

MRG-059-ZO: Irradiation of [Ru(bpy)₃]Cl₂ * 6 H₂O in the ChemSpeed robot - repetition of experiments from -ZN with not reproducible results

Date: 2025-01-21

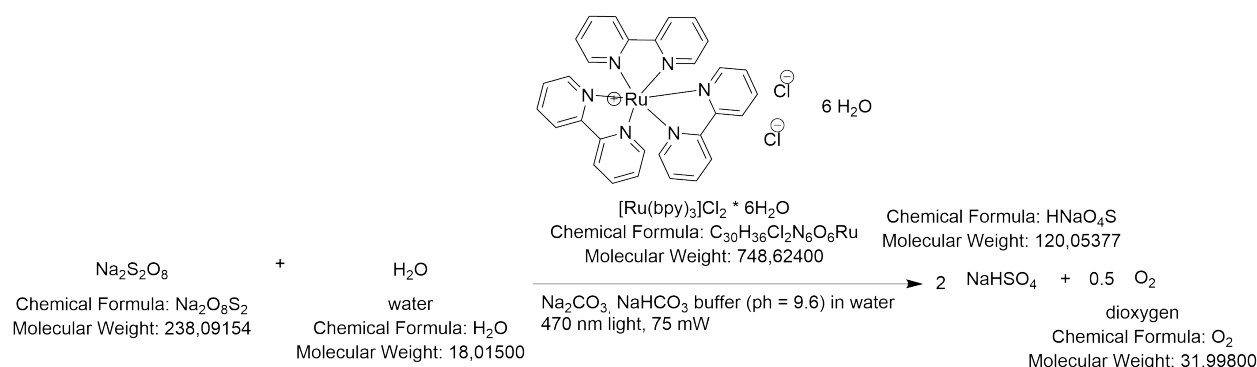
Tags: Radiation O2 [Ru(bpy)₃]Cl₂*6 H₂O AE reference analytics HTE MRG

Category: HTE

Status: Done

Created by: Michael Ringleb

Reaction scheme/sample structure



Literature/reference experiments

Literature	https://doi.org/10.1021/acscatal.6b02595
Reproduction	/
Related experiment	HTE - AE-414: Preparation of stock solutions for the irradiation of [Ru(bpy)₃]Cl₂ * 6 H₂O HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O₂ and T spot HTE - AE-406: MRG-059-ZM: Irradiation of [Ru(bpy)₃]Cl₂ * 6 H₂O in the ChemSpeed robot - reproduction of standard conditions - new vial HTE - MRG-059-ZN: Irradiation of [Ru(bpy)₃]Cl₂ * 6 H₂O in the ChemSpeed robot - screening of pH, [Ru], [Ox] and Irradiance

Reagents/Parameters table

for content of the stated solutions see [HTE - AE-422: Preparation of stock solutions for the irradiation of \[Ru\(bpy\)₃\]Cl₂ * 6 H₂O](#)

for calculations see: [AE-422-HTE.xlsx](#)

Experiment	Reproduction	V [Ru] [mL] ...			
1	-1,				
2	-1				
1	-2				
2	-2				
1	-3				
98	-1		Blank		
99	-1		Standard condtions		
99	-2		Standard conditions		

