

# NB-346: 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), D<sub>2</sub>O, 365 nm, 50 mW/cm<sup>2</sup>, 20 °C

Date: 2025-11-12

Tags: O<sub>2</sub> Test Calibration NB Firing  
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>

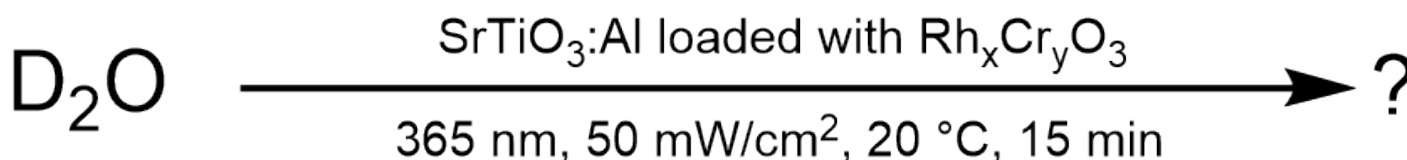
Status: Done

Created by: Nadzeya Brezhneva

## Objectives

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 (EA-358, 0.5 mg/mL, D<sub>2</sub>O), 365 nm LED, 50 mW/cm<sup>2</sup>, 20 °C (changing dispersion medium).

## Reaction scheme



ChemDraw file linked: [NB-346-SrTiO3-photocatalytic H2O splitting.cdxml](#)

## Literature/reference experiments

|                     |  |
|---------------------|--|
| Literature          | /  |
| Reproduction        | /  |
| Similar experiments | <a href="#">SrTiO3 - NB-316: Liquid phase H2 and O2 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</a> |

## Reagents

| Name                                | CAS Number / Experiment Number | Inventor y 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 (for calibration)            | /              | /                  |
| D <sub>2</sub> O, Eurisotop, 99.90% | 7789-20-0                      | C121243           | /             | /           | /                         | /                        | /                  | 1.11           | 25 (for suspension preparation) | /              | /                  |

|   |  |   |   |   |       |       |   |   |           |      |   |
|---|--|---|---|---|-------|-------|---|---|-----------|------|---|
| Al:SrTiO <sub>3</sub><br>RhCrOx<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 | 12.42 | / | / | /         | /    | / |
| Hydrogen                                    | 1333-74-0  | / | / | / | /     | /     | / | / | 1 balloon | ca.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] |
|--------------------|---|---|-----------------|--------------------|-------------------------------|
| 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  |

|  |                                    |
|--|------------------------------------|
| <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. 0h:10 min:6s<br>2. 19:49:06     |
| <b>Irradiation stop:</b><br><b>1. Firesting [relative to start log]</b><br><b>2. Unisense</b>  | 1. 0h: 25 min: 48 s<br>2. 20:04:49 |

## 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  |
|------------|--------------|--|---|---|
| 12.11.2025 |              | 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><br>Important steps and deviations are listed below                             | /   | /   |
|            | 8:07-39      | <a href="#">Conditioning of H<sub>2</sub> sensor</a>   | <b>NB-346-Logger1</b><br>1.7 mV at the end of polarization procedure, stable signal | <a href="#">NB-346.ulog</a><br><a href="#">NB-346-Logger1-pre-polarization.csv</a><br><a href="#">NB-346-Logger1-pre-polarization.bmp</a> |
|            | ca. 15:10-20 | 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. | /   | /   |
|            |              | Important note: water was used as a medium for preliminary calibration of H <sub>2</sub> sensor before performing experiment in D <sub>2</sub> O.  |   |   |

|  |           |   |                              |   |
|--|-----------|---|------------------------------|---|
|  | 15:38     | Start of O2 logging.  | NB-346-Ch2-1                 | 2025-11-12_153850_NB-346-Ch2-1.txt<br>2025-11-12_153850_NB-346-Ch2-1.png  |
|  | 15:38     | Start of H2 logging.  | NB-346-Logger2               | NB-346.ulong<br>NB-346-Logger2-calibration.csv<br>NB-346-Logger2-calibration step.bmp<br>NB-346-Logger-2point calibration.bmp |
|  | 15:41     | Degassing was started.  | /                            | 20251112_154212-degassing of water.jpg  |
|  | 16:07     | Cannula was moved to gas phase above the liquid level.  | /                            | /   |
|  | 16:09     | Introducing H2 sensor into the reactor under Ar flow.   | /                            | /   |
|  | 16:17     | 0 ppm was taken.  | /                            | /   |
|  | 16:21     | Removing cannula, closing the valve.  | /                            | /   |
|  | 16:22     | H2 bubbling of the reactor was started  | /                            | 20251112_162312-H2 bubbling.jpg   |
|  | 16:41     | 1.000.000 ppm point was taken   | 830 mV                       | /   |
|  | 16:43     | New 1.000.000 ppm point was taken and calibration was saved   | 832 mV, slope: 1.054, 790 uM | 20251112_164009-H2 table.jpg  |
|  | 16:46     | Stop of H2 logging.   | /                            | /   |
|  | 16:47     | Stop of O2 logging.   | /                            | /   |
|  | ca. 17:00 | Deassembling the setup, drying the reactor with acetone and compressed air .  | /                            | /   |
|  |           | <b>Sample preparation</b>   |                              |   |
|  | ca. 18:30 | Weighing <b>EA-358</b> photocatalyst in a 50 mL vial.   | Creamy solid.                | /   |
|  | 18:35     | Addition of 25 mL D2O to the vial via graduated cylinder.   | /                            | 20251112_183257-D2O bottle.jpg  |
|  | 18:37-40  | 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. | /                            | 20251112_184305-suspension after vortex.jpg   |
|  |           | Continue in <a href="#">Protocol - In-situ hydrogen and oxygen measurment in H2/O2 reactor</a> from step 6  |                              |   |

