

# NB-336: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C V (reproduction NB-316)

Date: 2025-11-10

Tags: O<sub>2</sub> Test Calibration Future NB  
Firing O<sub>2</sub> sensor H<sub>2</sub> SrTiO<sub>3</sub>  
troubleshooting Unisense  
RhCrO<sub>3</sub>:Al:SrTiO<sub>3</sub> H<sub>2</sub> Sensor  
temperature In situ Trace range robust  
oxygen sensor photocatalysis Unisense  
normal range

Category: SrTiO<sub>3</sub>

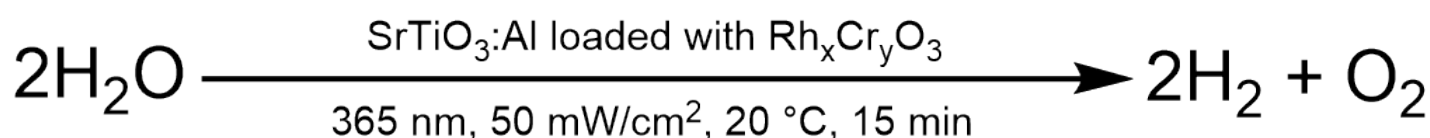
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Reproduction of NB-316: simultaneous detection of H<sub>2</sub> and O<sub>2</sub> evolution in liquid phase for irradiated suspension of Rh,CrOx:Al:SrTiO<sub>3</sub> suspension (EA-358 sample, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 15 min (reference conditions).

## Reaction scheme



ChemDraw file linked: [NB-316-SrTiO3-photocatalytic H2O splitting.cdxml](#)

## Literature/reference experiments

Literature	/
Reproduction	SrTiO <sub>3</sub> - NB-316: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrOx,Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C
Similar experiments	SrTiO <sub>3</sub> - NB-313: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrOx,Al:SrTiO <sub>3</sub> (NB-301, 0.5 mg/mL), 365 nm, 50 mW SrTiO <sub>3</sub> - NB-315: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrOx,Al:SrTiO <sub>3</sub> (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C

## Reagents

Name	CAS Number / Experiment Number	Inventory number	Amount [mmol]	Equivalents	Mass <sub>theo</sub> [mg]	Mass <sub>exp</sub> [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Pressure [bar]	Concentration [mM]
milli-Q H <sub>2</sub> O	/	/	/	/	/	/	/	0.998	25 + 25 (for calibration)	/	/

Al:SrTiO <sub>3</sub> RhCrO <sub>x</sub> (EA-358)	SrTiO <sub>3</sub> - EA-358: Modification of Al:SrTiO <sub>3</sub> (EA-354) via deposition of Rh, Cr oxide co- catalyst, 350°C, 1h, Upscaling (3.33x)	/	/	/	12.50	12.67	/	/	/	/	/
Hydrogen	1333-74-0	/	/	/	/	/	/	/	2 balloons (ca. 2 L)	approx. 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	<a href="#">Equipment - Advanced irradiation setup V1.0 I</a>
Irradiation setup number	<a href="#">Equipment - Irradiation setup 4 (CEEC II, E002)</a>

	Light Source Name	Power Source Name	Wavelength [nm]	Power Setting [mW]	Analog Setting [0.00 - 10.00]
<b>First light source</b>	<a href="#">Light Source - UHP LED 365 nm-4</a>	<a href="#">Power Sources - BLS-18000-1 4</a>	365	56	0.19

<b>Used beam combiner [Name or None]</b>	/
<b>Irradiation distance [cm]</b>	6.5
<b>Thermostat temperature [°C]</b>	20

<b>Stirring speed [rpm]</b>	500
<b>Irradiation start:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 600 s 2. 17:45:13
<b>Irradiation stop:</b> <b>1. Firesting [relative to start log]</b> <b>2. Unisense</b>	1. 1508 s 2. 18:00:21

## O<sub>2</sub>/H<sub>2</sub> 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 <sub>2</sub> sensor	Equipment - Robust probe for liquid O <sub>2</sub> measurment	Protocol - In-situ hydrogen and oxygen measurment in H <sub>2</sub> /O <sub>2</sub> reactor
Used H <sub>2</sub> sensor	Equipment - H <sub>2</sub> UniAmp Sensor - Normal range - 2.1 x 80 mm needle	Protocol - In-situ hydrogen and oxygen measurment in H <sub>2</sub> /O <sub>2</sub> reactor

