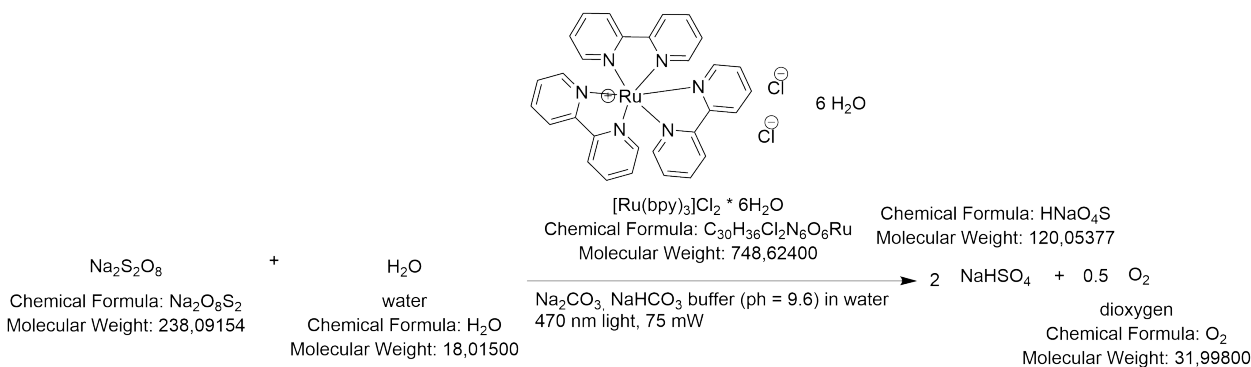


AE-406: MRG-059-ZM: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot - reproduction of standard conditions - new vial

Date: 2024-12-13
Tags: Radiation O2 [Ru(bpy)3]Cl2*6 H2O AE reference analytics HTE MRG
Category: HTE
Status: Done
Created by: Alexander Eith

Reaction scheme/sample structure



Literature/reference experiments

Literature	https://doi.org/10.1021/acscatal.6b02595
Reproduction	/
Related experiment	see below HTE - AE-405: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O I HTE - AE-404: Manufacturing, Test and calibration of HTE vial with O2 and T spot

Reagents

Constant parameters

Name	Abbreviation of solution	CAS Number / Experiment Number	Amount [mmol]	concentration used	Molar mass [g/mol]	Volume [mL]	concentration obtained
Sodium carbonate (anhydrous) solution	Na2CO3	497-19-8	0.267	0.8 M	105.988	0.334	0.0314 M

Sodium bicarbonate solution	NaHCO ₃	144-55-8	0.583	0.9 M	84.006	0.648	0.0686 M
Sodium persulfate solution	Ox	7775-27-1	0.051	60 mmol/L	238.09	0.850	6 mM
[Ru(bpy) ₃]Cl ₂ * 6 H ₂ O	Ru	50525-27-4	0.085 µmol	20 µmol/L	748.62	0.425	10 µM
Milli-Q water	/	7732-18-5	/	/	18.015	6.243	/

*final volume of all reaction solutions is 8.5 mL

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	75	0.18

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

Date	Time	Step	Observations
12.12	13:25	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	
	- 14:05	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	
			Degassing was not started. The python code was not loaded correctly into the robot
		The waiting times in the program where skipped and the vial was emptied by the robot.	
		Afterwards the robot was stopped and the computer was restarted	

		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"	
	approx. 14:50	from this point on the execution was done fully automatic according to the table with steps executed by the automated platform in the Protocol - Operation of automated workflow for investigation of oxygen evolution	The priming of the tubings was skipped
		MRG-059-ZM-1-1 was started	septum was pierced 9 times at this point
		MRG-059-ZM-1-2 was started	at this point the septum in the vial lid was pierced 18 times
		MRG-059-ZM-1-3 was started	at this point the septum in the vial lid was pierced 27 times
		MRG-059-ZM-1-4 was started	at this point the septum in the vial lid was pierced 36 times
	approx. 22:00	ZK-1-4 end	at this point the septum in the vial lid was pierced 45 times
13.12	8:35	The lid of the vial was exchanged	
	8:45	MRG-059-ZM-1-5 was started	septum was pierced 0 times at this point
		MRG-059-ZM-1-6 was started	at this point the septum in the vial lid was pierced 9 times
		MRG-059-ZM-1-7 was started	at this point the septum in the vial lid was pierced 18 times

		MRG-059-ZM-1-8 was started	at this point the setpum in the vial lid was pierced 27 times
		ZK-1-8 end	at this point the setpum in the vial lid was pierced 36 times
13.12	16:00	The lid of the vial was exchanged	
	16:05	MRG-059-ZM-1-9 was started	septum was pierced 0 times at this point
		MRG-059-ZM-1-10 was started	at this point the setpum in the vial lid was pierced 9 times
		MRG-059-ZM-1-11 was started	at this point the setpum in the vial lid was pierced 18 times
		MRG-059-ZM-1-12 was started	at this point the setpum in the vial lid was pierced 27 times
		ZK-1-12 end	at this point the setpum in the vial lid was pierced 36 times