|  |          |   |  |  |
|--|----------|---|--|--|
|  | 18:45    | The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) . | /  | /  |
|  | 18:50    | 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   | /  |
|  | 18:54    | Start of O2 logging.  | <b>NB-346-Ch2-2</b>  | 2025-11-12_185427_NB-346-Ch2-2.txt<br>2025-11-12_185427_NB-346-Ch2-2.png                       |
|  | 18:58    | The degassing was started   | Checking rotation of stirring bar, stop-start of stirring when the stirring bar was not rotated properly --> Change in O2 curve slope during degassing (afterwards, no problems with stirring were observed, everything was OK). | 20251112_185959-degassing of the suspension.jpg  |
|  | 19:30    | Cannula was transferred to gas phase, above the suspension.   | /  | /  |
|  | 19:34    | H <sub>2</sub> sensor was added in Ar counterflow.  | /  | 20251112_193815-after immersing H2 sensor.jpg  |
|  | 19:37    | The degassing was stopped by removing the cannula and closing the valve.  | /  | /  |
|  | 19:38    | Stop of O2 logging.   | /  | /  |
|  | 19:39    | Start of O2 logging.  | <b>NB-346-Ch2-3</b>  | 2025-11-12_193900_NB-346-Ch2-3.txt<br>2025-11-12_193900_NB-346-Ch2-3.png                       |
|  | 19:39    | Start of H2 logging.  | <b>NB-346-Logger3</b>  | NB-346.ulong<br>NB-346-Logger3-during irradiation.csv<br>NB-346-Logger3-during irradiation.bmp |
|  | 19:39-49 | Equilibration time.   | /  | /  |
|  | 19:49    | The irradiation was started   | /  | 20251112_195028-after start of irradiation.jpg   |

|  |              |   |  |   |
|--|--------------|---|--|---|
|  | 20:04        | The irradiation was stopped.                  | /  | /   |
|  | 20:04-14     | Equilibration time.                           | /  | /   |
|  | 20:14        | Stop of O2 and H2 logging.                    | /  | /   |
|  | ca.<br>20:25 | Deassembling the setup, cleaning the reactor. | <p>Tips of the sensors were covered with attached photocatalyst particles. Seems like the particles were less adhesive to the reactor surface than in previous experiments with H<sub>2</sub>O.</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> | <a href="#">20251112_201719-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-346-Logger2

| Date       | Time  | Sample name    | Analysis method              | Analytical device  | Solvent | Raw Data  | Python script                              | Processed Data  | Comparative Data | Interpretation   |
|------------|-------|----------------|------------------------------|--|---------|---|--|---|------------------|--|
| 12.11.2025 | 8:07  | NB-346-Logger1 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water   | <a href="#">NB-346.ulong</a><br><a href="#">NB-346-Logger1-pre-polarization.csv</a>   | /  | <a href="#">NB-346-Logger1-pre-polarization.bmp</a>   | /                | Pre-polarization, stable signal, ca. 1.7 mV.                               |
|            | 15:38 | NB-346-Logger2 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water   | <a href="#">NB-346.ulong</a><br><a href="#">NB-346-Logger2-calibration.csv</a>        | /  | <a href="#">NB-346-Logger2-calibration step.bmp</a><br><a href="#">NB-346-Logger2-point calibration.bmp</a> | /                | Calibration in water, 10 <sup>6</sup> point - response 832 mV, slope 1.054 |
|            | 19:39 | NB-346-Logger3 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | D2O     | <a href="#">NB-346.ulong</a><br><a href="#">NB-346-Logger3-during irradiation.csv</a> | <a href="#">NB-346-O2 and H2 curves.py</a> | <a href="#">NB-346-Logger3-during irradiation.bmp</a><br><a href="#">NB-346-O2 and H2 curves.png</a>        | /                | H2 evolution during irradiation of D2O-based suspension.                   |

