

# NB-348: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C VII (reproduction NB-316)

Date: 2025-11-13

Tags: O<sub>2</sub> Test Calibration NB Firing  
O<sub>2</sub> sensor H<sub>2</sub> SrTiO<sub>3</sub> Unisense  
RhCrO<sub>3</sub>:Al:SrTiO<sub>3</sub> H<sub>2</sub> Sensor  
temperature In situ Trace range robust  
oxygen sensor photocatalysis Unisense  
normal range

Category: SrTiO<sub>3</sub>

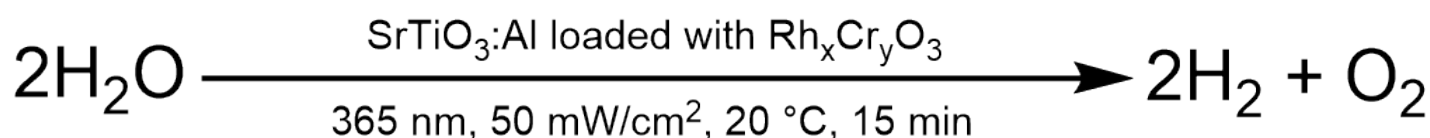
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Reproduction of NB-316: simultaneous detection of H<sub>2</sub> and O<sub>2</sub> evolution in liquid phase for irradiated suspension of Rh,CrOx:Al:SrTiO<sub>3</sub> suspension (EA-358 sample, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 15 min (reference conditions).

## Reaction scheme



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

## Literature/reference experiments

Literature	/
Reproduction	<a href="#">SrTiO<sub>3</sub> - NB-316: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C</a>
Similar experiments	<a href="#">SrTiO<sub>3</sub> - NB-313: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (NB-301, 0.5 mg/mL), 365 nm, 50 mW</a> <a href="#">SrTiO<sub>3</sub> - NB-315: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C</a>

## Reagents

Name	CAS Number / Experiment Number	Inventor y number	Amount [mmol]	Equivalents	Mass <sub>theo</sub> [mg]	Mass <sub>exp</sub> [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Pressure [bar]	Concentration [mM]
milli-Q H <sub>2</sub> 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)	/	/	/	12.5	12.45	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (ca. 1 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	<a href="#">Equipment - Advanced irradiation setup V1.0 I</a>
Irradiation setup number	<a href="#">Equipment - Irradiation setup 4 (CEEC II, E002)</a>

	Light Source Name	Power Source Name	Wavelength [nm]	Power Setting [mW]	Analog Setting [0.00 - 10.00]
First light source	<a href="#">Light Source - UHP LED 365 nm-4</a>	<a href="#">Power Sources - BLS-18000-1 4</a>	365	56	0.19

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	20

<b>Stirring speed [rpm]</b>	500
<b>Irradiation start:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 603 s 2. 17:37:25
<b>Irradiation stop:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 1520 s 2. 17:52:42

## O<sub>2</sub>/H<sub>2</sub> 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 <sub>2</sub> sensor	Equipment - Robust probe for liquid O <sub>2</sub> measurment	Protocol - In-situ hydrogen and oxygen measurment in H <sub>2</sub> /O <sub>2</sub> reactor
Used H <sub>2</sub> sensor	Equipment - H <sub>2</sub> UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurment in H <sub>2</sub> /O <sub>2</sub> reactor

## Procedure/observations

Date	Time	Step	Observations	Pictures/Files
13.11.2025		The experiment was done according to <a href="#">Protocol - In-situ hydrogen and oxygen measurment in H<sub>2</sub>/O<sub>2</sub> reactor</a> Important steps and deviations are listed below	/	/
	12:14-14:00	Conditioning of H <sub>2</sub> sensor	<b>NB-348-Logger1</b> ca. 1.8 mV at the end of polarization procedure	<a href="#">NB-348.ulog</a> <a href="#">NB-348-Logger1-pre-polarization.csv</a> <a href="#">NB-348-Logger1-pre-polarization.bmp</a>
	14:10	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), done according to <a href="#">Protocol - Liquid phase calibration of H<sub>2</sub> UniAmp sensor with H<sub>2</sub> bubbling</a> .	/	/

