

# NB-349: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>:Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), D<sub>2</sub>O, 365 nm, 50 mW/cm<sup>2</sup>, 20 °C (reproduction NB-346)

Date: 2025-11-13  
Tags: O<sub>2</sub> Test Calibration NB Firesting O<sub>2</sub> sensor H<sub>2</sub> SrTiO<sub>3</sub> Unisense RhCrO<sub>x</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>

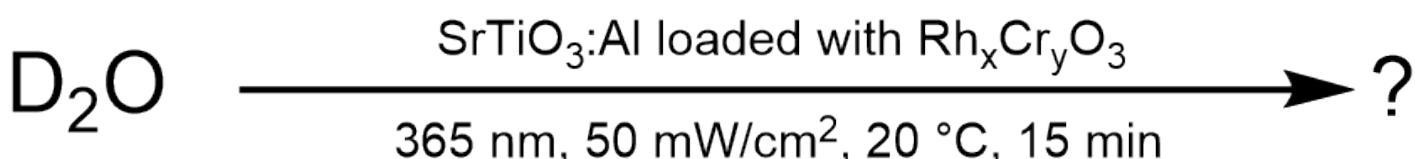
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Reproduction of NB-346: simultaneous detection of H<sub>2</sub> and O<sub>2</sub> evolution in liquid phase for irradiated suspension of Rh,CrO<sub>x</sub>:Al:SrTiO<sub>3</sub> suspension (EA-358, 0.5 mg/mL, D<sub>2</sub>O), 365 nm LED, 50 mW/cm<sup>2</sup>, 20 °C (changing dispersion medium).

## Reaction scheme



ChemDraw file linked: [NB-346-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	SrTiO <sub>3</sub> - NB-346: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> :Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), D <sub>2</sub> O, 365 nm, 50 mW/cm <sup>2</sup> , 20 °C
Similar experiments	SrTiO <sub>3</sub> - NB-316: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> :Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C

## Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	Mass <sub>theo</sub> [mg]	Mass <sub>exp</sub> [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Concentration [mM]
D <sub>2</sub> O, Eurisotop, 99.90%	7789-20-0	C121243	/	/	/	/	/	1.11	25 (for suspension preparation)	
Al:SrTiO <sub>3</sub> RhCrO <sub>x</sub> (EA-358)	SrTiO <sub>3</sub> - EA-358: Modification of Al:SrTiO <sub>3</sub> (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	12.50	12.53	/	/	/	/

# 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	<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]
<b>First light source</b>	<a href="#">Light Source - UHP LED 365 nm-4</a>	<a href="#">Power Sources - BLS-18000-14</a>	365	56	0.19

<b>Used beam combiner [Name or None]</b>	/
<b>Irradiation distance [cm]</b>	6.5
<b>Thermostat temperature [°C]</b>	20
<b>Stirring speed [rpm]</b>	500
<b>Irradiation start:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 0 h : 10 min : 5 s 2. 20:16:18
<b>Irradiation stop:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 0 h : 25 min : 40 s 2. 20:31:52

# 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> measurement	Protocol - In-situ hydrogen and oxygen measurement 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 measurement in H <sub>2</sub> /O <sub>2</sub> reactor

## Procedure/observations

Date	Time	Step	Observations	Pictures/Files
13.11.2025		Calibration from experiment SrTiO <sub>3</sub> - NB-348: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> ,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) was used. Calibration was performed in H <sub>2</sub> O.		
		<b>Sample preparation</b>		
	19:04	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	19:06	Addition of 25 mL D <sub>2</sub> O to the vial via graduated cylinder.	/	/
	19:09-12	The suspension was vortexed for 3 min ( Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251113_191500-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H <sub>2</sub> /O <sub>2</sub> reactor from step 6		
	19:15	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s).	/	/
	19:20	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	/
	19:29	Degassing was started.	/	20251113_193027-degassing of the suspension.jpg

