

# NB-365: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrO<sub>x</sub> (EA-358, 0.5 mg/mL), D<sub>2</sub>O, 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 70 min, degassing

Date: 2025-12-03

Tags: O<sub>2</sub> Test Calibration Future NB  
Firesting Irradiation O<sub>2</sub> sensor H<sub>2</sub>  
advanced irrad setup troubleshooting  
Unisense H<sub>2</sub> Sensor temperature In situ  
Trace range robust oxygen sensor  
photocatalysis

Category: SrTiO<sub>3</sub>

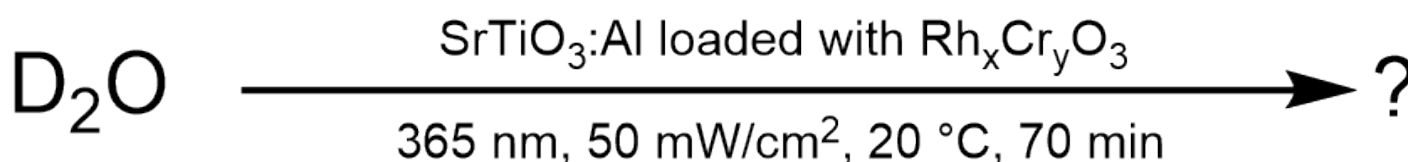
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

Simultaneous detection of O<sub>2</sub> and H<sub>2</sub> evolution in gas phase for irradiated suspension of Rh<sub>x</sub>Cr<sub>y</sub>O<sub>3</sub>:Al:SrTiO<sub>3</sub> suspension (EA-358, 0.5 mg/mL, D<sub>2</sub>O), 365 nm LED, 50 mW/cm<sup>2</sup>, 20 °C during 70 min.

## Reaction scheme



ChemDraw file linked: [NB-365-SrTiO3-D2O.cdxml](#)

## Literature/reference experiments

|                     |  |
|---------------------|--|
| Literature          | /  |
| Reproduction        | /  |
| Similar experiments | <a href="#">SrTiO<sub>3</sub> - NB-363: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrO<sub>x</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 70 min, degassing (reproduction NB-362)</a><br><a href="#">SrTiO<sub>3</sub> - NB-362: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrO<sub>x</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 70 min, degassing</a><br><a href="#">SrTiO<sub>3</sub> - NB-361: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrO<sub>x</sub> (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 15 min, degassing</a> |

## 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] |
|--------------------------|--------------------------------|------------------|---------------|-------------|---------------------------|--------------------------|--------------------|----------------|----------------------|----------------|
| milli-Q H <sub>2</sub> O | /                              | /                | /             | /           | /                         | /                        | /                  | 0.998          | 25 (for calibration) | /              |

|   |  |         |   |   |       |       |   |      |                                       |       |
|---|--|---------|---|---|-------|-------|---|------|---------------------------------------|-------|
| D <sub>2</sub> O,<br>Eurisotop,<br>99.90%               | 7789-20-0  | C121237 | / | / | /     | /     | / | 1.11 | 25 (for<br>suspension<br>preparation) | /     |
| Al:SrTiO <sub>3</sub><br>RhCrO <sub>x</sub><br>(EA-358) | SrTiO <sub>3</sub> -<br>EA-358:<br>Modification of<br>Al:SrTiO <sub>3</sub><br>(EA-354) via<br>deposition of<br>Rh, Cr oxide<br>co-catalyst,<br>350°C, 1h,<br>Upscaling<br>(3.33x) | /       | / | / | 12.50 | 12.52 | / | /    | /                                     | /     |
| Hydrogen  | 1333-74-0  | /       | / | / | /     | /     | / | /    | 1 balloon<br>(ca. 2 L)                | ca. 1 |

## 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 |

|  |                          |
|--|--------------------------|
| <b>Thermostat temperature [°C]</b>   | 20                       |
| <b>Stirring speed [rpm]</b>  | 500                      |
| <b>Irradiation start:</b><br><b>1. Firesting [relative to start log]</b><br><b>2. Unisense</b> | 1. 605 s<br>2. 18:55:17  |
| <b>Irradiation stop:</b><br><b>1. Firesting [relative to start log]</b><br><b>2. Unisense</b>  | 1. 4825 s<br>2. 20:05:35 |

