

NB-339: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C VI (reproduction NB-316)

Date: 2025-11-11
Tags: O₂ Test Calibration Future NB
Firesting O₂ sensor H₂ SrTiO₃ Unisense
RhCrO_x:Al:SrTiO₃ H₂ Sensor
temperature In situ Trace range robust
oxygen sensor photocatalysis Unisense
normal range

Category: SrTiO₃

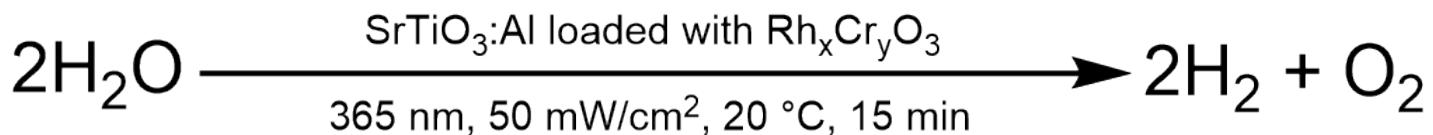
Status: Done

Created by: Nadzeya Brezhneva

Objectives

Reproduction of NB-316: simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh,CrO_x:Al:SrTiO₃ suspension (EA-358 sample, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C, 15 min (reference conditions).

Reaction scheme



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

Literature/reference experiments

| Literature | / |
|---------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Reproduction | SrTiO₃ - NB-316: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C SrTiO₃ - NB-319: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) SrTiO₃ - NB-320: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) I SrTiO₃ - NB-329: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) II SrTiO₃ - NB-331: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) III |
| Similar experiments | SrTiO₃ - NB-315: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C |

Reagents

| Name | CAS Number / Experiment Number | Inventor y number | Amoun t [mmol] | Equivale nts | Mass _{theo} [mg] | Mass _{exp} [mg] | Molar mass [g/mol] | Density [g/ml] | Volume [ml] | Pressure [bar] | Concentratio n [mM] |
|------|--------------------------------|-------------------|----------------|--------------|---------------------------|--------------------------|--------------------|----------------|-------------|----------------|---------------------|
| | | | | | | | | | | | |

| | | | | | | | | | | | |
|---------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------|---|---|---|------|-------|---|-------|--------------------------|-----------|---|
| milli-Q H ₂ O | / | / | / | / | / | / | / | 0.998 | 25 +25 (for calibration) | / | / |
| Al:SrTiO ₃ RhCrO _x (EA-358) | SrTiO ₃ - EA-358: Modification of Al:SrTiO ₃ (EA-354) via deposition of Rh, Cr oxide co-catalyst, 350°C, 1h, Upscaling (3.33x) | / | / | / | 12.5 | 12.52 | / | / | / | / | / |
| 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 measurement 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-18000-1 4 | 365 | 56 |

| | |
|-----------------------------------|---|
| Used beam combiner [Name or None] | / |
|-----------------------------------|---|

| | |
|----------------------------------------------------------------------------------|-----------------------------------|
| Irradiation distance [cm] | 6.5 |
| Thermostat temperature [°C] | 20 |
| Stirring speed [rpm] | 500 |
| Irradiation start: 1. Firesting [relative to start log] 2. Unisense | 1. 0 h:10 min:1 s 2. 18:18:58 |
| Irradiation stop: 1. Firesting [relative to start log] 2. Unisense | 1. 0 h:26 min:42 s 2. 18:35:39 |

O₂/H₂ 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 O ₂ measurement | Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor |
| Used H ₂ sensor | Equipment - H ₂ UniAmp Sensor - Normal range - 2.1 x 80 mm needle | Protocol - In-situ hydrogen and oxygen measurement in H ₂ /O ₂ reactor |

Procedure/observations

| Date | Time | Step | Observations | Pictures/Files |
|------------|-------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------|
| 11.11.2025 | | The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below | / | / |
| | 10:38-11:59 | Conditioning of H ₂ sensor | NB-339-Logger1 1.7 mV at the end of polarization procedure | NB-339.ulog NB-339-Logger1.csv NB-339-Logger1-pre-polarization.bmp |
| | 13:30-35 | Assembling the setup for calibration (25 mL of water was added using graduated cylinder), done according to Protocol - Liquid phase calibration of H₂ UniAmp sensor with H₂ bubbling . | / | / |

