

NB-349: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), D₂O, 365 nm, 50 mW/cm², 20 °C (reproduction NB-346)

Date: 2025-11-13

Tags: O₂ Test Calibration NB Firing
O₂ sensor H₂ SrTiO₃ Unisense
RhCrO₃:Al:SrTiO₃ H₂ Sensor
temperature In situ Trace range robust
oxygen sensor photocatalysis Unisense
normal range

Category: SrTiO₃

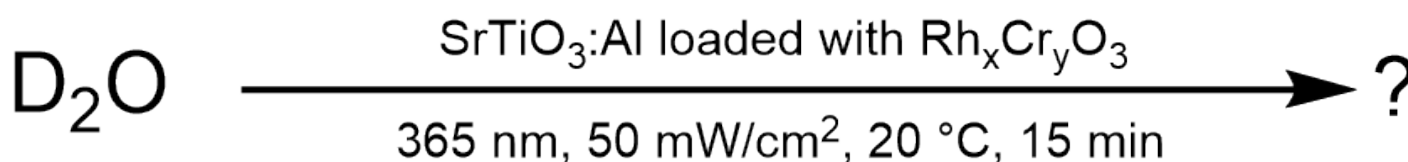
Status: Done

Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-346: simultaneous detection of H₂ and O₂ evolution in liquid 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 (changing dispersion medium).

Reaction scheme



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

Literature/reference experiments

Literature	/
Reproduction	SrTiO ₃ - NB-346: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), D ₂ O, 365 nm, 50 mW/cm ² , 20 °C
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	Inventory number	Amount [mmol]	Equivalents	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Concentration [mM]
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	12.53	/	/	/	/

Excel sheet for reagent calculation

/

Irradiation Parameters

Power measurement was performed using [Power Meter - 843-R-USB + 919P-020-12](#) in [Equipment - Advanced power measurment 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. 0 h : 10 min : 5 s 2. 20:16:18
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 0 h : 25 min : 40 s 2. 20:31:52

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 measurment	Protocol - In-situ hydrogen and oxygen measurment 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 measurment in H ₂ /O ₂ reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
13.11.2025		Calibration from experiment SrTiO ₃ - NB-348: Liquid phase H ₂ and O ₂ of RhCrOx,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C VII (reproduction NB-316) was used. Calibration was performed in H ₂ O.		
		Sample preparation		
	19:04	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	19:06	Addition of 25 mL D ₂ O to the vial via graduated cylinder.	/	/
	19:09-12	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251113_191500-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H ₂ /O ₂ reactor from step 6		
	19:15	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	19:20	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	/
	19:29	Degassing was started.	/	20251113_193027-degassing of the suspension.jpg

	19:38	Start of O2 logging.	NB-349-Ch2-1	2025-11-13_193836_NB-349-Ch2-1.txt 2025-11-13_193836_NB-349-Ch2-1.png
	19:57	Cannula was transferred to gas phase, above the suspension.	/	/
	19:59	H ₂ sensor was added in Ar counterflow.	/	/
	20:02	The degassing was stopped by removing the cannula and closing the valve.	/	/
	20:06	Stop of O2 logging.	/	/
	20:06	Start of O2 logging.	NB-349-Ch2-2	2025-11-13_200612_NB-349-Ch2-2.txt 2025-11-13_200612_NB-349-Ch2-2.png
	20:06	Start of H2 logging.	NB-349-Logger1	NB-349.ulog NB-349-Logger1.csv NB-349-Logger1.bmp
	20:06-16	Equilibration time.	/	/
	20:16	The irradiation was started	/	20251113_201650-after start of irradiation.jpg
	20:31	The irradiation was stopped.	/	/
	20:31-41	Equilibration time.	/	/
	20:41	Stop of O2 and H2 logging.	/	/

