AE-251: Test for leakage in chemspeed for automated oxygen measurment during irradiadtion with a 20x punctured septum

Date: 2024-04-03
Tags: O2 AE HTE Cooperation Michael Ringleb
Status: Done
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Literature/reference experiments

Literature	/
Reproduction	/
Related experiment	Experiment - AE-244: First test for automated oxygen measurment during irradiadtion using a chemspeed Experiment - AE-250: Test for leakage in chemspeed for automated oxygen measurment during irradiadtion with a 40x punctured septum Experiment - JSC-622: HTE vial for O2 measurements

Reagents

Name	CAS Number / Experiment Number	Amount [mmol]	Equivalents	$Mass_{theo}$ [mg]	$Mass_{exp}$ [mg]	Molar mass [g/mol]	Volume [ml]
dest. water	7732-18-5	/	/	/	/	/	18

Irradiation Parameters

	Light Source Name	Wavelength [nm]	Power Setting [mW]
First light source	Light Source - LCS-0470-50-22	470	/ (Power source was set to 1.10/10)

Used beam combiner [Name or None]	none
Irradiation distance [cm]	approx. 10 cm
Thermostat temperature [°C]	not used

Stirring speed [rpm]	not known

Procedure/observations

Date	Time	Step	Observations
03.04	9:42	The setup from Experiment - AE-250: Test for leakage in chemspeed for automated oxygen measurment during irradiadtion with a 40x punctured septum was used the vial was equipped with a new septum	general setup.jpg General Note: since the chemspeed robot moves above the whole setup all tubing, cables etc. should be kept below the metal frame, if that is not possible ask somebody how is experienced with a ChemSpeed robot, with the setup is ok
		The stirring was turned on	
		The robot was strated	For code used to run the chemspeed: ask Michael Ringleb
		The robot punctured the septum 20 times	robot-during-puncturing.jpg Video available, ask me, since upload is not possible (Uni Jena cloud 20240403_094537.mp4) Approx. 15 min
		The chemspeed robot added 18 mL of water into the vial though a slitted septum	For code used to run the chemspeed: ask Michael Ringleb
	9:58	The logging of the oxygen measurment was started (AE-251-1-Ch1)	
	9:59 - 10:29	The degassing was started and the sample was degassed for 12 min using a python skript not integrated into the ChemSpeed protocol, but acsessed via the ChemSpeed protocol, to control the valve of the argon line. Since the no equlibrium was reached, the sample was degassed for another 12 min using the skript. Between the two degassing steps approx. 5 min no degassing was done. For the second degassing the pressure on the argon line was increased to approx. 1 bar	For code used to run the valve: ask Michael Ringleb Slow degassing, even after pressure incerease.
	10:29	After the degassing stopped the O2 measument logging was stopped.	AE-251-1-Ch1.png, AE-251-1-Ch1.txt
	10:29	The logging of the oxygen measurment was started (AE-251-2-Ch1)	
	10:30	The light source was turned on. Power setting: 1.10/10.	
	10:30	The skript used for the chemspeed was continued, after the ChemSpeed robot stopped due to a programmed stop to turn the light on manual	For code used to run the chemspeed: ask Michael Ringleb

	10:30 - 10:50	The sample was stirred and irradiated for 20 min	
	10:50	The logging of the O2 measument was stopped.	AE-251-2-Ch1.png, AE-251-2-Ch1.txt
	10:50	The chemspeed was stopped manually before the water was removed.	
	10:52	The vial was removed from the setup.	
	10:55	Fotos of the septum were taken	

Analysis

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Processed Data	Interpretation
03.04	9:03	AE-251-1-Ch1	Optical O2 detection	Firesting	water	AE-251-1-Ch1.txt	AE-251-1-Ch1.png	Degassing did work quite slow. Down to c(O2) = -2 µmol/L.
	8:54	AE-251-1-Ch1	Optical O2 detection	Firesting	water	AE-251-2-Ch1.txt	AE-251-2-Ch1.png AE-251.png	First decrease in c(O2) can be seen, likely artefact from stopping the degassing (?). Artefact (abrupt increase in C(O2)) of turning on the light source can be seen. Afterwards a short plateu. Afterwards nearly linear increase of C(O2) from -1.4 to 0.6 µmol/L in 18 min. During the linear increase: significant bumps are observed, so that a not so smoth line is obtained. Used for plotting: AE-251_plot.py AE-251-2-Ch1.txt

Result

Low leakage rate with approx. 0.1 µmol/L*min, in margin of error identical to the rate observed in Experiment - AE-244: First test for automated oxygen measurment during irradiadtion using a chemspeed with approx. 0.1 µmol/L*min.

Future recommendations

Old procedure	Problem	Suggested new procedure
Degassing for 12 min	Slow degassing	generally increase degassing time to 20(?) min Observe, if problem occures again.

Linked experiments

- JSC-622: HTE vial for O2 measurements

- AE-244: First test for automated oxygen measurment during irradiadtion using a chemspeed

- AE-250: Test for leakage in chemspeed for automated oxygen measurment during irradiadtion with a 40x punctured septum

Linked items

Equipment - Irradiation Set-Up

Light Source - UHP LED 470 nm

Protocol - Irradiation setup

Attached files

AE-251.png
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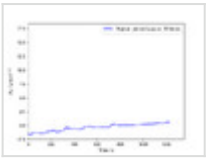

AE-251_plot.py
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AE-251-2-Ch1.txt
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general-setup.jpg
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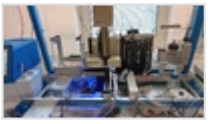

AE-251-2-Ch1.png
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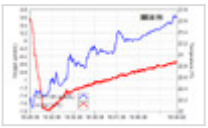

AE-251-2-Ch1.txt

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AE-251-1-Ch1.png
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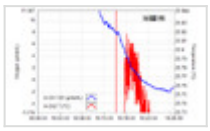

AE-251-1-Ch1.txt
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septum-after-test-II.jpg
sha256: 1a5b357399fde0378664d17fa4a0643d327e780eb88771a0c05ba2b83f2299b4

robot-during-puncturing.jpg
sha256: 9e016d7a124c2bffddcfc915505f2efac0eb62b1ebe1dc12d783806bbd4a1e1f

septum-after-test.jpg
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Unique eLabID: 20240403-378afcb8c8c98fc47007e71d034f3dfeee800f3b
Link: https://elab.water-splitting.org/experiments.php?mode=view&id=927