Operation of automated workflow for investigation of oxygen evolution - as of 03.07.2024

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Tags: HTE Irradiation MRG
Category: Protocol
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Goal

Description of the workflow necessary to operate the automated setup for the investigation of oxygen evolution.

Prerequisites and preparation

1. Preparation of the solutions necessary for the planned reaction set. Also note the necessary volumes of the solution for each reaction (necessary for later input into "experiments.yml").

E.g.:

Name	CAS Number / Experiment Number	Amount [mmol]	concentration	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	Volume [ml]
Sodium bicarbonate	Experiment - AE-270: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O (**AE-270-1**)	0.868	1.09 M	/	/	/	84.006	0.8
Sodium carbonate (anhydrous)	Experiment - AE-270: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O (**AE-270-2**)	0.632	0.79 M	/	/	/	105.988	0.8
Sodium persulfate	Experiment - AE-270: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O (**AE-270-3**)	48	60 mM	/	/	/	238.09	0.8
[Ru(bpy)3]Cl2 * 6 H2O	Experiment - AE-270: Preparation of stock solutions for the irradiation of [Ru(bpy)3]Cl2 * 6 H2O (**AE-270-4**)	0.24 µM	0.3 µM	/	/	/	748.62	0.8
Milli-Q water	7732-18-5	/	/	/	/	/	18.015	5.3

2. Determine the parameters under which the reaction shall be conducted:

E.g.:

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

3. Think about a determination scheme for the reactions which are performed. (E.g.: First reaction = MRG-055-A, Second = MRG-055-B...)

Steps for user to do

Step number	Step description
0	Get the firesting, connect it to the Laptop and load the configuration: "240717_Instrument_Settings_HTE_Vial_10mL_Liquid_Phase"

0.1	Connect firesting to group laptop (USB connection) and open PyroWorkbench on laptop (ignore the update prompt)
0.2	Goto Instrument-->Firesting O2 (2 Channels) (A) --> Load instrument configuration --> FireSting Data --> 240717_Instrument_Settings_HTE_Vial_10mL_Liquid_Phase (until 15.10.24) or 241015_Instruments_Settings_HTE_Vial_Liquid_Phase (from 16.10.24) or 241113_Instruments_Settings_HTE_Vial_Liquid_Phase (from 18.11.24) or xxxx.x.x.x.x.. (from xxx) -->Load
0.3	Disconnect Firesting from the laptop --> ready to go
1	Setup has to be installed. See: Overview-of-connections-in-setup.jpg, do not connect the magnetic stirrer's plug to the electrical outlet just yet
2	Switch the programmable power supply on and also press the "on" button to start it (initial voltage should be 0 V)
3	Switch the lamp power supply (13 A) on and place the lever on the front to "Trigger mode", make sure once more, that it i plugged into the right power outlet (slave outlet of the master/slave power outlet(white)
4	Open the argon valve on the wall and check if the reservoir bottle is filled with Milli-Q-water to the top
6	Placing necessary solutions in respective Chemspeed 13 mL vials
7	Placing Chemspeed vials at the respective places of the stock solution rack (see AutoSuite program for details about the position).
8	Placing a rare earth metal stirring bar in the reaction vial, closing it with a slit septum lid (**fully close the lid (screw it in as hard as** possible))
9	Also bend the degassing needle (small blue canula) and disconnect it from the luer adapter from the argon line. Then insert it to the vial so that the canula tip inside the vial is slightly above the level of the sensor spots (to ensure that no backflow from the vial into the canula happens during filling of the vial) (see photo). Furthermore, the needle should be inserted through the septum not close or in the slit area: Not like this but rather in this way (close to the edge of the septum between the lines of the star-shaped puncture in the septum) (Degassing needle position_like this) to avoid high leakage rates evoked by septum collapse. At last, reconnect the luer adapter of the argon line back to the canula. **Also check to NOT scratch the sensor spots with the canula.**
10	Then insert the vial to the vial holder (has to clip in)
	Connect the plug of the magnetic stirrer to the elctrical outlet and check if the stirring speed on the stirrer is set to 400 rpm
11	Open python program (click on Visual studio Code symbol in the taskbar) and defining the experiments which should be performed in "experiment.yml" (click on experiment.yml and enter the values for the experiments("buffer_solution_1" = sodium carbonate, "buffer_solution_2" = sodium bicarbonate). After four experiments with parameter run : true, add one with run : false (necessary to change the lid later on) --> save the yml (ctrl+s)
13	open Anaconda on the IPC of the robot
14	in Anaconda --> Environments --> base(root) --> press green triangle --> Open Terminal.