|       |              |                      |  |       |                                    |                            |   |   |  |
|-------|--------------|----------------------|--|-------|------------------------------------|----------------------------|---|---|--|
| 15:38 | NB-346-Ch2-1 | Optical O2 detection | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel | water | 2025-11-12_153850_NB-346-Ch2-1.txt | /                          | 2025-11-12_153850_NB-346-Ch2-1.png                                | / | Degassing of water followed by calibration of H2 sensor. |
| 18:54 | NB-346-Ch2-2 | Optical O2 detection | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel | D2O   | 2025-11-12_185427_NB-346-Ch2-2.txt | /                          | 2025-11-12_185427_NB-346-Ch2-2.png                                | / | Degassing of D2O-based suspension.                       |
| 19:39 | NB-346-Ch2-3 | Optical O2 detection | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel | D2O   | 2025-11-12_193900_NB-346-Ch2-3.txt | NB-346-O2 and H2 curves.py | 2025-11-12_193900_NB-346-Ch2-3.png<br>NB-346-O2 and H2 curves.png | / | O2 evolution during irradiation of D2O-based suspension. |

## Results

Simultaneous H<sub>2</sub> and O<sub>2</sub> measurements of irradiated suspension of EA-358 (0.5 mg/mL in D2O) 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 with preliminary calibration of H<sub>2</sub> sensor in water.

Both evolution of H<sub>2</sub> and O<sub>2</sub> were observed but with lower rates and amounts produced than in the case of H<sub>2</sub>O-based suspensions.

## 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-344: 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, 150 mW/cm<sup>2</sup>, 20 °C

## 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 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-346-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.cdxml

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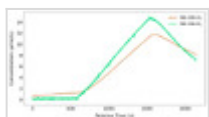
NB-346-SrTiO<sub>3</sub>-photocatalytic H<sub>2</sub>O splitting.png

sha256: 96c01d85c0fb58aa23ddc7986adb480cb5da9a3782e3486551b6a90bda3fb070



NB-346-O<sub>2</sub> and H<sub>2</sub> curves.png

sha256: aa225bff4d286fe0b04e4acf1ba7dc7ecbb3e9bab5f1566866ba6b6fea9ded7c



NB-346-O<sub>2</sub> and H<sub>2</sub> curves.py

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20251112\_154212-degassing of water.jpg

sha256: 258eb19f0adba2cbb5999cbdfec7d78a4ba19aed05c5538bdc18e4dc5ba19fe



20251112\_162312-H<sub>2</sub> bubbling.jpg

sha256: bab385aad7e490f47160128fe9ecfae64dde1fe3c5c190adef3a7dfdc9592db6



20251112\_164009-H<sub>2</sub> table.jpg

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20251112\_183257-D<sub>2</sub>O bottle.jpg

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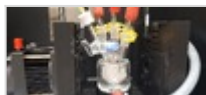
20251112\_184305-suspension after vortex.jpg

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20251112\_185959-degassing of the suspension.jpg

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20251112\_193815-after immersing H2 sensor.jpg

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20251112\_201719-after irradiation.jpg

sha256: 5c6b177b51b7607e7e272dc93e52b7b961f2b4cee270358b594bd09f6ccff18a



20251112\_195028-after start of irradiation.jpg

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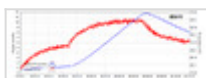


2025-11-12\_193900\_NB-346-Ch2-3.txt

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2025-11-12\_185427\_NB-346-Ch2-2.txt

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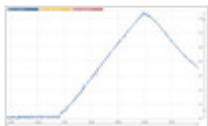


NB-346-Logger3-during irradiation.csv

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NB-346-Logger3-during irradiation.bmp

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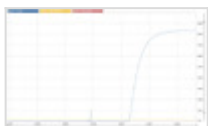


NB-346-Logger2-calibration.csv

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NB-346-Logger2-calibration step.bmp

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NB-346-Logger-2point calibration.bmp

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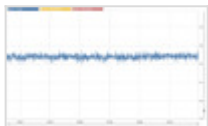


NB-346-Logger1-pre-polarization.csv

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NB-346-Logger1-pre-polarization.bmp

sha256: a5255ed6e865b0d3eb71594cdee641a125ffb9de97606442f38894f47b7397e3



NB-346.ulog

sha256: a28d2f8c4e43fea49a178437eaa890f6aa348bcb2df15afb476f375710a54081



Unique eLabID: 20251112-e1c473c58c2b5909781f35b512713bb1b5a9701d  
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3446>