MRG-059-ZC: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - pH Screening with Na2SiF6

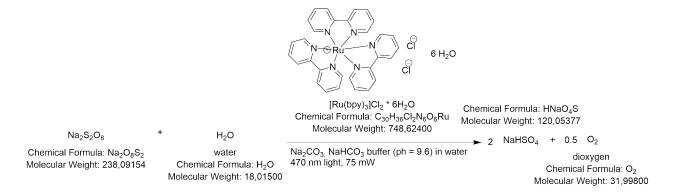
Date: 2024-09-11

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

Status: Done

Created by: Michael Ringleb

Reaction scheme/sample structure



Literature/reference experiments

Literature	https://doi.org/10.1021/acscatal.6b02595
Reproduction	
	see below
	AE-347: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O AE-JSC-321: Manufacturing and calibration of new 10 mL HTE with sensor spots I
Related experiment	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)
	MRG-059-ZB: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, fully automated peripherals - screening of light intensity

Reagents

Constant parameters

	Abbreviation of solution	CAS Number / Experiment Number	Amount [mmol]	concentration used	Molar mass [g/mol]	Volume [mL]	concentration obtained
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Sodium bicarbonate solution	NaHCO3 and NaHCO3/Na2CO3	144-55-8	see varied parameters table below	see varied parameters table below	84.006	see varied parameters table below	/
Sodium carbonate (anhydrous) solution	NaHCO3/Na2CO3	497-19-8	see varied parameters table below	see varied parameters table below	105.988	see varied parameters table below	/
Sodium hexafluoro silicate	Na2SiF6	16893-85-9	see varied parameters table below	see varied parameters table below	188.06	see varied parameters table below	/
Sodium persulfate solution	Ox	7775-27-1	0.051	60 mol/L	238.09	0.85	6 mM
[Ru(bpy)3]Cl2 * 6 H2O solution	Ru	50525-27-4	0.085 μmol	20 μmol/L	748.62	0.425	10 μΜ
Milli-Q water		7732-18-5	/	/	18.015	see varied parameters table below	/

Varied parameters

*final volume of all reaction solutions is 8.5 mL

Experiment determiner	pH value	Used CO3 solution	Used NaHCO3/ Na2CO3 concentratio n [mM]	Amount NaHCO3/ Na2CO3 [µmol]	concentration NaHCO3/ Na2CO3 in final solution [mmol/L]	Volume (NaHCO3/N a2CO3 solution) [mL]	Used Na2SiF6 concentratio n [mM]	Amount Na2SiF6 [µmol]	concentration Na2SiF6 in final solution [mmol/L]	Volume (Na2SiF6 solution) [mL]	volume Milli-Q water [mL]
MRG-059-ZC-1-1	9.2	NaHCO3/Na2CO 3	800	389.30/389.30	45.8/45.8	0.487	35	72.25	8.5	2.064	4.674
MRG-059-ZC-1-2	9.2	NaHCO3/Na2CO 3	800	389.30/389.30	45.8/45.8	0.487	35	72.25	8.5	2.064	4.674
MRG-059-ZC-2-1	8.6	NaHCO3/Na2CO 3	800	382.50/382.50	45.0/45.0	0.478	35	84.15	9.9	2.404	4.343
MRG-059-ZC-2-2	8.6	NaHCO3/Na2CO 3	800	382.50/382.50	45.0/45.0	0.478	35	84.15	9.9	2.404	4.343
MRG-059-ZC-2-3	8.6	NaHCO3/Na2CO 3	800	382.50/382.50	45.0/45.0	0.478	35	84.15	9.9	2.404	4.343
MRG-059-ZC-2-4	8.6	NaHCO3/Na2CO 3	800	382.50/382.50	45.0/45.0	0.478	35	84.15	9.9	2.404	4.343
MRG-059-ZC-3-1	8.6	NaHCO3	1000	850.00/0	100/0	0.85	35	0.00	0	0.0	6.375
MRG-059-ZC-3-2	8.6	NaHCO3	1000	850.00/0	100/0	0.85	35	0.00	0	0.0	6.375
MRG-059-ZC-3-3	8.6	NaHCO3	1000	850.00/0	100/0	0.85	35	0.00	0	0.0	6.375
MRG-059-ZC-4-1	7.5	NaHCO3	1000	812.60/0	95.6/0	0.813	35	37.40	4.4	1.069	5.344
MRG-059-ZC-4-2	7.5	NaHCO3	1000	812.60/0	95.6/0	0.813	35	37.40	4.4	1.069	5.344
MRG-059-ZC-5-1	7.0	NaHCO3	1000	765.00/0	90.0/0	0.765	35	85.00	10.0	2.429	4.031
MRG-059-ZC-5-2	7.0	NaHCO3	1000	765.00/0	90.0/0	0.765	35	85.00	10.0	2.429	4.031
MRG-059-ZC-6-1	6.0	NaHCO3	1000	697.00/0	82.0/0	0.697	35	153.00	18.8	4.371	2.157
MRG-059-ZC-6-2	6.0	NaHCO3	1000	697.00/0	82.0/0	0.697	35	153.00	18.8	4.371	2.157