15	in Terminal: change directory to folder with run.py (recently: "cd Documents"--> "cd photcat_hte" --> "cd photocat-hte" --> "cd experiments"
16	in Terminal: execute run.py ("python run.py")
17	Python and arduino (for degassing valve and lamp outlet) code available at: https://github.com/lamalab-org/photocat-hte
18	Open Chemspeed AutoSuite program (Desktop\Michael\HTE photocatalysis project\ tests for writing of csv from AutoSuite\"2024_08_02 - final_version_slower_emptying" (from 02.08.24) or "2024_07_03 - final_version_without degassing valve in autosuite"(until 02.08.24)), click the green triangle in the upper part ("Run Application in Executor in Real) --> the AutoSuite Executor is opened --> click start in the Executor program
19	Close the lid of the robotic platform but make sure to not squash/squeeze any electrical or inert gas lines!
20	From this point onwards, the automated setup will perform all steps required for the formulation of a photocatalytic reaction solution and it's activity test (for steps: see table below)
21	When the first degassing of a cohort (four experiments) is performed: Check if there are bubbles visible which indicate the flow of argon through the solution --> should normally be the case, just check to be extra sure
22	Turn the light in the room on and place signs which indicate that it should be left on (to ensure same experimental conditions for every conducted experiment)
23	After four experiments have been performed: Switch the lid of the vial for a new one (make a foto of the old lid and save it to the experiment) --> **also screw this new one in firmly**
24	Replace the experiments in "experiment.yml" with a new set of experiments, save them (ctrl + s) and execute "python run.py" again in the Terminal window of the Anaconda terminal
24.1	**Check if the tubing between the 4-needle head and the syringe pumps contains bubbles**
24.1.1	**If it contains bubbles:** Exit the dialogue window shown in the AutoSuite Executor window by clicking "ok" --> directly afterwards stop the AutoSuite Executor by pressing the "stop" button also stop the python script and restart it (to load the correct experiment) Click on the first macrotask ("Prime tubing ...") in the AutoSuite Executor and then restart the program by clicking onto the "Start" button (the one in the middle with the green triangle)
24.1.2	**If it does not contains bubbles:** Click the "ok" button in the dialogue window shown in the AutoSuite Executor --> the program will automatically continue
24.2	--> do prior points as often as necessary for the planned set of experiments (e.g. if you want to do 12 reactions in total you have to change the values in the "experiment.yml" twice) **--> always close the lid of robotic platform again after changing the vial lid.**

25	After all planned reactions have been performed: Stop python script (if it is still running (ctrl, shift, c at the same time)), Stop the program in the AutoSuite Executor, Disassemble the setup, Clean everything and bring it back to the right place
26	Close the argon valve on the wall
27	Export all result logs from the Chemspeed's IPC (path: "Documents\photcat_hte\photocat-hte\experiments\logs") (USB-stick necessary) and save the csv/json files to the elab experiment
28	Evaluate the reaction outcomes and note these

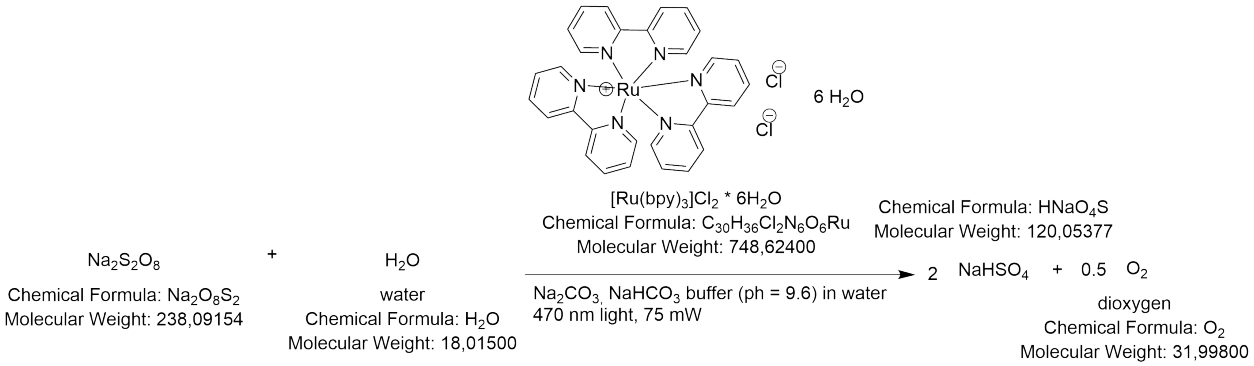
Steps in the automated protocol executed by the Chemspeed platform

see pictures of process below (picture 1, picture 2)

