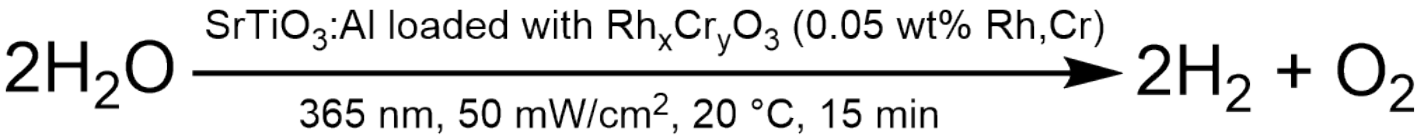
NB-357: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-341, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reproduction NB-354)

Date: 2025-11-21
Tags: O2 | Test | Calibration | 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-354: simultaneous detection of H_2 and O_2 evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:$SrTiO_3$ suspension (NB-341, **0.05 wt% Rh,Cr**, 0.5 mg/mL), 365 nm LED, 50 mW/cm^2, 20 °C.

Reaction scheme



ChemDraw file linked: NB-327-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	/
Similar experiments	SrTiO3 - NB-327: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-321, 0.05 wt% Rh, Cr, 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

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]
milli-Q H_2O	/	/	/	/	/	/	/	0.998	25.00	/

Al:SrTiO3 RhCrOx (NB-341)	SrTiO3 - NB-341: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%), fresh solutions of RhCl3 and Cr(NO3)3	/	/	/	12.50	12.50	/	/	/	/

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-18000-14	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. 605 s 2. 2:27:16
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1515 s 2. 2:42:26

O_2/H_2 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 O2 measurment	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor
Used H_2 sensor	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor

Procedure/observations

Date	Time	Step	Observations	Pictures/Files
		Calibration from SrTiO3 - NB-356: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C IX (reproduction NB-316) was used.	/	/
21.11.2025		**Sample preparation**		
	1:05	Weighing photocatalyst in a 50 mL vial.	Slightly creamy solid	/
		Important note: before weighing, the solid was dispersed inside the vial manually with Smartspatula - to break lumps manually, but to avoid mortaring.		
	1:06	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/

	1:09-12	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251121_011342-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	1:15	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	1:20	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	/
	1:29	Start of O2 logging.	**NB-357-Ch2-1**	2025-11-21_012927_NB-357-Ch2-1.txt 2025-11-21_012927_NB-357-Ch2-1.png
	1:32	The degassing was started	/	20251121_013310-degassing of the suspension.jpg
	2:07	Cannula was transferred to gas phase, above the suspension.	/	/
	2:10	H_2 sensor was added in Ar counterflow.	/	/
	2:13	The degassing was stopped by removing the cannula and closing the valve.	/	/
	2:17	Stop of O2 logging.	/	/
	2:17	Start of O2 logging.	**NB-357-Ch2-2**	2025-11-21_021711_NB-357-Ch2-2.txt 2025-11-21_021711_NB-357-Ch2-2.png
	2:17	Start of H2 logging.	**NB-357-Logger1**	NB-357.ulog NB-357-Logger1.csv NB-357-Logger1.bmp
	2:17-27	Equilibration time.	/	/
	2:27	The irradiation was started	/	20251121_022807-after start of irradiation.jpg
	2:42	The irradiation was stopped.	/	/

	2:42-52	Equilibration time.	/	/
	2:52	Stop of O2 and H2 logging.	/	/
	ca. 3:00	Deassembling the setup, cleaning the reactor.	Tips of the sensors and reactor were covered with attached photocatalyst particles, bubbles attached to the surface of the sensors tips. 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).	20251121_025513-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-356-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
21.11.2025	2:17	NB-357-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-357.ulog NB-357-Logger1.csv	NB-357-O2 and H2 curve.py	NB-357-Logger1.bmp NB-357-O2 and H2 curves.png	SrTiO3 - NB-354: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-341, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C	H2 evolution during irradiation.
	1:29	NB-357-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-21_012927_NB-357-Ch2-1.txt	/	2025-11-21_012927_NB-357-Ch2-1.png	/	Degassing of the suspension.
	2:17	NB-357-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-21_021711_NB-357-Ch2-2.txt	NB-357-O2 and H2 curve.py	2025-11-21_021711_NB-357-Ch2-2.png NB-357-O2 and H2 curves.png	SrTiO3 - NB-354: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-341, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C	O2 evolution during irradiation

Results

Reproduction of NB-354: simultaneous H_2 and O_2 measurements of irradiated suspension of NB-341 (0.05 wt% RhCr, 0.5 mg/mL) in O_2/H_2 photoreactor under 365 nm irradiation (50 mW/cm^2, 20 °C**,** 15 min) were performed.

Linked experiments

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

SrTiO3 - NB-322: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 100 mW/cm2, 20 °C (reproduction NB-318)

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

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

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

SrTiO3 - NB-331: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reprodution NB-316) I I I

SrTiO3 - NB-332: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reproduction NB-327)

SrTiO3 - NB-341: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%), fresh solutions of RhCl3 and Cr(NO3)3

SrTiO3 - NB-354: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-341, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

SrTiO3 - NB-356: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C IX (reproduction NB-316)

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-357-O2 and H2 curve.py
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NB-357-O2 and H2 curves.png
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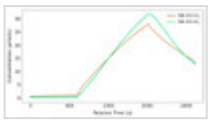

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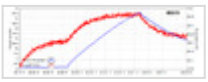

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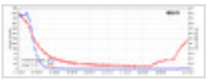

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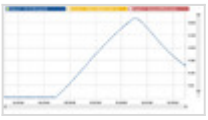

NB-357.ulog
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