

# 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

Date: 2025-10-29  
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 Liquid phase detection

Category: SrTiO<sub>3</sub>

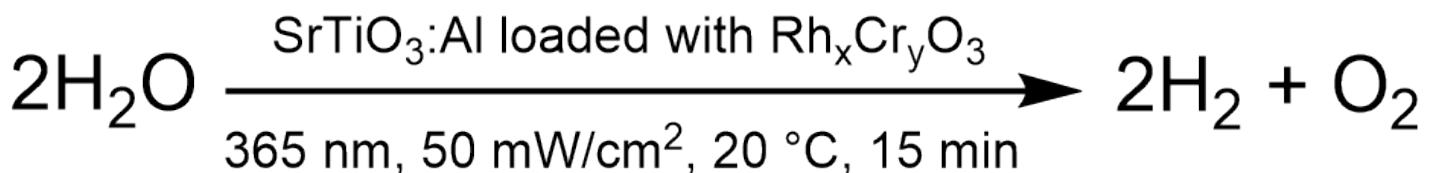
Status: Done

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, 0.5 mg/mL), 365 nm LED, 50 mW/cm<sup>2</sup>, 20 °C.

## Reaction scheme



ChemDraw file linked: [NB-329-SrTiO3-photocatalytic H<sub>2</sub>O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	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-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
Similar experiments	SrTiO <sub>3</sub> - NB-313: 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 (reproduction NB-316) II

## 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 (for calibration)	/	/

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	12.69	/	/	/	/	/	/
Hydrogen		/	/	/	/	/	/	/	2 balloons (ca. 2 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	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. 610 s 2. 19:47:02
<b>Irradiation stop:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 1562 s 2. 20:02:54

## 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
29.10.2025		Calibration data was used from experiment 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.	/	/
		<b>Sample preparation</b>		
	18:25	Weighing EA-358 photocatalyst in a 50 mL vial.	/	/
	ca. 18:32	Addition of 25 mL H <sub>2</sub> O to the vial via graduated cylinder.	/	/
	18:34-37	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	Milky white suspension	20251029_183725-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		
	18:40	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	/
	ca. 18:45	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	18:48	Start of O <sub>2</sub> logging.	<b>NB-329-Ch2-1</b>	2025-10-29_184846_NB-329-Ch2-1.txt 2025-10-29_184846_NB-329-Ch2-1.png
	18:52	The degassing was started	/	20251029_185556-during degassing of the suspension.jpg
	19:28	Cannula was transferred to gas phase, above the suspension.	/	/
	19:32	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	19:35	The degassing was stopped by removing the cannula and closing the valve.	/	/
	19:36	Stop of O <sub>2</sub> logging.	/	/
	19:36	Start of O <sub>2</sub> logging.	<b>NB-329-Ch2-2</b>	2025-10-29_193651_NB-329-Ch2-2.txt 2025-10-29_193651_NB-329-Ch2-2.png
	19:36	Start of H <sub>2</sub> logging.	<b>NB-329-Logger1</b>	NB-329.ulog NB-329-Logger1.csv NB-329-Logger1.bmp
	19:36-47	Equilibration time.	/	/
	19:47	The irradiation was started	19:56 and 20:00 - change in H <sub>2</sub> signal (no external impact on the system, most probably caused by attachment/detachment of bubble).	20251029_194850-after start of irradiation.jpg
	20:03	The irradiation was stopped.	/	/
	20:03-12	Equilibration time.	/	/

