NB-334: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C (reproduction NB-330)
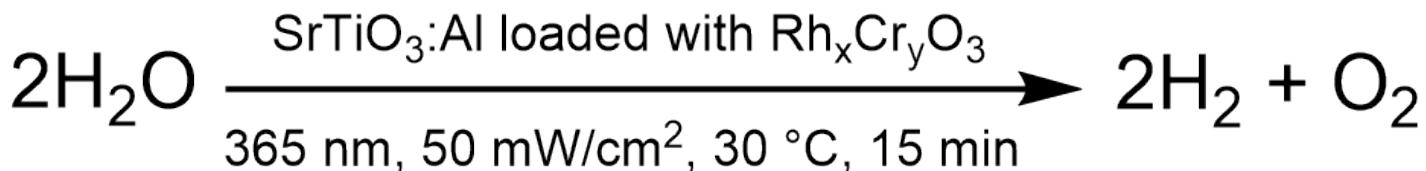
Objectives

Reproduction of NB-330: 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, 50 mW/cm^2, **30 °C**.

Reaction scheme

$$2H_2O \xrightarrow[\text{365 nm, 50 mW/cm}^2,\ 30\ °C,\ 15\ \text{min}]{\text{SrTiO}_3\text{:Al loaded with Rh}_x\text{Cr}_y\text{O}_3} 2H_2 + O_2$$

ChemDraw file linked: NB-330-SrTiO3-photocatalytic H2O splitting.cdxml

Literature/reference experiments

Literature	/
Reproduction	SrTiO3 - NB-330: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C
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]	Pressue [bar]	Concentration [mM]
milli-Q H$_2$O	/	/	/	/	/	/	/	0.998	25 + 25 (for calibration)	/	/
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	12.51	/	/	/	/	/

| Hydrogen | 1333-74-0 | / | / | / | / | / | / | / | 2 ballons (ca. 2 L) | ca. 1 | / |

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-18000-1 4	365	56	0.19

Used beam combiner [Name or None]	/
Irradiation distance [cm]	6.5
Thermostat temperature [°C]	30
Stirring speed [rpm]	500
Irradiation start: 1. Firesting [relative to start log] 2. Unisense	1. 601 s 2. 17:16:13

Irradiation stop: **1. Firesting [relative to start log]** **2. Unisense**	1. 1513 s 2. 17:41:21

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
05.11.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor Important steps and deviations are listed below	/	/
	10:08-11:15	Conditioning of H2 sensor	**NB-334-Logger1** 1.7 mV at the end of polarization procedure	NB-334.ulog NB-334-Logger1-pre-polarization.csv NB-334-Logger1-pre-polarization.bmp
	11:20-45	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 30 °C) done according to Protocol - Liquid phase calibration of H2 UniAmp sensor with H2 bubbling.	/	/
	11:45-12:14	Keeping the system at 30 °C (control by PT1000 sensor immersed in water inside the reactor).	/	/
	12:14	Start of O2 logging.	**NB-334-Ch2-1**	2025-11-05_121425_NB-334-Ch2-1.txt 2025-11-05_121425_NB-334-Ch2-1.png

	12:17	Degassing was started.	ca. 13:00 - increasing Ar flow a bit	20251105_121826-during degassing of water.jpg
	12:18	Start of H2 logging.	**NB-330-Logger2** offset -2 mV	NB-334.ulog NB-334-Logger2-calibration.csv NB-334-Logger-2point-calibration.bmp NB-334-calibration-step.bmp
	13:04	Moving cannula to the gas phase above the liquid	/	/
	13:05	Introducing H2 sensor into the reactor under Ar flow.	Offset -3 mV	/
	13:08	0 ppm was taken.	/	/
	13:10	Stop degassing by removing the cannula, closing the valve.	/	/
	13:17	H2 bubbling of the reactor was started by introducing the first H2 balloon.	ca. 13:20 - very slight moving of H2 sensor, slight change in the slope of the curve	20251105_131800-introducing 1 H2 balloon.jpg
	13:25	Introducing second H2 balloon.	/	20251105_132700-introducing 2nd H2 balloon.jpg
	13:31	1.000.000 ppm point was taken and calibration was saved	999 mV, slope: 1.315, 759.873 uM	20251105_133102-H2 table.jpg
	13:32	Stop of H2 logging.	/	/
	13:32	Stop of O2 logging.	/	/
	13:35	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation		
	15:45	Weighing **EA-358** photocatalyst in a 50 mL vial.	Creamy solid, manually breaking lumps inside the vial using Smartspatula	/
	15:50	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/

