

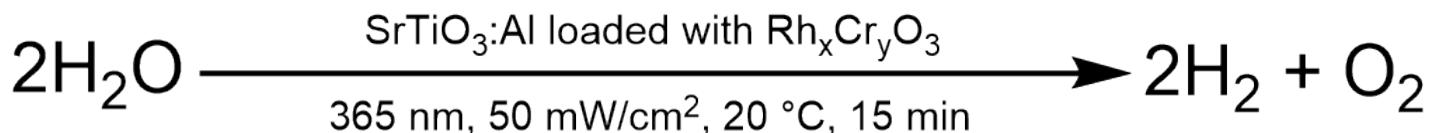
NB-320: Liquid phase H₂ and O₂ of RhCrO_x:Al:SrTiO₃ (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm², 20 °C (reproduction NB-316) I

Date: 2025-10-27
Tags: O₂ Test Calibration Future NB
Firesting O₂ sensor H₂ SrTiO₃
troubleshooting 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

Second reproduction of NB-316 (reference experiment): simultaneous detection of H₂ and O₂ evolution in liquid phase for irradiated suspension of Rh_xCr_yO₃:SrTiO₃ suspension (EA-358, 0.5 mg/mL), 365 nm LED, 50 mW/cm², 20 °C.

Reaction scheme



ChemDraw file linked: [NB-320-SrTiO3-photocatalytic H₂O splitting.cdxml](#)

Literature/reference experiments

Literature	/
Reproduction	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 SrTiO ₃ - NB-319: 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 (reproduction NB-316)
Similar experiments	SrTiO ₃ - NB-315: Liquid phase H ₂ and O ₂ of RhCrO _x :Al:SrTiO ₃ (NB-301, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C

Reagents

Name	CAS Number / Experiment Number	Inventor y number	Amount [mmol]	Equivalen ts	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Density (g/ml)	Volume [ml]	Pressure [bar]	Concentrati on [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)	/	/	/	12.50	12.80	/	/	/	/	/	/
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-18000-14	365	56	0.19

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. 600 s 2. 17:29:20
Irradiation stop: 1. Firesting [relative to start log] 2. Unisense	1. 1513 s 2. 17:44:33

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
27.10.2025		The experiment was done according to Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor Important steps and deviations are listed below	/	/
	9:27-10:27	Conditioning of H ₂ sensor	NB-320-Logger1 ca. 1.60 mV at the end of polarization procedure	NB-320.ulog NB-320-Logger1.csv NB-320-Logger1.bmp
		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 H ₂ bubbling.		

	11:11	Start of O ₂ logging.	NB-320-Ch2-1	2025-10-27_111158_NB-320-Ch2-1.txt 2025-10-27_111158_NB-320-Ch2-1.png
	11:12	Start of H ₂ logging.	NB-320-Logger2 -2 mV offset	NB-320.ulog NB-320-Logger2.bmp
		Important note: BOLA fitting for PT1000 sensor did not fit well, so the measurement was stopped and the setup was deassembled to eliminate the problem.		
	11:21	Start of O ₂ logging.	NB-320-Ch2-2	2025-10-27_112151_NB-320-Ch2-2.txt 2025-10-27_112151_NB-320-Ch2-2.png
	11:22	Start of H ₂ logging.	NB-320-Logger3	NB-320.ulog NB-320-Logger3-not saved calibration.csv NB-320-Logger3-calibration curve step.bmp
	11:22	Degassing was started.	/	/
	ca. 12:03	Removal cannula from water, keeping it in the gas phase above the liquid.	/	/
	12:05	Introducing H ₂ sensor into the reactor under Ar flow.	/	/
	12:15	H ₂ bubbling of the reactor was started	/	/
	12:21	Removing cannula from the reactor, closing the valve.	/	20251027_122208-first attempt of calibration.jpg
	12:31	Introducing second H ₂ balloon.	/	/
	ca. 12:40	1.000.000 ppm point was taken	853 mV, 775 uM	/
		Important note: But the 0 point was not taken, calibration needs to be redone.		
	ca. 12:50-13:00	Deassembling the setup, cleaning with acetone and compressed air.	/	/
	ca. 13:10-15	Assembling the setup for calibration once again.	/	/

