

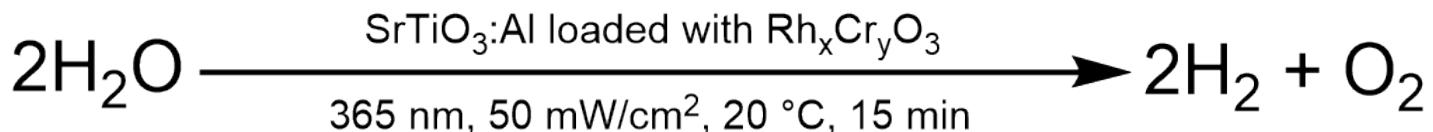
# NB-356: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>:Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C IX (reproduction NB-316)

Date: 2025-11-20  
Tags: O<sub>2</sub> Test Calibration Future NB  
Firesting O<sub>2</sub> sensor H<sub>2</sub> SrTiO<sub>3</sub>  
troubleshooting 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,CrO<sub>x</sub>: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-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	SrTiO <sub>3</sub> - NB-320: 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 (reproduction NB-316) I SrTiO <sub>3</sub> - NB-329: 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 (reproduction NB-316) II SrTiO <sub>3</sub> - NB-331: 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 (reproduction NB-316) III 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) SrTiO <sub>3</sub> - NB-339: 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 VI (reproduction NB-316) 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)
Similar experiments	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

## 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]	Pressure [bar]	Concentration [mM]
milli-Q H <sub>2</sub> O	/	/	/	/	/	/	/	0.998	25 + 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)	/	/	/	12.5	12.60	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (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 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

<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> 1. Firesting [relative to start log] 2. Unisense	1. 600 s 2. 23:10:16
<b>Irradiation stop:</b> 1. Firesting [relative to start log] 2. Unisense	1. 1516 s 2. 23:25:28

## O<sub>2</sub>/H<sub>2</sub> sensor equipment

	<b>Equipment</b>	<b>Used protocol</b>
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
20.11.2025		The experiment was done according to <a href="#">Protocol - In-situ hydrogen and oxygen measurement in H<sub>2</sub>/O<sub>2</sub> reactor</a> Important steps and deviations are listed below	/	/
	10:22-11:36	Conditioning of H <sub>2</sub> sensor	<b>NB-356-Logger1</b> 1.69 mV at the end of polarization procedure	<a href="#">NB-356.ulog</a> <a href="#">NB-356-Logger1-pre-polarization.csv</a> <a href="#">NB-356-Logger1-pre-polarization.bmp</a>

	16:00-05	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 20 °C) done according to <a href="#">Protocol - Liquid phase calibration of H2 UniAmp sensor</a> with H2 bubbling.	/	/
	16:14	Start of O2 logging.	<b>NB-356-Ch2-1</b>	<a href="#">2025-11-20_161452_NB-356-Ch2-1.txt</a> <a href="#">2025-11-20_161452_NB-356-Ch2-1.png</a>
	16:16	Start of H2 logging.	<b>NB-356-Logger2</b> offset -2 mV	<a href="#">NB-356.ulog</a> <a href="#">NB-356-Logger2-calibration.csv</a> <a href="#">NB-356-Logger2-2point calibration.bmp</a> <a href="#">NB-356-Logger2-calibration step.bmp</a>
	16:18	Degassing was started.	/	<a href="#">20251120_161902-degassing of water.jpg</a>
	17:12	Moving cannula to gas phase.	/	/
	17:15	Introducing H2 sensor into the reactor under Ar flow.	/	/
	17:24	0 ppm was taken.	/	/
	17:26	H2 bubbling of the reactor was started	/	<a href="#">20251120_172742-H2 bubbling.jpg</a>
	17:45	1.000.000 ppm point was taken and calibration was saved	847 mV, slope: 1.076, 787 uM	<a href="#">20251120_174523-H2 table.jpg</a>
	17:46	Stop of H2 logging.	/	/
	17:46	Stop of O2 logging.	/	/
	ca. 18:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
	<b>Sample preparation</b>			
	21:40	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	21:44	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	21:46-49	The suspension was vortexed for 3 min ( <a href="#">Equipment - VWR® VV3, Vortex Mixer</a> , stage 4/6), covered with Al foil before further use.	/	<a href="#">20251120_215000-suspension after vortex.jpg</a>

