

MRG-059-ZN: Irradiation of [Ru(bpy)₃]Cl₂ * 6 H₂O in the ChemSpeed robot - screening of pH, [Ru], [Ox] and Irradiance

Date: 2025-01-10

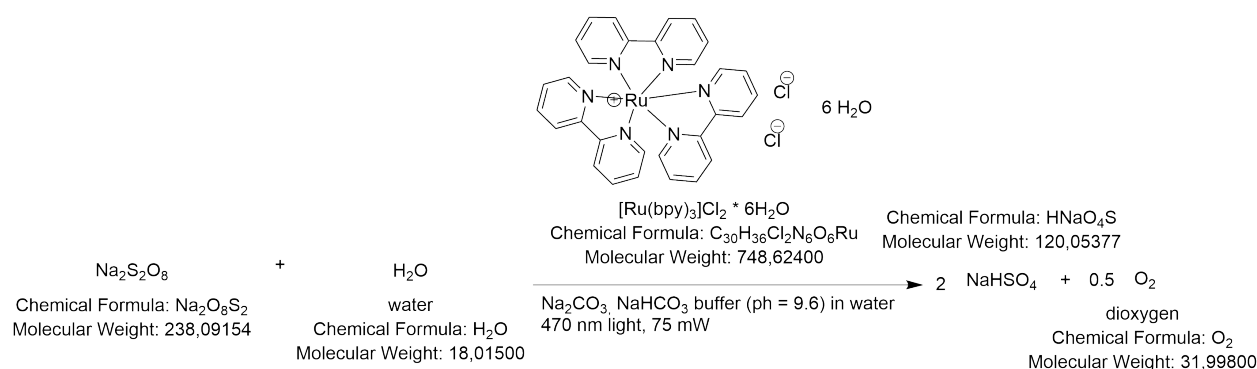
Tags: Radiation O2 [Ru(bpy)₃]Cl₂*6 H₂O AE reference analytics HTE MRG

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

Reagents/Parameters table

for content of the stated solutions see [HTE - AE-414: Preparation of stock solutions for the irradiation of \[Ru\(bpy\)₃\]Cl₂ * 6 H₂O](#)

for calculations see: [AE-41x-HTE.xlsx](#)

Run	Used [Ru] solution	V [Ru] [mL]	obtained conc. [μM]	Used [Ox] solution	V [Ox] [mL]	obtained conc. [mM]	Used [NaHCO ₃] solution	V [NaHCO ₃] [mL]	obtained conc. [M]	Used [Na ₂ CO ₃] solution	V [Na ₂ CO ₃] [mL]	obtained conc. [M]	obtained pH according to HTE - AE-413: Titration of Na ₂ CO ₃ with NaHCO ₃ and vice versa	V [H ₂ O] [mL]	power setting [V]	obtained power output [mW] AE-341: Power measurement with different power settings from Joy-It with 470 nm
1	Ru III	0.425	50	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.8000	0.18	75
2	Ru III	0.850	100	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.3750	0.18	75
3	Ru I	0.425	2	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.8000	0.18	75
4	Ru I	0.213	1	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	7.013	0.18	75
5a+b	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.18	75
6	Ru II	0.850	20	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.375	0.18	75
7	Ru II	0.213	5	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	7.013	0.18	75
8	Ru II	0.425	10	Ox III	0.708	10	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.517	0.18	75
5c	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.18	75
9	Ru II	0.425	10	Ox I	0.283	1	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.942	0.18	75
10	Ru II	0.425	10	Ox I	0.850	3	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.375	0.18	75
11	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.08	38
12	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.12	54
5d	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.18	75
13	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.30	115
14	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.800	0.80	285
5e	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	7.013	0.18	75
15	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.833	0.0980	Na ₂ CO ₃ III	0.347	0.00204	8.6	6.471	0.18	75
16	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.753	0.0886	Na ₂ CO ₃ II	0.324	0.0114	9.2	6.573	0.18	75
5f+g	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ II	0.248	0.0292	9.6	6.800	0.18	75
17	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.372	0.0439	Na ₂ CO ₃ I	0.478	0.0562	10.0	6.800	0.18	75
5h	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.602	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	7.013	0.18	75
18	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ I	0.231	0.0196	Na ₂ CO ₃ I	0.685	0.0804	10.4	6.735	0.18	75
5i	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ I	0.836	0.0708	Na ₂ CO ₃ I	0.248	0.0292	9.6	6.566	0.18	75
19	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ III	0.650	0.00382	Na ₂ CO ₃ I	0.818	0.0962	10.9	6.182	0.18	75
20	Ru II	0.425	10	Ox III	0.425	6	NaHCO ₃ II	0.753	0.0886	Na ₂ CO ₃ II	0.324	0.0114	9.2	6.573	0.18	75
21-neg. control		0			0			0			0			8.500	0.18	75
Run	Used [Ru] solution	V [Ru] [mL]	obtained conc. [μM]	Used [Ox] solution	V [Ox]	obtained conc. [mM]	Used [NaHCO ₃] solution	V [NaHCO ₃]	obtained conc. [M]	Used [Na ₂ CO ₃] solution	V [Na ₂ CO ₃]	obtained conc. [M]	obtained pH according to HTE - AE-413: Titration of Na ₂ CO ₃ with NaHCO ₃ and vice versa	V [H ₂ O] [mL]	power setting [V]	obtained power output [mW] AE-341: Power measurement with different power settings from Joy-It with 470 nm