	13:19	Start of O ₂ logging.	NB-320-Ch2-3	2025-10-27_131923_NB-320-Ch2-3.txt 2025-10-27_131923_NB-320-Ch2-3.png
	13:20	Start of H ₂ logging.	NB-320-Logger4 (used calibration)	NB-320.ulog NB-320-Logger4-used calibration.csv NB-320-Logger4-calibration info.png NB-320-Logger4-calibration curve step.bmp NB-320-Logger4-calibration 2 points.bmp
	13:24	Start degassing.	/	20251027_132610-degassing of water.jpg
	13:51	Moving cannula to the gas phase above the liquid.	/	/
	13:53	Introducing H ₂ sensor under Ar flow.	/	/
	13:56	0 point was taken.	0.4 mV	/
	14:02	Introducing H ₂ balloon.	/	20251027_140423-introducing H2 balloon.jpg
	14:10	Introducing 2nd H ₂ balloon.	/	20251027_141044-2nd balloon.jpg
	14:21	1.000.000 ppm point was taken and calibration was saved.	814 mV, 775 μ M, slope 1.049	20251027_142159-H2 table.jpg
	14:23	Stop of O ₂ and H ₂ loggings.	/	/
	ca. 14:30	Deassembling the setup, drying the reactor with acetone and compressed air .	/	/
		Sample preparation	/	/
	14:55	Weighing EA-358 photocatalyst in a 50 mL vial.	Creamy solid.	/
	15:00	Addition of 25 mL H ₂ O to the vial via graduated cylinder.	/	/
	15:07-10	The suspension was vortexed for 3 min (Equipment - VWR® VV3, Vortex Mixer , stage 4/6), covered with Al foil before further use.	/	20251027_151031-suspension after vortex.jpg
		Continue in Protocol - In-situ hydrogen and oxygen measurement in H₂/O₂ reactor from step 6		

	ca. 15:20	The suspension was transferred to the reactor using glass pipette (preliminary the vial was manually shaken ca. 15 s) .	/	/
	15:30-50	Eliminating leakage in the water cooling system.	Tubing was loosened and not completely sealed to the olive, leakage of water appeared when LAUDA was on --> problem was eliminated.	/
	ca. 15:55	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	/
	15:57	The degassing was started	/	20251027_160356-degassing of the suspension.jpg
	16:05	Start of O₂ logging.	NB-320-Ch2-4	2025-10-27_160525_NB-320-Ch2-4.txt 2025-10-27_160525_NB-320-Ch2-4.png
	16:26	Cannula was transferred to gas phase, above the suspension.	/	/
	16:28	H ₂ sensor was added in Ar counterflow.	/	20251027_163001-introducing H2 sensor under Ar flow.jpg
	16:31	The degassing was stopped by removing the cannula and closing the valve.	/	/
	16:35	Stop of O₂ logging.	/	/
	16:35	Start of O₂ logging.	NB-320-Ch2-5	2025-10-27_163556_NB-320-Ch2-5.txt 2025-10-27_163556_NB-320-Ch2-5.png
	16.35	Start of H₂ logging.	NB-320-Logger5	NB-320.ulog
	16:35-45	Equilibration time.	16:39 - putting side cover in the box --> change in O ₂ value, the stirring was stopped	/
	16:47	Switching stirring of the suspension ON again.	/	/

	16:51	Stop of O ₂ and H ₂ loggings.	Too high O ₂ leakage, BOLA fittings of all sensors need to be checked.	/
	17:00	Start O ₂ logging.	NB-320-Ch2-6	2025-10-27_170003_NB-320-Ch2-6.txt 2025-10-27_170003_NB-320-Ch2-6.png
	17:01	Start degassing once again.	/	/
	17:12	Removing cannula from the liquid phase, placing above the suspension in the gas phase.	/	/
	17:13	Introducing H ₂ sensor under Ar.	/	/
	17:16	Stop degassing, closing the valve.	/	20251027_171842-after introducing H2 sensor to the suspension.jpg
	17:18	Stop O ₂ logging.	/	/
	17:19	Start H ₂ logging.	NB-320-Logger6 (H₂ logging for irradiation)	NB-320.ulog NB-320-Logger6-during irradiation.csv NB-320-Logger6-during irradiation.bmp
	17:19	Start O ₂ logging.	NB-320-Ch2-7 (O₂ logging for irradiation)	2025-10-27_171920_NB-320-Ch2-7.txt 2025-10-27_171920_NB-320-Ch2-7.png
	17:19-29	Equilibration time.	Leakage rate ca. 0.5 um in 10 min	/
	17.29	The irradiation was started	Step in H ₂ curve at ca. 17:42	20251027_172931-after start of irradiation.jpg
	17:44	The irradiation was stopped.	/	/
	17:44-54	Equilibration time.	/	/
	17.54	Stop of O ₂ and H ₂ logging.	/	/

	ca. 18:00	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).	/
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Analysis

