

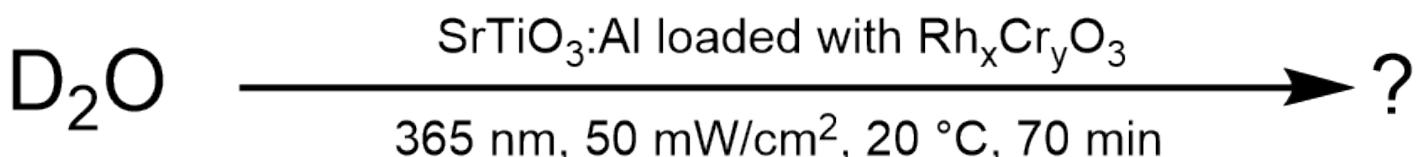
NB-366: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (EA-358, 0.5 mg/mL), D₂O, 365 nm, 50 mW/cm², 20 °C, 70 min, degassing (reproduction NB-365)

Date: 2025-12-03
Tags: O₂ Test Calibration NB Firesting Irradiation O₂ sensor H₂ advanced irrad setup 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-366-SrTiO3-D2O.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	SrTiO ₃ - NB-365: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), D ₂ O, 365 nm, 50 mW/cm ² , 20 °C, 70 min, degassing
Similar experiments	SrTiO ₃ - NB-364: 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-363)

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]
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	12.53	/	/	/	/	/
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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-18000-1 4	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. 23:16:10
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 4815 s 2. 0:26:20

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		Calibration from experiment SrTiO ₃ - NB-365: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), D ₂ O, 365 nm, 50 mW/cm ² , 20 °C, 70 min, degassing was used.		
		Sample preparation		
	21:25	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	21:26	Addition of 25 mL D ₂ O to the vial via graduated cylinder.	/	/
	21:30	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251203_213448-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor from step 6		
	21:40	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s).	Checking stirring --> OK	/
	21:45	Assembling the setup.	/	/

	21:50	Start of O2 logging.	NB-366-Ch2-1	2025-12-03_215026_NB-366-Ch2-1.txt 2025-12-03_215026_NB-366-Ch2-1.png
	21:52	The degassing was started	/	20251203_215259-degassing of the suspension.jpg
	22:27	The degassing was stopped by removing the cannula and closing the valve.	/	/
	22:29	Stop of O2 logging.	/	/
	22:29	Start of O2 logging.	NB-366-Ch2-2	2025-12-03_222957_NB-366-Ch2-2.txt 2025-12-03_222957_NB-366-Ch2-2.png
	22:30	Start of H2 logging.	NB-366-Logger1-prep	NB-366.ulog NB-366-Logger1-prep.bmp
	22:37	Stop of O2 and H2 loggings.	Too high O ₂ leakage rate --> check BOLA fittings of all sensors	
	22:47	Start degassing.		
	22:48	Start of O2 logging.	NB-366-Ch2-3	2025-12-03_224810_NB-366-Ch2-3.txt 2025-12-03_224810_NB-366-Ch2-3.png
	23:03	Stop of degassing.	/	/
	23:05	Stop of O2 logging.	/	/
	23:06	Start of O2 logging.	NB-366-Ch2-4	2025-12-03_230605_NB-366-Ch2-4.txt 2025-12-03_230605_NB-366-Ch2-4.png
	23:06	Start of H2 logging.	NB-366-Logger1	NB-366.ulog NB-366-Logger1.csv NB-366-Logger1.bmp
	23:06-16	Equilibration time.	/	/
	23:16	The irradiation was started	/	20251203_231625-after start of irradiation.jpg
04.12.2025	0:26	The irradiation was stopped.	/	/
	0:26-36	Equilibration time.	/	/

	0:36	Stop of O ₂ and H ₂ logging.	/	20251204_003738-after irradiation.jpg
	0:50	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	22:30	NB-366-Logger1-prep	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-366.ulog	/	NB-366-Logger1-prep.bmp	/	High O ₂ leakage during pre-reaction conditioning was observed -> the setup needed to be checked, H ₂ logging needed to be restarted.
	23:06	NB-366-Logger1	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-366.ulog NB-366-Logger1.csv	NB-366-O2 and H ₂ curve.py	NB-366-Logger1.bmp NB-366-O2 and H ₂ curves.png	SrTiO ₃ - NB-365: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), D ₂ O, 365 nm, 50 mW/cm ² , 20 °C, 70 min, degassing	H ₂ evolution during irradiation.
	21:50	NB-366-Ch2-1	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_215026_NB-366-Ch2-1.txt	/	2025-12-03_215026_NB-366-Ch2-1.png	/	Degassing of the suspension.
	22:29	NB-366-Ch2-2	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_222957_NB-366-Ch2-2.txt	/	2025-12-03_222957_NB-366-Ch2-2.png	/	High O ₂ leakage rate during pre-reaction conditioning was observed -> the setup needed to be checked
	22:48	NB-366-Ch2-3	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_224810_NB-366-Ch2-3.txt	/	2025-12-03_224810_NB-366-Ch2-3.png	/	Repeat of the suspension degassing.
	23:06	NB-366-Ch2-4	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-03_230605_NB-366-Ch2-4.txt	NB-366-O2 and H ₂ curve.py	2025-12-03_230605_NB-366-Ch2-4.png NB-366-O2 and H ₂ curves.png	SrTiO ₃ - NB-365: Gas phase H ₂ and O ₂ measurements of Al:SrTiO ₃ RhCrO _x (EA-358, 0.5 mg/mL), D ₂ O, 365 nm, 50 mW/cm ² , 20 °C, 70 min, degassing	O ₂ evolution during irradiation.

Results

Reproduction of NB-365: 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. Problems with stirring were eliminated.

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-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)

SrTiO₃ - 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

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](#)

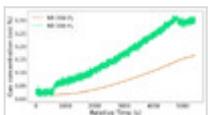
Protocol - [Gas phase calibration of H₂ UniAmp sensor](#)

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

Attached files

NB-366-O₂ and H₂ curves.png

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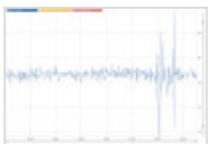


NB-366-O2 and H2 curve.py

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NB-366-Logger1-prep.bmp

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NB-366-SrTiO3-D20.cdxml

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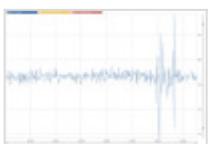
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NB-366-Logger1-prep.bmp

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20251203_213448-suspension after vortex.jpg

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20251203_215259-degassing of the suspension.jpg

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20251204_003738-after irradiation.jpg

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20251203_231625-after start of irradiation.jpg

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NB-366.ulog

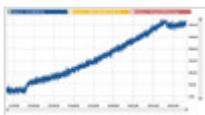
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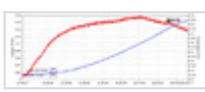


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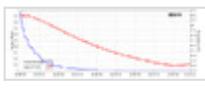


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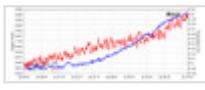


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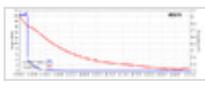
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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3599>