Run	Used [Ru] solution	V [Ru] [mL]	obtained conc. [μM]	Used [Ox] solution	V [Ox] [mL]	obtained conc. [mM]	Used [NaHCO3] solution	V [NaHCO3] [mL]	obtained conc. [M]	Used [Na2CO3] solution	V [Na2CO3] [mL]	obtained conc. [M]	obtained pH according to HTE - AE-413: Titration of Na2CO3 with NaHCO3 and vice versa	V [H2O] [mL]	power setting [V]	obtained power output [mW] AE-341: Power measurment with different power settings from Joy-It with 470 nm
1a+b	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 II	0.602	0.0708	Na2CO3 I	0.248	0.0292	9.6	6.800	0.18	75
2	Ru II	0.850	20	Ox III	0.425	6	NaHCO3 II	0.602	0.0708	Na2CO3 I	0.248	0.0292	9.6	6.375	0.18	75
3	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 II	0.602	0.0708	Na2CO3 I	0.248	0.0292	9.6	6.800	0.12	54
4	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 II	0.372	0.0438	Na2CO3 I	0.478	0.0292	10.0	6.800	0.18	75
8A	/	0.000	0	Ox III	0.425	6	NaHCO3 II	0.602	0.0708	Na2CO3 I	0.248	0.0292	9.6	7.225	0.18	75
5	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 I	0.231	0.0196	Na2CO3 I	0.683	0.0292	10.4	6.735	0.18	75
1c	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 I	0.836	0.0708	Na2CO3 I	0.248	0.0292	9.6	6.566	0.18	75
6	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 III	0.649	0.0382	Na2CO3 I	0.818	0.0292	10.9	6.183	0.18	75
7	Ru II	0.425	10	Ox III	0.425	6	NaHCO3 II	0.753	0.0886	Na2CO3 II	0.324	0.0292	9.2	6.573	0.18	75
9A	/	/	0	/	/	0	/	/	0	/	/	0	7.0	8.5	0.18	75

*final volume of all reaction solutions is 8.5 mL

no change of lid after 1c

Irradiation Parameters

Power measured using [\[Power Meter\] 843-R-USB + 919P-020-12](#) unless specified otherwise.

Oxygen sensor	Light Source Name	Wavelength [nm]	Power Setting [mW]	Analog input control voltage [V] using Equipment - Joy-it JT-RD6006 DC POWER SUPPLY
FireSting Fiber-Optic Oxygen Meter	Light Source - LCS-0470-50-22	470	see table above	see table above

Used beam combiner [Name or None]	none
Irradiation distance [cm]	9.5
Thermostat temperature [°C]	/
Stirring speed [rpm]	400
Start time irradiation [s]	see csv/json
End time irradiation [s]	see csv/json

Procedure/observations

Experiment was done by AE and MRG

Run Table

Run 1

Experiment	Date	Start Time	End Time	Number of piercing	Comment	Pictures
MRG-059-ZO-1A	22.01.25	11:00	12:20	9	/	/

Set-up changes: Change of Rul to Rull solution, change of lid

Run 2

Experiment	Date	Start Time	End Time	Number of piercing	Comment	Pictures
MRG-059-ZO-...	22.01.25	11:00	12:20	9	/	/

....

Date	Time	Step	Observations
22.01.25	10:10	The Calibration HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O2 and T spot was used The protocol for a fully automated workflow (Protocol - Operation of automated workflow for investigation of oxygen evolution) was utilized with a rare earth metal stir bar	
	- 10:40	everything was set up according to the Protocol - Operation of automated workflow for investigation of oxygen evolution	
		The python script is loaded as described in Protocol - Operation of automated workflow for investigation of oxygen evolution and the "experiment.yml" is initialized with four experiments (see reagents table for volumes of reactants) with the "run"-parameter = "true" and one at the end of the queue with "run" = "false"	
		The python script is executed with "python run.py"	
		AutoSuite program was started -->priming of tubings	