		Continue in <a href="#">Protocol - In-situ hydrogen and oxygen measurement in H<sub>2</sub>/O<sub>2</sub> reactor</a> from step 6		
	22:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	22:05	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	/
	22:13	<a href="#">Start of O<sub>2</sub> logging.</a>	<b>NB-356-Ch2-2</b>	<a href="#">2025-11-20_221307_NB-356-Ch2-2.txt</a> <a href="#">2025-11-20_221307_NB-356-Ch2-2.png</a>
	22:15	The degassing was started	/	<a href="#">20251120_221526-degassing of the suspension.jpg</a>
	22:50	Cannula was transferred to gas phase, above the suspension.	/	/
	22:54	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	22:58	The degassing was stopped by removing the cannula and closing the valve.	/	/
	22:59	<a href="#">Stop of O<sub>2</sub> logging.</a>	/	/
	23:00	<a href="#">Start of O<sub>2</sub> logging.</a>	<b>NB-356-Ch2-3</b>	<a href="#">2025-11-20_230014_NB-356-Ch2-3.txt</a> <a href="#">2025-11-20_230014_NB-356-Ch2-3.png</a>
	23:00	<a href="#">Start of H<sub>2</sub> logging.</a>	<b>NB-356-Logger3</b>	NB-356.ulog NB-356-Logger3-during irradiation.csv NB-356-Logger3-during irradiation.bmp
	23:00-10	Equilibration time.	/	/
	23:10	The irradiation was started	/	<a href="#">20251120_231241-after start of irradiation.jpg</a>
	23:25	The irradiation was stopped.	/	/
	23:25-35	Equilibration time.	/	/

	23:35	Stop of O2 and H2 logging.	/	/
	ca. 23:50	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>	20251121_000151-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-356-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
20.11.2025	10:22	NB-356-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-356.ulog NB-356-Logger1-pre-polarization.csv	/	NB-356-Logger1-pre-polarization.bmp	/	Pre-polarization of the sensor.
	16:16	NB-356-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-356.ulog NB-356-Logger2-calibration.csv	/	NB-356-Logger2-2point calibration.bmp NB-356-Logger2-calibration step.bmp	/	Calibration of H2 sensor, signal at 10^6 ppm - 847 mV, slope 1.076
	23:00	NB-356-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-356.ulog NB-356-Logger3-during irradiation.csv	NB-356-O2 and H2 curve.py	NB-356-Logger3-during irradiation.bmp NB-356-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	H2 evolution during irradiation.
	16:14	NB-356-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-20_161452_NB-356-Ch2-1.txt	/	2025-11-20_161452_NB-356-Ch2-1.png	/	Degassing of water followed by calibration of H2 sensor.
	22:13	NB-356-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-20_221307_NB-356-Ch2-2.txt	/	2025-11-20_221307_NB-356-Ch2-2.png	/	Degassing of the suspension.
	23:00	NB-356-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-20_230014_NB-356-Ch2-3.txt	NB-356-O2 and H2 curve.py	2025-11-20_230014_NB-356-Ch2-3.png NB-356-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	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) were performed.

## Linked 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

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)

SrTiO<sub>3</sub> - NB-339: 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 VI (reproduction NB-316)

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)

SrTiO<sub>3</sub> - NB-353: 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 VIII (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](#)

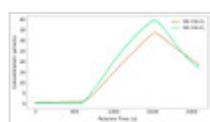
Protocol - [Liquid phase calibration of H<sub>2</sub> UniAmp sensor](#)

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

## Attached files

NB-356-O2 and H2 curves.png

sha256: b88c3616b07aef9846109f298e02b33218f62bd346bc99a6c985fc8334c07792



NB-356-O2 and H2 curve.py

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20251121\_000151-after irradiation.jpg

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20251120\_231241-after start of irradiation.jpg

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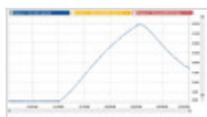


NB-356.ulog

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NB-356-Logger3-during irradiation.bmp

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NB-356-Logger3-during irradiation.csv

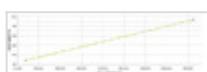
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NB-356-Logger2-2point calibration.bmp

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NB-356-Logger2-calibration step.bmp

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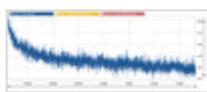


NB-356-Logger1-pre-polarization.csv

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NB-356-Logger1-pre-polarization.bmp

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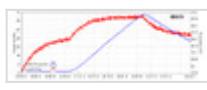


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2025-11-20\_221307\_NB-356-Ch2-2.txt

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3516>