Analysis

Quantitative Interpretation

Comparison of rates

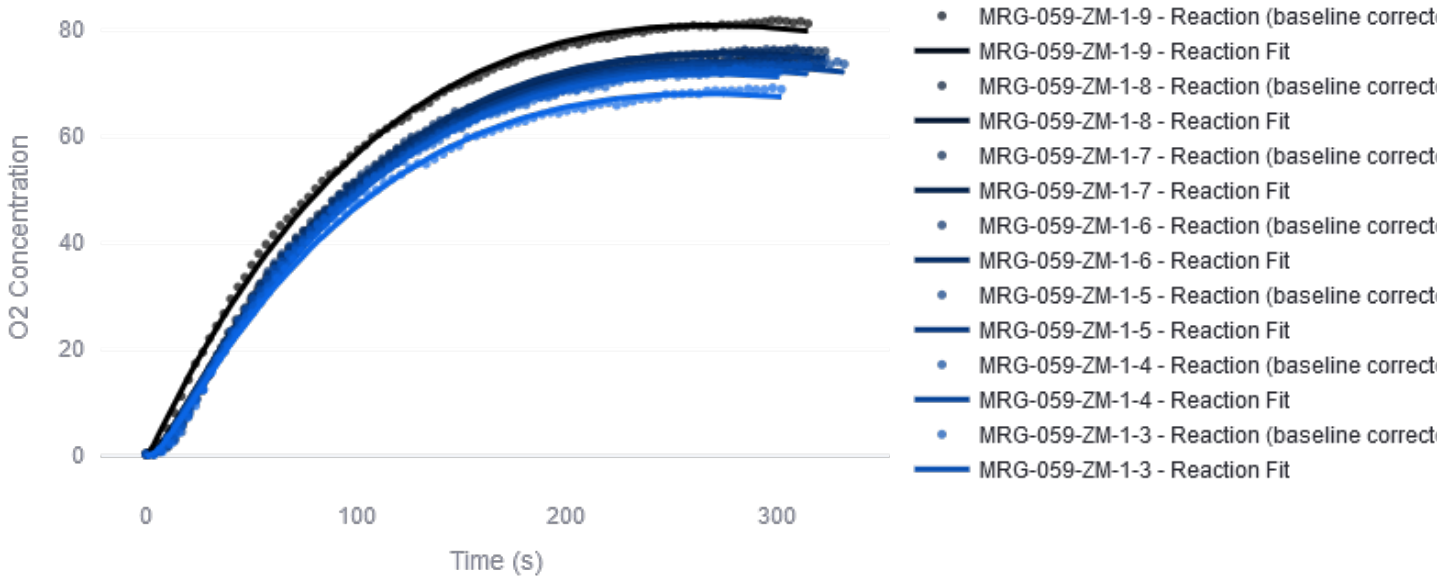
experiment determiner	rate constant (utilizing Jacobs fit inside the program on the IPC)	more information
MRG-059-ZM-1-1	0.0145	lid changed
MRG-059-ZM-1-2	0.0141	

MRG-059-ZM-1-3	0.0144	
MRG-059-ZM-1-4	0.0139	
MRG-059-ZM-1-5	0.0135	lid changed
MRG-059-ZM-1-6	0.0134	
MRG-059-ZM-1-7	0.0134	
MRG-059-ZM-1-8	0.0136	
MRG-059-ZM-1-9	0.0136	lid changed
MRG-059-ZM-1-10	0.0145	
MRG-059-ZM-1-11	0.0142	
MRG-059-ZM-1-12	0.0143	