| | | | | |
|--|-----------|--------------------------------------------------------------------------------------------------|----------------------------------|--------------------------------------------------------------------------------------|
| | 13:43 | Start of O2 logging. | NB-339-Ch2-1 | 2025-11-11_134338_NB-339-Ch2-1.txt 2025-11-11_134338_NB-339-Ch2-1.png |
| | 13:43 | Start of H2 logging. | NB-339-Logger2 | NB-339.ulog NB-339-Logger2-calibration.csv NB-339-Logger2-calibration step.bmp |
| | 13:46 | Degassing was started. | / | / |
| | 14:48 | Moving cannula to gas phase above the liquid. | / | / |
| | 14:50 | Introducing H2 sensor into the reactor under Ar flow. | / | / |
| | 14:51 | 0 ppm was taken. | / | / |
| | 14:57 | H2 bubbling of the reactor was started | / | 20251111_145912-first H2 balloon.jpg |
| | 15:04 | Introducing second H2 balloon. | / | 20251111_150542-two H2 balloons.jpg |
| | 15:13 | 1.000.000 ppm point was taken (841 mV). | / | / |
| | 15:16 | Previous 1.000.000 ppm point was removed, new point (842 mV) was taken and calibration was saved | 842 mV, slope: 1.065, 791.627 uM | 20251111_151134-H2 table.jpg |
| | 15:17 | Stop of H2 logging. | / | / |
| | 15:17 | Stop of O2 logging. | / | / |
| | ca. 15:30 | Deassembling the setup, drying the reactor with acetone and compressed air . | / | / |
| | | Sample preparation | | |
| | 16:45 | Weighing EA-358 photocatalyst in a 50 mL vial. | / | / |
| | 16:47 | Addition of 25 mL H2O to the vial via graduated cylinder. | / | / |

| | | | | |
|--|-----------|---------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------|
| | 16:52-55 | The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer , stage 4/6), covered with Al foil before further use. | / | 20251111_165549-suspension after vortex.jpg |
| | | Continue in Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor from step 6 | | |
| | 17:00 | The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) . | / | / |
| | 17:05 | 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 | / |
| | 17:14 | Start of O₂ logging. | NB-339-Ch2-2 | 2025-11-11_171421_NB-339-Ch2-2.txt 2025-11-11_171421_NB-339-Ch2-2.png |
| | 17:16 | The degassing was started | / | 20251111_171624-degassing of the suspension.jpg |
| | 17:59 | Cannula was transferred to gas phase, above the suspension. | / | / |
| | 18:02 | H ₂ sensor was added in Ar counterflow. | / | / |
| | ca. 18:05 | The degassing was stopped by removing the cannula and closing the valve. | / | / |
| | 18:08 | Stop of O₂ logging. | / | / |
| | 18:09 | Start of O₂ logging. | NB-339-Ch2-3 | 2025-11-11_180857_NB-339-Ch2-3.txt 2025-11-11_180857_NB-339-Ch2-3.png |
| | 18:09 | Start of H₂ logging. | NB-339-Logger3 | NB-339.ulog NB-339-Logger3-during irradiation.csv NB-339-Logger3-during irradiation.bmp |
| | 18:09-19 | Equilibration time. | / | / |
| | 18:19 | The irradiation was started | / | / |
| | 18:36 | The irradiation was stopped. | / | / |

| | | | | |
|--|-----------|-----------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---|
| | 18:36-46 | Equilibration time. | / | / |
| | 18:44 | Stop of O2 and H2 logging. | / | / |
| | ca. 19:00 | Deassembling the setup, cleaning the reactor. | Tips of the sensors and reactor were covered with attached photocatalyst particles. 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). | |

