

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

Date: 2025-11-12
Tags: O₂ Test Calibration NB Firesting O₂ sensor H₂ SrTiO₃ Unisense RhCrO_x:Al:SrTiO₃ H₂ Sensor temperature In situ Trace range robust oxygen sensor photocatalysis Unisense normal range

Category: SrTiO₃

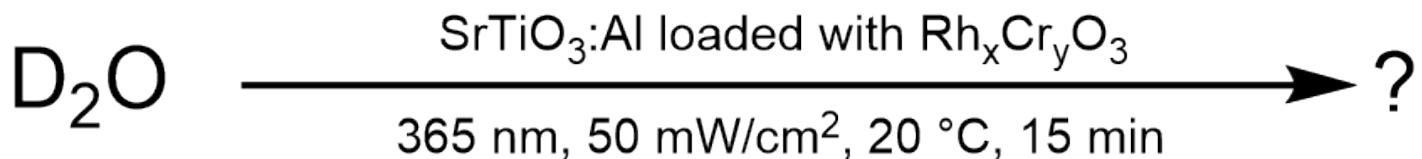
Status: Done

Created by: Nadzeya Brezhneva

Objectives

Simultaneous detection of H₂ and O₂ evolution in liquid 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 (changing dispersion medium).

Reaction scheme



ChemDraw file linked: [NB-346-SrTiO3-photocatalytic H₂O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO ₃ - NB-316: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C

Reagents

Name	CAS Number / Experiment Number	Inventor y number	Amount [mmol]	Equivale nts	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density [g/ml]	Volume [ml]	Pressure [bar]	Concentrati on [mM]
milli-Q H ₂ O	/	/	/	/	/	/	/	0.998	25 (for calibration)	/	/
D ₂ O, Eurisotop, 99.90%	7789-20-0	C121243	/	/	/	/	/	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	12.42	/	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	1 balloon	ca.1	/	/

Excel sheet for reagent calculation

/

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. 0h:10 min:6s 2. 19:49:06
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 0h: 25 min: 48 s 2. 20:04:49

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
12.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	8:07-39	Conditioning of H ₂ sensor	NB-346-Logger1 1.7 mV at the end of polarization procedure, stable signal	NB-346.ulog NB-346-Logger1-pre-polarization.csv NB-346-Logger1-pre-polarization.bmp
	ca. 15:10-20	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), done according to Protocol - Liquid phase calibration of H₂ UniAmp sensor with H₂ bubbling.	/	/
		Important note: water was used as a medium for preliminary calibration of H ₂ sensor before performing experiment in D ₂ O.		

	15:38	Start of O2 logging.	NB-346-Ch2-1	2025-11-12_153850_NB-346-Ch2-1.txt 2025-11-12_153850_NB-346-Ch2-1.png
	15:38	Start of H2 logging.	NB-346-Logger2	NB-346.ulog NB-346-Logger2-calibration.csv NB-346-Logger2-calibration step.bmp NB-346-Logger-2point calibration.bmp
	15:41	Degassing was started.	/	20251112_154212-degassing of water.jpg
	16:07	Cannula was moved to gas phase above the liquid level.	/	/
	16:09	Introducing H2 sensor into the reactor under Ar flow.	/	/
	16:17	0 ppm was taken.	/	/
	16:21	Removing cannula, closing the valve.	/	/
	16:22	H2 bubbling of the reactor was started	/	20251112_162312-H2 bubbling.jpg
	16:41	1.000.000 ppm point was taken	830 mV	/
	16:43	New 1.000.000 ppm point was taken and calibration was saved	832 mV, slope: 1.054, 790 μ M	20251112_164009-H2 table.jpg
	16:46	Stop of H2 logging.	/	/
	16:47	Stop of O2 logging.	/	/
	ca. 17:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
	Sample preparation			
	ca. 18:30	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	18:35	Addition of 25 mL D2O to the vial via graduated cylinder.	/	20251112_183257-D2O bottle.jpg
	18:37-40	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer , stage 4/6), covered with Al foil before further use.	/	20251112_184305-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		

	18:45	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	18:50	Assembling the setup.	Currently, stopper instead of H ₂ sensor, PT100, PT1000 and O ₂ robust probe are inside the reactor immersed in the liquid phase	/
	18:54	Start of O ₂ logging.	NB-346-Ch2-2	2025-11-12_185427_NB-346-Ch2-2.txt 2025-11-12_185427_NB-346-Ch2-2.png
	18:58	The degassing was started	Checking rotation of stirring bar, stop-start of stirring when the stirring bar was not rotated properly --> Change in O ₂ curve slope during degassing (afterwards, no problems with stirring were observed, everything was OK).	20251112_185959-degassing of the suspension.jpg
	19:30	Cannula was transferred to gas phase, above the suspension.	/	/
	19:34	H ₂ sensor was added in Ar counterflow.	/	20251112_193815-after immersing H2 sensor.jpg
	19:37	The degassing was stopped by removing the cannula and closing the valve.	/	/
	19:38	Stop of O ₂ logging.	/	/
	19:39	Start of O ₂ logging.	NB-346-Ch2-3	2025-11-12_193900_NB-346-Ch2-3.txt 2025-11-12_193900_NB-346-Ch2-3.png
	19:39	Start of H ₂ logging.	NB-346-Logger3	NB-346.ulog NB-346-Logger3-during irradiation.csv NB-346-Logger3-during irradiation.bmp
	19:39-49	Equilibration time.	/	/
	19:49	The irradiation was started	/	20251112_195028-after start of irradiation.jpg

