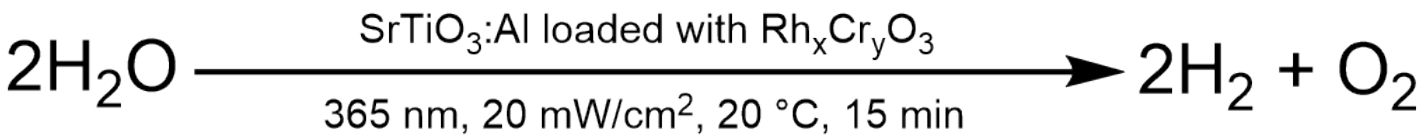
NB-345: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 20 mW/cm2, 20 °C (reproduction NB-325) I I

Date: 2025-11-12
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

Second reproduction of NB-325: simultaneous detection of H_2 and O_2 evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:$SrTiO_3$ suspension (EA-358, 0.5 mg/mL), 365 nm LED, **20 mW/cm²**, 20 °C.

Reaction scheme



ChemDraw file linked: NB-325-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	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-337: 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) I
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

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 (EA-358)	SrTiO3 - EA-358: Modification of Al:SrTiO3 (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	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-324: Measuring power output of UHP-365 nm #4 with 18A-4 in advanced irradiation setup I I

	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	22	0.07

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. 2:23:47
Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1515 s 2. 2:38:57

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
12.11.2025		Calibration from SrTiO3 - NB-339: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VI (reproduction NB-316) was used		
		Sample preparation		
	1:10	Weighing **EA-358** photocatalyst in a 50 mL vial.	Creamy solid.	/
	1:15	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/
	1:18-21	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251112_012130-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	1:30	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	1:35	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	/
	1:36	Start of O2 logging.	**NB-345-Ch2-1**	2025-11-12_013647_NB-345-Ch2-1.txt 2025-11-12_013647_NB-345-Ch2-1.png

	1:39	The degassing was started	/	20251112_014003-degassing of the suspension.jpg
	2:05	Cannula was transferred to gas phase, above the suspension.	/	/
	2:07	H_2 sensor was added in Ar counterflow.	/	/
	2:10	The degassing was stopped by removing the cannula and closing the valve.	/	/
	2:13	Stop of O2 logging.	/	/
	2:13	Start of O2 logging.	**NB-345-Ch2-2**	2025-11-12_021341_NB-345-Ch2-2.txt 2025-11-12_021341_NB-345-Ch2-2.png
	2:13	Start of H2 logging.	**NB-345-Logger1**	NB-345.ulog NB-345-Logger1.csv NB-345-Logger1.bmp
	2:13-23	Equilibration time.	/	/
	2:23	The irradiation was started	/	/
	2:38	The irradiation was stopped.	/	/
	2:38-48	Equilibration time.	/	/
	2:48	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. 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).	/

Analysis

Used calibration for Firesting: 20250611-BOLA-fitting-liquid-phase-trace-oxygen-sensor-H2-O2 reactor.ini

Used calibration for UniSense: NB-339-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
12.11.2025	2:13	NB-345-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-345.ulog NB-345-Logger1.csv	NB-345-O2 and H2 curves.py	NB-345-Logger1.bmp NB-345-O2 and H2 curves.png	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	H2 evolution during irradiation, some small spikes in H2 curve.
	1:36	NB-345-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-12_013647_NB-345-Ch2-1.txt	/	2025-11-12_013647_NB-345-Ch2-1.png	/	Degassing of the suspension.
	2:13	NB-345-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-12_021341_NB-345-Ch2-2.txt	NB-345-O2 and H2 curves.py	2025-11-12_021341_NB-345-Ch2-2.png NB-345-O2 and H2 curves.png	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	O2 evolution during irradiation.

Results

Simultaneous H_2 and O_2 measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O_2/H_2 photoreactor under 365 nm irradiation (20 mW/cm^2, 20 °C**,** 15 min, reproduction of NB-325) were performed.

The problem of sharp increase/decrease in O2 and H2 signals was eliminated.

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-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-337: 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) I

SrTiO3 - NB-339: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C VI (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

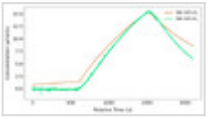
NB-325-SrTiO3-photocatalytic H2O splitting.png
sha256: 84aba7b2eaa7e7c6c75ec4bc4edf260ee29035205566e821b254e3f00db612f8

NB-325-SrTiO3-photocatalytic H2O splitting.cdxml
sha256: 1e25f76a703054a4e3a1c95ca0b8c004e2e67c7a646758e715ac13662fa83a8c

NB-345-O2 and H2 curves.py

sha256: 977f34736b27b2d1a8b198c425bf7a688213a4cb55a45d22c74179055f574029

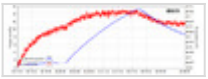
NB-345-O2 and H2 curves.png
sha256: 93b379c2daa19a77efa0e4756e7296c43d83ee83859da141b0ef9cf9476003c6



20251112_014003-degassing of the suspension.jpg
sha256: 0cd1612499883380a97288ef5c29e9a8a24aadf6c7ac0eca12bb35b7bf26ad0e

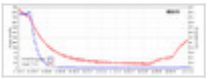
20251112_012130-suspension after vortex.jpg
sha256: 189ea9bb328fecb2fe15a184bc82d30dac3d2ec54938d6b2272df28fee734c88

2025-11-12_021341_NB-345-Ch2-2.png
sha256: a9868e3da46e95306cada16c32a4e53a7187e76076e06752f8d82d9f101efb08



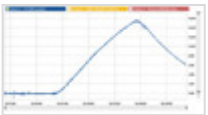
2025-11-12_021341_NB-345-Ch2-2.txt
sha256: 81b1268f3a17f872b3e4d2d21efbc91483a48509d7375972f15ce4beb871880f

2025-11-12_013647_NB-345-Ch2-1.png
sha256: e8a07c69587948925dc23394543836b98d2a8f6f8051e39acd08988e2e5423af



2025-11-12_013647_NB-345-Ch2-1.txt
sha256: 05511859cacfeb46ee777dfebf614e9417636f28004e94bae78db353d5758f2e

NB-345-Logger1.bmp
sha256: 7e4f88d08db73db362f82c0983ddf0d81456cacd8c2b77dc3e56f3e357f4d220



NB-345.ulog
sha256: 8c8c4cf95e1031e296ef966a1b925fe2012791794cec35487ab65c07215fedb6

NB-345-Logger1.csv
sha256: 125596b9cdc3cc6147872d458299b3ce7969b269507c41e58ad404271c106b72

Unique eLabID: 20251112-97cf9f89462666fff6e597a0a137d476e85965a8
Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3424