Analysis

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

Used calibration for UniSense: NB-339-Logger2

| Date | Time | Sample name | Analysis method | Analytical device | Solvent | Raw Data | Python script | Processed Data | Comparative Data | Interpretation |
|------------|-------|----------------|------------------------------|------------------------------------------------------------------|---------|--------------------------------------------------------------------------------------|-------------------------------------------|-------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------|
| 11.11.2025 | 10:38 | NB-339-Logger1 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water | NB-339.ulog NB-339-Logger1.csv | / | NB-339-Logger1-pre-polarization.bmp | / | Conditioning of H2 sensor, 1.7 mV at the end of polarization. |
| | 13:43 | NB-339-Logger2 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water | NB-339.ulog NB-339-Logger2-calibration.step.csv | / | NB-339-Logger2-calibration.step.bmp | / | 2 point calibration, at 10 ⁻⁶ ppm: 842 mV, slope: 1.065, 791.627 uM |
| | 18:09 | NB-339-Logger3 | electrochemical H2 detection | Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle | water | NB-339.ulog NB-339-Logger3-during irradiation.csv | NB-339-O2 and H2 curve.py | NB-339-Logger3-during irradiation.bmp | 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 | Smooth curve, small artefact during irradiation, but questionable correlation with O2 formation rate. |
| | 13:43 | NB-339-Ch2-1 | Optical O2 detection | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel | water | 2025-11-11_134338_NB-339-Ch2-1.txt | / | 2025-11-11_134338_NB-339-Ch2-1.png | / | Degassing followed by calibration of H2 sensor. |
| | 17:14 | NB-339-Ch2-2 | Optical O2 detection | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel | water | 2025-11-11_171421_NB-339-Ch2-2.txt | / | 2025-11-11_171421_NB-339-Ch2-2.png | / | Degassing of the suspension. |

| | | | | | | | | | | |
|--|-------|--------------|----------------------------------|----------------------------------------------------------------------|-------|----------------------------------------------------|---------------------------------------------------------|----------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| | 18:09 | NB-339-Ch2-3 | Optical O ₂ detection | Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel | water | 2025-11-11_180857_NB-339-Ch2-3.txt | NB-339-O ₂ and H ₂ curve.py | 2025-11-11_180857_NB-339-Ch2-3.png | SrTiO ₃ - NB-316: Liquid phase H ₂ and O ₂ of RhCrO _x ,Al:SrTiO ₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C | O ₂ evolution during irradiation, steps during irradiation, questionable formation rate and correlation with H ₂ formation. |
|--|-------|--------------|----------------------------------|----------------------------------------------------------------------|-------|----------------------------------------------------|---------------------------------------------------------|----------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|

Results

Simultaneous H₂ and O₂ measurements of irradiated suspension of EA-358 (0.5 mg/mL) in O₂/H₂ photoreactor under 365 nm irradiation (50 mW/cm², 20 °C, 15 min, reference conditions, reproduction of NB-316) were performed.

Questionable formation rates of H₂ and O₂, should be monitored further, poor reproduction with NB-316.

Future recommendations

| Old procedure | Problem | Suggested new procedure |
|---------------|----------------------------------------------------------------|--------------------------------------------------------------------|
| / | Questionable O ₂ and H ₂ formation rate. | Should be monitored further, another reproductions should be done. |

Linked experiments

[SrTiO₃ - NB-313: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(NB-301, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C](#)

[SrTiO₃ - NB-315: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(NB-301, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C](#)

[SrTiO₃ - NB-316: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C](#)

[SrTiO₃ - NB-319: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C \(reproduction NB-316\)](#)

[SrTiO₃ - NB-320: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C \(reproduction NB-316\) I](#)

[SrTiO₃ - NB-329: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C \(reproduction NB-316\) II](#)

[SrTiO₃ - NB-331: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C \(reproduction NB-316\) III](#)

[SrTiO₃ - NB-336: Liquid phase H₂ and O₂ of RhCrO_x,Al:SrTiO₃ \(EA-358, 0.5 mg/mL\), 365 nm, 50 mW/cm², 20 °C V \(reproduction NB-316\)](#)

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](#)

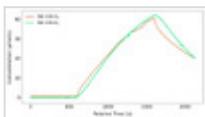
Protocol - [Liquid phase calibration of H2 UniAmp sensor](#)

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

Attached files

NB-339-O2 and h2 curve.png

sha256: a10e222d2cc72c6c1f1c38fdca2df05ecaa9413dc098352aa2a5ce276ed32cf0



NB-339-O2 and H2 curve.py

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20251111_151134-H2 table.jpg

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20251111_165549-suspension after vortex.jpg

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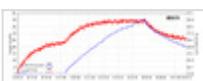


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2025-11-11_171421_NB-339-Ch2-2.txt

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NB-339.ulog

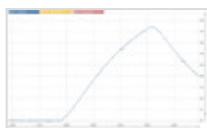
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NB-339-Logger3-during irradiation.csv

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NB-339-Logger3-during irradiation.bmp

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NB-339-Logger2-calibration.csv

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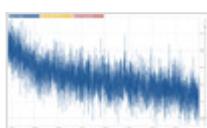
NB-339-Logger2-calibration step.bmp

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NB-339-Logger1-pre-polarization.bmp

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NB-339-Logger1.csv

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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3411>