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]	1
Stirring speed [rpm]	400
Start time irradiation [s]	see csv/json
End time irradiation [s]	see csv/json

Procedure/observations

Date	Time	Step	Observations
		The Experiment AE-JSC-321: Manufacturing and calibration of new 10 mL HTE with sensor spots I 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	used rare earth metal stir bar.jpg; canula position; utilized vial; position of reagent vials
		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	

		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	at this point the setpum in the vial lid was pierced 0 times
11.09.24	9:10-9:41	all the above steps were done during the execution of MRG-059-ZB lid changed, refill of water, refill of Ru and Ox with newly made solutions (see: AE-347: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O)> now: mix of the old and new solution placing of vial with NaHCO3 and Na2CO3 in robot platform (place of buffer_solution_1) placing o vial with Na2SiF6 in robot platform (place of buffer_solution_2)	
	9:41	MRG-059-ZC-1-1 was started with priming prior to reaction	at this point the setpum in the vial lid was pierced 0 times> rate =see comparison of rates table below
	ca.: 11:03	MRG-059-ZC-2-1 was started	at this point the setpum in the vial lid was pierced 11 times> rate =see comparison of rates table below
	ca. 12:05	MRG-059-ZC-1-2 was started	at this point the setpum in the vial lid was pierced 22 times> rate =see comparison of rates table below
	ca. 13:07	MRG-059-ZC-2-2 was started	at this point the setpum in the vial lid was pierced 33 times> rate =see comparison of rates table below> potentially repetition of ZC-2-1 and 2-2 needed after look at data
	ca. 14:09	end of ZC-2-2	at this point the setpum in the vial lid was pierced 44 times> pictures of septum top, bottom
	16:45-16:52	change of vial with NaHCO3/Na2CO3 to a vial with only NaHCO3> see table: varied parameters furthermore: refill of vial with Na2SiF6	
	16:55	MRG-059-ZC-6-1 was started> problems at degassing stage> autostop was triggered> need to be repeated> this experiment was named: ZC-6-1-degassing_stopped	at this point the setpum in the vial lid was pierced 0 times> rate =see comparison of rates table below

