

# NB-358: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrO<sub>x</sub>,Al:SrTiO<sub>3</sub> (NB-342, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C (reproduction NB-355)

Date: 2025-11-21

Tags: O<sub>2</sub> Test Calibration Future NB  
Firesting O<sub>2</sub> sensor H<sub>2</sub> SrTiO<sub>3</sub> 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>

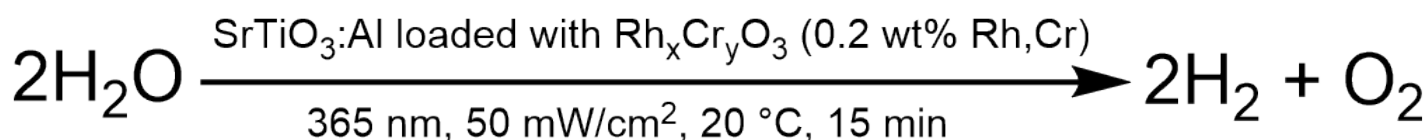
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Reproduction of NB-355: simultaneous detection of H<sub>2</sub> and O<sub>2</sub> evolution in liquid phase for irradiated suspension of Rh<sub>x</sub>Cr<sub>y</sub>O<sub>3</sub>:Al:SrTiO<sub>3</sub> suspension (NB-342, 0.2 wt% Rh,Cr, 0.5 mg/mL), 365 nm LED, 50 mW/cm<sup>2</sup>, 20 °C.

## Reaction scheme



ChemDraw file linked: [NB-328-SrTiO3-photocatalytic H2O splitting.cdxml](#)

## Literature/reference experiments

|                     |   |
|---------------------|---|
| Literature          | /   |
| Reproduction        | SrTiO <sub>3</sub> - NB-355: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> ,Al:SrTiO <sub>3</sub> (NB-342, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C   |
| Similar experiments | SrTiO <sub>3</sub> - NB-316: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> ,Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C<br>SrTiO <sub>3</sub> - NB-357: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> ,Al:SrTiO <sub>3</sub> (NB-341, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C (reproduction NB-354) |

## 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] | Concentration [mM] |
|--------------------------|--------------------------------|------------------|---------------|-------------|---------------------------|--------------------------|--------------------|----------------|-------------|--------------------|
| milli-Q H <sub>2</sub> O | /                              | /                | /             | /           | /                         | /                        | /                  | 0.998          | 25          | /                  |

|                                 |   |   |   |   |       |       |   |   |   |   |
|---------------------------------|---|---|---|---|-------|-------|---|---|---|---|
| Al:SrTiO3<br>RhCrOx<br>(NB-342) | SrTiO3 - NB-342:<br>Modification of<br>EA-354<br>(SrTiO3:Al,<br>upscaled batch)<br>with Rh, Cr oxide<br>cocatalyst (0.2<br>wt%), fresh<br>solutions of<br>RhCl3 and<br>Cr(NO3)3 | / | / | / | 12.50 | 12.51 | / | / | / | / |
|---------------------------------|---|---|---|---|-------|-------|---|---|---|---|

## 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] |
|--------------------|---|---|-----------------|--------------------|-------------------------------|
| First light source | <a href="#">Light Source - UHP LED 365 nm-4</a> | <a href="#">Power Sources - BLS-18000-1 4</a> | 365             | 56                 | 0.19                          |

|                                   |     |
|-----------------------------------|-----|
| Used beam combiner [Name or None] | /   |
| Irradiation distance [cm]         | 6.5 |
| Thermostat temperature [°C]       | 20  |
| Stirring speed [rpm]              | 500 |

|   |                         |
|---|-------------------------|
| <b>Irradiation start:</b><br><b>1. Firing [relative to start log]</b><br><b>2. Unisense</b> | 1. 605 s<br>2. 6:34:13  |
| <b>Irradiation stop:</b><br><b>1. Firing [relative to start log]</b><br><b>2. Unisense</b>  | 1. 1515 s<br>2. 6:49:22 |

## O<sub>2</sub>/H<sub>2</sub> sensor equipment

|                            | Equipment  | Used protocol  |
|----------------------------|--|--|
| Used Firing                | Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel (Firing 2)             | Protocol - Operation of Firing Fiber-Optic Oxygen Meter 2 Channel Software                   |
| Used O <sub>2</sub> sensor | Equipment - Robust probe for liquid O <sub>2</sub> measurement               | Protocol - In-situ hydrogen and oxygen measurement 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 measurement in H <sub>2</sub> /O <sub>2</sub> reactor |

## Procedure/observations

| Date       | Time    | Step  | Observations        | Pictures/Files                              |
|------------|---------|---|---------------------|---|
|            |         | Calibration from SrTiO <sub>3</sub> - NB-356: Liquid phase H <sub>2</sub> and O <sub>2</sub> of RhCrO <sub>x</sub> /Al:SrTiO <sub>3</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm <sup>2</sup> , 20 °C IX (reproduction NB-316) was used. | /                   | /   |
| 21.11.2025 |         | <b>Sample preparation</b>   |                     |   |
|            | 5:00    | Weighing photocatalyst in a 50 mL vial (the vial was covered with Al foil before further use) (preliminary, the lumps of the solid were broken with Smartspatula inside the vial).  | Slightly grey solid | /   |
|            | 5:04    | Addition of 25 mL H <sub>2</sub> O to the vial via graduated cylinder.  | /                   | /   |
|            | 5:07-10 | The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer, stage 4/6), covered with Al foil before further use.   | /                   | 20251121_051831-suspension after vortex.jpg |
|            |         | Continue in Protocol - In-situ hydrogen and oxygen measurement in H <sub>2</sub> /O <sub>2</sub> reactor from step 6  |                     |   |