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

Used calibration for UniSense: NB-320-Logger4

(for feasability, the succcesful O₂ and H₂ loggings used for calibration and photocatalytic test are highlighted with green)

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Python script	Processed Data	Comparative Data	Interpretation
27.10.2025	9:27-10:27	NB-320-Logger1	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-320.ulog NB-320-Logger1.csv	/	NB-320-Logger1.bmp	/	Pre-polarization
	11:12	NB-320-Logger2	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-320.ulog	/	NB-320-Logger2.bmp	/	Unsuccessful.
	11:22	NB-320-Logger3	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-320.ulog NB-320-Logger3-not saved calibration.csv	/	NB-320-Logger3-calibration curve step.bmp	/	0 point was not taken
	13:20	NB-320-Logger4	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-320.ulog NB-320-Logger4-used calibration.csv	/	NB-320-Logger4-calibration info.png NB-320-Logger4-calibration curve step.bmp NB-320-Logger4-calibration 2 points.bmp	SrTiO3 - NB-316: Liquid phase H2 and O2 of RhCrOx,Al:SrTiO3 (EA-358, 0.5 mg/mL), 365 nm, 50 mW/cm ² , 20 °C	Used calibration for H2 sensor 10 ⁻⁶ ppm - 814 mV, slope 1.049
	16:35	NB-320-Logger5	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-320.ulog	/	/	/	Unsuccesful.

	17:19	NB-320-Logger6	electrochemical H2 detection	Equipment - H2 UniAmp Sensor - Normal range - 2.1 x 80 mm needle	water	NB-320.ulog NB-320-Logger6-during irradiation.csv	NB-320-O2 and H2 curve.py	NB-320-Logger6-during irradiation.bmp NB-320-O2 and H2 curves.png	/	H2 logging for the photocatalytic test Clean response of H2 sensor, step in signal at ca. 17.42, ca. 47 µM H2 at the end of irradiation.
	11:11	NB-320-Ch2-1	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_111158_NB-320-Ch2-1.txt	/	2025-10-27_111158_NB-320-Ch2-1.png	/	First attempt, BOLA for PT1000 was not tight, needed to be redone.
	11:21	NB-320-Ch2-2	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_112151_NB-320-Ch2-2.txt	/	2025-10-27_112151_NB-320-Ch2-2.png	/	First attempt of degassing in the first attempt of calibration.
	13:19	NB-320-Ch2-3	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_131923_NB-320-Ch2-3.txt	/	2025-10-27_131923_NB-320-Ch2-3.png	/	Degassing for the calibration of H2 sensor used further in the experiment.
	16:05	NB-320-Ch2-4	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_160525_NB-320-Ch2-4.txt	/	2025-10-27_160525_NB-320-Ch2-4.png	/	First degassing of the photocatalyst suspension.
	16:35	NB-320-Ch2-5	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_163556_NB-320-Ch2-5.txt	/	2025-10-27_163556_NB-320-Ch2-5.png	/	Change in O2 value during stirring and after stop of stirring. High O2 leakage rate afterwards, elimination of the problem was required.
	17:00	NB-320-Ch2-6	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_170003_NB-320-Ch2-6.txt	/	2025-10-27_170003_NB-320-Ch2-6.png	/	2nd degassing of the photocatalyst suspension after eliminating leakage problem.
	17:19	NB-320-Ch2-7	Optical O2 detection	Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel	water	2025-10-27_171920_NB-320-Ch2-7.txt	NB-320-O2 and H2 curve.py	2025-10-27_171920_NB-320-Ch2-7.png NB-320-O2 and H2 curves.png	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	O2 logging for the photocatalytic test. Increase in O2 value during irradiation, clean response, O2 value ca. 37 µM at the end of irradiation

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, reproduction of [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](#)) were performed.

H₂ level at the end of irradiation - ca. 47 umol/L, O₂ level - ca. 37 umol/L.

Future recommendations

Old procedure	Problem	Suggested new procedure
/	High leakage rate in O ₂ measurements.	Check BOLA fitting of all sensors (should be tight and fit well to all sensors), check how well the GL14/NS14 and GL18/NS14 adapters are greased.

