

NB-319: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316)

Date: 2025-10-21

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

Category: SrTiO₃

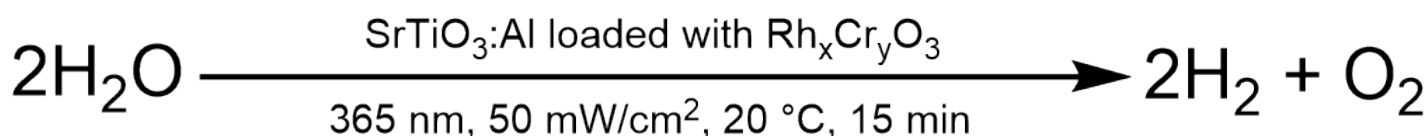
Status: Done

Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-316: simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh_xCrO_y:Al:SrTiO₃ suspension (EA-358, 0.5 mg/mL), 365 nm LED, 50 mW/cm², 20 °C.

Reaction scheme



ChemDraw file linked: [NB-319-SrTiO3-photocatalytic H2O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	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
Similar experiments	SrTiO ₃ - NB-315: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C

Reagents

Name	CAS Number / Experiment Number	Inventor y number	Amount [mmol]	Equivalents	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Pressure [bar]	Concentration [mM]
milli-Q H ₂ O	/	/	/	/	/	/	/	0.998	25 + 25 (for calibration)		/
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	12.74	/	/	/		/

Hydrogen	1333-74-0	/	/	/	/	/	/	/	2 balloons (ca. 2 L)	approx. 1	/
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Excel sheet for reagent calculation

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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. 600 s 2. 17:36:32

Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1518 s 2. 17:51:50
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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
21.10.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	10:50	Conditioning of H ₂ sensor	NB-319-Logger1 1.90 mV at the end of polarization procedure	NB-319.ulog NB-319-Logger1-pre-polarization.csv NB-319-pre-polarization.bmp
	ca. 14:10	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 H ₂ bubbling.	/	
	14:31	Start of O ₂ logging.	NB-319-Ch2-1	2025-10-21_143122_NB-319-Ch2-1.txt 2025-10-21_143122_NB-319-Ch2-1.png

	14:31	Start of H2 logging.	NB-319-Logger2 offset -2 mV	NB-319.ulog NB-319-Logger2-calibration.csv NB-319-2-point-calibration.bmp NB-319-Logger2-calibration step.bmp
	14:35	Degassing was started.	/	20251021_143611-degassing of water.jpg
	15:05	Introducing H2 sensor into the reactor under Ar flow.	/	/
	15:06	0 ppm was taken.	/	/
	15:07	H2 bubbling of the reactor was started	/	/
	15:18	Introducing second H2 balloon.	/	/
	15:31	1.000.000 ppm point was taken and calibration was saved	852 mV, slope: 1.096, 778 uM	20251021_153143-H2 table.jpg
	15:34	Stop of H2 logging.	/	/
	15:34	Stop of O2 logging.	/	/
	ca. 15:40-16:00	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	16:23	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	20251021_162408-weighed photocatalyst.jpg
	16:25	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	16:27-30	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251021_163026-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		

	16:30-40	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	16:40	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	/
	16:45	Start of O2 logging.	NB-319-Ch2-2	2025-10-21_164532_NB-319-Ch2-2.txt 2025-10-21_164532_NB-319-Ch2-2.png
	16:49	The degassing was started	16:52 - touching cabel of the O2 robust probe	20251021_164626-before degassing and irradiation.jpg
	ca. 17:15	Cannula was transferred to gas phase, above the suspension.	/	/
	17:19	H ₂ sensor was added in Ar counterflow.	/	20251021_172048-after introducing H2 sensor.jpg
	17:24	The degassing was stopped by removing the cannula and closing the valve.	/	/
	17:26	Stop of O2 logging.	/	/
	17:26	Start of O2 logging.	NB-319-Ch2-3	2025-10-21_172632_NB-319-Ch2-3.txt 2025-10-21_172632_NB-319-Ch2-3.png
	17:26	Start of H2 logging.	NB-319-Logger3	NB-319.ulog NB-319-Logger3-during irradiation.csv NB-319-Logger3-during irradiation.bmp
	17:26-36	Equilibration time.	/	/
	17:36	The irradiation was started	/	20251021_173644-after start of irradiation.jpg
	17:51	The irradiation was stopped.	/	/
	17:51-18:01	Equilibration time.	/	/
	18:01	Stop of O2 and H2 logging.	/	/