	20:04	The irradiation was stopped.	/	/
	20:04-14	Equilibration time.	/	/
	20:14	Stop of O2 and H2 logging.	/	/
ca. 20:25		Deassembling the setup, cleaning the reactor.	<p>Tips of the sensors were covered with attached photocatalyst particles. Seems like the particles were less adhesive to the reactor surface than in previous experiments with H₂O.</p> <p>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).</p>	20251112_201719-after irradiation.jpg

Analysis

Used calibration for Firesting: [20250611-BOLA-fitting-liquid-phase-trace-oxygen-sensor-H2-O2 reactor.ini](#)

Used calibration for UniSense: NB-346-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
12.11.2025	8:07	NB-346-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-346.ulog NB-346-Logger1-pre-polarization.csv	/	NB-346-Logger1-pre-polarization.bmp	/	Pre-polarization, stable signal, ca. 1.7 mV.
	15:38	NB-346-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-346.ulog NB-346-Logger2-calibration.csv	/	NB-346-Logger2-calibration step.bmp NB-346-Logger2-point calibration.bmp	/	Calibration in water, 10^6 point - response 832 mV, slope 1.054
	19:39	NB-346-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	D2O	NB-346.ulog NB-346-Logger3-during irradiation.csv	NB-346-O2 and H2 curves.py	NB-346-Logger3-during irradiation.bmp NB-346-O2 and H2 curves.png	/	H2 evolution during irradiation of D2O-based suspension.

	15:38	NB-346-Ch2-1	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-12_153850_NB-346-Ch2-1.txt	/	2025-11-12_153850_NB-346-Ch2-1.png	/	Degassing of water followed by calibration of H ₂ sensor.
	18:54	NB-346-Ch2-2	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	D2O	2025-11-12_185427_NB-346-Ch2-2.txt	/	2025-11-12_185427_NB-346-Ch2-2.png	/	Degassing of D2O-based suspension.
	19:39	NB-346-Ch2-3	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	D2O	2025-11-12_193900_NB-346-Ch2-3.txt	NB-346-O ₂ and H ₂ curves.py	2025-11-12_193900_NB-346-Ch2-3.png NB-346-O₂ and H₂ curves.png	/	O ₂ evolution during irradiation of D2O-based suspension.

Results

Simultaneous H₂ and O₂ measurements of irradiated suspension of EA-358 (0.5 mg/mL in D2O) in O₂/H₂ photoreactor under 365 nm irradiation (50 mW/cm², 20 °C, 15 min) were performed with preliminary calibration of H₂ sensor in water.

Both evolution of H₂ and O₂ were observed but with lower rates and amounts produced than in the case of H₂O-based suspensions.

Linked experiments

SrTiO₃ - NB-316: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C

SrTiO₃ - NB-318: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm², 20 °C

SrTiO₃ - NB-344: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 150 mW/cm², 20 °C

Linked resources

Equipment - [VWR® VV3, Vortex Mixer](#)

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

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

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

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

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

Attached files

NB-346-SrTiO3-photocatalytic H2O splitting.cdxml

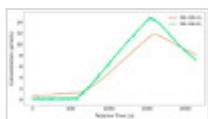
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NB-346-SrTiO3-photocatalytic H2O splitting.png

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NB-346-O2 and H2 curves.png

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NB-346-O2 and H2 curves.py

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20251112_164009-H2 table.jpg

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20251112_183257-D2O bottle.jpg

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20251112_184305-suspension after vortex.jpg

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20251112_185959-degassing of the suspension.jpg

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20251112_193815-after immersing H2 sensor.jpg

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20251112_201719-after irradiation.jpg

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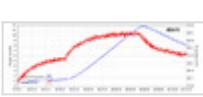
20251112_195028-after start of irradiation.jpg

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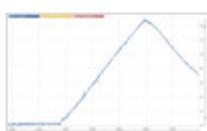


NB-346-Logger3-during irradiation.csv

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NB-346-Logger3-during irradiation.bmp

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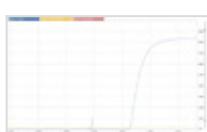


NB-346-Logger2-calibration.csv

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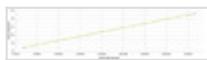
NB-346-Logger2-calibration step.bmp

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NB-346-Logger-2point calibration.bmp

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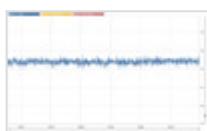


NB-346-Logger1-pre-polarization.csv

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NB-346.ulog

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3446>