

# NB-361: Gas phase H2 and O2 measurements of Al:SrTiO3 RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C, 15 min, degassing

Date: 2025-11-25

Tags: O2 Test Calibration Future NB  
Firing Irradiation O2 sensor H2  
advanced irr ad setup Unisense H2  
Sensor temperature In situ Trace range  
robust oxygen sensor photocatalysis

Category: SrTiO3

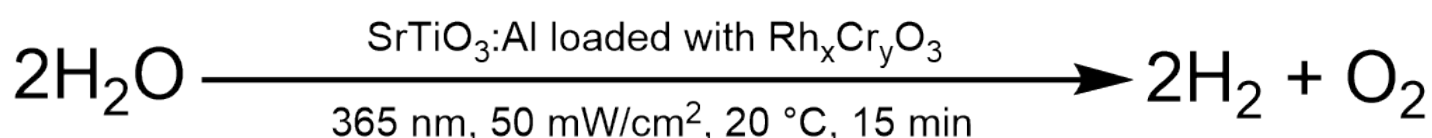
Status: Need to be redone

Created by: Nadzeya Brezhneva

## Objectives

Simultaneous detection of O2 and H2 evolution in gas 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-361-SrTiO3-photocatalytic H2O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	<a href="#">SrTiO3 - NB-304: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firing O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing</a> <a href="#">SrTiO3 - NB-312: Gas phase H2 and O2 measurements with Unisense H2 sensor, Firing O2 robust probe in irradiated Al:SrTiO3 RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing (reproduction NB-304)</a>

## 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]
milli-Q H <sub>2</sub> O	/	/	/	/	/	/	/	/	25	/
Hydrogen	<a href="#">1333-74-0</a>	/	/	/	/	/	/	/	1 balloon (approx. 1 L)	approx. 1

Al:SrTiO3 RhCrOx (EA-358)	SrTiO3 - EA-358: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co- catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	12.50	12.61	/	/	/	/
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## 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 measurment 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]
First light source	<a href="#">Light Source - UHP LED 365 nm-4</a>	<a href="#">Power Sources - BLS-18000-14</a>	365	56	0.19

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	20
Stirring speed [rpm]	500

<b>Irradiation start:</b> <b>1. Firing [relative to start log]</b> <b>2. Unisense</b>	1. 600 s 2. 15:57:22
<b>Irradiation stop:</b> <b>1. Firing [relative to start log]</b> <b>2. Unisense</b>	1. 1512 s 2. 16:12:34

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

	Equipment	Used protocol
Used Firing	Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel (Firing 2)	Protocol - Operation of Firing 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
25.11.2025		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	/	/
		Important note: replacing 10 mm BOLA fitting to 2 mm BOLA fitting for H <sub>2</sub> sensor.		
	10:58-12:47	Conditioning of H <sub>2</sub> sensor	<b>NB-361-Logger1</b> 1.6 mV at the end of polarization procedure	<a href="#">NB-361.uog</a> <a href="#">NB-361-Logger1-pre-polarization.csv</a> <a href="#">NB-361-Logger1-pre-polarization.bmp</a>
	12:50	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 H<sub>2</sub> UniAmp sensor</a> with H <sub>2</sub> bubbling.	/	/

	13:13	Start of O2 logging.	NB-361-Ch2-1	2025-11-25_131321_NB-361-Ch2-1.txt 2025-11-25_131321_NB-361-Ch2-1.png
	13:13	Start of H2 logging.	NB-361-Logger2 offset - 2 mV	NB-361.ulong NB-361-Logger2-calibration.csv NB-361-Logger2-calibration step.bmp NB-361-Logger2-2point calibration.bmp
	13:15	Degassing was started.		20251125_131615-degassing water.jpg
	13:55	0 ppm was taken.	/	/
	13:57	H2 bubbling of the reactor was started	/	20251125_135812-H2 bubbling.jpg
	14:05	1.000.000 ppm point was taken	866 mV	20251125_140453-H2 table.jpg
	14:08	The previous point was removed, new point was added and calibration was saved	867 mV, slope: 0.009, 96002 Pa	/
	14:09	Stop of H2 logging.	/	/
	14:09	Stop of O2 logging.	/	/
	14:15	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		<b>Sample preparation</b>		
	14:49	Weighing <b>EA-358</b> photocatalyst in a 50 mL vial.	Creamy solid.	/
	14:50	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	14:52-55	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.	/	20251125_145652-suspension after vortex.jpg
		Continue in <a href="#">Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor</a> from step 6		
	15:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/

	15:05	Assembling the setup.	/	/
	15:15	Start of O2 logging.	<b>NB-361-Ch2-2</b>	<a href="#">2025-11-25_151501_NB-361-Ch2-2.txt</a> <a href="#">2025-11-25_151501_NB-361-Ch2-2.png</a>
	15:18	The degassing was started	/	<a href="#">20251125_151834-degassing of the suspension.jpg</a>
	15:43	Cannula was transferred to gas phase, above the suspension.	/	/
	15:43	The degassing was stopped by removing the cannula and closing the valve.	/	/
	15:47	Stop of O2 logging.	/	/
	15:47	Start of O2 logging.	<b>NB-361-Ch2-3</b>	<a href="#">2025-11-25_154722_NB-361-Ch2-3.txt</a> <a href="#">2025-11-25_154722_NB-361-Ch2-3.png</a>
	15:47	Start of H2 logging.	<b>NB-361-Logger3</b>	<a href="#">NB-361.ulong</a> <a href="#">NB-361-Logger3-during irradiation.csv</a> <a href="#">NB-361-Logger3-during irradiation.bmp</a>
	15:47-57	Equilibration time.	/	/
	15:57	The irradiation was started	/	<a href="#">20251125_155928-after start of irradiation.jpg</a>
	16:12	The irradiation was stopped.	/	/
	16:12-22	Equilibration time.	/	/
	16:22	Stop of O2 and H2 logging.	/	/

	ca. 16:30	Deassembling the setup, cleaning the reactor.	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).	<a href="#">20251125_162352-after irradiation.jpg</a>
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## Analysis

