

# NB-332: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>:Al:SrTiO<sub>3</sub> (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C (reproduction NB-327)

Date: 2025-11-03  
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>

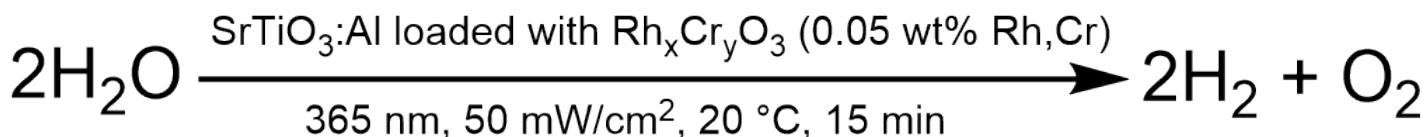
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Reproduction of NB-327: 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 (NB-321, **0.05 wt% Rh, Cr**, 0.5 mg/mL), 365 nm LED, 50 mW/cm<sup>2</sup>, 20 °C.

## Reaction scheme



ChemDraw file linked: [NB-327-SrTiO3-photocatalytic H2O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	SrTiO <sub>3</sub> - NB-327: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> :Al:SrTiO <sub>3</sub> (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C
Similar experiments	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)

## 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> (NB-321)	SrTiO <sub>3</sub> - NB-321: Modification of EA-354 (SrTiO <sub>3</sub> :Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)	/	/	/	12.50	12.51	/	/	/	/

# Excel sheet for reagent calculation

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## 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. 615 s 2. 22:29:35
<b>Irradiation stop:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 1525 s 2. 22:44:45

# 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
		Calibration from 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) was used.	/	/
03.11.2025		<b>Sample preparation</b>		
	ca.21:05	Weighing photocatalyst in a 50 mL vial.	Slightly creamy solid	/
		Important note: before weighing, the solid was dispersed inside the vial manually with Smartspatula - to break lumps manually, but to avoid mortaring.		
	21:13	Addition of 25 mL H <sub>2</sub> O to the vial via graduated cylinder.	/	/
	21:18-21	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251103_212138-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		
	ca. 21:25	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s).	/	/

	21:30	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	<a href="#">20251103_212855-before degassing and irradiation.jpg</a>
	21:30	<a href="#">Start of O2 logging.</a>	<b>NB-332-Ch2-1</b>	<a href="#">2025-11-03_213054_NB-332-Ch2-1.txt</a> <a href="#">2025-11-03_213054_NB-332-Ch2-1.png</a>
	21:32	The degassing was started	/	<a href="#">20251103_213303-degassing of the suspension.jpg</a>
	22:09	Cannula was transferred to gas phase, above the suspension.	/	/
	22:11	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	ca. 22:17	The degassing was stopped by removing the cannula and closing the valve.	/	/
	22:18	<a href="#">Stop of O2 logging.</a>	/	/
	22:19	<a href="#">Start of O2 logging.</a>	<b>NB-332-Ch2-2</b>	<a href="#">2025-11-03_221920_NB-332-Ch2-2.txt</a> <a href="#">2025-11-03_221920_NB-332-Ch2-2.png</a>
	22:19	<a href="#">Start of H2 logging.</a>	<b>NB-332-Logger1</b>	<a href="#">NB-332.ulog</a> <a href="#">NB-332-Logger1.csv</a> <a href="#">NB-332-Logger1.bmp</a>
	22:19-29	Equilibration time.	/	/
	22:29	The irradiation was started	/	<a href="#">20251103_223003-after start of irradiation.jpg</a>
	22:44	The irradiation was stopped.	/	/
	22:44-54	Equilibration time.	/	/
	22:54	<a href="#">Stop of O2 and H2 logging.</a>	/	/

	ca. 23:00	Deassembling the setup, cleaning the reactor.	Tips of the sensors and reactor were covered with attached photocatalyst particles, bubbles attached to the surface of the sensors tips. 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-331-Logger3

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
03.11.2025	22:19	NB-332-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-332.ulog NB-332-Logger1.csv	NB-332-O2 and H2 curve.py	NB-332-Logger1.bmp NB-332-O2 and H2 curve.png	SrTiO3 - NB-327: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C	Several artifacts during measurements.
	21:30	NB-332-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-03_213054_NB-332-Ch2-1.txt	/	2025-11-03_213054_NB-332-Ch2-1.png	/	Degassing of the suspension.
	22:19	NB-332-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-03_221920_NB-332-Ch2-2.txt	NB-332-O2 and H2 curve.py	2025-11-03_221920_NB-332-Ch2-2.png NB-332-O2 and H2 curve.png	SrTiO3 - NB-327: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C	Slight step at ca. 22:41.

## Results

Simultaneous H<sub>2</sub> and O<sub>2</sub> measurements of irradiated suspension of NB-321 (0.05 wt% RhCr, 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) were performed, reproduction NB-327.

H<sub>2</sub> level at the end of irradiation - ca. 34 umol/L, O<sub>2</sub> level - ca. 33 umol/L.

## 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-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-321: Modification of EA-354 (SrTiO<sub>3</sub>:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)

SrTiO<sub>3</sub> - NB-322: 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 (reproduction NB-318)

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

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-327: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,Al:SrTiO<sub>3</sub> (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C

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) |||

## 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 - [Hydrogen Measurement Using GC](#)

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](#)

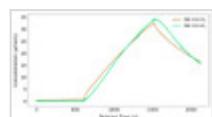
## Attached files

NB-332-O2 and H<sub>2</sub> curve.py

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NB-332-O2 and H2 curve.png

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20251103\_213303-degassing of the suspension.jpg

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20251103\_212855-before degassing and irradiation.jpg

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20251103\_212138-suspension after vortex.jpg

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20251103\_223003-after start of irradiation.jpg

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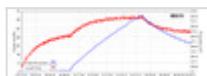
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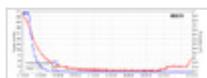


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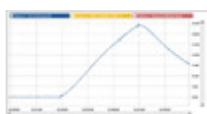


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NB-332-Logger1.bmp

sha256: 670967f7ac98296202c822e619745008c0492e5ce2b7dafa19f655d52be4b844



NB-332.ulog

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NB-332-Logger1.csv

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3324>