Step number	Step description	approximate time required
0	**If it is the first reaction of the day**, the robot will rinse the required tubings to ensure that they are free of air bubbles and potential impurities.	7 min
	If it is not the first reaction of the day, the robot will directly start with the points below	
1	Dispensing respective amounts of Milli-Q water, buffer component 1 (e.g., sodium carbonate), buffer component 2 (e.g., sodium bicarbonate), sacrificial oxidant and ruthenium catalyst solution (in this order) into the reaction vial while the emerging reaction solution is stirred at 400 rpm	ca. 25 min
2	afterwards the solution is automatically deoxygenized by argon sparging	recently 20 min (depending on user input to python code)
3	then, the solution is allowed to equilibrate in order for the oxygen levels in the headspace and solution to balance	recently 15 min (depending on user inputto python code)
4	afterwards the solution is irradiated with a 470 nm LED to start the reaction	recently 10 min (depending on user input to python code)
5	after the end of irradiation, again an equilibration time is given	recently 5 min (depending on user input to python code)

6	afterwards, the vial is automatically emptied, washed with 9 mL of Milli-Q water and emptied again to prepare it for the next round of experiments	7 min
		total time for 1 experiment (ca. 1 h 20 min - 1 h 30 min)

Reaction scheme/sample structure



Literature/reference experiments

Literature	https://doi.org/10.1021/acscatal.6b02595

Comparison to previous protocol

Category	Old protocol	New protocol
Degassing	different outcome because septum was sealed differing strong	needle without tubing is inserted into the septum to release excess pressure even if septum is very tightly sealed
Septum lid	no instruction	tighten as much as possible
Degassing II	Green canula utilized --> some problems with bending, etc.	blue canula utilized --> should be pre-bent before inserted to vial (addition of instruction to be careful with touching the sensor spots with the canula)

Degassing III	Degassing valve control was previously included in the AutoSuite as RS232 communication device --> had the problem that the port was blocked for further communication (e.g. to switch the master/slave outlet)	Deleting of RS232 device from AutoSuite app --> Inclusion to the python code (sending of "1" (string) for opening of degassing valve and "0" for closing)
Stirring	500 rpm --> problems with recovering of spinning of stirrer after emptying of vial	400 rpm
Guidance for glas fibre cables	no guidance --> much bending pressure on the holders for the glass fibre probes	made holes in box at height of glass fibre probe holders --> no bending pressure to be expected
Control of lamp power supply	Problems with small "lightnings" (probably due to short circuit in switch on power supply (Trigger <-> Manual))	Introduction of master/slave outlet to control the power connection to the lamps power supply --> utilizing a USB elongation cable and a relay to switch the (inspiration source) --> picture of setup Python code sends "ON" or "OFF" to Arduino to switch the relay and hence turn power outlet on or off
degassing canula position	degassing canula was forced until the bottom of the vial --> potential problems with scratching of the sensor spots	canula only inserted until ca. 1/3 of the vial (looking from top of lid) --> less risk of scratching

Change from "2024_07_03 - final_version_without degassing valve in autosuite.app" to "2024_08_02 - final_version_slower_emptying.app"

- the emptying of the vials was decreased in velocity
- change of aspiration speed for aspirations from vial after end of post-reaction baseline from 20 mL/min to 10 mL/min
- hope that with that all the liquid gets taken up from the vial, avoiding rests of the previous reaction or rinsing water are carried over to next reaction

Linked experiment

- AE-JSC-321: Manufacturing and calibration of new 10 mL HTE with sensor spots I

Linked items

Archived - Protocol - Operation of automated workflow for investigation of oxygen evolution - as of 03.06.2024 until 18.06.2024

Archived - Protocol - Operation of automated workflow for investigation of oxygen evolution - as of 18.06.2024

Archived - Protocol - Operation of automated workflow for investigation of oxygen evolution - as of 27.06.2024 (from MRG-059-M)

Equipment - Irradiation Set-Up

Equipment - Firesting Fiber-Optic Oxygen Meter 2 Channel (Firesting 1)

Equipment - Joy-it JT-RD6006 DC POWER SUPPLY

Light Source - UHP LED 470 nm

Attached files

241212_Instruments_Settings_HTE_Vial_Liquid_Phase.ini
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needle_depth.jpg
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2024_08_02-final_version_slower_emptying.app

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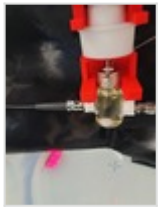

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program-overview.jpg
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degassing-valve-relay.jpg
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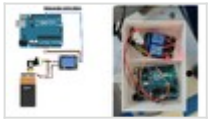

Power-outlet-relay.jpg
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vial-position-in-holder.jpg
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good-needle-position.jpg
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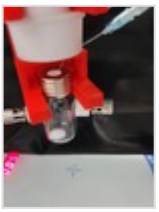

canula-to-be-utilized.jpg
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Comment

On Alexander Eith wrote:
* Adding, that master/slave socket needs to be turned on

Unique eLabID: 20240628-04a1e9200dd1b0ed41ff6a478b60c94d3933629f
Link: https://elab.water-splitting.org/database.php?mode=view&id=111