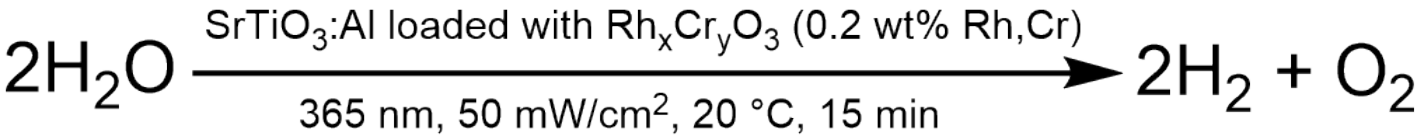
NB-355: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-342, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C

Date: 2025-11-19
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

Simultaneous detection of H_2 and O_2 evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:$SrTiO_3$ suspension (NB-342, **0.2 wt% Rh,Cr**, 0.5 mg/mL), 365 nm LED, 50 mW/cm^2, 20 °C.

Reaction scheme



ChemDraw file linked: NB-328-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	/
Similar 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-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 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	/
Al:SrTiO3 RhCrOx (NB-342)	SrTiO3 - NB-342: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%), fresh solutions of RhCl3 and Cr(NO3)3	/	/	/	12.50	12.54	/	/	/	/

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. 603 s 2. 22:18:33
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1510 s 2. 22:43:38

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-353: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VIII (reproduction NB-316) was used.	/	/
19.11.2025		**Sample preparation**		
	21:00	Weighing photocatalyst in a 50 mL vial (the vial was covered with Al foil before further use) (preliminary, the lumps of the solid were broken with Smartspatula inside the vial).	Slightly grey solid	/
	21:05	Addition of 25 mL H_2O to the vial via graduated cylinder.	/	/
	21:14-17	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251119_212245-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	21:25	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	21:30	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	/
	21:33	Start of O2 logging.	**NB-355-Ch2-1**	2025-11-19_213326_NB-355-Ch2-1.txt 2025-11-19_213326_NB-355-Ch2-1.png

	21:36	The degassing was started	/	20251119_213729-degassing of the suspension.jpg
	22:09	Cannula was transferred to gas phase, above the suspension.	/	/
	22:12	H_2 sensor was added in Ar counterflow.	/	/
	22:15	The degassing was stopped by removing the cannula and closing the valve.	/	/
	22:17	Stop of O2 logging.	/	/
	22:18	Start of O2 logging.	**NB-333-Ch2-2**	2025-11-19_221828_NB-355-Ch2-2.txt 2025-11-19_221828_NB-355-Ch2-2.png
	22:18	Start of H2 logging.	**NB-333-Logger1** ca. 22:26 - very slight movement of the holder to fit in the setup box completely	NB-355.ulog NB-355-Logger1.csv NB-355-Logger1.bmp
	22:18-28	Equilibration time.	/	/
	22:28	The irradiation was started	/	20251119_222851-after start of irradiation.jpg
	22:43	The irradiation was stopped.	/	/
	22:43-53	Equilibration time.	/	/
	22:53	Stop of O2 and H2 logging.	/	/
	ca. 23:00	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).	20251119_231806-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-353-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
19.11.2025	22:18	NB-355-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-355.ulog NB-355-Logger1.csv	NB-355-O2 and H2 curve.py	NB-355-Logger1.bmp NB-355-O2 and H2 curves.png	/	H2 evolution during irradiation.
	21:33	NB-355-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-19_213326_NB-355-Ch2-1.txt	/	2025-11-19_213326_NB-355-Ch2-1.png	/	Degassing of the suspension.
	22:18	NB-355-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-19_221828_NB-355-Ch2-2.txt	NB-355-O2 and H2 curve.py	2025-11-19_221828_NB-355-Ch2-2.png NB-355-O2 and H2 curves.png	/	O2 evolution during irradiation.

Results

Simultaneous H_2 and O_2 measurements of irradiated suspension of NB-342 (0.5 mg/mL, 0.2 wt% RhCr) 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-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-323: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%)

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-328: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 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-333: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C (reproduction NB-328)

SrTiO3 - NB-342: Modification of EA-354 (SrTiO3:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%), fresh solutions of RhCl3 and Cr(NO3)3

SrTiO3 - NB-353: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VIII (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 - In-situ hydrogen and oxygen measurment in H2/O2 reactor

Attached files

NB-355-O2 and H2 curve.py
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NB-355-O2 and H2 curves.png
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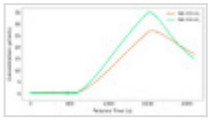

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20251119_213729-degassing of the suspension.jpg
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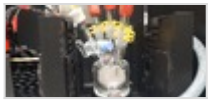

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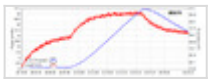

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NB-355-Logger1.bmp
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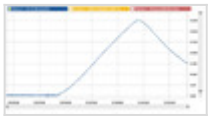

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NB-355.ulog
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