

NB-354: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (NB-341, 0.05 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

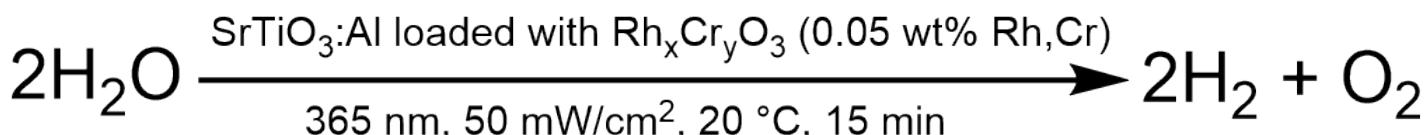
Status: Done

Created by: Nadzeya Brezhneva

Objectives

Simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:SrTiO₃ suspension (NB-341, 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-316: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C SrTiO ₃ - NB-327: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (NB-321, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 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 ₂ O	/	/	/	/	/	/	/	0.998	25	/

Al:SrTiO ₃ RhCrO _x (NB-341)	SrTiO ₃ - NB-341: Modification of EA-354 (SrTiO ₃ :Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.05 wt%), fresh solutions of RhCl ₃ and Cr(NO ₃) ₃	/	/	/	12.50	12.64	/	/	/	/	/
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Excel sheet for reagent calculation

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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-18000-1 4	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. 615 s 2. 22:29:35
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1525 s 2. 22:44:45

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
19.11.2025		Calibration from SrTiO ₃ - NB-353: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C VIII (reproduction NB-316) was used.	/	/
		Sample preparation		
	18:05	Weighing photocatalyst in a 50 mL vial.	Slightly creamy solid	/
	18:07	Addition of 25 mL H ₂ O to the vial via graduated cylinder.	/	/
	18:14-17	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.	/	20251119_182157-suspension after vortex.jpg
		Important note: after vortex, residual lumps of the solid were dispersed inside the vial manually with Smartspatula.		
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor from step 6		

	18:20	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	18:30	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	/
	18:33	Start of O ₂ logging.	NB-354-Ch2-1	2025-11-19_183310_NB-354-Ch2-1.txt 2025-11-19_183310_NB-354-Ch2-1.png
	18:34	The degassing was started	/	20251119_183800-degassing of the suspension.jpg
	19:10	Cannula was transferred to gas phase, above the suspension.	/	/
	19:13	H ₂ sensor was added in Ar counterflow.	/	/
	19:23	The degassing was stopped by removing the cannula and closing the valve.	/	/
	19:27	Stop of O ₂ logging.	/	/
	19:27	Start of O ₂ logging.	NB-354-Ch2-2	2025-11-19_192710_NB-354-Ch2-2.txt 2025-11-19_192710_NB-354-Ch2-2.png
	19:27	Start of H ₂ logging.	NB-354-Logger1	NB-354.ulog NB-354-Logger1.bmp
	19:29	Stop of O ₂ and H ₂ loggings.	Adjusting position of sensors sensors and reactor in the setup	/
	19:31	Start of O ₂ logging.	NB-354-Ch2-3	2025-11-19_193117_NB-354-Ch2-3.txt 2025-11-19_193117_NB-354-Ch2-3.png
	19:31	Start of H ₂ logging.	NB-354-Logger2	NB-354.ulog NB-354-Logger2-during irradiation.csv NB-354-Logger2-during irradiation.bmp
	19:31-41	Equilibration time.	/	/
	19:41	The irradiation was started	/	20251119_194218-after start of irradiation.jpg
	19:56	The irradiation was stopped.	/	/
	19:56-20:06	Equilibration time.	/	/
	20:06	Stop of O ₂ and H ₂ logging.	/	/

	ca. 20:15	Deassembling the setup, cleaning the reactor.	<p>Tips of the sensors and reactor were covered with attached photocatalyst particles, bubbles attached to the surface of the sensors tips.</p> <p>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).</p>	20251119_201739-after irradiation.jpg
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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-353-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
19.11.2025	19:27	NB-354-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-354.ulog	/	NB-354-Logger1.bmp	/	Adjusting sensors position is needed.
	19:31	NB-354-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-354.ulog NB-354-Logger2-during irradiation.csv	NB-354-O2 and H2 curve.py	NB-354-Logger2-during irradiation.bmp NB-354-O2 and H2 curves.png	/	H ₂ evolution during irradiation.
	18:33	NB-354-Ch2-1	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-19_183310_NB-354-Ch2-1.txt	/	2025-11-19_183310_NB-354-Ch2-1.png	/	Degassing of the suspension.
	19:27	NB-354-Ch2-2	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-19_192710_NB-354-Ch2-2.txt	/	2025-11-19_192710_NB-354-Ch2-2.png	/	Adjusting sensors position is needed.
	19:31	NB-354-Ch2-3	Optical O ₂ detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-19_193117_NB-354-Ch2-3.txt	NB-354-O2 and H2 curve.py	2025-11-19_193117_NB-354-Ch2-3.png NB-354-O2 and H2 curves.png	/	O ₂ evolution during irradiation.

Results

Simultaneous H₂ and O₂ measurements of irradiated suspension of NB-341 (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.

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-322: Liquid phase H₂ and O₂ of RhCrO_x,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 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)

SrTiO₃ - 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

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

SrTiO₃ - NB-332: 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 (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-353: Liquid phase H₂ and O₂ of RhCrO_x,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 measurement in H₂/O₂ reactor

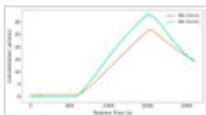
Attached files

NB-354-O2 and H2 curve.py

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NB-354-O2 and H2 curves.png

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20251119_201739-after irradiation.jpg

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20251119_194218-after start of irradiation.jpg

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20251119_183800-degassing of the suspension.jpg

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20251119_182157-suspension after vortex.jpg

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NB-354.ulog

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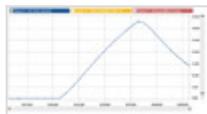
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NB-354-Logger2-during irradiation.bmp

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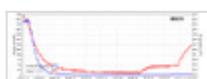


NB-354-Logger2-during irradiation.csv

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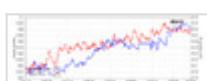


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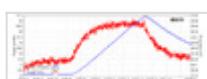


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