Used calibration for Firesting: [20250910-BOLA fitting-gas phase-4-neck photoreactor-trace oxygen robust probe-Ch2.ini](#)

Used calibration for UniSense: NB-361-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
25.11.2025	10:58	NB-361-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	<a href="#">NB-361.ulong</a> <a href="#">NB-361-Logger1-pre-polarization.csv</a>	/	<a href="#">NB-361-Logger1-pre-polarization.bmp</a>	/	Pre-polarization of H2 sensor.
	13:13	NB-361-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	<a href="#">NB-361.ulong</a> <a href="#">NB-361-Logger2-calibration.csv</a>	/	<a href="#">NB-361-Logger2-calibration step.bmp</a> <a href="#">NB-361-Logger2-2point calibration.bmp</a>	/	Calibration of H2 sensor, 10 <sup>6</sup> ppm corresponds to 867 mV. slope 0.009
	15:47	NB-361-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	<a href="#">NB-361.ulong</a> <a href="#">NB-361-Logger3-during irradiation.csv</a>	<a href="#">NB-361-O2 and H2 curve.py</a>	<a href="#">NB-361-Logger3-during irradiation.bmp</a> <a href="#">NB-361-O2 and H2 curves.png</a>	/	H2 evolution during irradiation.
	13:13	NB-361-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	<a href="#">2025-11-25_131321_NB-361-Ch2-1.txt</a>	/	<a href="#">2025-11-25_131321_NB-361-Ch2-1.png</a>	/	Degassing of water followed by calibration of H2 sensor.
	15:15	NB-361-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	<a href="#">2025-11-25_151501_NB-361-Ch2-2.txt</a>	/	<a href="#">2025-11-25_151501_NB-361-Ch2-2.png</a>	/	Degassing of the photocatalyst suspension.
	15:47	NB-361-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	<a href="#">2025-11-25_154722_NB-361-Ch2-3.txt</a>	<a href="#">NB-361-O2 and H2 curve.py</a>	<a href="#">2025-11-25_154722_NB-361-Ch2-3.png</a> <a href="#">NB-361-O2 and H2 curves.png</a>	/	O2 evolution during irradiation.

## Results

Simultaneous H<sub>2</sub> and O<sub>2</sub> measurements (gas phase) 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.

## Future recommendations

Old procedure	Problem	Suggested new procedure
/	Very low values of produced O <sub>2</sub> and H <sub>2</sub> .	Increase the duration of irradiation while performing O <sub>2</sub> /H <sub>2</sub> measurements in gas phase

## Linked experiments

SrTiO<sub>3</sub> - NB-312: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements with Unisense H<sub>2</sub> sensor, Firesting O<sub>2</sub> robust probe in irradiated Al:SrTiO<sub>3</sub> RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing (reproduction NB-304)

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)

## Linked resources

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

Equipment - [Robust probe for liquid O<sub>2</sub> measurment](#)

Equipment - [Advanced irradiation chamber V1.0 I](#)

Equipment - [H<sub>2</sub> UniAmp Sensor - Normal range - 2.1 x 80 mm needle](#)

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

Protocol - [Operation of Firesting Fiber-Optic Oxygen Meter 2 Channel Software](#)

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

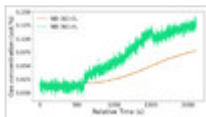
Protocol - [Gas phase calibration of H<sub>2</sub> UniAmp sensor](#)

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

## Attached files

NB-361-O<sub>2</sub> and H<sub>2</sub> curves.png

sha256: 32f2f7f17bc983cd69b8c26b6a39cc1d7ecbe02e8c0745d3aa05809e668ba600



NB-361-O2 and H2 curve.py

sha256: 010c7e965503d47476aad7780eae7ac173d1c9aa6e18f2f3d9f814fccf427d5d

NB-361-SrTiO3-photocatalytic H2O splitting.cdxml

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20251125\_135812-H2 bubbling.jpg

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20251125\_140453-H2 table.jpg

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20251125\_145652-suspension after vortex.jpg

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20251125\_151834-degassing of the suspension.jpg

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20251125\_155928-after start of irradiation.jpg

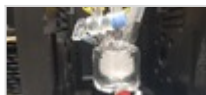
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NB-361.ulog

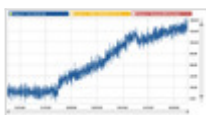
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NB-361-Logger3-during irradiation.csv

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NB-361-Logger3-during irradiation.bmp

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NB-361-Logger2-calibration.csv

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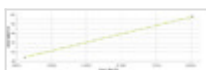
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NB-361-Logger2-2point calibration.bmp

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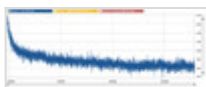


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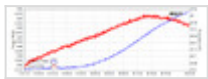


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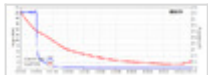


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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3551>