*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	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

Date	Time	Step	Observations
13.01	10:35	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	
	- 11:05	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	
	11:10	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	
		MRG-059-ZN-1A was started	septum was pierced 0 times at this point
		MRG-059-ZN-2A was started	at this point the setpum in the vial lid was pierced 9 times
		MRG-059-ZN-1B was started	at this point the setpum in the vial lid was pierced 18 times
		MRG-059-ZN-2B was started	at this point the setpum in the vial lid was pierced 27 times
	approx. 16:15	ZN-2B end	at this point the setpum in the vial lid was pierced 36 times
	16:45	MRG-059-ZN-3A was started	septum was pierced 0 times at this point
		MRG-059-ZN-4A was started	at this point the setpum in the vial lid was pierced 9 times
		MRG-059-ZN-3B was started	at this point the setpum in the vial lid was pierced 18 times
		MRG-059-ZN-4B was started	at this point the setpum in the vial lid was pierced 27 times
	approx. 21:00	ZN-4B end	at this point the setpum in the vial lid was pierced 36 times
14.01.25	7:30	The lid of the vial was exchanged and the ruthenium solution was exchanged as well (Ru II instead of Ru I)	
	7:30	MRG-059-ZN-5A was started	septum was pierced 0 times at this point

	ca. 8:50	MRG-059-ZN-6A was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 10:10	MRG-059-ZN-6B was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 11:30	MRG-059-ZN-5B was started	at this point the setpum in the vial lid was pierced 27 times
	ca. 12:50	ZN-5B end	at this point the setpum in the vial lid was pierced 36 times
	13:45	The lid of the vial was exchanged	
	13:55	MRG-059-ZN-7A was started	septum was pierced 0 times at this point
	14:30		degassing canula was not connected to the tubing for argon --> no degassing --> experiment was not conducted
15.01	7:15	refill of water bottle, NaHCO ₃ and Na ₂ CO ₃ vials	
	ca. 7:20	MRG-059-ZN-7A was started	
	ca. 8:40	MRG-059-ZN-5C was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 10:00	MRG-059-ZN-8A was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 11:20	MRG-059-ZN-7B was started	at this point the setpum in the vial lid was pierced 27 times
	ca. 12:40	MRG-059-ZN-8B was started	at this point the setpum in the vial lid was pierced 36 times
	ca. 14:00	ZN-8B end	at this point the setpum in the vial lid was pierced 45 times
	ca. 14:30	MRG-059-ZN-9A was started	septum was pierced 0 times at this point
		MRG-059-ZN-10A was started	at this point the setpum in the vial lid was pierced 9 times
		MRG-059-ZN-10B was started	at this point the setpum in the vial lid was pierced 18 times