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

|                            | <b>Equipment</b>   | <b>Used protocol</b>  |
|----------------------------|--|---|
| 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

| <b>Date</b> | <b>Time</b> | <b>Step</b>  | <b>Observations</b>                    | <b>Pictures/Files</b>   |
|-------------|-------------|--|--|---|
| 03.12.2025  |             | 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><br>Important steps and deviations are listed below | /                                      | /   |
|             | 14:28-15:27 | Conditioning of H <sub>2</sub> sensor  | <b>NB-365-Logger1</b><br>offset - 2 mV | <a href="#">NB-365.uog</a><br><a href="#">NB-365-Logger1.csv</a><br><a href="#">NB-365-Logger1-pre-polarization.bmp</a> |

|  |                 |  |                                  |  |
|--|-----------------|--|----------------------------------|--|
|  | ca.<br>15:30-40 | Assembling the setup for calibration (25 mL of water was added using graduated cylinder), (LAUDA set to 20 °C) done according to <a href="#">Protocol - Liquid phase calibration of H2 UniAmp sensor</a> with H2 bubbling. | /                                | /  |
|  | 15:59           | Start of O2 logging.   | <b>NB-365-Ch2-1</b>              | <a href="#">2025-12-03_155900_NB-365-Ch2-1.txt</a><br><a href="#">2025-12-03_155900_NB-365-Ch2-1.png</a>   |
|  | 15:59           | Start of H2 logging.   | <b>NB-365-Logger2</b>            | <a href="#">NB-365.ulong</a><br><a href="#">NB-365-Logger2.csv</a><br><a href="#">NB-365-Logger2-calibration step.bmp</a><br><a href="#">NB-365-Logger2-2point calibration.bmp</a> |
|  | 16:02           | Degassing was started.   | /                                | <a href="#">20251203_160306-degassing of water.jpg</a>   |
|  | 16:48           | Stop degassing.  |                                  |  |
|  | 16:49           | 0 ppm was taken.   | /                                | /  |
|  | 16:56           | H2 bubbling of the reactor was started   | /                                | <a href="#">20251203_165730-H2 bubbling.jpg</a>  |
|  | 17:04           | 1.000.000 ppm point was taken and calibration was used.  | 905 mV, slope<br>0.009, 96956 Pa | <a href="#">20251203_170405-H2 table.jpg</a>   |
|  | 17:08           | Stop of H2 logging.  | /                                | /  |
|  | 17:08           | Stop of O2 logging.  | /                                | /  |
|  | ca. 17:15       | Deassembling the setup, drying the reactor with acetone and compressed air .   | /                                | /  |
|  |                 | <b>Sample preparation</b>  |                                  |  |
|  | 17:45           | Weighing <b>EA-358</b> photocatalyst in a 50 mL vial.  | Creamy solid.                    | /  |
|  | 17:46           | Addition of 25 mL D <sub>2</sub> O to the vial via graduated cylinder.   | /                                | <a href="#">20251203_174628-D2O reagent bottle.jpg</a>   |
|  | 17:47-50        | 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.  | /                                | <a href="#">20251203_175117-suspension after vortex.jpg</a>  |
|  |                 | Continue in <a href="#">Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor</a> from step 6   |                                  |  |
|  | 18:00           | The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .  | /                                | /  |