	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 uM	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 .	/	/
		<b>Sample preparation</b>		
	16:25	Weighing <b>EA-358</b> 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 <sub>2</sub> sensor, PT100, PT1000 and O <sub>2</sub> robust probe are inside the reactor immersed in the liquid phase	
	16:47	Start of O2 logging.	<b>NB-348-Ch2-2</b>	<a href="#">2025-11-13_164739_NB-348-Ch2-2.txt</a> <a href="#">2025-11-13_164739_NB-348-Ch2-2.png</a>
	16:49	The degassing was started	/	<a href="#">20251113_165040-degassing of the suspension.jpg</a>
	17:17	Cannula was transferred to gas phase, above the suspension.	/	/
	17:19	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	17:24	The degassing was stopped by removing the cannula and closing the valve.	/	/
	17:26	Stop of O2 logging.	/	/
	17:27	Start of O2 logging.	<b>NB-348-Ch2-3</b>	<a href="#">2025-11-13_172722_NB-348-Ch2-3.txt</a> <a href="#">2025-11-13_172722_NB-348-Ch2-3.png</a>
	17:27	Start of H2 logging.	<b>NB-348-Logger3</b>	<a href="#">NB-348.ulog</a> <a href="#">NB-348-Logger3-during irradiation.csv</a> <a href="#">NB-348-Logger3-during irradiation.bmp</a>
	17:27-37	Equilibration time.	/	/
	17:37	The irradiation was started	/	<a href="#">20251113_174011-after start of irradiation.jpg</a>
	17:52	The irradiation was stopped.	/	/
	17:52-18:02	Equilibration time.	/	/

	18:02	Stop of O2 and H2 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).	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 H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-348.ulong NB-348-Logger1-pre-polarization.csv	/	NB-348-Logger1-pre-polarization.bmp	/	Pre-polarization of the H2 sensor.
	14:33	NB-348-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-348.ulong NB-348-Logger2-calibration.csv	/	NB-348-Logger2-calibration step.bmp NB-348-Logger2-2point calibration curve.bmp	/	Calibration, 10 <sup>6</sup> ppm corresponds to 847 mV, slope 1.074
	17:27	NB-348-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-348.ulong NB-348-Logger3-during irradiation.csv	NB-348-O2 and H2 curve.py	NB-348-Logger3-during irradiation.bmp NB-348-O2 and H2 curve.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	H2 evolution during irradiation of the suspension, small step in signal during irradiation.
	14:33	NB-348-Ch2-1	Optical O2 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 H2 sensor.

	16:47	NB-348-Ch2-2	Optical O2 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 O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-13_172722_NB-348-Ch2-3.txt	NB-348-O2 and H2 curve.py	2025-11-13_172722_NB-348-Ch2-3.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	O2 evolution during irradiation.

## Results

Reproduction of NB-316 - simultaneous H<sub>2</sub> and O<sub>2</sub> measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O<sub>2</sub>/H<sub>2</sub> photoreactor under 365 nm irradiation (50 mW/cm<sup>2</sup>, 20 °C, 15 min, reference conditions) - was performed.

## 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

SrTiO3 - NB-336: Liquid phase H2 and O2 of RhCrOx,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 RhCrOx,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](#)

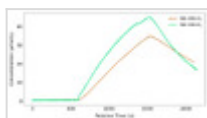
## Attached files

NB-348-O2 and H2 curve.py

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NB-348-O2 and H2 curve.png

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20251113\_143712-degassing of water.jpg

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20251113\_153640-H2 table.jpg

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20251113\_163014-suspension after vortex.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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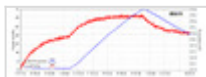


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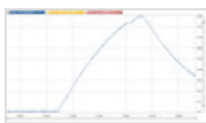


NB-348-Logger3-during irradiation.csv

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NB-348-Logger3-during irradiation.bmp

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NB-348-Logger2-calibration.csv

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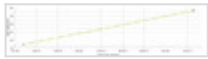
NB-348-Logger2-calibration step.bmp

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

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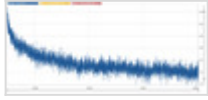


NB-348-Logger1-pre-polarization.csv

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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>