	ca. 21:00	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>	20251113_211928-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-348-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
13.11.2025	20:06	NB-349-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	D2O	NB-349.ulong NB-349-Logger1.csv	NB-349-O2 and H2 curve.py	NB-349-Logger1.bmp NB-349-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/cm2, 20 °C	H ₂ evolution during irradiation of D ₂ O-based suspension.
	19:38	NB-349-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	D2O	2025-11-13_193836_NB-349-Ch2-1.txt	/	2025-11-13_193836_NB-349-Ch2-1.png	/	Degassing of D ₂ O-based suspension.
	20:06	NB-346-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	D2O	2025-11-13_200612_NB-349-Ch2-2.txt	NB-349-O2 and H2 curve.py	2025-11-13_200612_NB-349-Ch2-2.png NB-349-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/cm2, 20 °C	O ₂ evolution during irradiation of D ₂ O-based suspension.

Results

Reproduction of NB-346: 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

Prep work - [NB-234: Liquid phase calibration of O2 robust probe in H2/O2 photoreactor](#)

Prep work - [AE-536: Liquid phase calibration in Argon of normal range H2 sensor](#)

SrTiO₃ - [NB-315: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 \(NB-301, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C](#)

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

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

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

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

SrTiO₃ - [NB-348: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C VII \(reproduction NB-316\)](#)

Linked resources

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

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

Equipment - [H2 UniAmp Sensor - Low range - 2.1 x 80 mm needle](#)

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

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

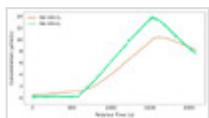
Protocol - [Liquid phase calibration of H2 UniAmp sensor](#)

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

Attached files

NB-349-O2 and H2 curves.png

sha256: d8c7c1db20b516ed59d68b3a498694bd3deca1b3f5da94e7b51dfdded6ff6a96



NB-349-O2 and H2 curve.py

sha256: e8abce5dc4578e7ebcd398a30922770851974ad80229f2abe4d06a5faed7f676

20251113_191500-suspension after vortex.jpg

sha256: 7dd59169a84ea57d264f250fb5ed30a24b764668614648ab8ebb8c5dc7fd1151



20251113_201650-after start of irradiation.jpg

sha256: 011bb854fa6bf45259f6e34965c741ebae9806030c1b001a460b4c710c48fd08



20251113_211928-after irradiation.jpg

sha256: bf56de9229299444cb402884d2b59af4a0f90d52c1ca7beb4929eb4670134aab



20251113_193027-degassing of the suspension.jpg

sha256: 25180bb96cd99eddf435c3b0e1d29c401f59ef80c2ae5945b0b3e4c44b1b4848



2025-11-13_193836_NB-349-Ch2-1.png

sha256: 4fb85c9301110c8b7369ad40c0fa1a4e435eb41ec151bfa29e600e84c125f9d4

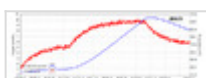


2025-11-13_193836_NB-349-Ch2-1.txt

sha256: 1851195a14db053787491b959f07aa67a30612179d67d79a8018a70acd867395

2025-11-13_200612_NB-349-Ch2-2.png

sha256: d0e619c18e223d49ff1b6664baa1bf0189782a3acd5841863e5058b632a76d8b



2025-11-13_200612_NB-349-Ch2-2.txt

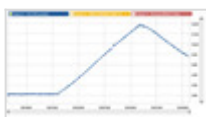
sha256: 7432c62cded8e1de107d82229af6a3900768038ccb8d89a2d5565c078253c205

NB-349-Logger1.csv

sha256: e0f24fe3762328b4929c2c67d90ed4104dda733d485ed572d6f2d93bc2e37c27

NB-349-Logger1.bmp

sha256: c2a138e9d697c85abd91efa5327dcd8e55eb87c614faa3957085b462be4bb47f



NB-349.ulog

sha256: 4f0a38c0b54ae33ea50536c133165cdd50efae79d2b3a36da032dcacc9305ac8



Unique eLabID: 20251113-2e44ef3b05eded3aa9489d4959cd66b0888926b1
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3467>