## Procedure/observations

Date	Time	Step	Observations	Pictures/Files
10.11.2025		The experiment was done according to <a href="#">Protocol - In-situ hydrogen and oxygen measurment in H<sub>2</sub>/O<sub>2</sub> reactor</a> Important steps and deviations are listed below	/	/
	11:29-12:50	Conditioning of H <sub>2</sub> sensor	<b>NB-336-Logger1</b> 1.7 mV at the end of polarization procedure	<a href="#">NB-336.ulong</a> <a href="#">NB-336-Logger1-pre-polarization.csv</a> <a href="#">NB-336-Logger1-pre-polarization.bmp</a>
	ca. 13:00-20	Assembling the setup for calibration (25 mL of water was added using graduated cylinder), done according to <a href="#">Protocol - Liquid phase calibration of H<sub>2</sub> UniAmp sensor</a> with H <sub>2</sub> bubbling.	Now PT100 is recognized with Firesting without problems, no errors	/

	13:46	Start of O2 logging.	NB-336-Ch2-1	2025-11-10_134636_NB-336-Ch2-1.txt 2025-11-10_134636_NB-336-Ch2-1.png
	13:46	Start of H2 logging.	NB-336-Logger2 offset -2 mV	NB-336.ulong NB-336-Logger2-calibration.csv NB-336-2-point calibration.bmp NB-336-Logger2-calibration step.bmp
	13:49	Degassing was started.	/	20251110_135012-degassing of water.jpg
	14:23	Moving cannula to gas phase, above liquid.	/	/
	14:25	Introducing H2 sensor into the reactor under Ar flow.	/	/
	14:26	0 ppm was taken.	/	/
	14:36	H2 bubbling of the reactor was started	First needle of the H2 balloon - no bubbling, Replacing the needle with the new one - start bubbling	/
	14:44	Introducing second H2 balloon.	/	20251110_144451-two H2 balloons.jpg
	14:52	1.000.000 ppm point was taken and calibration was saved	826 mV, slope: 1.043, 790.065 uM	20251110_145047-H2 table.jpg
	14:53	Stop of H2 logging.	/	/
	15:00	Stop of O2 logging.	/	/
	ca. 15:20	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		<b>Sample preparation</b>		
	16:35	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	16:42	Addition of 25 mL H2O to the vial via graduated cylinder.	/	/

	16:42-45	The suspension was vortexed for 3 min ( <a href="#">Equipment - VWR® VV3, Vortex Mixer</a> , stage 4/6), covered with Al foil before further use.	breaking residual lumps manually with Smartspatula in the vial.	<a href="#">20251110_164831-suspension after vortex.jpg</a>
		Continue in <a href="#">Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor</a> from step 6		
	ca. 16:50	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	16:55	Assembling the setup.	Currently, stopper instead of H <sub>2</sub> sensor, PT100, PT1000 and O <sub>2</sub> robust probe are inside the reactor immersed in the liquid phase	<a href="#">20251110_165652-before degassing and irradiation.jpg</a>
	16:58	<a href="#">Start of O2 logging.</a>	<b>NB-336-Ch2-2</b>	<a href="#">2025-11-10_165836_NB-336-Ch2-2.txt</a> <a href="#">2025-11-10_165836_NB-336-Ch2-2.png</a>
	17:00	The degassing was started	/	<a href="#">20251110_170124-degassing of the suspension.jpg</a>
	17:28	Cannula was transferred to gas phase, above the suspension.	/	/
	17:30	H <sub>2</sub> sensor was added in Ar counterflow.	/	/
	17:34	The degassing was stopped by removing the cannula and closing the valve.	/	/
	17:34	<a href="#">Stop of O2 logging.</a>	/	/
	17:35	<a href="#">Start of O2 logging.</a>	<b>NB-336-Ch2-3</b>	<a href="#">2025-11-10_173512_NB-336-Ch2-3.txt</a> <a href="#">2025-11-10_173512_NB-336-Ch2-3.png</a>
	17:35	<a href="#">Start of H2 logging.</a>	<b>NB-336-Logger3</b>	<a href="#">NB-336.ulong</a> <a href="#">NB-336-Logger3-during irradiation.csv</a> <a href="#">NB-336-Logger3-during irradiation.bmp</a>
	17:35-45	Equilibration time.	/	/