	19:38	Start of O2 logging.	<b>NB-349-Ch2-1</b>	2025-11-13_193836_NB-349-Ch2-1.txt 2025-11-13_193836_NB-349-Ch2-1.png
	19:57	Cannula was transferred to gas phase, above the suspension.	/	/
	19:59	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	20:02	The degassing was stopped by removing the cannula and closing the valve.	/	/
	20:06	Stop of O2 logging.	/	/
	20:06	Start of O2 logging.	<b>NB-349-Ch2-2</b>	2025-11-13_200612_NB-349-Ch2-2.txt 2025-11-13_200612_NB-349-Ch2-2.png
	20:06	Start of H2 logging.	<b>NB-349-Logger1</b>	NB-349.ulog NB-349-Logger1.csv NB-349-Logger1.bmp
	20:06-16	Equilibration time.	/	/
	20:16	The irradiation was started	/	20251113_201650-after start of irradiation.jpg
	20:31	The irradiation was stopped.	/	/
	20:31-41	Equilibration time.	/	/
	20:41	Stop of O2 and H2 logging.	/	/

ca. 21:00		Deassembling the setup, cleaning the reactor.		Tips of the sensors were covered with attached photocatalyst particles. Seems like the particles were less adhesive to the reactor surface than in previous experiments with H <sub>2</sub> O. 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_211928-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	20:06	NB-349-Logger1	electrochemical H <sub>2</sub> detection	Equipment - H <sub>2</sub> UniAmp Sensor - Normal range - 2.1 x 80 mm needle	D2O	NB-349.ulog NB-349-Logger1.csv	NB-349-O2 and H <sub>2</sub> curve.py	NB-349-Logger1.bmp NB-349-O2 and H <sub>2</sub> curves.png	SrTiO <sub>3</sub> - NB-346: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> :Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), D2O, 365 nm, 50 mW/cm <sup>2</sup> , 20 °C	H <sub>2</sub> evolution during irradiation of D <sub>2</sub> O-based suspension.
	19:38	NB-349-Ch2-1	Optical O <sub>2</sub> detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	D2O	2025-11-13_193836_NB-349-Ch2-1.txt	/	2025-11-13_193836_NB-349-Ch2-1.png	/	Degassing of D <sub>2</sub> O-based suspension.
	20:06	NB-346-Ch2-2	Optical O <sub>2</sub> detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	D2O	2025-11-13_200612_NB-349-Ch2-2.txt	NB-349-O2 and H <sub>2</sub> curve.py	2025-11-13_200612_NB-349-Ch2-2.png NB-349-O2 and H <sub>2</sub> curves.png	SrTiO <sub>3</sub> - NB-346: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> :Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), D2O, 365 nm, 50 mW/cm <sup>2</sup> , 20 °C	O <sub>2</sub> evolution during irradiation of D <sub>2</sub> O-based suspension.

## Results

Reproduction of NB-346: simultaneous H<sub>2</sub> and O<sub>2</sub> measurements of irradiated suspension of EA-358 (0.5

mg/mL in D<sub>2</sub>O) in O<sub>2</sub>/H<sub>2</sub> photoreactor under 365 nm irradiation (50 mW/cm<sup>2</sup>, 20 °C, 15 min) were performed with preliminary calibration of H<sub>2</sub> sensor in water.

Both evolution of H<sub>2</sub> and O<sub>2</sub> were observed but with lower rates and amounts produced than in the case of H<sub>2</sub>O-based suspensions.

## Linked experiments

Prep work - NB-234: Liquid phase calibration of O<sub>2</sub> robust probe in H<sub>2</sub>/O<sub>2</sub> photoreactor

Prep work - AE-536: Liquid phase calibration in Argon of normal range H<sub>2</sub> sensor

SrTiO<sub>3</sub> - NB-315: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,Al:SrTiO<sub>3</sub> (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C

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

SrTiO<sub>3</sub> - NB-318: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-344: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 150 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-346: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), D<sub>2</sub>O, 365 nm, 50 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-348: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,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)

## Linked resources

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

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

Equipment - [H2 UniAmp Sensor - Low range - 2.1 x 80 mm needle](#)

Equipment - [Irradiation setup 4 \(CEEC II, E002\)](#)

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

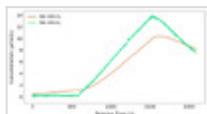
Protocol - [Liquid phase calibration of H2 UniAmp sensor](#)

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

## Attached files

NB-349-O2 and H2 curves.png

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NB-349-O2 and H2 curve.py

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20251113\_201650-after start of irradiation.jpg

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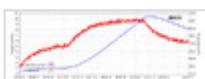


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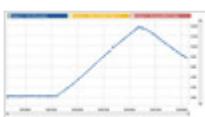
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NB-349-Logger1.csv  
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NB-349-Logger1.bmp  
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NB-349.ulog  
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Unique eLabID: 20251113-2e44ef3b05edded3aa9489d4959cd66b0888926b1  
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3467>