation			
	16:25	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	16:27	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	16:27-30	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251113_163014-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		

	ca. 16:40	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	
	16:45	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	
	16:47	Start of O ₂ logging.	NB-348-Ch2-2	2025-11-13_164739_NB-348-Ch2-2.txt 2025-11-13_164739_NB-348-Ch2-2.png
	16:49	The degassing was started	/	20251113_165040-degassing of the suspension.jpg
	17:17	Cannula was transferred to gas phase, above the suspension.	/	/
	17:19	H ₂ sensor was added in Ar counterflow.	/	/
	17:24	The degassing was stopped by removing the cannula and closing the valve.	/	/
	17:26	Stop of O ₂ logging.	/	/
	17:27	Start of O ₂ logging.	NB-348-Ch2-3	2025-11-13_172722_NB-348-Ch2-3.txt 2025-11-13_172722_NB-348-Ch2-3.png
	17:27	Start of H ₂ logging.	NB-348-Logger3	NB-348.ulog NB-348-Logger3-during irradiation.csv NB-348-Logger3-during irradiation.bmp
	17:27-37	Equilibration time.	/	/
	17:37	The irradiation was started	/	20251113_174011-after start of irradiation.jpg
	17:52	The irradiation was stopped.	/	/
	17:52-18:02	Equilibration time.	/	/

	18:02	Stop of O ₂ and H ₂ logging.	/	/
	ca. 18:20	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>	20251113_181137-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-348-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
13.11.2025	12:14	NB-348-Logger1	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-348.ulog NB-348-Logger1-pre-polarization.csv	/	NB-348-Logger1-pre-polarization.bmp	/	Pre-polarization of the H ₂ sensor.
	14:33	NB-348-Logger2	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-348.ulog NB-348-Logger2-calibration.csv	/	NB-348-Logger2-calibration step.bmp NB-348-Logger2-2point calibration curve.bmp	/	Calibration, 10 ⁶ ppm corresponds to 847 mV, slope 1.074
	17:27	NB-348-Logger3	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-348.ulog NB-348-Logger3-during irradiation.csv	NB-348-O2 and H ₂ curve.py	NB-348-Logger3-during irradiation.bmp NB-348-O2 and H ₂ curve.png	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)	H ₂ evolution during irradiation of the suspension, small step in signal during irradiation.
	14:33	NB-348-Ch2-1	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-13_143340_NB-348-Ch2-1.txt	/	2025-11-13_143340_NB-348-Ch2-1.png	/	Degassing of water followed by calibration of H ₂ sensor.

	16:47	NB-348-Ch2-2	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-13_164739_NB-348-Ch2-2.txt	/	2025-11-13_164739_NB-348-Ch2-2.png	/	Degassing of the suspension.
	17:27	NB-348-Ch2-3	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-13_172722_NB-348-Ch2-3.txt	NB-348-O ₂ and H ₂ curve.py	2025-11-13_172722_NB-348-Ch2-3.png	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	O ₂ evolution during irradiation.

Results

Reproduction of NB-316 - 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) - was performed.

Linked experiments

[SrTiO₃ - NB-313: 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-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](#)

[SrTiO₃ - 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 \(reproduction NB-316\)](#)

[SrTiO₃ - NB-339: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C VI \(reproduction NB-316\)](#)

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](#)

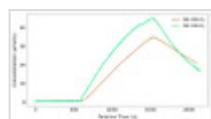
Attached files

[NB-348-O₂ and H₂ curve.py](#)

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NB-348-O2 and H2 curve.png

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20251113_153640-H2 table.jpg

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20251113_163014-suspension after vortex.jpg

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20251113_165040-degassing of the suspension.jpg

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20251113_181137-after irradiation.jpg

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20251113_174011-after start of irradiation.jpg

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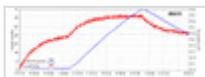


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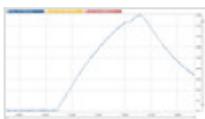


NB-348-Logger3-during irradiation.csv

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NB-348-Logger3-during irradiation.bmp

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NB-348-Logger2-calibration.csv

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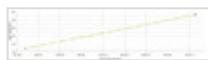
NB-348-Logger2-calibration step.bmp

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NB-348-Logger2-2point calibration curve.bmp

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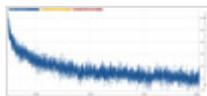


NB-348-Logger1-pre-polarization.csv

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NB-348-Logger1-pre-polarization.bmp

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NB-348.ulog

sha256: dba5e90e51d937bc0765088ed92524211b507061dc7d57e8c4c7688de7efd33d



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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3466>