	ca. 18:10	Deassembling the setup, cleaning the reactor.	Tips of the sensors and reactor were covered with attached photocatalyst particles. 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).	20251021_180427-after irradiation.jpg
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Analysis

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

Used calibration for UniSense: NB-319-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
21.10.2025	10:50	NB-319-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-319.ulong NB-319-Logger1-pre-polarization.csv	/	NB-319-pre-polarization.bmp	/	Pre-polarization of H2 sensor.
	14:31	NB-319-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-319.ulong NB-319-Logger2-calibration.csv	/	NB-319-Logger2-calibration step.bmp NB-319-2-point-calibration.bmp	/	Calibration of H2 sensor under degassed conditions. 852 mV, slope 1.096, 778 uM
	17:26	NB-319-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-319.ulong NB-319-Logger3-during irradiation.csv	NB-319-O2 and H2 curve.py	NB-319-Logger3-during irradiation.bmp NB-319-O2 and H2 curves.png	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	Clean response of H2 sensor, but with sharp increase and decrease in the signal upon switching irradiation on and off, H2 value at the end of irradiation ca. 52 uM.
	14:31	NB-319-Ch2-1	Optical O2 detection	Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-21_143122_NB-319-Ch2-1.txt	/	2025-10-21_143122_NB-319-Ch2-1.png	/	Degassing of water before calibration of H2 sensor.
	16:45	NB-319-Ch2-2	Optical O2 detection	Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-21_164532_NB-319-Ch2-2.txt	/	2025-10-21_164532_NB-319-Ch2-2.png	/	Degassing of the photocatalyst suspension.
	17:26	NB-319-Ch2-3	Optical O2 detection	Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-21_172632_NB-319-Ch2-3.txt	NB-319-O2 and H2 curve.py	2025-10-21_172632_NB-319-Ch2-3.png NB-319-O2 and H2 curves.png	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	O2 response during photocatalytic test. O2 value at the end of irradiation ca. 35 uM.

Results

Simultaneous H₂ and O₂ measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O₂/H₂ photoreactor under 365 nm irradiation (50 mW/cm², 20 °C, 15 min) were performed (reproduction of NB-316).

H₂ level at the end of irradiation - 52 umol/L, O₂ level - 37 umol/L.

Future recommendations

Old procedure	Problem	Suggested new procedure
SrTiO3 - NB-313: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 0.5 mg/mL), 365 nm, 50 mW	Sharp increase in H ₂ signal after start and stop of irradiation	Adjust position of the H ₂ sensor tip relatively to the light source so that the open part of the tip should be shadowed (label the position of the tip on the GL18/NS14 adapter with the marker or also on the reactor itself, e.g.)
/	Disturbance of the system during calibration of H ₂ sensor (touching cables of O ₂ robust probe, moving cannula for Ar purging).	Avoid disturbing of the system during calibration of H ₂ sensor during H ₂ purging, then the raise in signal will be smoother (some step may appear after introducing the 2nd H ₂ balloon).

