

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

Date: 2024-04-03
Tags: O2 AE HTE Cooperation Michael Ringleb
Status: Done
Created by: Alexander Eith

Literature/reference experiments

Literature	/
Reproduction	/
Related experiment	Experiment - AE-244: First test for automated oxygen measurment during irradiadtion using a chemspeed 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 1.1)

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	8:10	In the chemspeed in ZAF 108 the Protocol - Irradiation setup was set up: Therefor the 470 nm light source was placed in a 3D printed stand in a plastic box. The powersource was installed behind the reaction vial.	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
		In the chemspeed the oxygen measurment was set up.	general setup.jpg
		The vial was installed in the desired position and euipped with a metal cannula connected to argon	For more details, ask Michael Ringleb, general setup.jpg
		The stirring was turned on	
		The robot was strated	For code used to run the chemspeed: ask Michael Ringleb
		The robot punctured the septum 40 times	took approx. 20 min
	approx. 9:00	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
	9:03	The logging of the oxygen measurment was started (AE-250-1-Ch1)	
	9:04 - 9:16	The degassing was started and the sample was degassed for 12 min using a python skript not integrated into the ChemSpeed protocol, but accessed via the ChemSpeed protocol, to control the valve of the argon line	For code used to run the valve: ask Michael Ringleb Very strong bubbling. Some water was bubled out of the vial
	9:16	After the degassing stopped the O2 measument logging was stopped.	AE-250-1-Ch1.png , 2024-04-03_090341_AE-250-1-Ch1.txt
	9:16	The logging of the oxygen measurment was started (AE-250-2-Ch1)	
	9:16	The light source was turned on. Power setting: 1.10/10.	irrad setup.jpg , approx. 20 s after start of the measurment
	9:17	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
	9:17 - 9:37	The sample was stirred and irradiated for 20 min	
	9:37	The logging of the O2 measument was stopped.	AE-250-2-Ch1.png , 2024-04-03_091640_AE-250-2-Ch1.txt

	9:37	The chemspeed was stopped manually before the water was removed.	
	9:40	The vial was removed from the setup.	
	9:42	Fotos of the septum were taken	septum after test.jpg septum after test II.jpg

Analysis

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Processed Data	Interpretation
03.04	9:03	AE-250-1-Ch1	Optical O2 detection	Firesting	water	2024-04-03_090341_AE-250-1-Ch1.txt	AE-250-1-Ch1.png	Degassing did work very fast. Down to c(O2) = -3 µmol/L.
	9:16	AE-250-2-Ch1	Optical O2 detection	Firesting	water	2024-04-03_091640_AE-250-2-Ch1.txt	AE-250-2-Ch1.png AE-250_plot.png	First artefact of turning on the light source can be seen. Afterwards a short plateau. Afterwards nearly linear increase of C(O2) from -1 to 17 µmol/L in 18 min. Used for plotting: AE-250_plot.py 2024-04-03_091640_AE-250-2-Ch1.txt

Result

High leakage rate with approx. 1 µmol/L*min, significantly higher than for [Experiment - AE-244: First test for automated oxygen measurment during irradiadtion using a chemspeed](#) with approx. 0.1 µmol/L*min.

Linked experiments

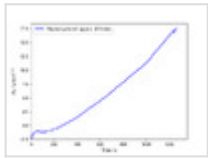
- [JSC-622: HTE vial for O2 measurements](#)
- [AE-244: First test for automated oxygen measurment during irradiadtion using a chemspeed](#)

Linked items

- Equipment - [Irradiation Set-Up](#)
- Light Source - [UHP LED 470 nm](#)
- Protocol - [Irradiation setup](#)

Attached files

- 2024-04-03_091640_AE-250-2-Ch1.txt
sha256: d9fdbf8a1bc965140e22005e7fcc8d33443c877a548392105ba89885e867aede
- AE-250_plot.png
sha256: 383b3da1d6b3107838a17967627352b0a0179cb6f2dbaf62df7fa0b2d936f3a4

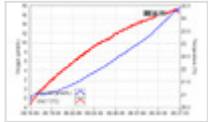


AE-250_plot.py

sha256: 333a3a3078c642dc9dc23181a8d6df31602f3c9e48012ee18834e7b0409e53bb

AE-250-2-Ch1.png (Untitled)

sha256: dc828ec4af9af9df4a3678c98ba4982d8f491a521eb82809ffb8b43e15e13351

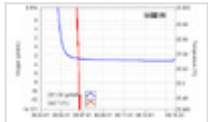


2024-04-03_091640_AE-250-2-Ch1.txt

sha256: 7c59e0df4a5290abb9c4e202f7529f6af276182ea2179c427b7e6f1216e948dd

AE-250-1-Ch1.png

sha256: 22308bc1f8e37beb59610d933187ed1d25a7325036ecb536160e213320d98e82



2024-04-03_090341_AE-250-1-Ch1.txt

sha256: e85f235ae3100ae6335cc85f2575217872689e1648a3575bc64b6bd42c643799

septum-after-test-ll.jpg

sha256: 4698ce9bb9af9fefa8bdd35b1be236bcaeab71100ab233eeaad7f6357d8c9805



septum-after-test.jpg

sha256: 24cb702d522f8df9c638538fc5e54688f2a3e5861705615b5d74128e15d3ef26



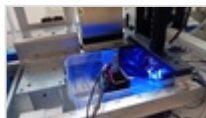
general-setup.jpg

sha256: 97ca3905b0ab4251ff8ba661471528fd09516554b2db3cdb40fd61d156ac838d



irrad-setup.jpg

sha256: cb9ef07a8d74995d5c544d6bcada92187f55f7a2d2bc5dfa90409353b749cee7



Comment

On Jacob Schneidewind wrote:

Notes:

- * removing scheme heading
- * related experiments: linking experiment for preparation of HTE vial/calibration
- * deleting second light source entry
- * in the future: adding stirring speed in irradiation table
- * procedure table: adding a note that no parts of the set-up should extend above a certain threshold (cm?) to not get in the way of the robot
- * uploading code/csv files that control robot operation, ChemSpeed code?
- * procedure table: missing time stamps, specifying time it took to perform 40 punctures
- * procedure table: for O2 measurements, linking files of measurement (.txt and .png) in the observations section
- * procedure table: specifying that a separate program was used to control the argon valve, not integrated into ChemSpeed control
- * procedure table: specifying time when the light was turned on relative to starting point of O2 measurement
- * procedure table: explaining pause and continuation of ChemSpeed script
- * procedure table: replacing "equilibration" with "stirred and irradiated"



Unique eLabID: 20240403-be76ad4d7a51b43910f14ebb8cfa1921cd679d5c
Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=926>