12.09.24	6:45-6:51	no apparative problem visible> change of lid and also of degassing canula this time the degassing canula was inserted less deep into the vial to avoid the backflow of liquid into it during the filling of the vial	
	6:51	MRG-059-ZC-3-1 was started	at this point the setpum in the vial lid was pierced 0 times> rate =see comparison of rates table below
	ca. 8:14	MRG-059-ZC-6-1 was started> problems at degassing stage> autostop was triggered> this experiment was named: ZC-6-1-degassing_stopped2 potential explanation: fluoride in the solution messes with the sensor	at this point the setpum in the vial lid was pierced 8 times> rate =see comparison of rates table below
	9:45-9:51	change of lid, refill H2O, NaHCO3, Na2SiF6	
	9:56	MRG-059-ZC-3-2 was started	at this point the setpum in the vial lid was pierced 0 times> rate =see comparison of rates table below
	ca. 11:12	MRG-059-ZC-4-1 was started	at this point the setpum in the vial lid was pierced 8 times> rate =see comparison of rates table below
	ca. 12:34	MRG-059-ZC-3-3 was started	at this point the setpum in the vial lid was pierced 18 times> rate =see comparison of rates table below
	ca. 13:49	MRG-059-ZC-4-2 was started	at this point the setpum in the vial lid was pierced 26 times> rate =see comparison of rates table below
	ca. 15:13	end of ZC-4-2	at this point the setpum in the vial lid was pierced 36 times> pictures of septum top, bottom
		change of lid and refill of Ru vial	
	ca. 15:13	MRG-059-ZC-5-1 was started	at this point the setpum in the vial lid was pierced 0 times> rate =see comparison of rates table below

	ca. 15:40	did not work (potentially too much fluorine in the solution for the sensor spots to work properly) > no secod try because it will most probably not work! > skip 5-2> directly to 2-3 and so on	
	-15:55	change of lid, change of vial with NaHCO3 for vial with Na2CO3/NaHCO3 and refill Ox and Na2SiF6 vial	
	ca. 15:55	MRG-059-ZC-2-3 was started repetition of ZC-2-1 and 2-2	at this point the setpum in the vial lid was pierced 0 times> rate =see comparison of rates table below
	ca. 17:18	MRG-059-ZC-1-3 was started repetition of ZC-1-1 and 1-2	at this point the setpum in the vial lid was pierced 11 times> rate =see comparison of rates table below
	ca. 18:41	MRG-059-ZC-2-4 was started	at this point the setpum in the vial lid was pierced 22 times> rate =see comparison of rates table below
	ca. 20:04	MRG-059-ZC-1-4 was started	at this point the setpum in the vial lid was pierced 33 times> rate =see comparison of rates table below
	ca. 21:07	end of ZC-1-4	at this point the setpum in the vial lid was pierced 44 times> pictures of septum top, bottom
13.09.24	8:15-8:45	disassembly of setup	

Analysis

Quantitative Interpretation

Comparison of rates

experiment determiner	pH value	Used CO3 solution	rate constant (utilizing Jacobs fit)	more information
MRG-059-ZC-1-1	9.2	NaHCO3/Na2CO3		lid (and septum) change beforehand
MRG-059-ZC-2-2	8.6	NaHCO3/Na2CO3		septum pierced 11 times beforehand
MRG-059-ZC-1-2	9.2	NaHCO3/Na2CO3		septum pierced 22 times beforehand
MRG-059-ZC-2-2	8.6	NaHCO3/Na2CO3		septum pierced 33 times beforehand
MRG-059-ZC-6-1	6.0	NaHCO3		lid (and septum) change beforehand > did not work
MRG-059-ZC-3-1	8.6	NaHCO3		septum pierced 0 times beforehand
MRG-059-ZC-6-1	6.0	NaHCO3		did not work
MRG-059-ZC-3-2	8.6	NaHCO3		septum pierced 0 times beforehand
MRG-059-ZC-4-1	7.5	NaHCO3		septum pierced 8 times beforehand

MRG-059-ZC-3-3	8.6	NaHCO3	septum pierced 18 times beforehand
MRG-059-ZC-4-2	7.5	NaHCO3	septum pierced 26 times beforehand
MRG-059-ZC-5-1	7.0	NaHCO3	lid (and septum) change beforehand
MRG-059-ZC-2-3	8.6	NaHCO3/Na2CO3	lid (and septum) change beforehand
MRG-059-ZC-1-3	9.2	NaHCO3/Na2CO3	septum pierced 11 times beforehand
MRG-059-ZC-2-4	8.6	NaHCO3/Na2CO3	septum pierced 22 times beforehand
MRG-059-ZC-1-4	9.2	NaHCO3/Na2CO3	septum pierced 33 times beforehand