		MRG-059-ZN-9B was started	at this point the setpum in the vial lid was pierced 27 times
		ZN-9B end	at this point the setpum in the vial lid was pierced 36 times
16.01.	7:10	changed lid, change Oxl to OxIII, refill water bottle, refill NaHCO ₃ vial, refill Ru vial	
	7:35	saw that oxygen sensor was scratched (see pictures) --> potential explanation for problems from this point on	scratched_oxygen_sensor(1), scratched_oxygen_sensor(2)
	7:44	MRG-059-ZN-11A was started	septum was pierced 0 times at this point
	ca. 09:05	MRG-059-ZN-12A was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 10:25	MRG-059-ZN-11B was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 11:45	MRG-059-ZN-12B was started	at this point the setpum in the vial lid was pierced 27 times
	ca. 13:05	MRG-059-ZN-5D was started	at this point the setpum in the vial lid was pierced 36 times
	ca. 14:25	ZN-5D end	at this point the setpum in the vial lid was pierced 45 times
	14:30	change of lid	
	14:40	MRG-059-ZN-13A was started	septum was pierced 0 times at this point
	16:00	MRG-059-ZN-14A was started	at this point the setpum in the vial lid was pierced 9 times
	17:20	MRG-059-ZN-13B was started	at this point the setpum in the vial lid was pierced 18 times

	18:40	MRG-059-ZN-5E was started	at this point the setpum in the vial lid was pierced 27 times
	20:00	MRG-059-ZN-14B was started	at this point the setpum in the vial lid was pierced 36 times
	21:20	ZN-14B end	at this point the setpum in the vial lid was pierced 45 times
17.01.25	7:20	change of lid and refill of NaHCO ₃ (II) vial, change of Na ₂ CO ₃ vial from I to III	
	7:25	MRG-059-ZN-15A was started	septum was pierced 0 times at this point
	ca. 8:45	MRG-059-ZN-15B was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 10:05	ZN-15B end	at this point the setpum in the vial lid was pierced 18 times
	10:35	changed lid, change Na ₂ CO ₃ III to Na ₂ CO ₃ II and refill it, refill NaHCO ₃ II with newly prepared solution (NaHCO ₃ IIB), refill Ru vial, refill Ox vial	
	ca. 10:40	MRG-059-ZN-16A was started	septum was pierced 0 times at this point
	ca. 12:00	MRG-059-ZN-5F was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 13:20	MRG-059-ZN-16B was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 14:40	MRG-059-ZN-5G was started	at this point the setpum in the vial lid was pierced 27 times
	ca. 16:00	ZN-5G end	at this point the setpum in the vial lid was pierced 36 times

		<p>realization, that wrong NaCO₃ solution was placed inside the robot for the previous experiments --> Na₂CO₃I was in the robot, Na₂CO₃II should have been inside --> these experiments have to be changed in the table above and offer more experimental data</p> <p>--> the experiments for 16 will be repeated with the right solutions as 20A and 20B.</p>	
	17:30	MRG-059-ZN-17A was started	septum was pierced 0 times at this point
	ca. 18:50	MRG-059-ZN-5H was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 20:10	MRG-059-ZN-17B was started	at this point the setpum in the vial lid was pierced 18 times
	ca. 21:30	ZN-17B end	at this point the setpum in the vial lid was pierced 27 times
18.01.25	7:20	change of lid, refill of water, Ru vial and Ox vial, change of NaHCO ₃ vial (II --> I)	
	ca. 7:40	MRG-059-ZN-18A was started	septum was pierced 0 times at this point
	ca. 9:00	MRG-059-ZN-5I was started	at this point the setpum in the vial lid was pierced 9 times
	ca. 10:20	MRG-059-ZN-18B was started	at this point the setpum in the vial lid was pierced 18 times
	ca.11:40	ZN-18B end	at this point the setpum in the vial lid was pierced 27 times
	ca. 12:20	change of lid, change of NaHCO ₃ I --> NaHCO ₃ III, refill of Na ₂ CO ₃ vial	light was turned of intermittently --> possible explanation for unexpected behaviour
	ca. 12:30	MRG-059-ZN-19A was started	septum was pierced 0 times at this point
	ca. 13:50	MRG-059-ZN-19B was started	at this point the setpum in the vial lid was pierced 9 times

	ca. 14:10	change of NaHCO ₃ III --> NaHCO ₃ II and Na ₂ CO ₃ I - -> Na ₂ CO ₃ II	
	ca. 15:15	MRG-059-ZN-20A was started without prior lid change	septum was pierced 18 times at this point
	ca. 16:35	MRG-059-ZN-20B was started	at this point the setpum in the vial lid was pierced 27 times
	ca. 17:55	MRG-059-ZN-21-neg_control was started	at this point the setpum in the vial lid was pierced 36 times
	ca. 19:15	ZN-21-neg_control end	at this point the setpum in the vial lid was pierced 45 times