|  |           |  |  |  |
|--|-----------|--|--|--|
|  | ca. 18:10 | Assembling the setup.  | /  | /  |
|  | 18:16     | Start of O2 logging.   | <b>NB-365-Ch2-2</b>  | <a href="#">2025-12-03_181612_NB-365-Ch2-2.txt</a><br><a href="#">2025-12-03_181612_NB-365-Ch2-2.png</a>                                     |
|  | 18:17     | The degassing was started  | /  | /  |
|  | 18:41     | The degassing was stopped by removing the cannula and closing the valve. | /  | /  |
|  | 18:44     | Stop of O2 logging.  | /  | /  |
|  | 18:44     | Start of O2 logging.   | <b>NB-365-Ch2-3</b>  | <a href="#">2025-12-03_184510_NB-365-Ch2-3.txt</a><br><a href="#">2025-12-03_184510_NB-365-Ch2-3.png</a>                                     |
|  | 18:45     | Start of H2 logging.   | <b>NB-365-Logger3</b>  | <a href="#">NB-365.uog</a><br><a href="#">NB-365-Logger3-during irradiation.csv</a><br><a href="#">NB-365-Logger3-during irradiation.bmp</a> |
|  | 18:45-55  | Equilibration time.  | /  | <a href="#">20251203_185234-before irradiation.jpg</a>   |
|  | 18:55     | The irradiation was started  | Problems with stirring<br>19:38 - restart stirring<br>19:51 - restart stirring<br>20:00 - restart stirring                 | <a href="#">20251203_185730-after start of irradiation.jpg</a>   |
|  | 20:05     | The irradiation was stopped.   | /  | /  |
|  | 20:05-15  | Equilibration time.  | /  | /  |
|  | 20:15     | Stop of O2 and H2 logging.   | /  | <a href="#">20251203_201647-after irradiation.jpg</a>  |
|  | ca. 20:30 | Deassembling the setup, cleaning the reactor.                            | Seems that particles are less adhesive to the walls of the reactor than in the case of H <sub>2</sub> O-based suspensions. | /  |

# Analysis

Used calibration for Firing: 20250910-BOLA fitting-gas phase-4-neck photoreactor-trace oxygen robust probe-Ch2.ini

Used calibration for UniSense: NB-365-Logger2

| Date       | Time  | Sample name    | Analysis method              | Analytical device  | Solvent | Raw Data   | Python script             | Processed Data   | Comparative Data   | Interpretation  |
|------------|-------|----------------|------------------------------|--|---------|--|---------------------------|--|--|---|
| 03.12.2025 | 14:28 | NB-365-Logger1 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water   | NB-365.ulog<br>NB-365-Logger1.csv                    | /                         | NB-365-Logger1-pre-polarization.bmp  | /  | Pre-polarization of the sensor.                                     |
|            | 15:59 | NB-365-Logger2 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water   | NB-365.ulog<br>NB-365-Logger2.csv                    | /                         | NB-365-Logger2-calibration step.bmp<br>NB-365-Logger2-2point calibration.bmp | /  | Calibration of H2 sensor, 10 <sup>6</sup> ppm corresponds to 905 mV |
|            | 18:45 | NB-365-Logger3 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water   | NB-365.ulog<br>NB-365-Logger3-during irradiation.csv | NB-365-O2 and H2 curve.py | NB-365-Logger3-during irradiation.bmp<br>NB-365-O2 and H2 curves.png         | SrTiO3 - NB-346: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), D2O, 365 nm, 50 mW/cm2, 20 °C | H2 evolution during irradiation.                                    |
|            | 15:59 | NB-365-Ch2-1   | Optical O2 detection         | Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel            | water   | 2025-12-03_155900_NB-365-Ch2-1.txt                   | /                         | 2025-12-03_155900_NB-365-Ch2-1.png   | /  | Degassing of water followed by calibration of H2 sensor.            |
|            | 18:16 | NB-365-Ch2-2   | Optical O2 detection         | Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel            | water   | 2025-12-03_181612_NB-365-Ch2-2.txt                   | /                         | 2025-12-03_181612_NB-365-Ch2-2.png   | /  | Degassing of the suspension.  |
|            | 18:44 | NB-365-Ch2-3   | Optical O2 detection         | Equipment - Firing Fiber-Optic Oxygen Meter 2 Channel            | water   | 2025-12-03_184510_NB-365-Ch2-3.txt                   | NB-365-O2 and H2 curve.py | 2025-12-03_184510_NB-365-Ch2-3.png<br>NB-365-O2 and H2 curves.png            | SrTiO3 - NB-346: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), D2O, 365 nm, 50 mW/cm2, 20 °C | O2 evolution during irradiation.                                    |

## Results

Simultaneous H<sub>2</sub> and O<sub>2</sub> measurements (gas phase) of irradiated suspension of EA-358 (0.5 mg/mL, D<sub>2</sub>O) in O<sub>2</sub>/H<sub>2</sub> photoreactor under 365 nm irradiation (50 mW/cm<sup>2</sup>, 20 °C, 70 min) were performed.