Comparison of [O2]

experiment determiner	pH value	Used CO3 solution	rate constant (from implement ed ODE fit from Jacob)	[O2] degassing	[O2] pre- reaction baseline	[O2] reaction	Δ [O2] with underlying oxygen "penetration" through septum	[O2] post reaction baseline	more information
				[µmol/L]	[µmol/L]	[µmol/L]	[µmol/L]	[µmol/L]	
MRG-059-ZC-1-1	9.2	NaHCO3/Na2CO3		248> 4.3	4.3> 7	7> 86 (ca. 10 min)	ca. 80	86 - ->79.5	lid (and septum) change beforehand

MRG-059-ZC-2-1	8.6	NaHCO3/Na2CO3	240> 3.2	3.2> 5.3	5.3> 44.8 (ca. 10 min)	ca. 40	44.8> 52	septum pierced 11 times beforehand
MRG-059-ZC-1-2	9.2	NaHCO3/Na2CO3	229> 3.1	3.1> 11.7	11.7> 109 (ca. 10 min)	ca. 100	109> 120	septum pierced 22 times beforehand
MRG-059-ZC-2-2	8.6	NaHCO3/Na2CO3	237> 3.4	3.4> 21.7	21.7> 130 (ca. 10 min)	ca. 110	130> 138	septum pierced 33 times beforehand
MRG-059-ZC-6-1- degassing_stopped	6.0	NaHCO3	109> 84 > 164					lid (and septum) change beforehand> did not work potentially problem with too much fluorine in the solution which messes with sensor spots
MRG-059-ZC-3-1	8.6	NaHCO3	228> 4.4	4.4> 11.2	11.2> 121 (ca. 5.5 min)> 126 (ca. 1 min)> fluctuation around 126 (ca. 1 min)> 122 (ca. 2.5 min)	ca.110	122> 112	septum pierced 0 times beforehand
MRG-059-ZC-6-1- degassing_stopped_2	6.0	NaHCO3	67> 76 - -> 59					did not work potentially problem with too much fluorine in the solution which messes with sensor spots
MRG-059-ZC-3-2	8.6	NaHCO3	244> 10.7	10.7> 29.3	29.3> 147 (ca. 6 min) > 156 (ca. 2.5 min) > fluctuation 154 - 156 (ca. 1.5 min)	ca. 120	155> 154	septum pierced 0 times beforehand
MRG-059-ZC-4-1	7.5	NaHCO3	229> 12	12> 71	71> 213 (ca. 10 min)	ca. 140	213> 226	septum pierced 8 times beforehand
MRG-059-ZC-3-3	8.6	NaHCO3	260> 11.5	11.5> 58	58> 196 (ca. 10 min)	ca. 140	196> 207	septum pierced 18 times beforehand
MRG-059-ZC-4-2	7.5	NaHCO3	227> 14.7	14.7> 53.8	53.8> 187	ca. 135	187> 207	septum pierced 26 times beforehand
MRG-059-ZC-5-1- degassing_stopped	7.0	NaHCO3	157> 144>148 > 130					lid (and septum) change beforehand potentially problem with too much fluorine in the solution which messes with sensor spots> but: seems as if in this case the sensor levelled out after a while> but; levelling out did not happen fast enoguh> run was stopped by degassing check

MRG-059-ZC-2-3	8.6	NaHCO3/Na2CO3	227> 7.2	7.2> 5.4	5.4> 30.7	ca. 25	30.7> 28	lid (and septum) change beforehand> decreasing O2 content during prereaction baseline> untypical! (vial = very tightly closed as potential reason)
MRG-059-ZC-1-3	9.2	NaHCO3/Na2CO3	219> 4.7	4.7> 4.4	4.4> 82.5	ca. 80	82.5> 71	septum pierced 11 times beforehand> decreasing O2 content during prereaction baseline> untypical! (vial = very tightly closed as potential reason)
MRG-059-ZC-2-4	8.6	NaHCO3/Na2CO3	238> 5	5> 4.6	4.6> 34.3	ca. 30	34.3> 30.6	septum pierced 22 times beforehand> decreasing O2 content during prereaction baseline> untypical! (vial = very tightly closed as potential reason)
MRG-059-ZC-1-4	9.2	NaHCO3/Na2CO3	251> 5.5	5.5> 6	6> 88	ca. 80	88> 78	septum pierced 33 times beforehand> decreasing O2 content during prereaction baseline> untypical! (vial = very tightly closed as potential reason)