	10:55	from this point on the execution was done fully automatic (except the changing of lids and refill of stock vials) according to the table with steps executed by the automated platform in the Protocol - Operation of automated workflow for investigation of oxygen evolution	
	ca. 11:00	MRG-059-ZO-1A was started	septum was pierced 0 times at this point
	ca. 12:20	MRG-059-ZO-2A was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 13:40	MRG-059-ZO-1B was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 15:00	MRG-059-ZO-2B was started	at this point the setpum in the vial lid was pierced 27 times
	approx. 16:20	ZN-2B end	at this point the setpum in the vial lid was pierced 36 times
	17:05	change of lid, refill Rull, OxIII, NaHCO ₃ II, refill Milli Q water	
	17:20	MRG-059-ZO-3A was started	septum was pierced 0 times at this point
		MRG-059-ZO-4A was started	at this point the setpum in the vial lid was pierced 9 times
		MRG-059-ZO-3B was started	at this point the setpum in the vial lid was pierced 18 times
		MRG-059-ZO-4B was started	at this point the setpum in the vial lid was pierced 27 times
		MRG-059-ZO-8A was started	at this point the setpum in the vial lid was pierced 36 times
		ZO-8A end	at this point the setpum in the vial lid was pierced 45 times
	7:30	The lid of the vial was exchanged, also change of NaHCO ₃ II --> NaHCO ₃ I	
	7:36	MRG-059-ZO-5A was started	septum was pierced 0 times at this point
	ca. 8:56	MRG-059-ZO-5B was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 10:16	ZO-5B end	at this point the setpum in the vial lid was pierced 18 times

	10:23	MRG-059-ZO-1C was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 11:43	ZO-1C end	at this point the setpum in the vial lid was pierced 27 times
		changed NaHCO ₃ I --> NaHCO ₃ III, refilled Na ₂ CO ₃ I	
	11:46	MRG-059-ZO-6A was started	at this point the setpum in the vial lid was pierced 27 times
	ca. 13:06	MRG-059-ZO-6B was started	at this point the setpum in the vial lid was pierced 36 times
	ca. 14:26	ZO-6B end	at this point the setpum in the vial lid was pierced 45 times
	15:00	The lid was changed, changed NaHCO ₃ III to NaHCO ₃ II and Na ₂ CO ₃ I to Na ₂ CO ₃ II, refilled water	
	15:13	MRG-059-ZO-7A was started	septum was pierced 0 times at this point
	ca. 16:30	MRG-059-ZO-7B was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 17:50	MRG-059-ZO-9A was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 19:10	ZO-9A end	at this point the setpum in the vial lid was pierced 27 times

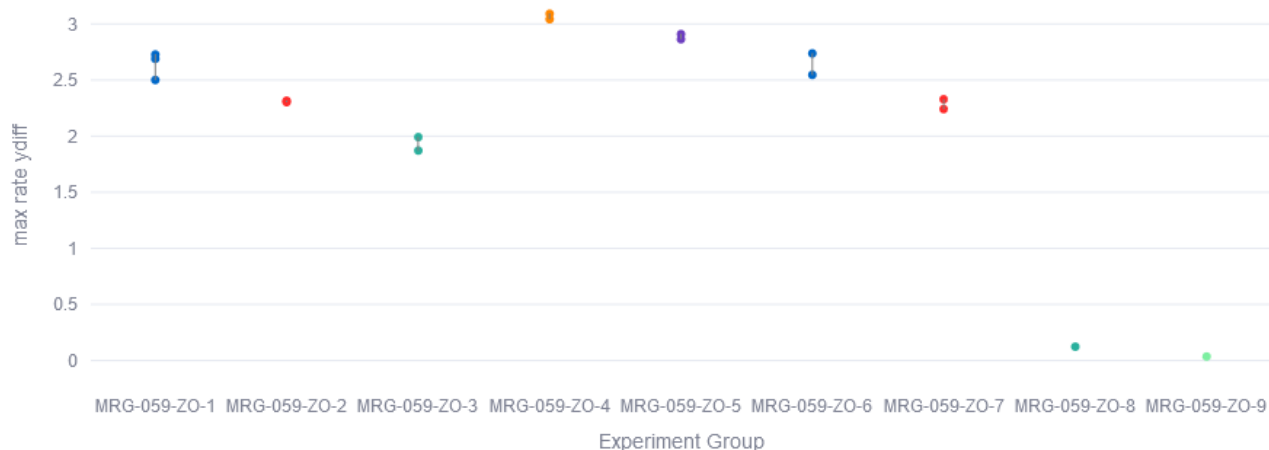
Results

Analysis

done with hte_streamlit (<https://github.com/jschneidewind/hte-streamlit>)