|  |         |   |  |  |
|--|---------|---|--|--|
|  | 5:25    | The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) . | /  | /  |
|  | 5:30    | 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 | /  |
|  | 5:33    | Start of O2 logging.  | <b>NB-358-Ch2-1</b>  | <a href="#">2025-11-21_053309_NB-358-Ch2-1.txt</a><br><a href="#">2025-11-21_053309_NB-358-Ch2-1.png</a> |
|  | 5:38    | The degassing was started   | /  | <a href="#">20251121_053914-degassing of the suspension.jpg</a>  |
|  | 6:13    | Cannula was transferred to gas phase, above the suspension.   | /  | /  |
|  | 6:15    | H <sub>2</sub> sensor was added in Ar counterflow.  | /  | /  |
|  | 6:18    | The degassing was stopped by removing the cannula and closing the valve.  | /  | /  |
|  | 6:23    | Stop of O2 logging.   | /  | /  |
|  | 6:24    | Start of O2 logging.  | <b>NB-358-Ch2-2</b>  | <a href="#">2025-11-21_062408_NB-358-Ch2-2.txt</a><br><a href="#">2025-11-21_062408_NB-358-Ch2-2.png</a> |
|  | 6:24    | Start of H2 logging.  | <b>NB-358-Logger1</b>  | <a href="#">NB-358.ulong</a><br><a href="#">NB-358-Logger1.csv</a><br><a href="#">NB-358-Logger1.bmp</a> |
|  | 6:24-34 | Equilibration time.   | /  | /  |
|  | 6:34    | The irradiation was started   | /  | <a href="#">20251121_063439-after start of irradiation.jpg</a>   |
|  | 6:49    | The irradiation was stopped.  | /  | /  |
|  | 6:49-59 | Equilibration time.   | /  | /  |
|  | 6:59    | Stop of O2 and H2 logging.  | /  | /  |

|  |          |   |  |   |
|--|----------|---|--|---|
|  | ca. 7:10 | 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). | <a href="#">20251121_070103-after irradiation.jpg</a> |
|--|----------|---|--|---|

## Analysis

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

Used calibration for UniSense: NB-356-Logger2

| Date       | Time | Sample name    | Analysis method              | Analytical device  | Solvent | Raw Data   | Python script                             | Processed Data  | Comparative Data  | Interpretation                   |
|------------|------|----------------|------------------------------|--|---------|--|---|---|---|----------------------------------|
| 21.11.2025 | 6:24 | NB-358-Logger1 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water   | <a href="#">NB-358.ulong</a><br><a href="#">NB-358-Logger1.csv</a> | <a href="#">NB-358-O2 and H2 curve.py</a> | <a href="#">NB-358-Logger1.bmp</a><br><a href="#">NB-358-O2 and H2 curves.png</a>                 | SrTiO3 - NB-355: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-342, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C | H2 evolution during irradiation. |
|            | 5:33 | NB-358-Ch2-1   | Optical O2 detection         | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel         | water   | <a href="#">2025-11-21_053309_NB-358-Ch2-1.txt</a> /               | /   | <a href="#">2025-11-21_053309_NB-358-Ch2-1.png</a> /  | /   | Degassing of the suspension.     |
|            | 6:24 | NB-358-Ch2-2   | Optical O2 detection         | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel         | water   | <a href="#">2025-11-21_062408_NB-358-Ch2-2.txt</a>                 | <a href="#">NB-358-O2 and H2 curve.py</a> | <a href="#">2025-11-21_062408_NB-358-Ch2-2.png</a><br><a href="#">NB-358-O2 and H2 curves.png</a> | SrTiO3 - NB-355: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (NB-342, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm2, 20 °C | O2 evolution during irradiation. |

## Results

Reproduction of NB-355: simultaneous H<sub>2</sub> and O<sub>2</sub> measurements of irradiated suspension of NB-342 (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) were performed.

## Linked experiments

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

SrTiO<sub>3</sub> - NB-318: 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, 100 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-322: 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, 100 mW/cm<sup>2</sup>, 20 °C (reproduction NB-318)

SrTiO<sub>3</sub> - NB-323: Modification of EA-354 (SrTiO<sub>3</sub>:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%)

SrTiO<sub>3</sub> - NB-325: 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, 20 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-326: 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, 20 mW/cm<sup>2</sup>, 20 °C (reproduction NB-325)

SrTiO<sub>3</sub> - NB-331: 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 (reproduction NB-316) I I I

SrTiO<sub>3</sub> - NB-333: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (NB-323, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C (reproduction NB-328)

SrTiO<sub>3</sub> - NB-342: Modification of EA-354 (SrTiO<sub>3</sub>:Al, upscaled batch) with Rh, Cr oxide cocatalyst (0.2 wt%), fresh solutions of RhCl<sub>3</sub> and Cr(NO<sub>3</sub>)<sub>3</sub>

SrTiO<sub>3</sub> - NB-355: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (NB-342, 0.2 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-356: 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 IX (reproduction NB-316)

SrTiO<sub>3</sub> - NB-357: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (NB-341, 0.05 wt% Rh, Cr, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C (reproduction NB-354)

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

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

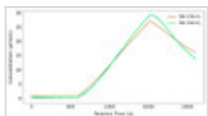
## Attached files

NB-358-O2 and H2 curve.py

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NB-358-O2 and H2 curves.png

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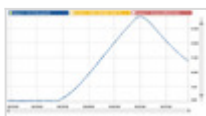
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NB-358-Logger1.bmp

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NB-358.ulog

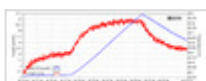
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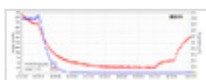
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