Conclusions:

- experiments with much (>2 mL) Na2SiF6 at low pH did not work properly --> potentially a
 problem with the degassing and the sensor (sensor showed odd values for [O2] at beginning
 of degassing)
- --> should be left out for future experiments
 - reproducibility not as good as in experiments until MRG-059-ZA --> 3 possible reasons:
- 1. setup stood for a few weeks and was not utilized;
- 2. random order of experiments does have influence on outcome because the setup/sensors alter over the course of one experiment row (4 experiments) --> more information first available, when Jacob has executed the rate evaluation;
- 3. the needle position during the degassing is important and it should not be put too deep into the vial to avoid backflow of solution into the canula and the syringe for degassing
 - for future: only work with carbonate buffer and test the edges of its buffer capacity but not with the fluorosilicate buffer as it produces problem with the sensors

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 [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, with manual light control
- AE-272: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, with manual light control I
- MRG-059-A: Irradiation of [Ru(bpy)3]Cl2*6 H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals
- MRG-059-B: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals II
- MRG-059-C: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals III
- MRG-059-D: Irradiation of [Ru(bpy)3]Cl2*6 H2O in the ChemSpeed robot, using a rare earth metal stirbar, fully automated peripherals IV
- MRG-059-E: Irradiation of [Ru(bpy)3]Cl2*6~H2O in the ChemSpeed robot, using a rare earth metal stirbar, fully automated peripherals V
- MRG-059-F: Irradiation of [Ru(bpy)3]Cl2*6 H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals VI
- MRG-059-Q: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O in the ChemSpeed robot, using a rare earth metal stir

bar, fully automated peripherals - 4th try I

- MRG-059-R: Irradiation of [Ru(bpy)3]CI2 * 6 H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals 4th try II
- MRG-059-S: Irradiation of [Ru(bpy)3]CI2 * 6 H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals 4th try III
- MRG-059-T: Irradiation of [Ru(bpy)3]Cl2*6~H2O in the ChemSpeed robot, using a rare earth metal stir bar, fully automated peripherals 4th try IV
- MRG-059-U: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O 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 [Ru(bpy)3]Cl2 * 6 H2O
- MRG-059-V: Irradiation of [Ru(bpy)3]Cl2 * 6 H2O 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)

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-ZC-data.zip

sha256: 03967ff59e35fc2a150946e28dde722075fb577f62b747426ad4bbf4aa0bb03d

ZC-12_3and4_top.jpg

sha256: 543be469d119793ceb8b9953615addd5c7e4e21c7655f396ab8e5b00f74c5a1e



ZC-12 3and4 top2.jpg

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ZC-12_3and4_bottom.jpg

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ZC-12_3and4_bottom2.jpg

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ZC-3_2and3_4_1and2_top.jpg

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ZC-3 2and3 4 1and2 top2.jpg

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ZC-3_2and3_4_1and2_bottom.jpg sha256: b02c1bd860811d69606abeaad908e891fd20ce9b22220beaf8ec2637c31ce92e



ZC-3_2and3_4_1and2_bottom2.jpg sha256: f4e395556ff00bf1146b81df460fe43b8ac4a1ad69b73577be07d003567ea383



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ZC-12_1and2_top2.jpg sha256: 59b51a91b9fc7b3f5089f51882a9fb9b24ddd1643ef19e343804d8b10e033298



ZC-12_1and2_bottom.jpg sha256: 49e0b8175c0132809f2e0531003bd4bfc551d24520104c02a282f77a2c0d1aba



ZC-12_1and2_bottom2.jpg sha256: 329ed350cce1c9bce45cb12dab103cb2a57499edf406f7e482094ba80fec7572





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