	17:45	The irradiation was started	/	<a href="#">20251110_174514.after start of irradiation.jpg</a>
	18:00	The irradiation was stopped.	/	/
	18:00-10	Equilibration time.	/	/
	18:10	<a href="#">Stop of O2 and H2 logging.</a>	/	/
	ca. 18:20	Deassembling the setup, cleaning the reactor.	<p>Tips of the sensors and reactor were covered with attached photocatalyst particles.</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>	/

## Analysis

Used calibration for Firing: [20250611-BOLA-fitting-liquid-phase-trace-oxygen-sensor-H2-O2 reactor.ini](#)

Used calibration for UniSense: NB-336-Logger2

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
10.11.2025	11:29	NB-336-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	<a href="#">NB-336.ulong</a> <a href="#">NB-336-Logger1-pre-polarization.csv</a>	/	<a href="#">NB-336-Logger1-pre-polarization.bmp</a>	/	Conditioning of sensor.
	13:46	NB-336-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	<a href="#">NB-336.ulong</a> <a href="#">NB-336-Logger2-calibration.csv</a>	/	<a href="#">NB-336-2-point calibration.bmp</a> <a href="#">NB-336-Logger2-calibration step.bmp</a>	/	2-point calibration, slope 1.043

	17:35	NB-336-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-336.ulog NB-336-Logger3-during irradiation.csv	NB-336-O2 and H2 curves.py	NB-336-Logger3-during irradiation.bmp NB-336-O2 and H2 curves.png	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	Sharp increase and decrease at the beginning and end of irradiation.
	13:46	NB-336-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-10_134636_NB-336-Ch2-1.txt	/	2025-11-10_134636_NB-336-Ch2-1.png	/	Degassing followed by calibration of H2 sensor.
	16:58	NB-336-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-10_165836_NB-336-Ch2-2.txt	/	2025-11-10_165836_NB-336-Ch2-2.png	/	Degassing of the suspension.
	17:35	NB-336-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-11-10_173512_NB-336-Ch2-3.txt	NB-336-O2 and H2 curves.py	2025-11-10_173512_NB-336-Ch2-3.png NB-336-O2 and H2 curves.png	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	Change in O2 value during irradiation, very slow O2 decrease rate after switching irradiation off.

## Results

Reproduction of NB-316 was performed. Simultaneous H<sub>2</sub> and O<sub>2</sub> measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O<sub>2</sub>/H<sub>2</sub> photoreactor under 365 nm irradiation (50 mW/cm<sup>2</sup>, 20 °C, 15 min, reference conditions) were performed.

Sharp increase/decrease in the H2 signal after start and end of irradiation, Slow decrease rate of O2 in post-irradiation equilibration.

## Future recommendations

Old procedure	Problem	Suggested new procedure
/	Sharp increase in H2 signal.	Adjust the position of H2 sensor in the reactor.

## Linked experiments

SrTiO3 - NB-313: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 0.5 mg/mL), 365 nm, 50 mW

SrTiO3 - NB-315: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-301, 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

## Linked resources

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](#)

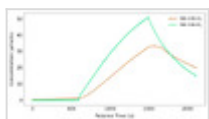
Protocol - [Liquid phase calibration of H2 UniAmp sensor](#)

Protocol - [In-situ hydrogen and oxygen measurment in H2/O2 reactor](#)

## Attached files

NB-336-O2 and H2 curves.png

sha256: 4f092b7d39ba7d4c54155efebda7474993d2e2dbb3d3e1bc9fab82bbad6e52d5



NB-336-O2 and H2 curves.py

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20251110\_135012-degassing of water.jpg

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20251110\_144451-two H2 balloons.jpg

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20251110\_145047-H2 table.jpg

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20251110\_164831-suspension after vortex.jpg

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20251110\_165652-before degassing and irradiation.jpg

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20251110\_170124-degassing of the suspension.jpg

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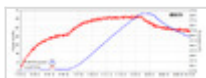


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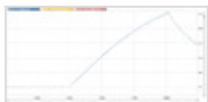


NB-336-Logger3-during irradiation.csv

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NB-336-Logger3-during irradiation.bmp

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NB-336-Logger2-calibration.csv

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NB-336-2-point calibration.bmp

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NB-336-Logger2-calibration step.bmp

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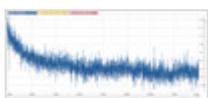


NB-336-Logger1-pre-polarization.csv

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NB-336-Logger1-pre-polarization.bmp

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NB-336.ulog

sha256: 6d18a988aa09d7b2918e8e8a32c348990100b991df574fe417b029c09467f115



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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3403>