## Future recommendations

| Old procedure | Problem   | Suggested new procedure                   |
|---------------|---|---|
| /             | Not constant stirring of the suspension during irradiation. | Replace PTFE stirring bar with a new one. |

## Linked experiments

SrTiO<sub>3</sub> - NB-312: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements with Unisense H<sub>2</sub> sensor, Firesting O<sub>2</sub> robust probe in irradiated Al:SrTiO<sub>3</sub> RhCrOx (NB-289, 0.5 mg/mL), 365 nm, 50 mW, 1 h, degassing

SrTiO<sub>3</sub> - NB-346: Liquid phase H<sub>2</sub> and O<sub>2</sub> of RhCrOx,Al:SrTiO<sub>3</sub> (EA-358, 0.5 mg/mL), D<sub>2</sub>O, 365 nm, 50 mW/cm<sup>2</sup>, 20 °C

SrTiO<sub>3</sub> - NB-361: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 15 min, degassing

SrTiO<sub>3</sub> - NB-362: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 70 min, degassing

SrTiO<sub>3</sub> - NB-363: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 70 min, degassing (reproduction NB-362)

SrTiO<sub>3</sub> - NB-364: Gas phase H<sub>2</sub> and O<sub>2</sub> measurements of Al:SrTiO<sub>3</sub> RhCrOx (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm<sup>2</sup>, 20 °C, 70 min, degassing (reproduction NB-363)

## Linked resources

Equipment - [Firesting Fiber-Optic Oxygen Meter 2 Channel \(Firesting 2\)](#)

Equipment - [Robust probe for liquid O<sub>2</sub> measurment](#)

Equipment - [Advanced irradiation chamber V1.0 I](#)

Equipment - [H<sub>2</sub> UniAmp Sensor - Normal range - 2.1 x 80 mm needle](#)

Equipment - [Irradiation setup 4 \(CEEC II, E002\)](#)

Protocol - [Operation of Firesting Fiber-Optic Oxygen Meter 2 Channel Software](#)

Protocol - [Getting hydrogen from hydrogen bottle in CEEC II E014](#)

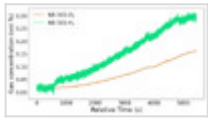
Protocol - [Gas phase calibration of H<sub>2</sub> UniAmp sensor](#)

Protocol - [In-situ hydrogen and oxygen measurment in H<sub>2</sub>/O<sub>2</sub> reactor](#)

## Attached files

NB-365-O<sub>2</sub> and H<sub>2</sub> curves.png

sha256: 0c0943f18a13eff8f11ba7066a1ce1ced9c9005ed69c789fb0657a23763e0bde



NB-365-O2 and H2 curve.py

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NB-365-SrTiO3-D2O.png

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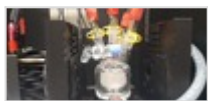


NB-365-SrTiO3-D2O.cdxml

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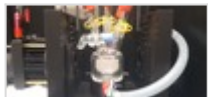
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20251203\_185234-before irradiation.jpg

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20251203\_175117-suspension after vortex.jpg

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20251203\_165730-H2 bubbling.jpg

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20251203\_160306-degassing of water.jpg

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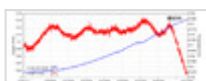


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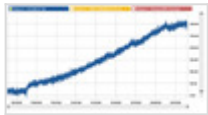


NB-365-Logger3-during irradiation.csv

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NB-365-Logger3-during irradiation.bmp

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NB-365-Logger2-calibration step.bmp

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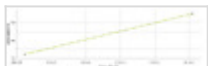


NB-365-Logger2.csv

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NB-365-Logger2-2point calibration.bmp

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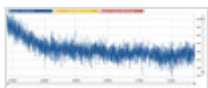


NB-365-Logger1.csv

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NB-365-Logger1-pre-polarization.bmp

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NB-365.ulog

sha256: fe5dde562eca7ae69b3864096e60b3520820cdf3a811d48ac4598386a579d453



Unique eLabID: 20251203-5892e45bc654e74c815381c17490e44914cfc45d  
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3595>