Good reproducible, generally not taken into account: -ZN-5-6 and -7, since wrong pH and [buffer] was used

max rate ydiff by Experiment Group

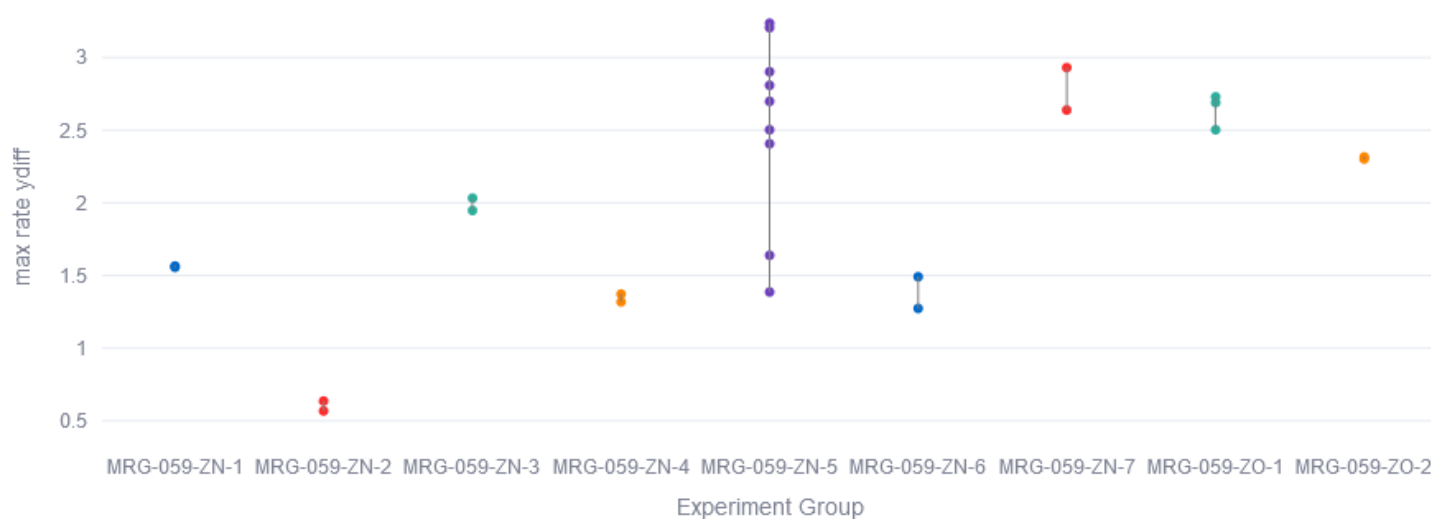


Evaluation of -ZN and -ZO screenings:

[Ru] screening: taken into account: -ZN-1 to -5, -ZN-7 and -ZO-1 to -2, not taken into account: -ZN-5-1 and -2 and -ZN-6, due to low rates, in reproduction expected rates, therefore most like ok to neglect

as expected. Highest rate for -ZN-7 and -ZN-5, into both directions slower rates (as expected).

