

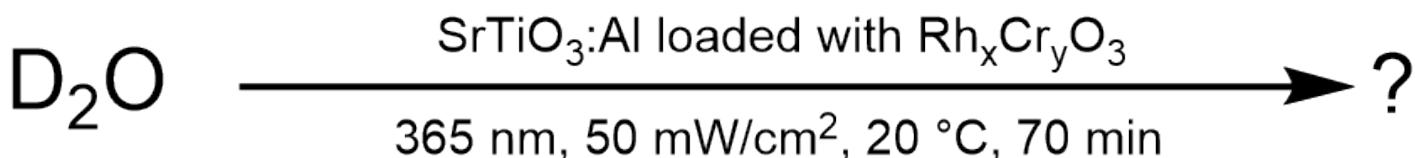
NB-367: Gas phase H₂ and O₂ measurements of Al:SrTiO₃ RhCrO_x (EA-358, 0.5 mg/mL), D₂O, 365 nm, 50 mW/cm², 20 °C, 70 min, degassing (reproduction NB-366)

Date: 2025-12-04
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

Reproduction of NB-366: simultaneous detection of O₂ and H₂ evolution in gas phase for irradiated suspension of Rh,CrO_x: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-366: 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 (reproduction NB-365)
Similar experiments	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 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]
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	12.59	/	/	/	/	/
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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. 2:23:46
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 4820 s 2. 3:34:01

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
04.12.2025		Calibration from 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		
	1:20	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	1:23	Addition of 25 mL D ₂ O to the vial via graduated cylinder.	/	/
	1:24-27	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251204_012821-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor from step 6		
	1:30	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s).	/	20251204_014529-degassing of the suspension.jpg

	1:30	Assembling the setup.	/	/
	1:42	Start of O2 logging.	/	2025-12-04_014243_NB-367-Ch2-1.txt 2025-12-04_014243_NB-367-Ch2-1.png
	1:45	The degassing was started	/	/
	2:10	The degassing was stopped by removing the cannula and closing the valve.	/	/
	2:13	Stop of O2 logging.	/	/
	2:13	Start of O2 logging.	/	2025-12-04_021341_NB-367-Ch2-2.txt 2025-12-04_021341_NB-367-Ch2-2.png
	2:13	Start of H2 logging.	/	NB-367.ulog NB-367-Logger1.csv NB-367-Logger1.bmp
	2:13-23	Equilibration time.	/	/
	2:23	The irradiation was started	/	20251204_022602-after start of irradiation.jpg
	3:34	The irradiation was stopped.	/	/
	3:34-44	Equilibration time.	/	/
	3:44	Stop of O2 and H2 logging.	/	/

	ca. 3:50	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.	/
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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-364-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
04.12.2025	2:13	NB-367-Logger1	electrochemical H ₂ detection	Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-367.ulog NB-367-Logger1.csv	NB-367-O2 and H ₂ curve.py	NB-367-Logger1.bmp NB-367-O2 and H ₂ curves.png	SrTiO ₃ - NB-366: 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 (reproduction NB-365)	Photocatalytic reaction.
	1:42	NB-367-Ch2-1	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-04_014243_NB-367-Ch2-1.txt	/	2025-12-04_014243_NB-367-Ch2-1.png	/	Degassing of the suspension
	2:13	NB-367-Ch2-2	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-12-04_021341_NB-367-Ch2-2.txt	NB-367-O2 and H ₂ curve.py	2025-12-04_021341_NB-367-Ch2-2.png NB-367-O2 and H ₂ curves.png	SrTiO ₃ - NB-366: 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 (reproduction NB-365)	O ₂ evolution during photocatalytic reaction

Results

Reproduction of NB-366: 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, successful.

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

SrTiO₃ - 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)

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](#)

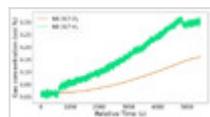
Attached files

NB-367-O2 and H2 curve.py

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NB-367-O2 and H2 curves.png

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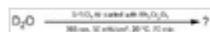


NB-366-SrTiO3-D20.cdxml

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NB-366-SrTiO3-D20.png

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20251204_014529-degassing of the suspension.jpg

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20251204_012821-suspension after vortex.jpg

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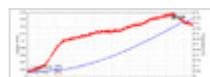
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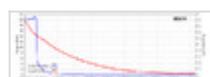


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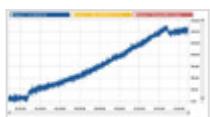


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NB-367-Logger1.bmp

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NB-367.ulog

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NB-367-Logger1.csv

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3603>