Linked experiments

Prep work - [AE-477: Testing of in-situ electrochemical hydrogen sensor and test H2 evolution reaction with RhCrO:Al:SrTiO3](#)

Prep work - [AE-482: Volume determination of GL14/NS14 flask](#)

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

Prep work - [AE-533: Liquid phase calibration in Argon of normal range H2 sensor](#)

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

Prep work - [NB-310: Measuring power output of UHP-365 nm #1 with 18A-2 in advanced irradiation setup](#)

[SrTiO3 - NB-162: Preparation of SrTiO3:Al with RhCr2-yO3 \(Osterloh route\), I attempt, 1000 C 10h](#)

SrTiO₃ - AE-483: Testing of in-situ electrochemical hydrogen sensor and test H₂ evolution reaction with RhCrO:Al:SrTiO₃

SrTiO₃ - AE-484: Testing of in-situ electrochemical hydrogen sensor and test H₂ evolution reaction with RhCrO:Al:SrTiO₃

SrTiO₃ - NB-AE-215 / AE-485: Gas phase H₂ measurements with H₂ sensor, SrTiO₃:Al loaded with RhxCrO₃ (NB-162-4) suspension (1 mg/ml), 365 nm UHP LED, check with GC measurements

SrTiO₃ - NB-AE-222: Gas phase H₂ measurements with Unisense H₂ sensor, GC calibration

SrTiO₃ - NB-AE-229: Liquid phase H₂ and O₂ measurements with Unisense H₂ sensor, Firesting probe in irradiated Al:SrTiO₃ RhCrOx (NB-162-4, 0.8 mg/mL), 365 nm, 50 mW, 1 h, degassing

SrTiO₃ - AE-534: Liquid phase H₂ and O₂ of RhCrOx,Al:SrTiO₃ (NB-162-4, 1 mg/mL), 365 nm, 50 mW

SrTiO₃ - AE-537: Liquid phase H₂ and O₂ of RhCrOx,Al:SrTiO₃ (NB-237, 1 mg/mL), 365 nm, 50 mW

SrTiO₃ - AE-538: Liquid phase H₂ and O₂ of RhCrOx,Al:SrTiO₃ (NB-237, 1 mg/mL), 365 nm, 50 mW

SrTiO₃ - NB-301: Modification of NB-285 (SrTiO₃:Al (from self-made SrTiO₃, Osterloh, no Al₂O₃, PVDF filter) 1000 C, 10 h) with Rh, Cr oxide cocatalyst I

SrTiO₃ - NB-313: Liquid phase H₂ and O₂ of RhCrOx,Al:SrTiO₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW

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

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](#)

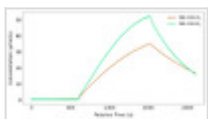
Protocol - [Liquid phase calibration of H₂ UniAmp sensor](#)

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

Attached files

NB-319-O2 and H2 curves.png

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NB-319-O2 and H2 curve.py

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Unisense-NB-319-Screenshot 2025-11-07 092928.png

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NB-319-Logger2-calibration.csv

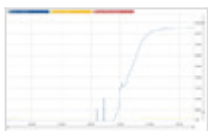
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NB-319-Logger1-pre-polarization.csv

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NB-319-Logger2-calibration step.bmp

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NB-319-2-point-calibration.bmp

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NB-319-SrTiO3-photocatalytic H2O splitting.cdxml

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NB-319-SrTiO3-photocatalytic H2O splitting.png

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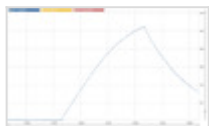


NB-319-Logger3-during irradiation.csv

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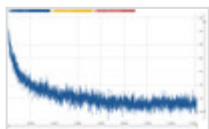
NB-319-Logger3-during irradiation.bmp

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NB-319-pre-polarization.bmp

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NB-319-calibration.bmp

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20251021_180427-after irradiation.jpg

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20251021_173644-after start of irradiation.jpg

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20251021_172048-after introducing H2 sensor.jpg

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20251021_165005degassing of the suspension.jpg

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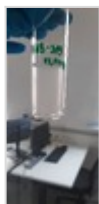
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20251021_163026-suspension after vortex.jpg

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20251021_153143-H2 table.jpg

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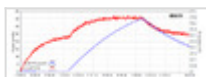


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NB-319.ulog

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3246>