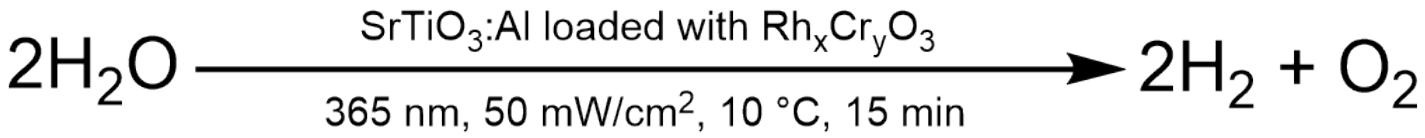
# NB-360: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C (reproduction NB-351) I

**Date:** 2025-11-24
**Tags:** O2 | Test | Calibration | NB | Firesting | O2 sensor | H2 | SrTiO3 | Unisense | RhCrO3:Al:SrTiO3 | H2 Sensor | temperature | In situ | H2/O2 reactor | Trace range robust oxygen sensor | photocatalysis | Unisense normal range
**Category:** SrTiO3
**Status:** Done
**Created by:** Nadzeya Brezhneva

## Objectives

Second reproduction of NB-359: 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/$cm^2$, **10 °C** (changing temperature relatively to reference conditions).

## Reaction scheme




ChemDraw file linked: NB-351-SrTiO3-photocatalytic H2O splitting.cdxml

## Literature/reference experiments

| | |
|---|---|
| **Literature** | / |
| **Reproduction** | SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C<br>SrTiO3 - NB-359: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C (reproduction NB-351) |
| **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 | 2 | / |
| 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 | 12.48 | / | / | / | / |

# 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]** | 10 |
| **Stirring speed [rpm]** | 500 |
| **Irradiation start:**<br>**1. Firesting [relative to start log]**<br>**2. Unisense** | 1. 604 s<br>2. 20:08:24 |
| **Irradiation stop:**<br>**1. Firesting [relative to start log]**<br>**2. Unisense** | 1. 1535 s<br>2. 20:23:56 |

# $O_2/H_2$ 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 |
|---|---|---|---|---|
| 24.11.2025 | | Calibration from experiment SrTiO3 - NB-359: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C (reproduction NB-351) was used. | | |
| | | **Sample preparation** | | |
| | 18:45 | Weighing EA-358 photocatalyst in a 50 mL vial. | Creamy solid. | / |
| | 18:49 | Addition of 25 mL H2O to the vial via graduated cylinder. | / | / |
| | 18:50-53 | The suspension was vortexed for 3 min ( Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use. | / | 20251124_185332-suspension after vortex.jpg |
| | | Continue in Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor from step 6 | | |
| | 18:55 | The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) . | / | / |
| | 19:00 | 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 | / |
| | 19:00-11 | Keeping suspension at 10 °C, T control by PT1000. | / | / |
| | 19:12 | Start of O2 logging. | **NB-360-Ch2-1** | 2025-11-24_191210_NB-360-Ch2-1.txt 2025-11-24_191210_NB-360-Ch2-1.png |

| | | | | |
|---|---|---|---|---|
| | 19:15 | The degassing was started | / | 20251124_191601-degassing of the suspension.jpg |
| | 19:47 | Cannula was transferred to gas phase, above the suspension. | / | / |
| | 19:49 | $H_2$ sensor was added in Ar counterflow. | / | / |
| | 19:52 | The degassing was stopped by removing the cannula and closing the valve. | offset for H2 sensor - 1mV | / |
| | 19:58 | Stop of O2 logging. | / | / |
| | 19:58 | Start of O2 logging. | **NB-360-Ch2-2** | 2025-11-24_195821_NB-360-Ch2-2.txt<br>2025-11-24_195821_NB-360-Ch2-2.png |
| | 19:58 | Start of H2 logging. | **NB-360-Logger1** | NB-360.ulog<br>NB-360-Logger1.csv<br>NB-360-Logger1.bmp |
| | 19:58-20:08 | Equilibration time. | / | / |
| | 20:08 | The irradiation was started | / | 20251124_200900-after start of irradiation.jpg |
| | 20:23 | The irradiation was stopped. | / | / |
| | 20:23-33 | Equilibration time. | / | / |
| | 20:33 | Stop of O2 and H2 logging. | / | / |
| | ca. 20.40 | Deassembling the setup, cleaning the reactor. | Tips of the sensors and reactor were covered with attached photocatalyst particles.<br>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). | 20251124_204350-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-359-Logger2