Results

Experiement	Light intensity [W/m ²]	c(Ru) [M]	c(Ox) [M]	c(buffer) [M]	pH	rate	annotations	max rate	max rate ydiff	rate constant
MRG-059-ZN-1A	6.637	0.00005	0.006	0.1	9.6	0.0119		1.3726065718542522	1.56684024772834	0.0023847090581694554
MRG-059-ZN-1B	6.637	0.00005	0.006	0.1	9.6	0.01122		1.4116023229990269	1.5567521274306269	0.10113379989859962
MRG-059-ZN-2A	6.637	0.0001	0.006	0.1	9.6	0.00902		0.5279846986364785	0.5689145211803568	0.0016765323703009489
MRG-059-ZN-2B	6.637	0.0001	0.006	0.1	9.6	0.00883		0.574786478128583	0.6381782133534027	0.0016138550036848723
MRG-059-ZN-3A	6.637	0.000002	0.006	0.1	9.6	0.01231		2.0485004485416285	2.033471461862278	0.10767370363596639
MRG-059-ZN-3B	6.637	0.000002	0.006	0.1	9.6	0.01196		1.8059572776799957	1.9476825877544672	0.0855823622009792
MRG-059-ZN-4A	6.637	0.000001	0.006	0.1	9.6	0.01137		1.291716359055795	1.3199809306185575	0.0024443484566659315
MRG-059-ZN-4B	6.637	0.000001	0.006	0.1	9.6	0.01142		1.3124245506374708	1.3727790333770906	0.07701253460838343
MRG-059-ZN-5A	6.637	0.00001	0.006	0.1	9.6	0.0092		1.3072630202969346	1.3885016460258668	0.13413820215079295
MRG-059-ZN-5B	6.637	0.00001	0.006	0.1	9.6	0.0106		1.4269653412030667	1.6399940244635447	0.9312796988755047
MRG-059-ZN-5C	6.637	0.00001	0.006	0.1	9.6	0.01409		2.8856829947457925	3.236932871395383	0.0027023709961994102
MRG-059-ZN-5D	6.637	0.00001	0.006	0.1	9.6	0.01229		2.241964979368425	2.4070964138684867	0.17347725328840866
MRG-059-ZN-5E	6.637	0.00001	0.006	0.1	9.6	0.01425		2.434552474452543	2.809120822449656	0.002689588753303034
MRG-059-ZN-5F	6.637	0.00001	0.006			0.01636	wrong Na ₂ CO ₃ stock solution used	2.692843210242308	2.9026728205040992	0.0023236281070955762
MRG-059-ZN-5G	6.637	0.00001	0.006			0.01643	wrong Na ₂ CO ₃ stock solution used	2.8443894366245837	3.2045976400612455	0.002497478545648613
MRG-059-ZN-5H	6.637	0.00001	0.006	0.1	9.6	0.0135		2.4593488164706416	2.699771250367079	0.16481287378640036
MRG-059-ZN-5I	6.637	0.00001	0.006	0.1	9.6	0.01252		2.3602741116996153	2.5028991175293385	0.14831854063085123
MRG-059-ZN-6A	6.637	0.00002	0.006	0.1	9.6	0.00899		1.2161975487147858	1.2754322660051896	0.2167747498592621
MRG-059-ZN-6B	6.637	0.00002	0.006	0.1	9.6	0.00976		1.273231632463744	1.4929582338447407	0.0017276247750278291
MRG-059-ZN-7A	6.637	0.000005	0.006	0.1	9.6	0.01377		2.365963903911414	2.6383979719581525	0.002684741680614671
MRG-059-ZN-7B	6.637	0.000005	0.006	0.1	9.6	0.0141		2.5278358513337835	2.9314146769843785	0.21594265804746307
MRG-059-ZN-8A	6.637	0.00001	0.01	0.1	9.6	0.01488		3.121338729142341	3.442367903379981	0.2668673048345261
MRG-059-ZN-8B	6.637	0.00001	0.01	0.1	9.6	0.01436		2.8308683839517848	3.3477837230559153	0.002915224421996032
MRG-059-ZN-9A	6.637	0.00001	0.001	0.1	9.6	0.00826		1.346624443785485	1.5283611518616635	0.10371765621910725
MRG-059-ZN-9B	6.637	0.00001	0.001	0.1	9.6	0.00828		1.444066893106493	1.595405059065614	0.0016590837752508691
MRG-059-ZN-10A	6.637	0.00001	0.003	0.1	9.6	0.01245		2.251276564515914	2.6119755252099313	0.002492314277435684
MRG-059-ZN-10B	6.637	0.00001	0.003	0.1	9.6	0.01247		2.365034869738274	2.538150181128327	0.0024725110959560453