max rate ydiff by Experiment Group

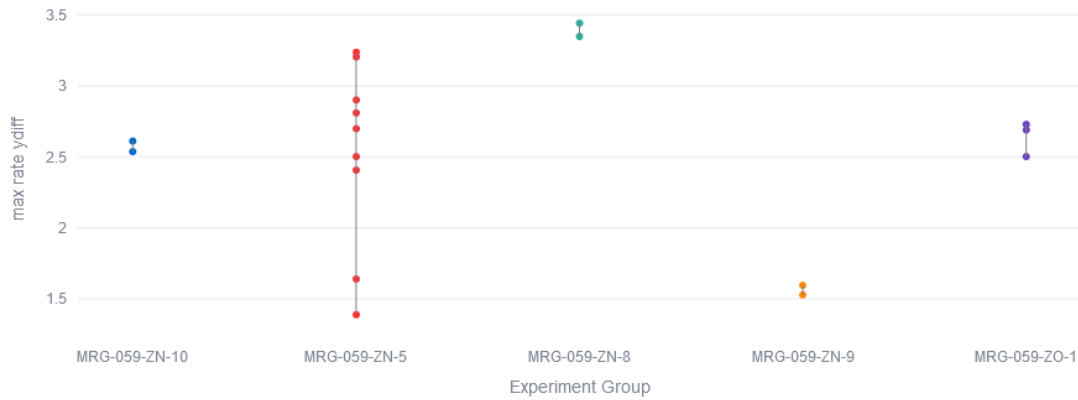


[Ox]: taken into account: -ZN-5, -ZN-8 to -10 and -ZO-1

not taken into account: -ZN-5-1 and -2

as expected, -5 and -10 quite similar, but also not much change in [Ox] (only doubled)

max rate ydiff by Experiment Group



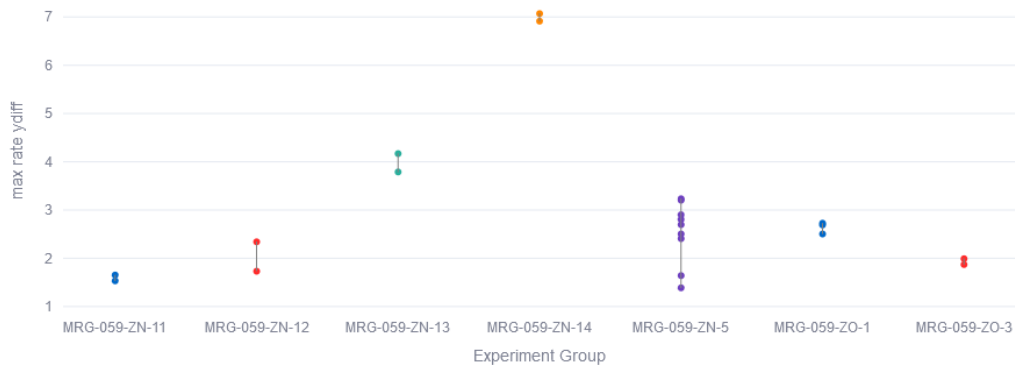
Irradiance:

Taken into account: -ZN-5(besides -1 and -2), -ZN-11 till -14 and -ZO-3

strongest influence on rate from investigated parameters. Higher irradiance means higher rate (as expected).

Repetition of -ZN-12 gave much less deviation, and at lower rate for -ZN, therefor in -ZN-12-2 most likely some error happened