	15:51-54	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251105_155807-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6		
	ca. 16:00	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	20251105_160440-suspension inside the reactor.jpg
	ca. 16:05-10	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	/
	16:10-25	Stirring of the suspension at 30 °C.	/	20251105_162423-assembling setup.jpg
	16:25	Start of O2 logging.	**NB-334-Ch2-2**	2025-11-05_162517_NB-334-Ch2-2.txt 2025-11-05_162517_NB-334-Ch2-2.png
	16:28	The degassing was started	16:43 - The glass connector popped up from the tubing in Ar line --> problem eliminated using screw driver and heat gun (to provide better fixing of the connector)	20251105_162942-degassing of the suspension.jpg
	16:54	Continuation of degassing.	/	/
	17:08	Cannula was transferred to gas phase, above the suspension.	/	/
	17:11	H_2 sensor was added in Ar counterflow.	/	20251105_175313-after introducing H2 sensor.jpg
	17:14	The degassing was stopped by removing the cannula and closing the valve.	/	/
	17:15	Stop of O2 logging.	/	/
	17:16	Start of O2 logging.	**NB-334-Ch2-3**	2025-11-05_171608_NB-334-Ch2-3.txt 2025-11-05_171608_NB-334-Ch2-3.png

	17:16	Start of H2 logging.	**NB-334-Logger3**	NB-334.ulog NB-334-Logger3-during irradiation.csv NB-334-Logger3-during irradiation.bmp
	17:16-26	Equilibration time.	/	/
	17:26	The irradiation was started	/	20251105_172634-after start of irradiation.jpg
	17:41	The irradiation was stopped.	/	/
	17:41-51	Equilibration time.	/	/
	17:51	Stop of O2 and H2 logging.	/	/
	ca. 17:55	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-334-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
05.11.2025	10:08	NB-334-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-334.ulog NB-334-Logger1-pre-polarization.csv	/	NB-334-Logger1-pre-polarization.bmp	/	1.7 mV at the end of polarization
	12:18	NB-334-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-334.ulog NB-334-Logger2-calibration.csv	/	NB-334-calibration-step.bmp NB-334-Logger-2point-calibration.bmp	/	Calibration with 1.000.000 ppm pint: slope of 1.315

Time	Name	Method	Equipment	Medium	Data files	Script	Image files	Linked	Notes
17:16	NB-334-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-334.ulog NB-334-Logger3-during irradiation.csv	NB-334-O2 and H2 curve.py	NB-334-Logger3-during irradiation.bmp NB-334-O2 and H2 curve.png	SrTiO3 - NB-330: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C	worked, clean signal response, ca. 58 uM at the end of irradiation
12:14	NB-334-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-05_121425_NB-334-Ch2-1.txt	/	2025-11-05_121425_NB-334-Ch2-1.png	/	O2 measurements during H2 calibration (degassing with Ar and purging with H2 afterwards).
16:25	NB-334-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-05_162517_NB-334-Ch2-2.txt	/	2025-11-05_162517_NB-334-Ch2-2.png	/	Degassing of the photocatalyst suspension.
17:16	NB-334-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel	water	2025-11-05_171608_NB-334-Ch2-3.txt	NB-334-O2 and H2 curve.py	2025-11-05_171608_NB-334-Ch2-3.png NB-334-O2 and H2 curve.png	SrTiO3 - NB-330: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C	worked, ca. 47 umol/L at the end of irradiation

Results

Successful reproduction of NB-330: 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 (50 mW/cm^2, **30 °C,** 15 min) were performed.

H_2 level at the end of irradiation - ca. 58 umol/L, O_2 level - ca. 47 umol/L.

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-330: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 30 °C

Linked resources

Equipment - VWR® VV3, Vortex Mixer

Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 2)

Equipment - Advanced power measurment chamber V1.0 I

Equipment - Irradiation setup 4 (CEEC II, E002)

Light Source - UHP LED 365 nm-4

Power Sources - BLS-18000-1 4

Protocol - Getting hydrogen from hydrogen bottle in CEEC II E014

Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor
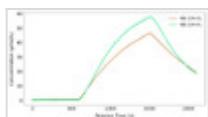
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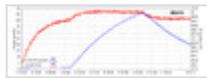

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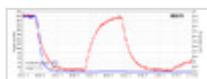
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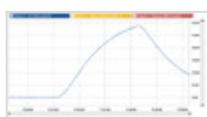
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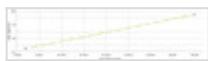
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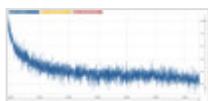

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