MRG-059-ZN-11A	3.363	0.00001	0.006	0.1	9.6	0.01063		1.5191964106963698	1.6522772559220749	0.9952833519597576
MRG-059-ZN-11B	3.363	0.00001	0.006	0.1	9.6	0.00898		1.3617860404521824	1.531652178524913	0.0016648580877988942
MRG-059-ZN-12A	4.779	0.00001	0.006	0.1	9.6	0.00972		1.5675177488651313	1.7349592030903467	0.11455826214587113
MRG-059-ZN-12B	4.779	0.00001	0.006	0.1	9.6	0.01265		2.031902634540831	2.344757685492076	0.0023751440564162714
MRG-059-ZN-13A	10.177	0.00001	0.006	0.1	9.6	0.01655		3.4536001964116245	3.7885936813240106	0.0032420883697277525
MRG-059-ZN-13B	10.177	0.00001	0.006	0.1	9.6	0.01682		4.22415209928913	4.17128480288723	0.17890052741383766
MRG-059-ZN-14A	25.221	0.00001	0.006	0.1	9.6	0.02476		6.699417430360139	7.071885607915305	0.1266951773227783
MRG-059-ZN-14B	25.221	0.00001	0.006	0.1	9.6	0.02543		6.838699507742303	6.915803386968304	0.2599749081766893
MRG-059-ZN-15A	6.637	0.00001	0.006	0.1	8.6	0.01049		2.1538228923311085	2.300010510001799	0.0020284638036083447
MRG-059-ZN-15B	6.637	0.00001	0.006	0.1	8.6	0.01046		2.1731825094762733	2.4986575631543606	0.0020020661488796897
MRG-059-ZN-16A	6.637	0.00001	0.006	0.127	9.56	0.01511	wrong Na2CO3 stock solution used	3.0281782257248735	3.4088405493294376	0.002608928991125947
MRG-059-ZN-16B	6.637	0.00001	0.006	0.127	9.56	0.0166	wrong Na2CO3 stock solution used	2.9769602394836467	3.284300066038602	0.0024530387235953176
MRG-059-ZN-17A	6.637	0.00001	0.006	0.1	10	0.01473		2.9202496719186684	3.301153067153039	0.18460900645574085
MRG-059-ZN-17B	6.637	0.00001	0.006	0.1	10	0.01381		2.62453843646667	2.919728153959134	0.0026573692647804447
MRG-059-ZN-18A	6.637	0.00001	0.006	0.1	10.4	0.01386		2.855095050351741	2.874486755876948	0.0029015723563683765
MRG-059-ZN-18B	6.637	0.00001	0.006	0.1	10.4	0.01405		2.983765152491678	3.263406331153049	0.0027300030199764636
MRG-059-ZN-19A	6.637	0.00001	0.006	0.1	10.9	0.01439		2.526709313612363	2.801883550879067	0.07690041366208857
MRG-059-ZN-19B	6.637	0.00001	0.006	0.1	10.9	0.01577		3.041565802723591	3.614131098978619	0.0029015723314568275
MRG-059-ZN-20A	6.637	0.00001	0.006	0.1	9.2	0.01396		2.5935545071403494	2.8900171467224487	0.31279195820445044
MRG-059-ZN-20B	6.637	0.00001	0.006	0.1	9.2	0.01341		2.6151901295296147	2.9216129302816216	0.4346106269514234
MRG-059-ZN-21neg_control	6.637	0	0	0	NA	0				