max rate ydiff by Experiment Group

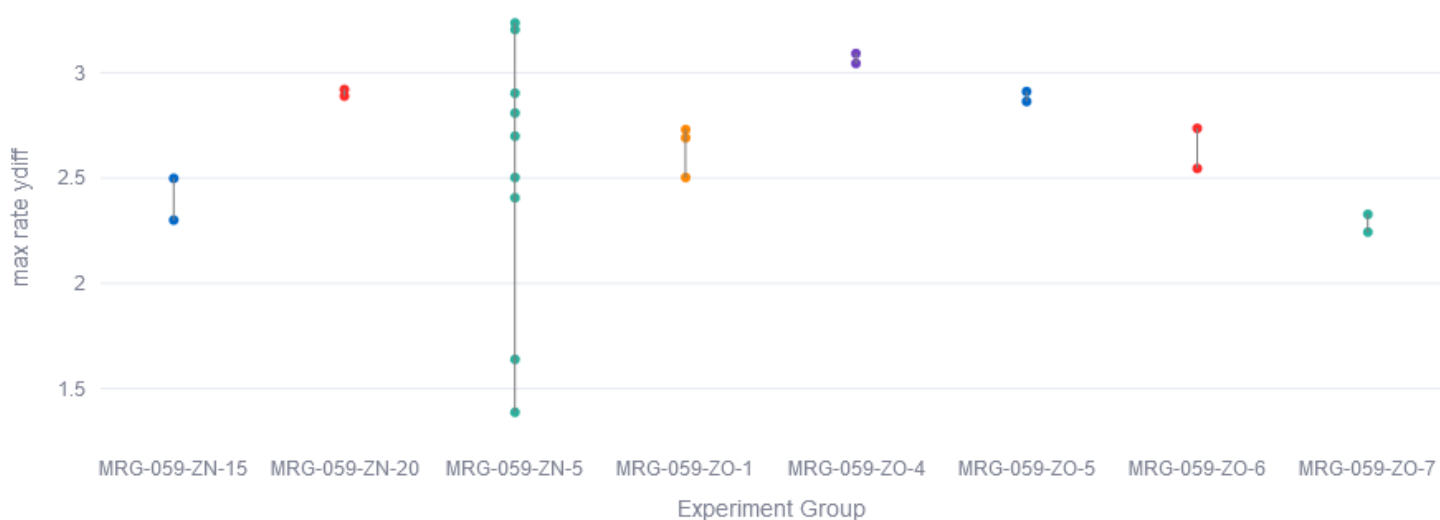


pH: taken into account: -ZN-5,-15 and -20, -ZO-1 and -4 to -6

not taken into account: -ZN-5-1 and -2, -ZN-16 to 19

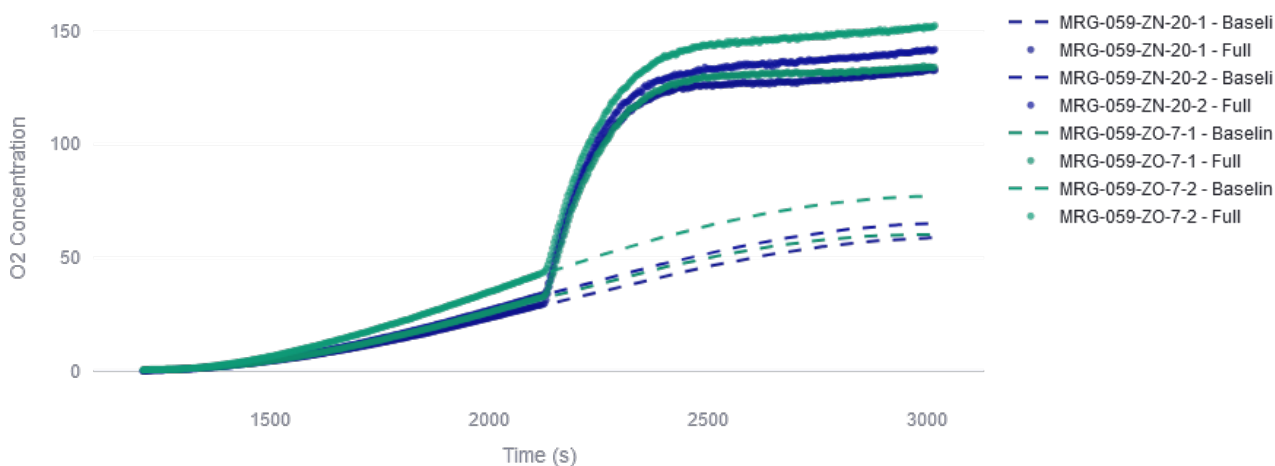
not as expected (steady increase to higher pH, from pH 5 to 11), but highest rate found for -ZO-4 (pH 10) than to higher pH decrease. No good explanation why different than reproted. Also decrease to lower pH.

max rate ydiff by Experiment Group



Problem with -ZN-20 and -ZO-7, which are both at pH = 9.2, since -ZN-20 much faster than -ZO-7. -ZO-7 would fit better in the trend, but no good reason, why it should be preferred other than that -ZN-20 was at the end of the -ZN runs and the runs before gave bad reproduction (so -ZN-20 could be a double fluke, even so that seems unlikely, but still possible)

