

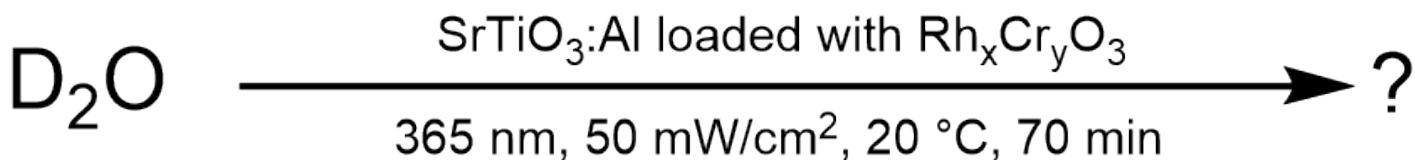
NB-365: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (EA-358, 0.5 mg/mL), D₂O, 365 nm, 50 mW/cm², 20 °C, 70 min, degassing

Date: 2025-12-03
Tags: O₂ Test Calibration Future NB
Firesting Irradiation O₂ sensor H₂
advanced irrad setup troubleshooting
Unisense H₂ Sensor temperature In situ
Trace range robust oxygen sensor
photocatalysis
Category: SrTiO₃
Status: Done
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, D₂O), 365 nm LED, 50 mW/cm², 20 °C during 70 min.

Reaction scheme



ChemDraw file linked: [NB-365-SrTiO3-D2O.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO ₃ - NB-363: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C, 70 min, degassing (reproduction NB-362) SrTiO ₃ - NB-362: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C, 70 min, degassing SrTiO ₃ - NB-361: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C, 15 min, degassing

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	/	/	/	/	/	/	/	0.998	25 (for calibration)	/

D ₂ O, Eurisotop, 99.90%	7789-20-0	C121237	/	/	/	/	/	1.11	25 (for suspension preparation)	/
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	12.52	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon (ca. 2 L)	ca. 1

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. 605 s 2. 18:55:17
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 4825 s 2. 20:05:35

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
03.12.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	14:28-15:27	Conditioning of H ₂ sensor	NB-365-Logger1 offset - 2 mV	NB-365.ulog NB-365-Logger1.csv NB-365-Logger1-pre-polarization.bmp

	ca. 15:30-40	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.	/	/
	15:59	Start of O2 logging.	NB-365-Ch2-1	2025-12-03_155900_NB-365-Ch2-1.txt 2025-12-03_155900_NB-365-Ch2-1.png
	15:59	Start of H2 logging.	NB-365-Logger2	NB-365.ulog NB-365-Logger2.csv NB-365-Logger2-calibration step.bmp NB-365-Logger2-2point calibration.bmp
	16:02	Degassing was started.	/	20251203_160306-degassing of water.jpg
	16:48	Stop degassing.		
	16:49	0 ppm was taken.	/	/
	16:56	H2 bubbling of the reactor was started	/	20251203_165730-H2 bubbling.jpg
	17:04	1.000.000 ppm point was taken and calibration was used.	905 mV, slope 0.009, 96956 Pa	20251203_170405-H2 table.jpg
	17:08	Stop of H2 logging.	/	/
	17:08	Stop of O2 logging.	/	/
	ca. 17:15	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
	Sample preparation			
	17:45	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	17:46	Addition of 25 mL D ₂ O to the vial via graduated cylinder.	/	20251203_174628-D2O reagent bottle.jpg
	17:47-50	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer , stage 4/6), covered with Al foil before further use.	/	20251203_175117-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	18:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/

	ca. 18:10	Assembling the setup.	/	/
	18:16	Start of O2 logging.	NB-365-Ch2-2	2025-12-03_181612_NB-365-Ch2-2.txt 2025-12-03_181612_NB-365-Ch2-2.png
	18:17	The degassing was started	/	/
	18:41	The degassing was stopped by removing the cannula and closing the valve.	/	/
	18:44	Stop of O2 logging.	/	/
	18:44	Start of O2 logging.	NB-365-Ch2-3	2025-12-03_184510_NB-365-Ch2-3.txt 2025-12-03_184510_NB-365-Ch2-3.png
	18:45	Start of H2 logging.	NB-365-Logger3	NB-365.ulog NB-365-Logger3-during irradiation.csv NB-365-Logger3-during irradiation.bmp
	18:45-55	Equilibration time.	/	20251203_185234-before irradiation.jpg
	18:55	The irradiation was started	Problems with stirring 19:38 - restart stirring 19:51 - restart stirring 20:00 - restart stirring	20251203_185730-after start of irradiation.jpg
	20:05	The irradiation was stopped.	/	/
	20:05-15	Equilibration time.	/	/
	20:15	Stop of O2 and H2 logging.	/	20251203_201647-after irradiation.jpg
	ca. 20:30	Deassembling the setup, cleaning the reactor.	Seems that particles are less adhesive to the walls of the reactor than in the case of H ₂ O-based suspensions.	/