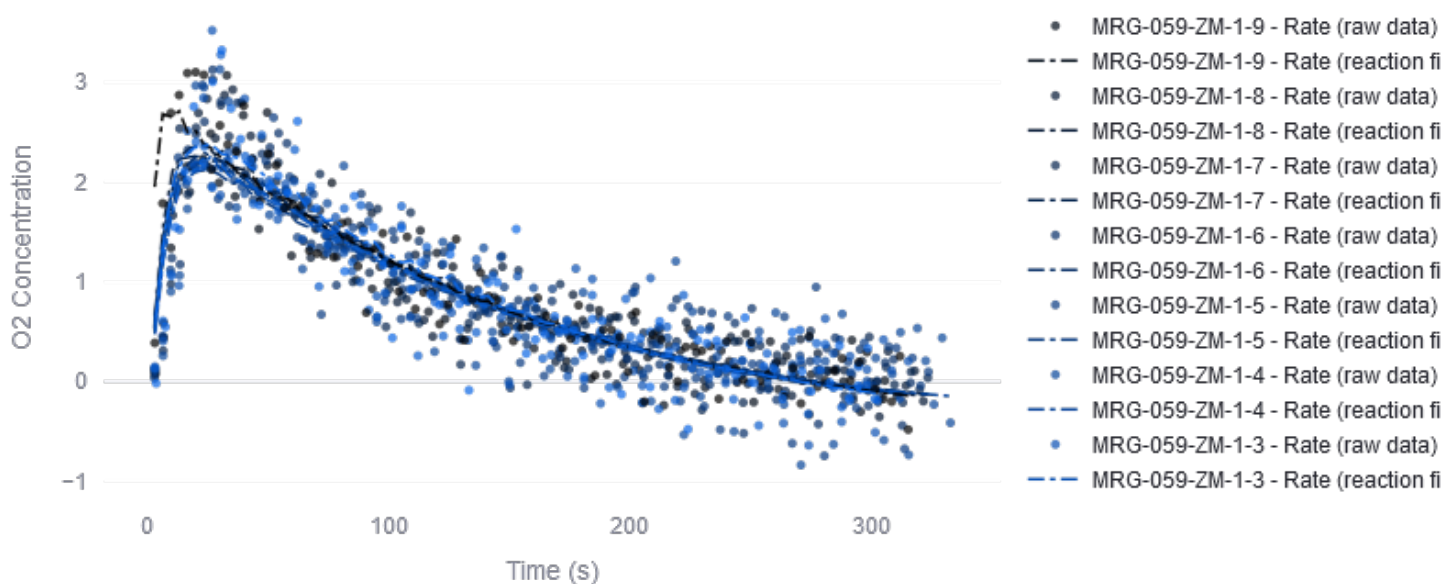
Results

Good reproducibility between runs. 1 outlier in the rate and also in the O2 concentration (ZM-1-10), But not in make rate y diff

HTE Data Visualization

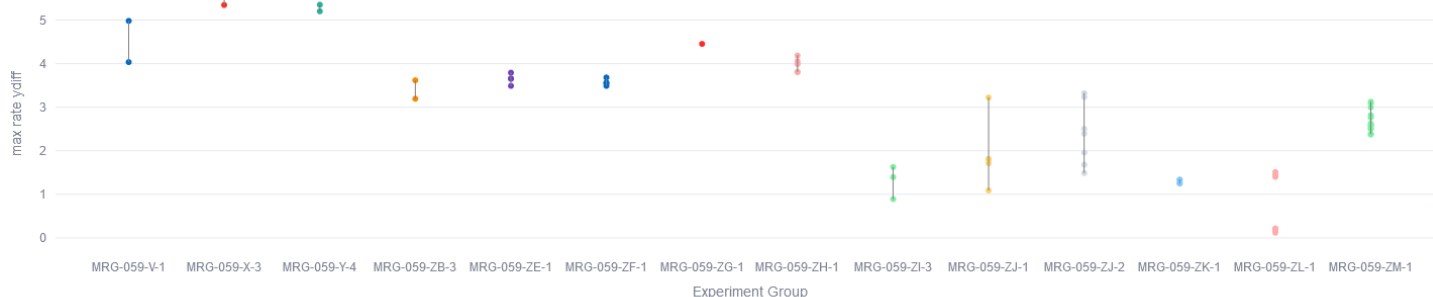


HTE Data Visualization



Span in make rate y diff from 2.379 (ZM-1-2) to 3.126 (ZM-1-12), approx. 24 % of highest value.

max rate ydiff by Experiment Group



Some deviation from former experiments. Cannot be but in a category (like ZB till ZH or ZI till ZL).

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 [Ru(bpy)₃]Cl₂ * 6 H₂O

- AE-266: Calibration of oxygen and temperature sensor spots in the 10 mL HTE vial

- AE-267: Irradiation of [Ru(bpy)₃]Cl₂ * 6 H₂O

- 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 $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{ H}_2\text{O}$ screenings (MRG-059-X, -Y, -Z)
- MRG-059-X: 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 sacrificial oxidant

- MRG-059-Y: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ in the ChemSpeed robot, fully automated peripherals - 2nd screening of $[\text{Ru}]$

- MRG-059-Z: 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 pH (9.2 - 10.4)

- MRG-059-ZA - Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ in the ChemSpeed robot - 2nd pH screening (5.8 -12)

- AE-342: Preparation of stock solutions for the irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ I

- MRG-059-ZB: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ in the ChemSpeed robot, fully automated peripherals - screening of light intensity

HTE - MRG-059-ZI: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ in the ChemSpeed robot, fully automated peripherals - screening of light intensity I

HTE - MRG-059-ZJ: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ 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 $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$

HTE - MRG-AE-059-ZK: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ in the ChemSpeed robot, fully automated peripherals - reproduction of standard conditions - new vial, new T calibration

HTE - AE-MRG-059-ZL: Irradiation of $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ 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 $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ I

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

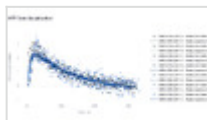
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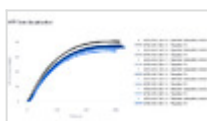
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Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=1594>