HTE Data Visualization



Linked experiments

- AE-262: Irradiation of PhPDA (AE-257), 1.5 mg/mg SDS, 2 mg/mL PhPDA after 3 d
- AE-265: Preparation of stock solutions for the irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$

- AE-266: Calibration of oxygen and temperature sensor spots in the 10 mL HTE vial
- AE-267: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$
- AE-271: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, with manual light control
- AE-272: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, with manual light control I
- MRG-059-A: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals
- MRG-059-B: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals II
- MRG-059-C: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals III
- MRG-059-D: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals IV
- MRG-059-E: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals V
- MRG-059-F: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals VI
- MRG-059-Q: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals - 4th try I
- MRG-059-R: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals - 4th try II
- MRG-059-S: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals - 4th try III
- MRG-059-T: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals - 4th try IV
- MRG-059-U: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals - 4th try V
- AE-JSC-321: Manufacturing and calibration of new 10 mL HTE with sensor spots I
- AE-323: Preparation of stock solutions for the irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$
- MRG-059-V: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ in the ChemSpeed robot, fully automated peripherals
- 1st screening of Ru-cat concentration

- MRG-059-W: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O screenings (MRG-059-X, -Y, -Z)
- MRG-059-X: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals
- 1st screening of sacrificial oxidant
- MRG-059-Y: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - 2nd screening of [Ru]
- MRG-059-Z: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals
- 1st screening of pH (9.2 - 10.4)
- MRG-059-ZA - Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - 2nd pH screening (5.8 -12)
- AE-341: Power measurment with different power settings from Joy-It with 470 nm
- AE-342: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O I
- MRG-059-ZB: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - screening of light intensity
- HTE - MRG-059-ZI: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - screening of light intensity I
- HTE - MRG-059-ZJ: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - reproduction of standard conditions - new vial (with ruthenium stock solution or directly weighed in)
- HTE - AE-379: Test and calibration of O2 vial for O2 and T measurment in irrad setup
- HTE - AE-383: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O
- HTE - MRG-AE-059-ZK: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - reproduction of standard conditions - new vial, new T calibration
- HTE - AE-MRG-059-ZL: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - reproduction of standard conditions - new calibration
- HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O2 and T spot
- HTE - AE-405: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O I
- HTE - AE-406: MRG-059-ZM: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - reproduction of standard conditions - new vial
- HTE - AE-413: Titration of Na2CO3 with NaHCO3 and vice versa

HTE - AE-414: Preparation of stock solutions for the irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$

HTE - MRG-059-ZN: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ in the ChemSpeed robot - screening of pH, $[\text{Ru}]$, $[\text{Ox}]$ and Irradiance

HTE - AE-422: Preparation of stock solutions for the irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$

Linked items

Equipment - [Irradiation Set-Up](#)

Equipment - [Joy-it JT-RD6006 DC POWER SUPPLY](#)

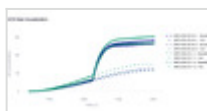
Light Source - [UHP LED 470 nm](#)

Protocol - [Operation of automated workflow for investigation of oxygen evolution - as of 03.07.2024](#)

Attached files

newplot24.png

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newplot23.png

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newplot22.png

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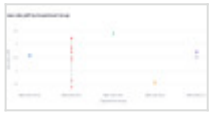
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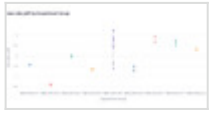
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MRG-059-ZO.zip

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AE-422-HTE.xlsx

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Comment

On Jacob Schneidewind wrote:

Notes:

- * reproduction: linking ZN experiment series
- * Adding annotation that colors in reagent table corresponding to runs
- * Using standard numbers for known conditions (reference experiments, 95, 96, 97 etc.)
- * Using only Excel sheet fully fledged out (with reproductions), no more overview in elab, with comments column
- * Changing to Run table structure
- * elab template for HTE



Unique eLabID: 20250121-594f52d7471dc9da919cc26b426e0c3b2ad57550
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=1654>