Analysis

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

Used calibration for UniSense: NB-365-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
03.12.2025	14:28	NB-365-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-365.ulog NB-365-Logger1.csv	/	NB-365-Logger1-pre-polarization.bmp	/	Pre-polarization of the sensor.
	15:59	NB-365-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-365.ulog NB-365-Logger2.csv	/	NB-365-Logger2-calibration step.bmp NB-365-Logger2-2point calibration.bmp	/	Calibration of H2 sensor, 10^{-6} ppm corresponds to 905 mV
	18:45	NB-365-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-365.ulog NB-365-Logger3-during irradiation.csv	NB-365-O2 and H2 curve.py	NB-365-Logger3-during irradiation.bmp NB-365-O2 and H2 curves.png	SrTiO3 - NB-346: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL, D2O, 365 nm, 50 mW/cm ² , 20 °C)	H2 evolution during irradiation.
	15:59	NB-365-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_155900_NB-365-Ch2-1.txt	/	2025-12-03_155900_NB-365-Ch2-1.png	/	Degassing of water followed by calibration of H2 sensor.
	18:16	NB-365-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_181612_NB-365-Ch2-2.txt	/	2025-12-03_181612_NB-365-Ch2-2.png	/	Degassing of the suspension.
	18:44	NB-365-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_184510_NB-365-Ch2-3.txt	NB-365-O2 and H2 curve.py	2025-12-03_184510_NB-365-Ch2-3.png NB-365-O2 and H2 curves.png	SrTiO3 - NB-346: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL, D2O, 365 nm, 50 mW/cm ² , 20 °C)	O2 evolution during irradiation.

Results

Simultaneous H₂ and O₂ measurements (gas phase) of irradiated suspension of EA-358 (0.5 mg/mL, D₂O) in O₂/H₂ photoreactor under 365 nm irradiation (50 mW/cm², 20 °C, 70 min) were performed.

Future recommendations

Old procedure	Problem	Suggested new procedure
/	Not constant stirring of the suspension during irradiation.	Replace PTFE stirring bar with a new one.

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

SrTiO₃ - NB-346: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), D₂O, 365 nm, 50 mW/cm², 20 °C

SrTiO₃ - 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

SrTiO₃ - NB-362: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C, 70 min, degassing

SrTiO₃ - NB-363: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C, 70 min, degassing (reproduction NB-362)

SrTiO₃ - NB-364: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C, 70 min, degassing (reproduction NB-363)

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](#)

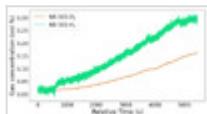
Protocol - [Gas phase calibration of H₂ UniAmp sensor](#)

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

Attached files

NB-365-O₂ and H₂ curves.png

sha256: 0c0943f18a13eff8f11ba7066a1ce1ced9c9005ed69c789fb0657a23763e0bde



NB-365-O2 and H2 curve.py

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NB-365-SrTiO3-D2O.png

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NB-365-SrTiO3-D2O.cdxml

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20251203_201647-after irradiation.jpg

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20251203_185730-after start of irradiation.jpg

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20251203_185234-before irradiation.jpg

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20251203_175117-suspension after vortex.jpg

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20251203_174628-D2O reagent bottle.jpg

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20251203_170405-H2 table.jpg

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20251203_165730-H2 bubbling.jpg

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20251203_160306-degassing of water.jpg

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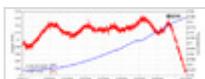


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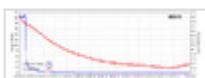


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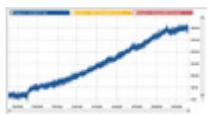


NB-365-Logger3-during irradiation.csv

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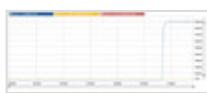
NB-365-Logger3-during irradiation.bmp

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NB-365-Logger2-calibration step.bmp

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NB-365-Logger2.csv

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NB-365-Logger2-2point calibration.bmp

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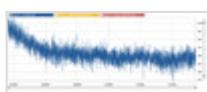


NB-365-Logger1.csv

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NB-365-Logger1-pre-polarization.bmp

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NB-365.ulog

sha256: fe5dde562eca7ae69b3864096e60b3520820cdf3a811d48ac4598386a579d453



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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3595>