/	0 point during H ₂ calibration was not saved.	Don't forget to take 0 point after introducing H ₂ sensor in the reactor and before introducing H ₂ balloon.
/	Stop stirring of the stirring plate when closing the irradiation box with the front cover.	Check the position of the stirring plate in the box. When closing the box before irradiation, no touching of the wall with the press button of the stirrer should appear.

Linked experiments

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)

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](#)

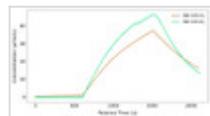
Protocol - [Liquid phase calibration of H₂ UniAmp sensor](#)

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

Attached files

NB-320-O2 and H2 curves.png

sha256: d9d7b2c215feeb0f98b8a672f92447f6783bcc820b5ba6441c70159a250157ac



NB-320-O2 and H2 curve.py

sha256: 06a3d6350deea81473a72eaf51a1df31fd3c06d91e50eb2d9e4d029d332ae65a

Unisense-NB-320Screenshot 2025-11-07 093103.png

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NB-320-SrTiO₃-photocatalytic H₂O splitting.cdxml

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20251027_175519-after irradiation.jpg

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20251027_172931-after start of irradiation.jpg

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20251027_160356-degassing of the suspension.jpg

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20251027_163001-introducing H₂ sensor under Ar flow.jpg

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20251027_171842-after introducing H₂ sensor to the suspension.jpg

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20251027_142159-H2 table.jpg

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20251027_151031-suspension after vortex.jpg

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20251027_141044-2nd balloon.jpg

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20251027_140423-introducing H2 balloon.jpg

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20251027_132610-degassing of water.jpg

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20251027_122208-first attempt of calibration.jpg

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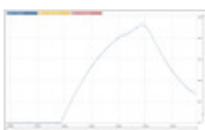


NB-320-Logger6-during irradiation.csv

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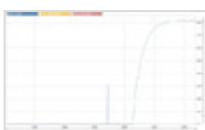
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NB-320-Logger4-calibration curve step.bmp

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NB-320-Logger4-used calibration.csv

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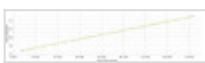
NB-320-Logger4-calibration info.png

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NB-320-Logger4-calibration 2 points.bmp

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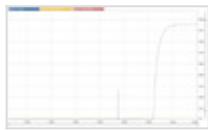


NB-320-Logger3-not saved calibration.csv

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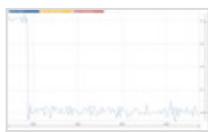
NB-320-Logger3-calibration curve step.bmp

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NB-320-Logger2.bmp

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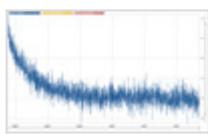


NB-320-Logger1.csv

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NB-320-Logger1.bmp

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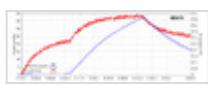


2025-10-27_171920_NB-320-Ch2-7.txt

sha256: de96e1bdd9f36bcf5eca4c49ba40aa6062977676d7c214a09215762ab51512c

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2025-10-27_170003_NB-320-Ch2-6.png

sha256: e33a1537d39ca1777655632a5e4584ff2c0dc3aaef4d61003fb289765b81243



2025-10-27_170003_NB-320-Ch2-6.txt

sha256: 6fd7b316bee332224c9235388c540c3cf3191ce135ea3fdd0319840f10ae0363

2025-10-27_163556_NB-320-Ch2-5.txt

sha256: eb020b90e9b8c2f57c4ca901a62942acf0634e7ae654b80f726dd8966496697b

2025-10-27_163556_NB-320-Ch2-5.png

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2025-10-27_160525_NB-320-Ch2-4.txt

sha256: f9c2719b2c3df350f311dbe63dd814f06473208ee5d3c0bd099c7e8223f12179

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2025-10-27_131923_NB-320-Ch2-3.txt

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2025-10-27_112151_NB-320-Ch2-2.txt

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2025-10-27_112151_NB-320-Ch2-2.png

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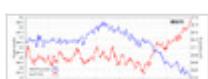


2025-10-27_111158_NB-320-Ch2-1.txt

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2025-10-27_111158_NB-320-Ch2-1.png

sha256: cdbf3e331e1c8301ab3c050122877558d01b67348055dcbebd57b16c81c41de7



NB-320.ulog

sha256: 66c7c58d62eb69cf90ebda6f243332da31f4636a26ca8e7d4a04ede9c2e13b05



Unique eLabID: 20251027-15f09fafef1c2e5c3939717d4c0df694186b1897

Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=3262>