

NB-361: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (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
Firesting Irradiation O₂ sensor H₂
advanced irrad setup Unisense H₂
Sensor temperature In situ Trace range
robust oxygen sensor photocatalysis

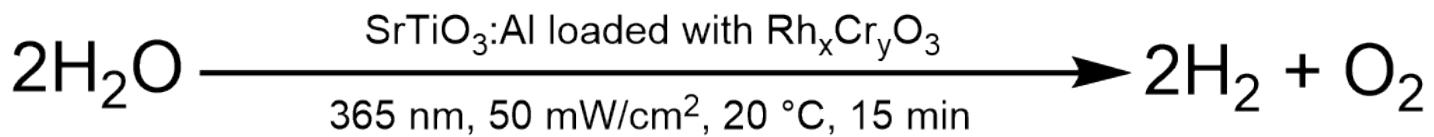
Category: SrTiO₃

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_xCr_yO₃:Al:SrTiO₃ suspension (EA-358, 0.5 mg/mL), 365 nm LED, 50 mW/cm², 20 °C.

Reaction scheme



ChemDraw file linked: [NB-361-SrTiO₃-photocatalytic H₂O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO ₃ - NB-304: Gas phase H ₂ and O ₂ measurements with Unisense H ₂ sensor, Firesting O ₂ robust probe in irradiated Al:SrTiO ₃ RhCrO _x (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing SrTiO ₃ - NB-312: Gas phase H ₂ and O ₂ measurements with Unisense H ₂ sensor, Firesting O ₂ robust probe in irradiated Al:SrTiO ₃ RhCrO _x (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing (reproduction NB-304)

Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	pressure [bar]
milli-Q H ₂ O	/	/	/	/	/	/	/	/	25	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (approx. 1 L)	approx. 1

Al:SrTiO ₃ RhCrO _x (EA-358)	SrTiO ₃ - EA-358: Modification of Al:SrTiO ₃ (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 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	Equipment - Advanced irradiation setup V1.0 I
Irradiation setup number	Equipment - Irradiation setup 4 (CEEC II, E002)

	Light Source Name	Power Source Name	Wavelength [nm]	Power Setting [mW]	Analog Setting [0.00 - 10.00]
First light source	Light Source - UHP LED 365 nm-4	Power Sources - BLS-18000-14	365	56	0.19

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

Irradiation start: 1. Firesting [relative to start log] 2. Unisense	1. 600 s 2. 15:57:22
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1512 s 2. 16:12:34

O₂/H₂ 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 ₂ sensor	Equipment - Robust probe for liquid O ₂ measurement	Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor
Used H ₂ sensor	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
25.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
		Important note: replacing 10 mm BOLA fitting to 2 mm BOLA fitting for H ₂ sensor.		
	10:58-12:47	Conditioning of H₂ sensor	NB-361-Logger1 1.6 mV at the end of polarization procedure	NB-361.ulog NB-361-Logger1-pre-polarization.csv NB-361-Logger1-pre-polarization.bmp
	12:50	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 20 °C) done according to Protocol - Liquid phase calibration of H₂ UniAmp sensor with H₂ 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.ulog 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 .	/	/
	Sample preparation			
	14:49	Weighing EA-358 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 (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251125_145652-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor 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.	NB-361-Ch2-2	2025-11-25_151501_NB-361-Ch2-2.txt 2025-11-25_151501_NB-361-Ch2-2.png
	15:18	The degassing was started	/	20251125_151834-degassing of the suspension.jpg
	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.	NB-361-Ch2-3	2025-11-25_154722_NB-361-Ch2-3.txt 2025-11-25_154722_NB-361-Ch2-3.png
	15:47	Start of H2 logging.	NB-361-Logger3	NB-361.ulog NB-361-Logger3-during irradiation.csv NB-361-Logger3-during irradiation.bmp
	15:47-57	Equilibration time.	/	/
	15:57	The irradiation was started	/	20251125_155928-after start of irradiation.jpg
	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).	20251125_162352-after irradiation.jpg
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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	NB-361.ulog NB-361-Logger1-pre-polarization.csv	/	NB-361-Logger1-pre-polarization.bmp	/	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	NB-361.ulog NB-361-Logger2-calibration.csv	/	NB-361-Logger2-calibration step.bmp NB-361-Logger2-2point calibration.bmp	/	Calibration of H2 sensor, 10^-6 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	NB-361.ulog NB-361-Logger3-during irradiation.csv	NB-361-O2 and H2 curve.py	NB-361-Logger3-during irradiation.bmp NB-361-O2 and H2 curves.png	/	H2 evolution during irradiation.
	13:13	NB-361-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-25_131321_NB-361-Ch2-1.txt	/	2025-11-25_131321_NB-361-Ch2-1.png	/	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	2025-11-25_151501_NB-361-Ch2-2.txt	/	2025-11-25_151501_NB-361-Ch2-2.png	/	Degassing of the photocatalyst suspension.
	15:47	NB-361-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-25_154722_NB-361-Ch2-3.txt	NB-361-O2 and H2 curve.py	2025-11-25_154722_NB-361-Ch2-3.png NB-361-O2 and H2 curves.png	/	O2 evolution during irradiation.

Results

Simultaneous H₂ and O₂ measurements (gas phase) of irradiated suspension of EA-358 (0.5 mg/mL) in O₂/H₂ photoreactor under 365 nm irradiation (50 mW/cm², 20 °C, 15 min) were performed.

Future recommendations

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

Linked experiments

SrTiO₃ - NB-312: Gas phase H₂ and O₂ measurements with Unisense H₂ sensor, Firesting O₂ robust probe in irradiated Al:SrTiO₃ RhCrO_x (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing (reproduction NB-304)

SrTiO₃ - EA-358: Modification of Al:SrTiO₃ (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 O₂ measurement](#)

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

Equipment - [H₂ 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](#)

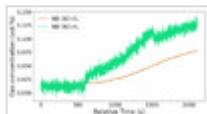
Protocol - [Gas phase calibration of H₂ UniAmp sensor](#)

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

Attached files

NB-361-O2 and H2 curves.png

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NB-361-O2 and H2 curve.py

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NB-361-SrTiO3-photocatalytic H2O splitting.cdxml

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NB-361-SrTiO3-photocatalytic H2O splitting.png

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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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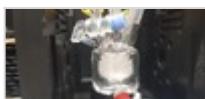
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20251125_162352-after irradiation.jpg

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NB-361.ulog

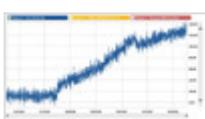
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NB-361-Logger3-during irradiation.csv

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NB-361-Logger3-during irradiation.bmp

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NB-361-Logger2-calibration.csv

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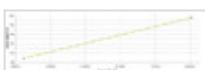
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NB-361-Logger2-2point calibration.bmp

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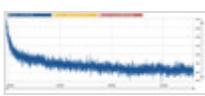


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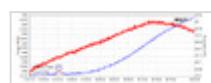


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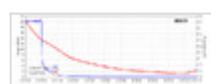


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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3551>