	20:12	Stop of O <sub>2</sub> and H <sub>2</sub> logging.	/	/
ca. 20:15-30		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).	20251029_201328-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-326-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
29.10.2025	19:36	NB-329-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-329.ulog NB-329-Logger1.csv	NB-329-O2 and H2 curve.py	NB-329-Logger1.bmp NB-329-O2 and H2 curves.png	SrTiO3 - NB-316: Liquid phase H2 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)	Clean response, but at 19:56 - change in signal intensity (decrease and afterwards increase), at 20:00 - again, most probably caused by bubble(s) attached/detached to/from the tip surface of the electrode, 52 uM H <sub>2</sub> at the end of irradiation.
	18:48	NB-329-Ch2-1	Optical O <sub>2</sub> detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-29_184846_NB-329-Ch2-1.txt	/	2025-10-29_184846_NB-329-Ch2-1.png	/	Degassing of the suspension.
	19:36	NB-329-Ch2-2	Optical O <sub>2</sub> detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-29_193651_NB-329-Ch2-2.txt	NB-329-O2 and H2 curve.py	2025-10-29_193651_NB-329-Ch2-2.png NB-329-O2 and H2 curves.png	SrTiO3 - NB-316: Liquid phase H2 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)	Changes in O <sub>2</sub> value during irradiation, 42 uM O <sub>2</sub> at the end of irradiation.

## Results

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) were performed.

H<sub>2</sub> level at the end of irradiation - 52 umol/L, O<sub>2</sub> level - 42 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-319: 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)

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

## Linked resources

Equipment - VWR® VV3, Vortex Mixer

Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)

Equipment - Irradiation setup 4 (CEEC II, E002)

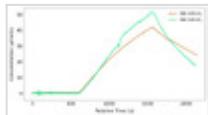
Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

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

## Attached files

NB-329-O2 and H2 curves.png

sha256: b2678d1613fc48456e3dad22a827cac8a6c9c5fb9da805521ce2c73549285728



NB-329-O2 and H2 curve.py

sha256: 9181c042ca5c1fc696f2f66ac713ea5d80ba6785014993d3053707d22bb4ed9d

NB-329-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.cdxml

sha256: a3caf200f519f799cf69e04a1ed54b96f1f160247d0afe3c6eab6f838aeafae8

NB-329-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.png

sha256: 0d37edf6c8cc83c6521ce326767eedaced8ca35d8ac61d0bbd91534b5bdfb0df



20251029\_201328-after irradiation.jpg

sha256: 6978293043b164917fed3df5e0d3ac799c72d03e98b52758d5115c7d22b73af7



20251029\_185556-during degassing of the suspension.jpg

sha256: 9809ae67bea3730ecd32e0bfc2540bd7e724c267b77d45426f2811bf08289b75



20251029\_183725-suspension after vortex.jpg

sha256: a5f6ec8b3af1c72b69071fa8caa50f66c63e789712a72fb39834a4fc59d90b5b



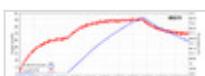
20251029\_194850-after start of irradiation.jpg

sha256: 330a59dace19fe5dd76fbecfcb56597efd0ecf1e4eb3c4511fbe42e6c72b8500



2025-10-29\_193651\_NB-329-Ch2-2.png

sha256: c032354064d4f18b11fa523dfec5831386bff52305593479082ba8eee7e977be



2025-10-29\_193651\_NB-329-Ch2-2.txt

sha256: e5aa0e4a11f4b09404f1d05b46427b8f1bd11b0374dfd4b203c342b6ad49ba56

2025-10-29\_184846\_NB-329-Ch2-1.png

sha256: 27d8e4d1bb2bf39e24b0d7c9e2b411c203ebb82eaa2e67d65b3d23795005d403

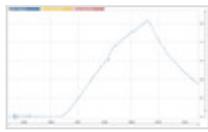


2025-10-29\_184846\_NB-329-Ch2-1.txt

sha256: d68439ad35d0c611e8be97737429599ef8b5e643400d7ef39a0d026890a46e97

NB-329-Logger1.bmp

sha256: 118b14fe146c4391889c9eb34c9f34f03a12733a97af4550c3ab222c17b15e4b



NB-329.ulog

sha256: 4b165abf4521ed092f34ebe940166048a10d54e12a981e201d49d2d3e74a819f

NB-329-Logger1.csv

sha256: ffd199e284108c0b8c597eee69baa6a3fae84bdacdb43637391e533bc29c7556



Unique eLabID: 20251029-018eeb71d9d1a900b0e4c393d39cd9f781a8f566

Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3299>