

NB-327: Liquid phase H₂ and O₂ of RhCrO_x:Al:SrTiO₃ (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C

Date: 2025-10-28

Tags: O₂ Test Calibration Future 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

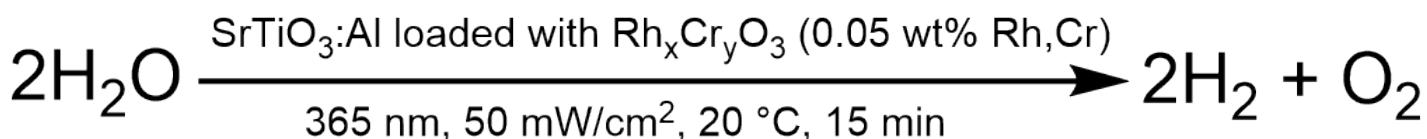
Status: Need to be redone

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Objectives

Simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:SrTiO₃ suspension (NB-321, **0.05 wt% Rh,Cr**, 0.5 mg/mL), 365 nm LED, 50 mW/cm², 20 °C.

Reaction scheme



ChemDraw file linked: [NB-327-SrTiO3-photocatalytic H₂O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO ₃ - NB-326: Liquid phase H ₂ and O ₂ of RhCrO _x :Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm ² , 20 °C (reproduction NB-325)

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]	Concentration [mM]
milli-Q H ₂ O	/	/	/	/	/	/	/	0.998	25 + 25 (for calibration)	/	/
Al:SrTiO ₃ RhCrO _x (NB-321)	SrTiO ₃ - NB-321: Modification of EA-354 (SrTiO ₃ :Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)	/	/	/	12.50	12.87	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	2 balloons (ca. 2 L)	ca. 1 bar	/

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-18000-14	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. 603 s 2. 22:08:19
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1513 s 2. 22:23:30

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
		Calibration from SrTiO ₃ - NB-326: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm ² , 20 °C (reproduction NB-325) was used.	/	/
28.10.2025		Sample preparation		
	20:30	Weighing photocatalyst in a 50 mL vial (the vial was covered with Al foil before further use).	Creamy solid	/
	21:06	Addition of 25 mL H ₂ O to the vial via graduated cylinder.	/	/
	21:06-09	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	Seems less optically dense than for the suspension with 0.1 wt% Rh,Cr catalyst EA-358	20251028_210937-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor from step 6		
	ca. 21:15-20	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	21:20	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	20251028_212335-before degassing.jpg

	21:25	Start of O2 logging.	NB-327-Ch2-1	2025-10-28_212536_NB-327-Ch2-1.txt 2025-10-28_212536_NB-327-Ch2-1.png
	21:30	The degassing was started	/	20251028_213111-suspension during degassing.jpg
	21:52	Cannula was transferred to gas phase, above the suspension.	/	/
	21:54	H ₂ sensor was added in Ar counterflow.	/	/
	21:57	The degassing was stopped by removing the cannula and closing the valve.	/	/
	21:58	Stop of O2 logging.	/	/
	21:58	Start of O2 logging.	NB-327-Ch2-2	2025-10-28_215816_NB-327-Ch2-2.txt 2025-10-28_215816_NB-327-Ch2-2.png
	21:59	Start of H2 logging.	NB-327-Logger1	NB-327.ulog NB-327-Logger1.csv NB-327-Logger1.bmp
	21:59-22:09	Equilibration time.	/	/
	22:09	The irradiation was started	/	20251028_220831-after start of irradiation.jpg
	22:23	The irradiation was stopped.	/	/
	22:23-33	Equilibration time.	/	/
	22:33	Stop of O2 and H2 logging.	/	/

	ca. 22:40	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).	/
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Analysis

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

Used calibration for UniSense: NB-326-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
28.10.2025	21:59	NB-327-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-327.ulog NB-327-Logger1.csv	NB-327-O2 and H2 curve.py	NB-327-Logger1.bmp NB-327-O2 and H2 curves.png	/	Clean response signal, slight decrease at 22:21. H2 value 34 uM H2
	21:25	NB-327-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-28_212536_NB-327-Ch2-1.txt	/	2025-10-28_212536_NB-327-Ch2-1.png	/	Degassing of the suspension.
	21:58	NB-327-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-28_215816_NB-327-Ch2-2.txt	NB-327-O2 and H2 curve.py	2025-10-28_215816_NB-327-Ch2-2.png NB-327-O2 and H2 curves.png	/	A bit sharp increase in O2 value after start of irradiation, sharp decrease after stop of irradiation. O2 value 32 uM.

Results

Simultaneous H₂ and O₂ measurements of irradiated suspension of NB-321 (0.05 wt% RhCr, 0.5 mg/mL) in O₂/H₂ photoreactor under 365 nm irradiation (50 mW/cm², 20 °C, 15 min) were performed.

H₂ level at the end of irradiation - 34 umol/L, O₂ level - 32 umol/L.

Future recommendations

Old procedure	Problem	Suggested new procedure
/	Sharp increases in O ₂ and H ₂ signals - maybe related either with the position of the sensors in the reactor or the optical density.	<ul style="list-style-type: none"> • Adjust the position of the sensors relatively to the light beam. • If necessary, destroy the lumps of the solid photocatalyst before adding water and vortexing to improve dispersion quality.

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-321: Modification of EA-354 (SrTiO₃:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%)

SrTiO₃ - NB-325: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm², 20 °C

SrTiO₃ - NB-326: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm², 20 °C (reproduction NB-325)

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](#)

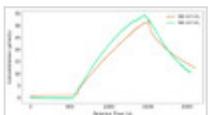
Protocol - [In-situ hydrogen and oxygen measurement in H₂/O₂ reactor](#)

Attached files

NB-327-O2 and H2 curve.py
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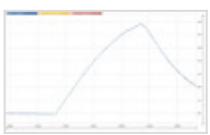
NB-327-O2 and H2 curves.png

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NB-327-Logger1.bmp

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NB-327-Logger1.csv

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NB-327-SrTiO3-photocatalytic H2O splitting.cdxml

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NB-327-SrTiO3-photocatalytic H2O splitting.png

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20251028_212335-before degassing.jpg

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20251028_213111-suspension during degassing.jpg

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20251028_210937-suspension after vortex.jpg

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20251028_220831-after start of irradiation.jpg

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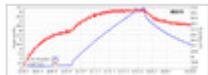


NB-327.ulog

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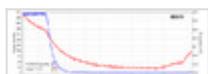


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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3289>