

# NB-337: 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, 20 mW/cm<sup>2</sup>, 20 °C (reproduction NB-325) I

Date: 2025-11-10  
Tags: O<sub>2</sub> Test Calibration Future 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>

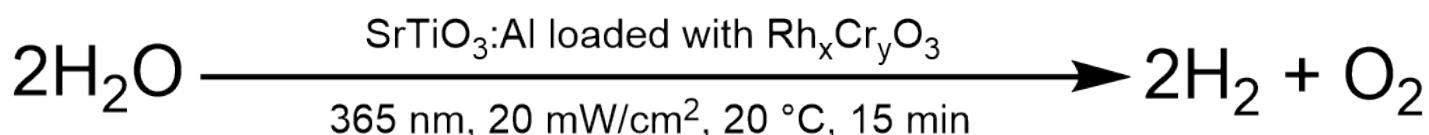
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Reproduction of NB-325: 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), 365 nm LED, 20 mW/cm<sup>2</sup>, 20 °C.

## Reaction scheme



ChemDraw file linked: [NB-325-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	SrTiO <sub>3</sub> - NB-326: 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, 20 mW/cm <sup>2</sup> , 20 °C (reproduction NB-325) SrTiO <sub>3</sub> - NB-325: 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, 20 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]
milli-Q H <sub>2</sub> O	/	/	/	/	/	/	/	0.998	25	/
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	12.68	/	/	/	/

# 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-324: Measuring power output of UHP-365 nm #4 with 18A-4 in advanced irradiation setup II](#)

	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	22	0.07

<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. 602 s 2. 21:41:42
<b>Irradiation stop:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 1527 s 2. 22:07:07

# 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
10.11.2025		Calibration from SrTiO <sub>3</sub> - NB-336: 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 V (reproduction NB-316) was used		
		<b>Sample preparation</b>		
	20:43	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	20:45	Addition of 25 mL H <sub>2</sub> O to the vial via graduated cylinder.	Breaking lumps manually using Smartspatula inside the vial.	/
	20:46-49	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251110_204943-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		
	20:50-55	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s).	/	/
	20:55-21:00	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	/

	21:00	Start of O2 logging.	<b>NB-337-Ch2-1</b>	<a href="#">2025-11-10_210048_NB-337-Ch2-1.txt</a> <a href="#">2025-11-10_210048_NB-337-Ch2-1.png</a>
	21:02	The degassing was started	/	<a href="#">20251110_210323-degassing of the suspension.jpg</a>
	21:32	Cannula was transferred to gas phase, above the suspension.	/	/
	21:34	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	21:39	The degassing was stopped by removing the cannula and closing the valve.	/	/
	ca. 21:40	Stop of O2 logging.	/	/
	21:41	Start of O2 logging.	<b>NB-337-Ch2-2</b>	<a href="#">2025-11-10_214137_NB-337-Ch2-2.txt</a> <a href="#">2025-11-10_214137_NB-337-Ch2-2.png</a>
	21:41	Start of H2 logging.	<b>NB-337-Logger1</b>	NB-337.ulog NB-337-Logger1.csv NB-337-Logger1.bmp
	21:41-51	Equilibration time.	/	/
	21:51	The irradiation was started	/	<a href="#">20251110_215343-after start of irradiation.jpg</a>
	22:07	The irradiation was stopped.	/	/
	22:07-16	Equilibration time.	/	/
	22:16	Stop of O2 and H2 logging.	/	/

	ca. 22:20	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).	/
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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-336-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
10.11.2025	21:41	NB-337-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	<a href="#">NB-337.ulog</a> <a href="#">NB-337-Logger1.csv</a>	<a href="#">NB-337-O2 and H2 curve.py</a>	<a href="#">NB-337-O2 and H2 curve.png</a>	<a href="#">SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C</a>	Sharp increase/decrease in H2 signal after start and stop of irradiation.
	21:00	NB-337-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	<a href="#">2025-11-10_210048_NB-337-Ch2-1.txt</a>	/	<a href="#">2025-11-10_210048_NB-337-Ch2-1.png</a>	/	Degassing of the suspension.
	21:41	NB-337-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	<a href="#">2025-11-10_214137_NB-337-Ch2-2.txt</a>	<a href="#">NB-337-O2 and H2 curve.py</a>	<a href="#">NB-337-O2 and H2 curve.png</a> <a href="#">2025-11-10_214137_NB-337-Ch2-2.png</a>	<a href="#">SrTiO3 - NB-325: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C</a>	Change in O2 value during irradiation.

## Results

Reproduction of NB-325 was performed: 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 (20 mW/cm<sup>2</sup>, 20 °C, 15 min) were performed.

Sharp increase/decrease in H<sub>2</sub> signal after start and end of irradiation.

## Future recommendations

Old procedure	Problem	Suggested new procedure
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/	Sharp increase/decrease in H2 signal.	Adjust the position of H2 sensor.
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## Linked experiments

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-318: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C

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

SrTiO3 - NB-326: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C (reproduction NB-325)

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)

## Linked resources

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

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

## Attached files

NB-325-SrTiO3-photocatalytic H2O splitting.cdxml  
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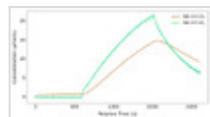
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NB-337-O2 and H2 curve.py

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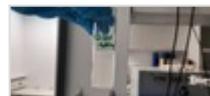
NB-337-O2 and H2 curve.png

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20251110\_204943-suspension after vortex.jpg

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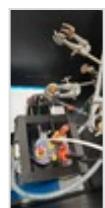
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20251110\_215343-after start of irradiation.jpg

sha256: cfb9df95414d4d3d0a6e2165cd5de642543621a86d7c783a886ac4af8a3c816f

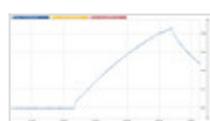


NB-337.ulog

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NB-337-Logger1.bmp

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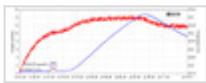


NB-337-Logger1.csv

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3405>