| Date | Time | Sample name | Analysis method | Analytical device | Solvent | Raw Data | Python script | Processed Data | Comparative Data | Interpretation |
|---|---|---|---|---|---|---|---|---|---|---|
| 24.11.2025 | 19:58 | NB-360-Logger1 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water | NB-360.ulog NB-360-Logger1.csv | NB-360-O2 and H2 curve.py | NB-360-Logger1.bmp | SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C | H2 evolution during irradiation, weird increase in signal during irradiation, change in baseline. |
|  | 19:12 | NB-360-Ch2-1 | Optical O2 detection | Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel | water | 2025-11-24_191210_NB-360-Ch2-1.txt | / | 2025-11-24_191210_NB-360-Ch2-1.png | / | Degassing of the suspension. |
|  | 19:58 | NB-360-Ch2-2 | Optical O2 detection | Equipment - Firesting Fiber-Opitc Oxygen Meter 2 Channel | water | 2025-11-24_195821_NB-360-Ch2-2.txt | NB-360-O2 and H2 curve.py | 2025-11-24_195821_NB-360-Ch2-2.png | SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C | $O_2$ evolution during irradiation. |

# Results

Second reproduction of NB-351, 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$^2$, 10 °C**,** 15 min) were performed. Strong increase in H2 signal during irradiation --> data not applicable for analysis.

## 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

SrTiO3 - 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)

SrTiO3 - NB-351: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C

SrTiO3 - NB-359: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm2, 10 °C (reproduction NB-351)

## 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

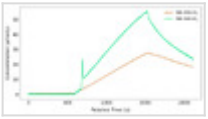
Protocol - Liquid phase calibration of H2 UniAmp sensor

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

## Attached files

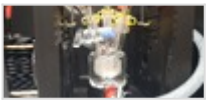
NB-360-O2 and H2 curve.py
sha256: c8f782a92a3c63c2ebe3d7974aed7d3e83e2c4ef36fa1284790fce888e96bf3f

NB-360-O2 and H2 curves.png
sha256: 4dcbc875b97d1f6531414166460aa31f8e381d91b52426261b1583666c5227ca

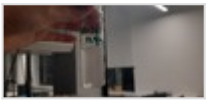


20251124_200900-after start of irradiation.jpg
sha256: 03a6ecad5ea7084097a1e8a27555eb1b915b7e703f4c26752b9ec25b4d8b3a02

20251124_204350-after irradiation.jpg
sha256: b1a827bd9db8f5135d75c6244e0e4645ef56b299f28cc99d52e2206ec7d99509



20251124_185332-suspension after vortex.jpg
sha256: 22b00d5aeb623b989e53e6184631f2deff1e73dcb66bbbfd77e22d5518a38a90

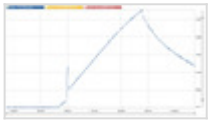


20251124_191601-degassing of the suspension.jpg
sha256: 96a98a56a6231d9a73d09a31e8cb9c0e93c1c8d3fef1a2055f583580319e9a02

NB-360-Logger1.csv
sha256: e52cab7c87910caefc720300069bee65c846920d6feee073ed1cfa3f508e9814

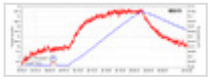
NB-360.ulog
sha256: 7403996e3d592b28a1d414eac39bb9bbbd2a5eb90ef4a249e7745e733c42051c

NB-360-Logger1.bmp
sha256: 347b2c9803e88d01500973ead0f98e6bf22b6effaf945e6e4674e991c91699f7

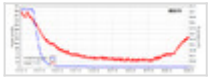


2025-11-24_195821_NB-360-Ch2-2.txt
sha256: 1a7580759adf5354ce949101c4d0775bcb77d32e145d0bac94c08b875ea21fcc

2025-11-24_195821_NB-360-Ch2-2.png
sha256: a2d881571a2be1dd984f0a2cc199050f064ed06f71eb4451dbcc5bd292ec283b



2025-11-24_191210_NB-360-Ch2-1.png
sha256: 1bedfbcebb647ec0af86dd74fb330c1d30cd95b51b17e6f381b17835f94c576f



2025-11-24_191210_NB-360-Ch2-1.txt
sha256: 84726997e332984dc33165f5a4e5408027bcbe2f094b2566e4eb128626152162

Unique eLabID: 20251124-06d37f1eb406b35265537de84353999dc98f845f
Link: https://elab.water-splitting.org/experiments.php?mode=view&id=3546