Analysis

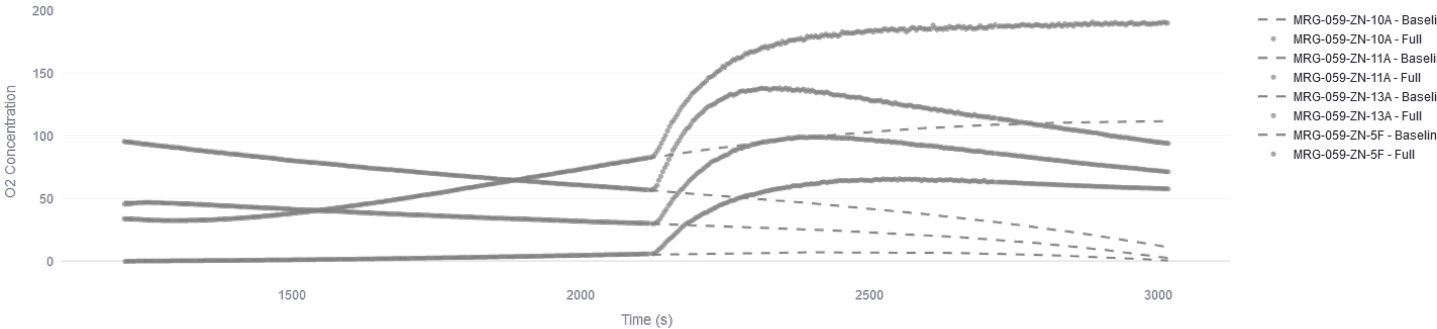
done with hte_streamlit (<https://github.com/jschneidewind/hte-streamlit>)

Generally good results.

Different baselines were observed. It is not clear why and when this different behaviours occur. It was seen, that AE did not put the degassing needle down far enough, which can (at least partially) explain the observed degassing, but also other experiments sometimes show high baselines or decreasing baselines during pre reaction baseline.

Some full reactions and thier baselines to highlight the differnt baseline behaviours:

HTE Data Visualization



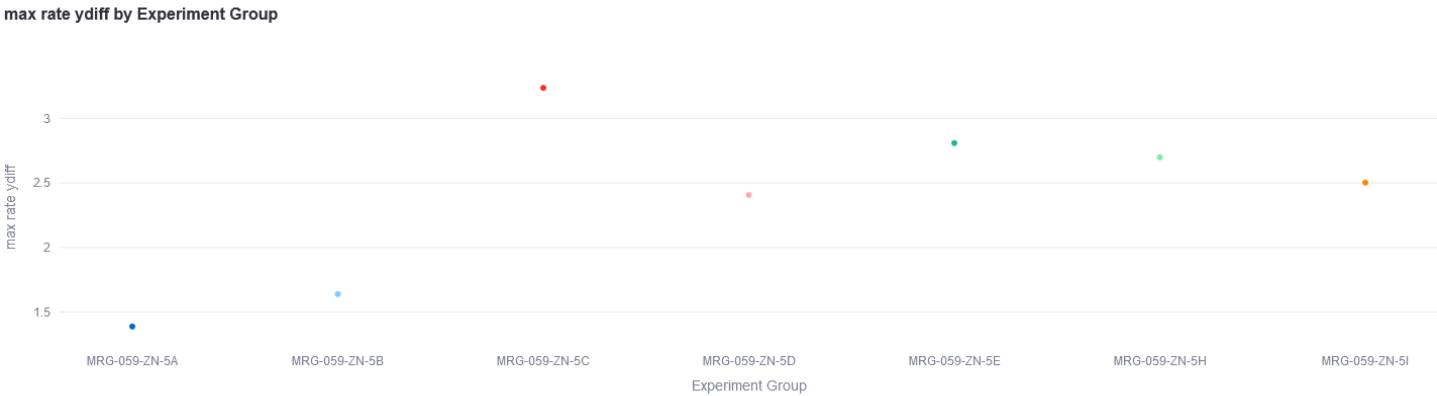
But the different degassing behaviours did not yield a clear trend in the observed rates, so that seems to not be a big problem.

Plot of all max rate ydiff:



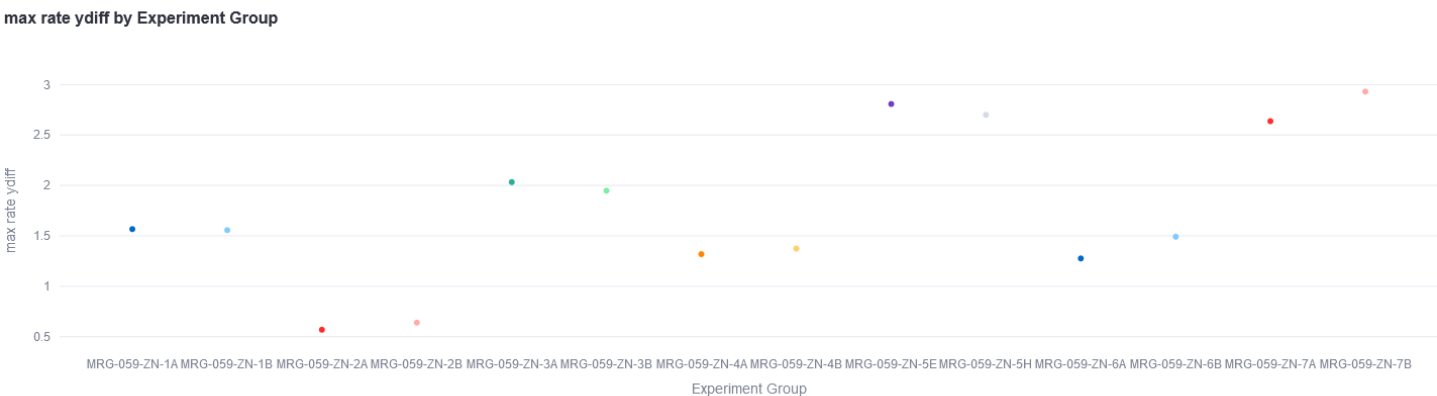
No extrem outliers observed. Detail discussion for changing of each parameter below:

Standard conditions:



A and B are slower than the other and C is bit faster than other. 5A and B has faster increase in baseline, this could explain lower rate

Screening of c(Ru):



Note that the order here is not in order of the Ru conc.

In general behaviour as expected. Peaks at 5 μ M. In both directions than decline only 6 and 1 don't fit the trend, but have nearly identical rates, No good explanation for that, but 6 in the same run as 5A/B, therefore maybe error in that run, which let to slower rates.

Screening c(Ox):



Note that the order here is not in order of the Ox conc.

Good reproducibility. Trend as expected.

Screening irradiance:



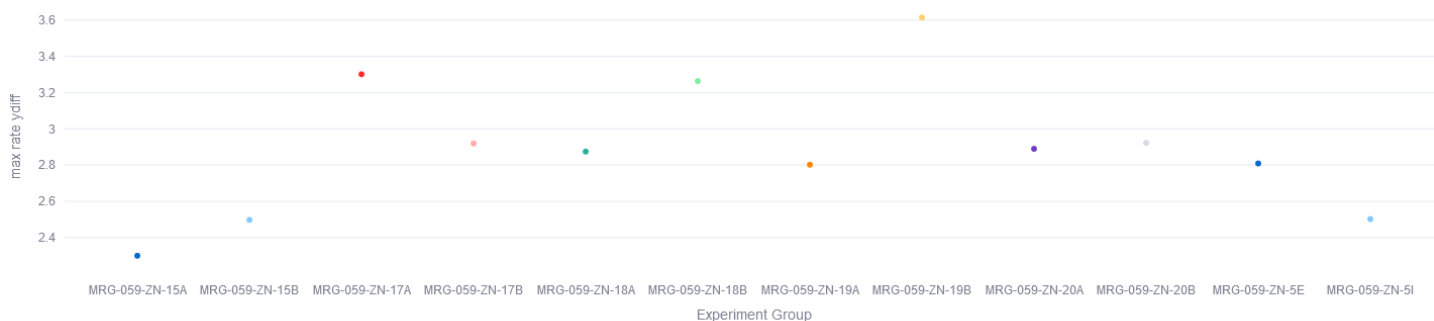
Note that the order here is not in order of the irradiance

Good reproducibility, except for 12, there larger error.

Trend makes sense (higher rates for higher intensity)

Screening pH value:

max rate ydiff by Experiment Group



Results look not so good, often not reproducibil results. Also trend not as expected, but likely due to high flutuation of results. No clear reason for that behaviour.

Blank

No O2 formation observed as expected

TL;dr

In general good results, some problems with reproducibility (5, 12) and issues with pH screening

Next steps

Repeating of not reproducible runs namly:

5A/B and 6 (1 run), 12 and 17 (1 run), and 18 to 20, seperate runs each

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 $[\text{Ru}(\text{bpy})_3]\text{Cl}_2 \cdot 6 \text{H}_2\text{O}$ 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 [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 [Ru(bpy)3]Cl2 * 6 H2O

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

MRG-059-ZN.zip

sha256: 6afb3c370ee46007c89520f7964ae84912882150128387f78d1e3d885d8da3c6

newplot16.png

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AE-41x-HTE.xlsx

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Unique eLabID: 20250110-023ce845f0dd6defc021c02d7333c01b5c441a19
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=1625>